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وزارة التعليم العالي والبحث العلمي
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IRAQI JOURNAL OF MARKET RESEARCH AND CONSUMER PROTECTION

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Information of...
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The journal publishes scientific researches in the field of social sciences that are directly related to the market and consumer in various fields including public health, marketing, economics, environment, agriculture, pure science and media.

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To enhance the level of scientific research in the field of market research and consumer protection and to achieve a distinguished international position through publish of scientific research in this field that aimed at to serving and developing the society.

Message:

To provide the appropriate journal and scientific references for researchers which working in the field of market research and consumer protection through the publication of scientific research in this field in English languages with emphasis on strict adherence to the standards used in scientific publishing.

Aims:

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(Alsoufi & Aziz, 2019) **Alsoufi & Aziz (2019) observed that...**	Alsoufi, M.A. & Aziz, R.A. (2019). Production of aspartame by immobilized thermolysin. *Iraqi Journal of Science*, 60(6), 1232-1239.
(Alsoufi *et al.*, 2019) **Alsoufi *et al.* (2019) observed that...**	Alsoufi, M.A., Aziz, R.A. & Hussein, Z.G. (2017). Effect of some artificial sweeteners consumption in biochemical parameters of rats. *Current Research in Microbiology and Biotechnology*, 5(3), 1095-1099.
(Ledenbach & Marshall, 2009) **Ledenbach & Marshall (2009) observed that...**	Ledenbach, L.H. & Marshall, R.T. (2009). *Microbiological Spoilage of Dairy Products*. 1st ed., Springer, Germany, p. 41-67.

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Issue contents

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Al-Abboodi & Al-lami

المجلة العراقية لبحوث السوق
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NEW METHDOLOGY OF SYNTHESIS PYRAZOLO- THIAZOLO DERIVATIVES WITH STUDY ANTIMICROBAL ACTIVITIES

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ABSTRACT

In this contribution, new derivatives of thiosemicarbazon, cyclic thiazolidinone, yilidine thiazolidinone, and parazolo-thiazole were synthesized, starting from the reaction of 2 -aminobenzothiazole with *p*-bromo phenacyl bromide to give the first step of 2(4-bromo phenyl) imidazole (2,1 -b) benzo thiazole (1). Compound (1) then was subjected under Viels Myer Haack to yield 3-carbaldehyde linking with imidazo-benzothiazol (2). Compound (2) condensed with thiosemicarbazide togivenewsynthesis of thiosemicarbazon derivative (3). Compound 4-Oxo-1,3-thiazolidine (4) was constituted from reacting compound (3) with ethyl chloroacetate, while Thiazolidinone derivative ((5 was formed by reacting compound (4) with aromatic aldehyde to give 4-Oxo-1,3-thiazolidine-5 benzylidene linking with imidazo-benzothiazol (5). Compound (5) was then reacted with hydrazine hydride to give fused ring of pyrazolo thaizolo derivatives (6). In the finally, azo methene groups were opening by two reagents are (acetic anhydride and 4-nitro benzoyl chloride) to give new derivatives of hydrazone contacted with imidazobenzothiazol (7) and (8) compound respectively. All the prepared compounds were identified by Fourier Transform Infrared (FT-IR), Proton nuclear magnetic resonance (^{1} H-NMR) and Carbon nuclear magnetic resonance (^{13}C-NMR) spectra. Some of the synthesized compounds were evaluated via biological activity.

Keywords: Imidazo (2,1 -b) benzothiazol , parazolo –thiazolo, Azomethene group, Antimicrobial Activities.

طريقه جديدة لتخليق مشتقات البايروزول - ثايازول مع دراسة نشاط مضادات الميكروبات

دعاء هاشم العبودي [1]، نعيمه اللامي [2]
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الخلاصة

في هذه المساهمه، خلقت مركبات جديده من ثاياسيماكاربازون، حلقات الثايازوليدينات، اليلد ثايازوليدينات وحلقات ملتحمه من بايروزو- ثايازول من تفاعل المادة الأولية 2 –امينو بنزوثايازول مع برومو فنسيل بروماید لتشكل في الخطوه الاولى 2-(4- برومو فنيل) ايميدازو (2-1-b) بنزوثايازول (1). المركب الاول خضع لتفاعل فلز –مايرهاك ليعطي مجموعه الالديهايد في الموقع الثالث مركب (2)، وفي الخطوة التاليه تم تكاثف ثاياسيماكاربازيد مع المركب الثاني لتعطي مشتقات جديده من ثاياسيماكاربازون (3). مشتقات الثاياسيماكاربازون اغلقت بواسطه مركب اثيل كلورو اسيتات لتشكل حلقه الثايازوليدين (4).4-اوكسو (1و3)ثايازوليدين 5-بنزيلدين (5) تكون من تفاعل المركب (4)مع مختلف الالديهايدات الاروماتيه.بعد ذلك عومل المركب (5) مع هيدرازين هدراید ليكون حلقات ملتحمه من البايرازول – ثايازول (6). في الخطوات الاخيره لهذا العمل تم فتح مجموعه الايزوميثين وذلك من خلال معاملة مركب (6) مع الكواشف

*** The research is extracted from the doctoral thesis of the first researcher.**

(استيك انهدرايد، 4 نترو بنزويل كلورايد) لتعطي مشتقات جديده من مركبات الهيدرازينو(7)و (8)على التوالي . جميع المركبات تم فحصها بواسطه FT-IR وبعض منها تم فحصها بواسطه ^{1}H-NMRو C-NMR ^{13}،وكذلك تم دراسه مضاد نشاط الميكروبات لهذه المشتقات الجديده.

الكلمات المفتاحية: ايميدازو (1،2 – b) بنزوثايازول، بايرازول–ثايازول .فتح اصره الايزوميثين، مضادات الميكروبات.

INTRODUCTION

Heterocyclic compounds are important major objectives of organic synthesis **(Karam *et al.*, 2022)**, the imidazo benzo (2, 1 -b) thiazole compound is a heterocyclic compound including divers' significant biological activities **(Khalil& Khalal, 2021)** such as, anti-cancer, antimicrobial **(Al-Sultani & Al-Lami, 2021 .(**Aldehydes are one type carbonyl compounds are widly found in food products. The formation carbaldehyde belong to oxidation of fatty acid and higher alcohols, aldol condensation, Strecker degradation, and Villismyer haack reaction. The studies have proved the functional group of carbonyls have a very biological active such as antibacterial, antioxidant, anti-fungal, and some them used as an anti-and analge **(Al-Lami, 2015).**

Thiosemecarbazons are most of the intermediates compounds for the organic synthesis, the imine bond (-N=CH-) in this compounds are useful in particular for the preparation of many heterocyclic compounds, it has S/N regoselective nucleophile can able synthesis new substituent derivatives from ring closure of thiosemicarbazone such as indol, thiazol, thiadiazol, traizol ect. All this compound have wide biological activities **(Dawood *et al.*, 2010)**. Thiazolidinone heterocyclic compounds contained of five-member ring with with two heteroatoms sulpher and nitrogen**, (Ayyash *et al* 2019)** thiazolidinone compound has a wide of pharmaceutical significance in various drugs for examples antimycobacterial, antimicrobial, anticancer. Also, many clinically used drugs contain thiazolidinone ring in their skeletons such as antibiotic actithiazic acid **(Hussein & Al-Lami, 2022)** 4-Oxo-1,3-thiazolidine-5 benzylidene carrying functional group as hydroxy, methoxy, nitro and chloro groups ect, linking with imidazo benzo (2,1 -b) thiazole derivatives were synthesized from thiazolidine and aromatic aldehyde, these compounds have biological antimicrobial activity against gram positive and gram negative bacteria, yeasts and mould **(Turgut *et al* .,2007)**. Parazolo-thiazolo is a fused Five-Five membered rings system without ring junction heteroatoms, More specifically, these ring systems have two heteroatoms of nitrogen in the five-membered ring and other fused ring has one heteroatom of nitrogen and sulpher **(Kasralikar *et al.*, 2019)**. The main types of fused pyrazolo thiazole showed in (Figure, 1).

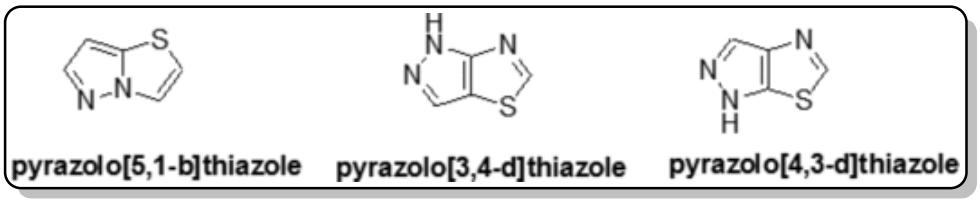


Fig (1): Types of pyrazolo thiazole.

These compounds have a wide spectrum of bioactivity such as antioxidants and anti-inflammatory agents. Some of them exhibit appreciable activity as virucides, bacteriocides, cytokine inducers, and immunomodulators. **(Kasralikar *et al.*, 2019)**. The studied proved a number of hydrazide compounds have been reported for their medical applications, hydrazide derivatives are present in many bioactive molecules and display a wide variety of biological

activities, such as antibacterial, antitubercular, antifungal, anticancer, anti-inflammatory, anticonvulsant, antiviral, and antiprotozoal action P. **(Preethi Kumari *et al* ., 2017).**

MATERIALS AND METHODS

Material and instrumentation

Corporations were supplied from diverse all chemicals such as Thomas baker, Merck, BDH, Sigma-Aldrich. End of reaction of all compounds were checked on aluminum –coated TLC plates 60 F245[E. MERCK] by using ethyl acetate and Petroleum ether and imagined under iodine vapor.

Melting points were determined on an electro thermal melting point (Stuart Germany), and they were uncorrected. Infrared spectra resolves were done as a KBR disk in range of (400-4000 cm^{-1}) FT-IR Shimadzu was used to record at university of Bagdad /College of science. The proton ^{1}H-NMR and ^{13}C-NMR spectra operating at 400 MH_Z and 100 MH_Z respectively in DMSO-d_6, measurements are performed at Collage Sharif University of Technology /Tehran/ Iran.

Preparation of 2(4-bromo phenyl) imidazo benzo [2,1 -b] thiazol (1) (Al-Sultani *et al.*, 2021)

Equimolar quantity (0.01 mol,3.2 gm) of 2-amino-benzothiazole, 4-bromophenyl phencyl bromide respectively were dissolved in abs. ethanol (40ml). The mixture was refluxed for 6 h. The solid compound was filtered and recrystallized by suitable solvent. All physical properties of compound **(1)** represent with molecular formula: $C_{14}H_9N_2S$ Br, Color: Off white, Yield :85%, M.P160: Re-crystallization solvent: Absolut ethanol.

Preparation of 2-(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3-carbaldyhade (2) (Al-Lami, 2015; Alyaa, 2020).

2-aminobenzothiazol (0.01 mol, 1.5 gm) dissolved in(15mL) of DMF with kept on temperature (0-5) ^{0}C, then added drops wise (1mL) phosphorous oxychloride ($POCl_3$) with stirring then refluxed about 5 h. After that the obtain solid compound was filtered and purified from ethanol. All physical properties to compound **(2)** represented with molecular formula: $C_{16}H_9N_2OBr$, Color: White, Yield :75%, M.P: 256 C^0, Re-crystallization solvent: Ethyl acetate.

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3-thiosemicarbazone (3) (AL-Khazraji& Ahmed, 2022; Mousa& Jassim, 2021).

an equimolar of aldehyde (0.01 mol, 1gm) and thiosimecarbazide (0.01 mol ,3.12gm) in absolute ethanol (20 mL) with 2-3 drops of glacial acetic acid, were refluxed for 5 h. The mixture was cooled to room temperature, and the solid product was washed with cold water, purified with ethanol to yield compounds(3). all physical properties to compound represented with molecular formula: $C_{16}H_{12}N_5OSBr$, Color: Yellow, Yield :70%, M.P: 195-197 ^{0}C, Re-crystallization solvent: Ethyl acetate

Synthesis of 2(4-bromopheny) limidazo (2, 1-b) benzothiazole-3-yl) methylenehydrazineyl-thiazol-4(5H)-one (4) (Khalil & Khalal, 2021).

A mixture of thiosemicarbazone (0.01mol,1.2 gm) with (0.01mol,0.5 mL) of ethyl chloroacetate and sodium acetate (0.04 mol) was also added in absolute ethanol (20mL) and the resulting mixture was refluxed for 7 h. The reaction monitored by TLC. The crude product was cooled at room temperature, then poured onto ice-water. The resulted precipitate was filtered, washed with water and dried.

Syntheses of 2(4-bromophenyl) imidazo (2,1-b) benzothiazole-(3- methylene hydrazineyl-(4-Oxo-1,3-thiazolidine)-5 benzylidene (5) (Kasralikar *et al.*, 2019)

Compound (4) (0.01 mol, 0.5gm) was mixed with (0.01mol, 0.3 gm) of aromatic aldehyde (*p*-Nitro benzaldehyde, O-hydroxy benzaldehyde with anhydrous sodium acetate (0.015 mol) dissolved in (10 mL) of glacial acetic acid. The mixture was heated under reflux for 7 h. The reaction was monitored by TLC. The crude product was cooled to room temperature, the resulted precipitate was filtered, washed with water, and dried to give compound (5). All physical properties of compound **(5)** are listed in (Table, 1).

Table (1): The physical properties of the compound [5P-5Q].

Comp. NO	M.F	Color	Yield (%)	M.P	Re- cryst. solvent
5P	$C_{23}H_{17}N_6SO_3$ Br	yellow	85	279	Chloroform
5Q	$C_{23}H_{18}N_5O_2S$ Br	yellow	75	330	ethanol

Synthesis of of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- methylene hydrazineyl - [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole] (6 P-6Q) (El-Hamouly *et al*., 2011).

A mixture of compound (5) -(4-Oxo-1,3-thiazolidine)-5 benzylidene (0.02 mol, 1gm) and hydrazine hydride (0.06 mol, 1mL) was refluxed in ethanol (20 mL) in the presence of few drops of hydrochloric acid for 5 hrs. The crude product was cooled to room temperature, the resulted precipitate was filtered, washed with water, dried. (Table 2) explained all physical properties to compound (6).

Table (2): The physical properties of the compound [6P-6Q].

Comp. No	M.F	Color	Yield(%)	M.P	Re-cryst.solvent
6P	$C_{23}H_{19}N_8O_2S$ Br	yellow	75	270	Chloroform
6Q	$C_{23}H_{20}N_7OS$ Br	yellow	85	380	ethanol

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- methyl acetate hydrazono-1-acytel [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole] (7P-7Q) (Zainab, 2008)

(0.5 gm,0.005 mol) of Schiff bases dissolved in (15 mL) of dry benzene then added (0.51 gm ,0.005 mol) from acetic anhydride dissolved in (10 mL) from benzene after that

refluxed about 2h. After that the obtain solid compound was filtered and purified from suitable solvent. (Table 3) explained all physical properties to compound (23).

Table (3): The physical properties of the compound [7P-7Q].

Comp.NO	M.F	Color	Yield (%)	M.P	Re-cryst.solvent
7P	$C_{27}H_{25}N_8O_5S$ Br	Brown	75	279	Chloroform
7Q	$C_{27}H_{26}N_7O_4S$ Br	yellow	75	280	ethanol

Synthesis of 2(4-bromophenyl) imidazo (2, 1-b) benzothiazole-3- chloro methyl -4-nitro benzo hydrazino [3-benzyl -5H-pyrazolo-[3,4-d]-1,3-thiazole](8P-8Q) (Zainab ,2008)

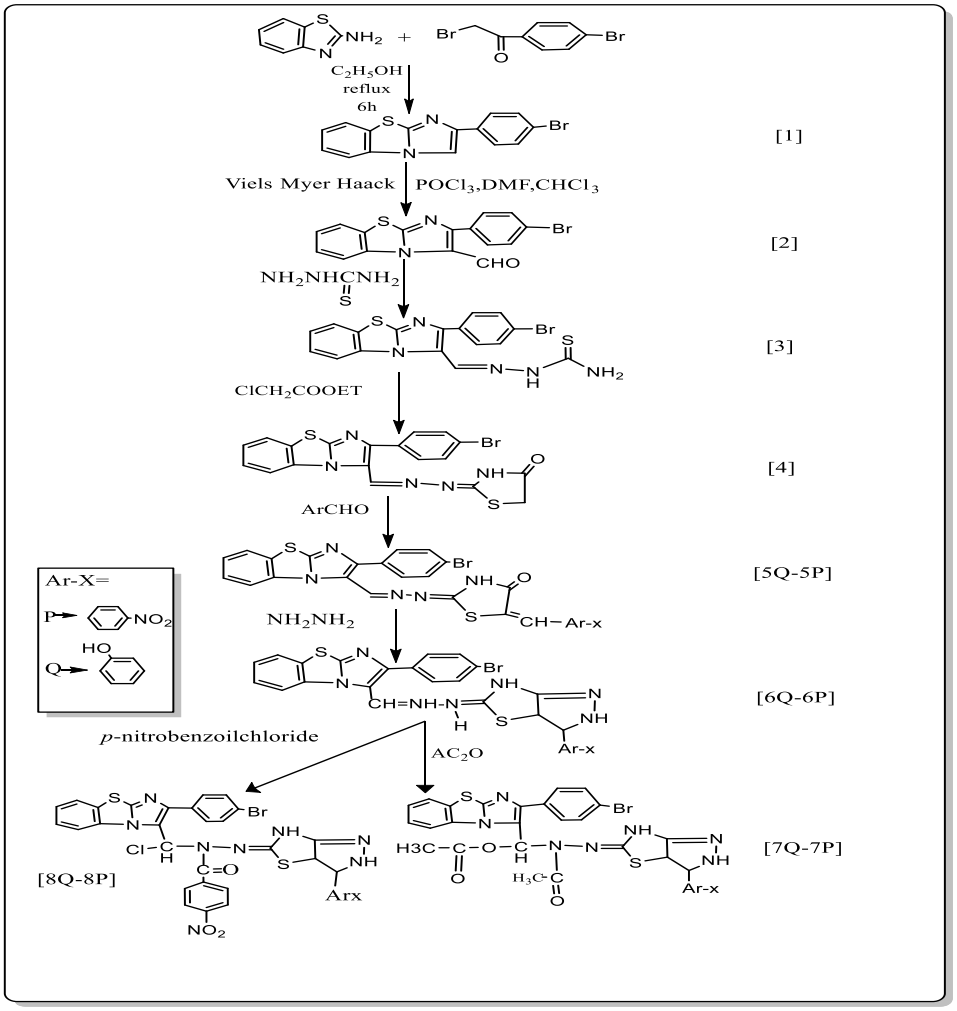
Schiff bases (0.005 mol, 0.5 gm) dissolved in (15 mL) of dry benzene then added (0.005 mol ,0.51 gm) from 4-nitro benzoyl chloride dissolved in (10 mL) from benzene after that refluxed about 3 hrs. After that the obtain solid compound was filtered and purified from suitable solvent. (Table 4) explained all physical properties to compound (24).

Table (4): The physical properties of the compound [8P-8Q].

Comp.NO	M.F	Color	Yield (%)	M.P	Re-cryst.solvent
8P	$C_{31}H_{26}N_{11}O_5S$ BrCl	yellow	75	285	Chloroform
8Q	$C_{31}H_{27}N_{10}O_4S$ BrCl	yellow	89	288	ethanol

RESULTS AND DISCUSSION

All the reactions are explained in Scheme 1



Scheme (1): Synthesis steps of compounds.

In this work, the imidazo benzo (2,1 –b) thiazole compound **(1)** was obtained from the reaction of 2- amino benzo thiazole with 4-(2- bromo phenacyl bromide) and characterization by FTIR spectrum showed bands at 1645 due to ν C=N imidazo ring, ν C=C aromatic at1575, other bands showed in(Table5)

Table (5): Characteristic absorption bands in FT-IR spectra of compound (1) in Cm^{-1}

Comp No.	ν(C-H) Aromatic	νC=N imidazo ring	ν C=C Arom.	Other bands
1	3051	1645	1575 1490	ν(C-N) 1336 ν(C-C)937 ν(C-Br)744

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compound (1) reacted with thiosemicarbazone to give formal derivatives **(2)** was showed absorption band at 1685 cm^{-1} due to ν C=O, at (1591) cm^{-1}due to ν C=N, other bands showed in (Table 6).

Table (6): Characteristic absorption bands in FT-IR spectra of compound (2)in Cm^{-1}

Comp No.	ν(C-H) Aromatic	ν C=O	νC=N imidazoring	ν C=C Arom.	Other bands
2	3076	1685	1591	1556 1433	ν(C-N) 1321 ν(C-C)927 ν(C-Br)781

Thiosemicarbazon compounds **(3)** was prepared by reacted formal compound with thiosemicarbazide were subjected to diagnosis by FT-IR was showed absorption band at (3367-3498) cm^{-1} belong to ν NH_2, (CH=N) at 1662cm^{-1}, ν (C=N) at1640 cm^{-1}.other bands in (Table 7).

Table (7): Characteristic absorption bands in FT-IR spectra of compound (3)in Cm^{-1}

Comp No.	ν NH_2	ν NH	ν CH=N	νC=N imidazo ring	ν C=C Arom.	Other bands
3	3367-3498	3161	1662	1640	1573 1490	ν (CH)aldehyde 1725 ν (CH)Arom3004 ν(C-N) 1323 ν(C-C)925 ν(C-Br)748

In **^{1}H-NMR** compound **(3)** was result as fellow: at 4.49 ppm (s,2H,NH_2), at (7.20-8.56)ppm, (m,8H,ArH), at (8.66)ppm (s,1H,CH=N), at (9.07)ppm (s,1H,NHC=S).

Thiosemecarbazone was reacted with ethylchloroacetate and anhydrous sodium acetate in ethanol to give five member ring is thiazolidinone ring compound **(4)** confirmed by the FT-IR, showed the disappearances of NH_2 group bands of thiosemicarbazons group and the appearances of new characteristic bands at (1708cm^{-1}) belong to the stretching vibration of (C=O) of lactam groups. All FT-IR spectra of compounds **(4)** are listed in (Table, 8).

Table (8): Characteristic absorption bands in FT-IR spectra of compound (4) in Cm^{-1}

Comp No.	ν NH	ν C=O Lactam	ν CH=N	ν C=N	ν C=C Arom	Other bands
4	3101	1708	1662	1652 1595	1595 1440	(CH)Arom.3076 ν(C-N) 1396 ν(C-C)939 ν(C-Br)748

Compound **(4)** Characteristic by ^{1}**H-NMR** the result as fellow: at (4.02-4.07) ppm (s,2H,CH_2), at (7.37 -8.48) ppm (m,8H,ArH), at (8.53)ppm (s,1H,CH=N), at (9.69)ppm (s,1H,NHC=S)

Thiozolidinone compound reacted with different aromatic aldehyde to give compound **(5P-5Q),** showed stretch band at1697-1699 cm^{-1} belong to (C=O),at 1604-1614 cm^{-1}due to (C=C)alkene,at1652-1666 cm^{-1} belong to ν (CH=N), other bands showed in(Table 9),

Table (9): Characteristic absorption bands in FT-IR spectra of compound (5P- 5Q)in Cm^{-1}

Comp No.	**ν NH**	**ν C=O**	**ν CH=N**	**ν C=C Alkene**	**ν C=N**	**Other bands**
5P	3191	1699	1652	1604	1564	CH Arom. 3004 ν(NO_2)Asym. 1512,Sym 1350, ν(C=C)Arom. 1463,1550 ν(C-N) 1319, ν(C-C)927, ν(C-S) 819, ν(C-Br)769
5Q	3137	1697	1666	1614	1573	ν(O-H)3434, ν(C=C)Arom. 1541,1440 ν(C-N) 1317, ν(C-C)925, ν(C-S) 788, ν(C-Br)748

Compound (5P) conformed by ^{1}H-NMR,^{13}C-NMR spectra all data appeared in (Table 10).

Table (10): Characteristic absorption bands in ^{1}H-NMR α^{13}CNMRspectra of compound(5P)

Comp.	**^{1}H-NMR**	**^{13}CNMR**
5P	at (7.38) ppm (s,1H,C=CH), at(7.40-8.42)ppm(m,8H,ArH), at (8.50) ppm(s,1H, CH=N) ,at(9.26)ppm (s,1H,NHC=S).	(21.54) ppm(C-NO_2) , (116.47-133.79) ppm(C=C) arom, at (149.01) ppm(=CH), at (150.76) ppm (CH=N), at (178.21) ppm carbonyl group

Compound (5) cyclic by hydrazine hydride to give pyrazolo –thiazolo derivation **(6Q-6P)** an characterization by FTIR and results as fellow: compound **(6p)** appeared band absorption at 1614-1600 cm^{-1} due to (C=N) pyrazolo- thiazolo ring ,1649-1662 cm^{-1} due to CH=N . all FT-IR spectra of compounds (6) are listed in (Table, 11).

Table (11): Characteristic absorption bandsin FT-IR spectra of compound(6p-6Q)inCm^{-1}

Comp No.	ν NH	ν CH=N	ν C=N pyrazo-thiazol and imidazo ring	ν C=C Arom	Other bands
6P	3149	1629	1595 1564	1550 1490	CH Ar.3070 ν(NO_2)Asym1531,Sym 1394, ν(C-N) 1317, ν(C-C)929, ν(C-S) 802, ν(C-Br)746
6Q	3137	1623	1600 1564	1598 1488	ν(O-H)3448, (CH)Arom 3074, ν(C-N) 1319, ν(C-C)931, ν(C-S) 802, ν(C-Br)748

Compound**(6P)** confirmed by **^{1}H-NMR**and results as fellow at (7.388.35) ppm (m,8H, ArH), at(8.55)ppm(s,1H,CH=N),at(9.60)ppm (s,1H,NHC=S). In compound (7) the action mechanism **(Zainab, 2008**) was Azomethen group opening by reagent (acetic anhydride) .see bellow to Scheme (2).

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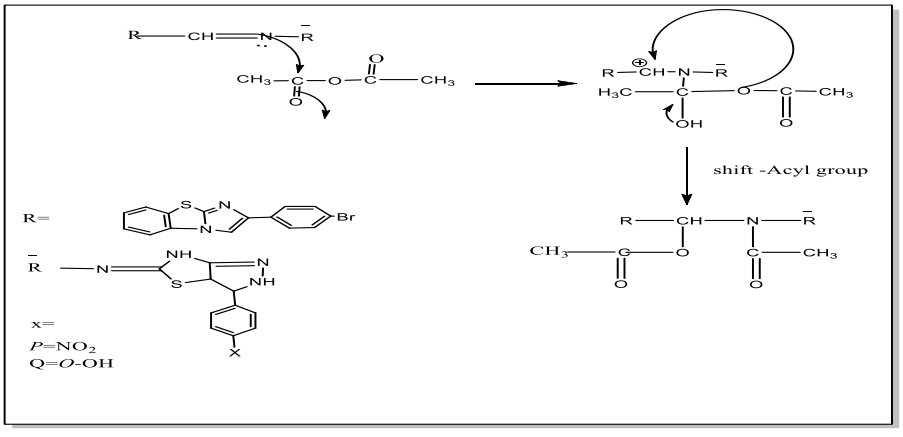

Scheme (2): Mechanism of compound (7).

Compound (7P-7Q) in FTIR spectra showed disappearances stretch bands belong Azomethen and appearance carbonyl lactams at (1685-1703) cm^{-1}, and carbonyl ester at (1718-1720). All FT-IR spectra of compounds (7P-7Q) appear in (Table12)

Table (12): Characteristic absorption bands in HNMR spectra of compound (7P-7Q)

Comp No.	ν NH	ν C=O Ester, Amide	ν C=N pyrazo thiazol,and imidazo ring	ν C=C Arom	Other bands
7P	3139	1718 1703	1627 1595	1490	ν CH Ar3011 ν(NO2)Asym 1531,Sym 1394, ν(C-N) 1317, ν(C-C)927, ν(C-S) 802, ν(C-Br)746
7Q	3228	1720 1685	1623 1575	1535	ν(O-H)3425 (CH)Ar.3028, ν(C-N) 1363, ν(C-C)925, ν(C-S) 802, ν(C-Br)750

Compound **7P** confirmed by **^{1}H-NMR** and results as fellow: at (2.27) ppm(s,3H,CH_3 lactam), At (2.27) ppm(s,3H,CH_3 lactone), at (7.43-8.35)ppm (m,8H,Ar-H) ,at(9.70)ppm (s,1H,NHC=S) Azomethen opening by reagent(4-nitro benzoyl chloride) to give (8P-8Q)compound, the FTIR spectra showed disappearances stretch bands belong to Azomethen group and appearance carbonyl lactam at (1693-1695) cm^{-1}.All FT-IR spectra of compounds(8P-8Q) appeared in (Table, 13)

Table (13): Characteristic absorption bands in HNMR spectra of compound (8P-8Q)

Comp No.	**ν NH**	**ν C=O Amide**	**ν C=N pyrazo thiazol,and imidazo ring**	**ν C=C Arom**	**Other bands.**
8P	3114	1693	1604 1595	1550 1492	ν CH Ar3062 ν(NO_2)Asym 1525,Sym 1348, ν(C-N) 1313, ν(C-C)931, ν(C-S) 800, ν(C-Br)717
8Q	3261	1695	1649 1573		ν(O-H)3438 (CH)Ar.3028, ν(NO_2)Asym 1502,Sym 1373 ν(C-N) 1363, ν(C-C)925, ν(C-S) 786, ν(C-Br)767

Biological Activities (Alyaa,2020)

In this work synthesized compounds have active moieties in their structures, therefor these compounds to possess a wide spectrum from biological activity. Some of the prepared compounds were tested against two type of bacteria (Staphylococcus aureus (+*ve*), and Escherichia coli(-*ve*), and two type of anti-fungal (Candia albicans, Asp. niger). As shown (Table 14).

Table (14): Anti-bacterial activity for some prepared compounds

Comp. NO.	Staphylococcus Aureus(+ve)	Escherichia coli (-ve)	Candia albicans	Asp.niger
5p	25mm	--	16mm	22mm
5Q	-	-	23mm	22mm
8p	14mm	25mm	-	-
8Q	-	25mm	23mm	-

CONCLUSIONS

From this work, we have successfully prepared new heterocyclic compound as (thiazolidinone and parazolo- thiazolo) rings by using start martials 2- amino benzothiazol ,fused ring have very important biological activity, all new derivations analytical and spectral data FT-IR and some of them analytical by (^{1}HNMR,^{13}CNMR)proved the proposed structures.

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Tayeh & AL- hadedee

المجلة العراقية لبحوث السوق
وحماية المستهلك

THE ANTIBACTERIAL ACTIVITY OF TIO_2 NANOPARTICLES PREPARED BY SOL-GEL METHOD FOR A GROUP OF GRAM-POSITIVE AND NEGATIVE BACTERIA

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ABSTRACT

Titanium dioxide nanoparticles TiO_2 NP were prepared by sol-gel method. TiO_2 NP was identified and characterized using scanning electron microscopy (SEM), ultraviolet spectroscopy (UV-vis), Fourier transform infrared (FTIR), X-ray diffraction (XRD) and atomic force microscopy (AFM). The SEM results showed an irregular spherical shape with different diameters (22.84-65.98) nm. The highest UV-Vis absorption was recorded at 345 nm wavelength. FTIR was used to find out the effective aggregates and the success of the process of forming TiO_2 NP bands. The first centered between (450 – 800 cm-1), which is attributed to the patterns of stretching vibrations of the titanium oxide bond (Ti-O Vibrations). While the XRD peaks appeared at angles 2Ɵ (27.32°, 35.89° 39.03°, 41.02°, 43.88°, 54.09°, 56.38°, 62.43°, 63.77°, 68.67°, 69.41°, 76.11°) at Crystal Planes (110) (101) (200) (111) (210) (211) ((220) (002) (310) (301) (112) and (202), (respectively, which corresponds to JCPDS standard tables), and this indicates the formation of rutile-type NPs TiO_2. The results of (AFM) ranged between (6-7 nm) and the mean height (Z-mean value) was (8 nm). The effectiveness of NP TiO_2 was tested at different concentrations (1, 0.75,0.5) mg/ml against a group of Gram-negative and Gram-positive bacteria:(Pseudomonas aeruginosa, Salmonella typhimurium, Bacillus subtilis, Staphylococcus aureus, E.coli) reached the highest zone of inhibition at a concentration of (1) mg/ml for Staphylococcus aureus, as the diameter of inhibition was (19.5).

Keywords: TiO_2, minimum inhibition, sol gel, Nanoparticles

[*]**The research is taken from a master's thesis by the first researcher.**

الفعالية المضادة لجسيمات التيتانيوم النانوية المحضرة بطريقة السول-جل لمجموعة من البكتريا السالبة و الموجبة لصبغة كرام

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الخلاصة

تم تحضير ثاني أوكسيد التيتانيوم النانوي TiO_2 NP بواسطة طريقة السول –جل، حيث تم تشخيص وتوصيف TiO_2 NP باستخدام المجهر الالكتروني الماسح (SEM) ,مطياف الاشعة فوق البنفسجية (UV-vis) ، تحويل فورير الأشعة تحت الحمراء(FTIR)، حيود الاشعة السينية (XRD) ومجهر القوة الذري (AFM). اظهرت نتائج (SEM) شكلاً كروي(Spherical Shape) غير منتظم و بأقطار مختلفة بلغت (22.84-65.98) nm.

وبلغ أعلى امتصاص لـ UV-Vis سجله عند الطول الموجي 345 nm. واستخدم (FTIR) لمعرفة المجاميع الفعالة ومدى نجاح عملية تكوينTiO_2 NP الحزمة الاولى تتمركز بين (cm^{-1} 800 – 450) والتي تعزى الى انماط اهتزازات التمدد (Stretching Vibrations) لاصرة اوكسيد معدن التيتانيوم Ti-O. في حين XRD ظهر قمم عند الزوايا

(39.03°,41.02°,43.88°,54.09°,56.38°,62.43°,63.77°,68.67°,69.41°,76.11°,35.89° ،27.32°)2Ɵ

عند المستويات البلورية (110) (101) (200) (111) (210) (211) (220) (002) (310) (301) (112) و(202) على التوالي والذي يتطابق مع الجداول القياسية (JCPDS) وهذا يشيرعلى تكوين TiO_2 NPمن نوع روتيل. أما نتائج (AFM) كانت تتراوح بين (6-7 nm) ومعدل ارتفاع (Z-mean value) (8 nm). أختبرت فعالية TiO_2 NP بتراكيز مختلفة (0.5, 0.75 ,1) ملغم/مل ضد مجموعة من البكتريا السالبة والموجبة لصبغة كرام

(*Bacillus subtilis, Staphylococcus aureus,* Escherichia *coli, Salmonella typhimurium Pseudomonas aeruginosa)*

بلغت أعلى منطقة تثبيط عند تركيز(1) ملغم/مل مل لبكتريا *Staphylococcus aureus* إذ كان قطر التثبيط (19.5).

الكلمات المفتاحية: ثاني أوكسيد التيتانيوم، أقل فعالية تثبيطية، سول-جل، جزيئات النانو.

INTRODUCTION

TiO_2NP is a semiconductor transition metal oxide that exhibits unique properties such as low cost, ease of handling, non-toxicity and resistance to chemical corrosion. These advantages make TiO_2NP a widely used material in solar cells, chemical sensors, self-cleaning surfaces, environmental cleaning applications and in the food industry **(Hamza *et al.,* 2013)**. TiO_2NP exists in both crystalline and amorphous forms and is mainly found in three polymorphic forms namely anatase, rutile and brookite. Anatase and rutile have a quadrangular structure, while brookite has a straight structure **(Jaafer *et al,* 2009)** Crystal phase, particle size, and particle shape all influence the physical and chemical characteristics of TiO_2 **(Fahem *et al.,* 2022)**. There are different methods that can be used to synthesize TiO_2NP **(Kim *et al.,* 2004)** such as spraying, chemical vapor deposition, microwaves and method Sol-gel **(Di Paole *et al.,* 2013)** which is one of the most promising technologies as this method Homogeneous samples at low cost produces **(Dai *et al.* , 2010)**.

MATERIAL AND METHODS

Preparation of TiO_2 NP by sol-gel method

TiO_2 NP was prepared by the sol-gel method by dissolving 12ml of titanium tetraisopyroxide (TTIP) in 100ml of ethanol and stirring the mixture for 30 minutes using a magnetic stirrer, 3ml of deionized water and 2ml of HCl were added into the solution drip and continued stirring for two hours to obtain a homogeneous solution at a pH of (3), then the solution was left for 24 hours, after which the gel was dried at 400 °C**(Ramalingam *et al,* 2019).**

Diagnosis and characterization of TiO_2 NP

The characterization process was carried out using a scanning electron microscope (SEM), a UV-visible spectrometer (UV), Fourier transform spectroscopy (FTIR), X-ray diffraction (XRD), and Atomic Force Microscopy (AFM) .

TiO_2 NP's minimum inhibitory concentration Test (MIC)

The Agar well diffusion method mentioned by **(De Oliveira *et al.*, 2014)** was followed, where the inhibition activity of TiO_2 NP was tested against the isolates used in the study by growing the isolates in 10 ml of the nutrient broth prepared at 37 °C for 24 hours **(Al-hadedee *& Awahd,* 2022)**, then spreading 0.1 ml of activated test bacteria on the surface of the a solid culture medium Muller Hinton Agar using a sterile glass diffuser (L-shape), a hole with diameter of 6 mm was made on the surface of the culture medium with a corkscrew, and 50 of microliters of the solution was placed in each hole with concentrations of (1, 0.75,0.5) mg /ml TiO_2 NP using a micropipette, then the plates were incubated at a temperature of 37 °C for 24 hours in the incubator, then the diameter of the corona was Measured Inhibition Zone.

Organisms used

E. coli, Pseudomonas aeruginosa, Salmonella typhimurium Bacillus subtilis, Staphylococcus aureus

RESULTS AND DISCUSSION

Diagnosis and characterization: SEM

(Figure, 1) shows the scanning electron microscopy images (FESEM Images) of the prepared TiO_2 NP at (100 kx and 200 kx) magnifications. The obtained results showed that the TiO_2 NP has a spherical shape. The average particle size ranges from 22.84-65.98 nm. And the difference in the size of the material in one sample indicates that it was formed at different times **(Geethalakshmi &Sarada, 2012)**.

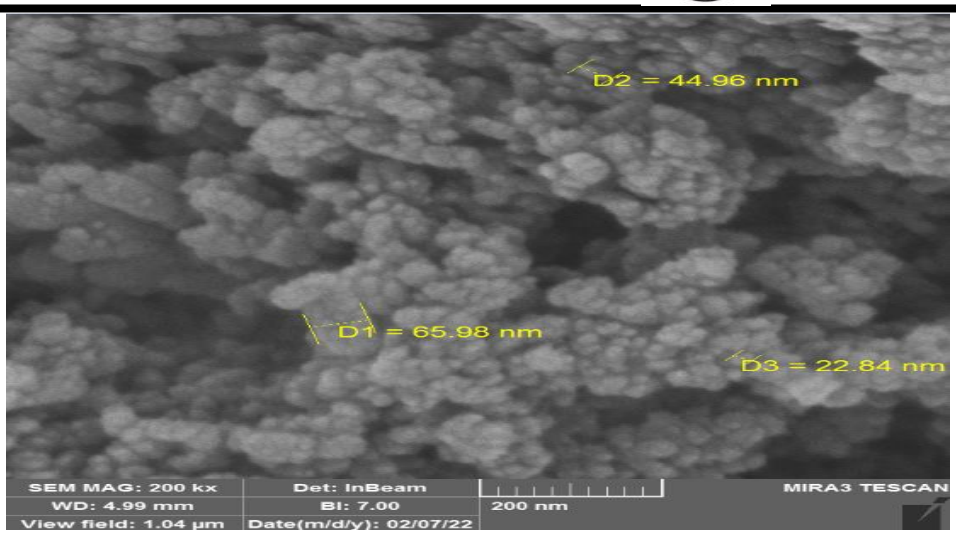

Figure (1): SEM images of TiO_2 NP

UV-visible spectrometer

Figure (2) shows the absorption spectrum of (TiO_2 NP) nanopowder prepared by the (sol-gel method). The results showed obtaining a prominent peak in the absorption spectrum at wavelength (345 nm) with an absorbance less than 1. Obtaining a strong peak at wavelength (345 nm) is within the wavelength range (200-1000 nm) confirms obtaining (TiO_2 NP) by the sol-gel method **(Vijayalakshmi &*Rajendran*, 2012)**.

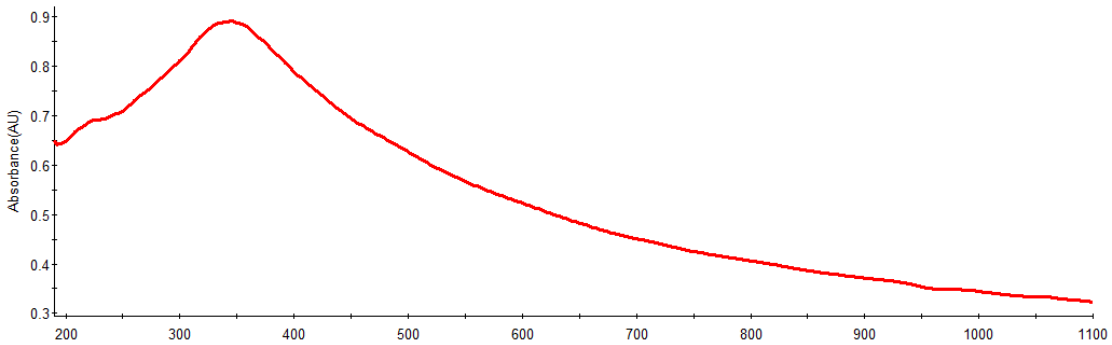

Figure (2): UV-Vis absorption spectral TiO_2 NP

Fourier transform spectroscopy (FTIR):

(Figure, 3) shows an Fourier transform spectroscopy (FTIR) examination of the prepared TiO_2 NP in order to determine the effective aggregates using the spectrometer (Shimadzu Japan-IR Affinity-1), by measuring the transmittance spectrum as a function of the wavenumber. number) within the range (400-4000 cm-1). The results showed the emergence of three diagnostic bands (Characteristic Bands), the first band is centered between (450-800 cm-1), which is attributed to the patterns of stretching vibrations of Ti-O Vibrations **(Sonali *et al.*, 2021; Yu *et al.*, 2006)**, the second band is centered around (1636 cm-1), which is due to the stretching vibrations of the (carboxyl-titanium) and hydroxyl (O-H) groups, respectively **(Ghaly *et al.*, 2011)**, while the third band It is represented by a broad band centered between (3000 - 3800 cm-1), which is due to the stretching vibrations of the

hydroxyl group (O-H) resulting from the moisture absorbed from the external environment by the prepared TiO_2 NP**(Sonali *et al.*, 2021).**

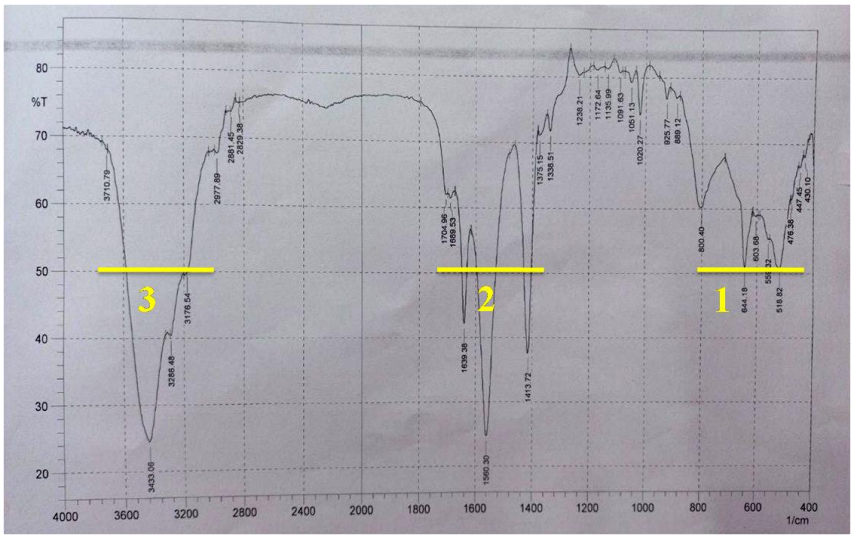

Figure (3): shows the FTIR spectrum diagram of TiO_2 NP particles

X-ray diffraction (XRD):

X-ray diffraction (XRD) of TiO_2 NP (Fig. 4) was carried out using an X-ray diffraction device (Shimadzu-6000) with a wavelength (λ = 1.54060 A°) and a potential difference (40 KV). The results of the X-ray diffraction (XRD) test showed that the diagnostic peaks were obtained Characteristic Peak of TiO_2 NPs at angles (2Θ = 27.32°, 35.89°, 39.03°,41.02°, 43.88°, 54.09°, 56.38°, 62.43°, 63.77°, 68.67°, 69.41°, 76.11°) at Crystal Planes (110) (101) (200) (111) (210) (211) (220) (002) (310) (301) (112) and (202) ,respectively, which indicate obtaining (TiO_2 NPs) type rutile with a tetragonal crystal structure of (Space Group) level (P42/mnm no.136), with dimensions (a = b = 4.6107 ˚A and c = 2.9732 ˚A) and crystal angles (α=β=γ=90°), which corresponds to the standard card (JCPDS 01-077-0443). No other additional peaks were detected, which indicates obtaining high purity TiO_2 NPs.

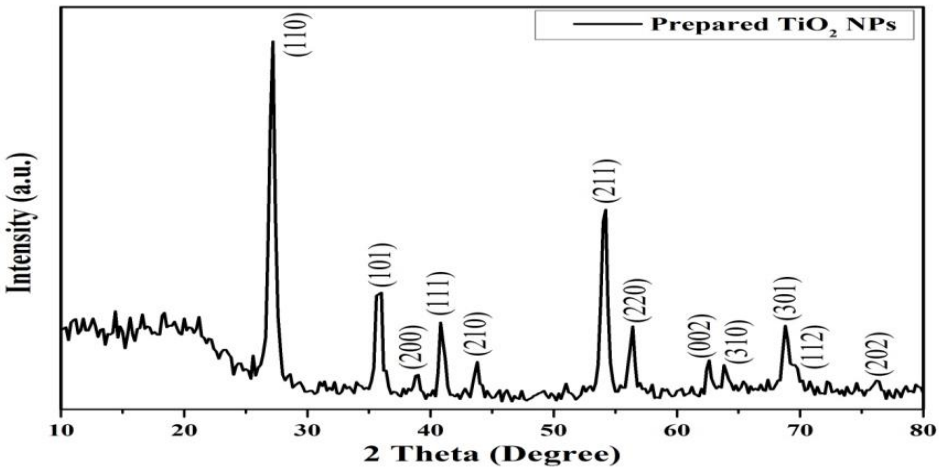

Figure (4): (XRD) TiO_2 NP prepared by (Sol-gel) method.

Atomic Force Microscopy AFM:

The surface morphology of the TiO_2 NP was studied by atomic force microscopy (AFM). Figure (a, b5) presents 2D and 3D atomic force microscope (AFM) images of TiO_2 nanoparticles prepared at (400 °C). The images showed that the TiO_2 NP powder has a high roughness surface with a granular microstructure and a non-flat texture, which consists of particles with diameters ranging between (6-7 nm) and a mean height (Z-mean value) of (8 nm), as shown in Figure (3). On the other hand, the atomic force microscopy (AFM) examination gives surface roughness values, as the root mean square (Sq) of TiO_2 NP is higher than (1 nm) with a value of (2.045 nm), and this indicates roughness High surface.

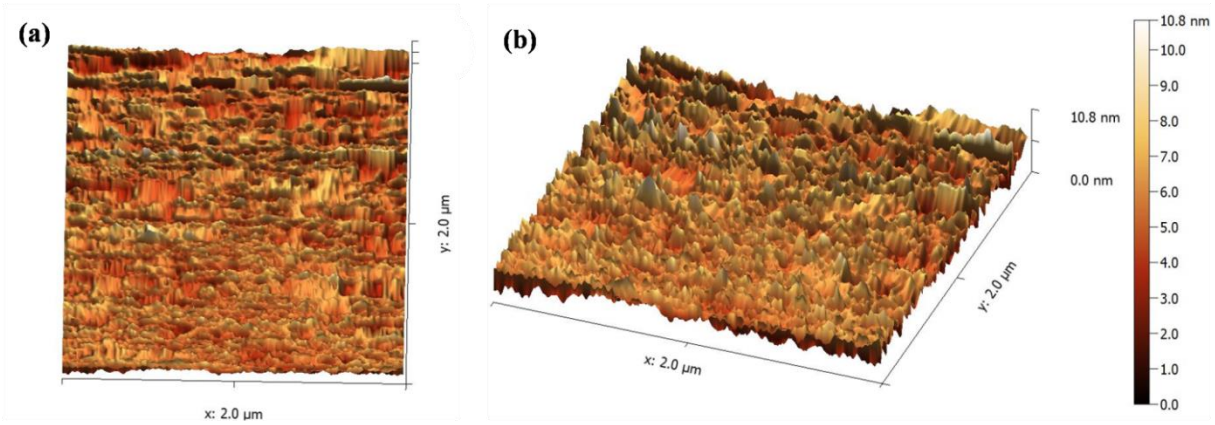

Figure (5): AFM images (a) 2D and (b) 3D of prepared and calcined NPs TiO_2 at (400 °C).

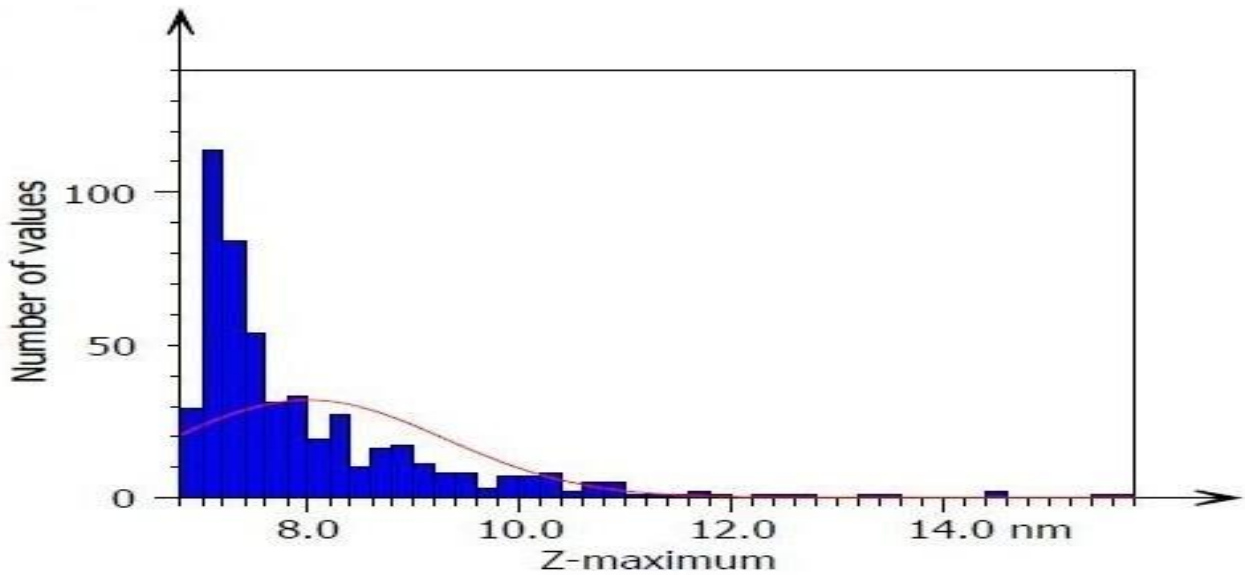

Figure (6): Z-mean value plot of the as-prepared TiO_2NPs

NP's minimum inhibitory concentration Test (MIC): TiO_2

The minimum inhibitory concentration (MIC) was determined by Agar well diffusion method at different concentrations (1, 0.75, 0.5) mg/ml. Its effect on inhibiting the growth of a group of bacteria was studied, some of which are Gram-positive and Gram-negative. The

results showed that the use of NP TiO_2 inhibited all types of bacteria Where the lowest value of the minimum inhibitory concentration was (0.5) mg/ml for Bacillus subtilis, as the diameter of inhibition was 9 mm, while the diameter of inhibition was (15.5, 13.5) mm, respectively, for the concentrations used (1,0.75) mg/ml. While the value of the minimum inhibitory concentration was (0.5) mg/ml for Staphylococcus aureus, and the diameter of inhibition was 9.5 mm, while the diameter of inhibition was (14.5, 19.5) mm, respectively, for the concentrations used (1, 0.75) mg/ml. As for the lowest value of the minimum inhibitory concentration was (0.5) mg/ml for E. coli bacteria, as the diameter of inhibition was 10 mm, while the diameter of inhibition was (17.5, 14) mm, respectively, for the concentrations used (1,0.75) mg/ml. The reason for the difference in inhibition on the types of positive and negative bacteria **(Ahmad *et al.*,2015)** where they found that NP TiO_2 invade bacterial cells by damaging the cell membrane in both positive and negative bacteria, causing cell leakage and death. And that the lowest value of the minimum inhibitory concentration was (0.5) mg/ml for Pseudomonas aeruginosa bacteria, as the diameter of inhibition was 10 mm, while the diameter of inhibition was (15.5,14) mm, respectively, for the concentrations used (1,0.75) mg/ml. While the lowest value for concentration the minimum inhibitor was (0.5) mg / ml for Salmonella typhimurium, as the diameter of inhibition was 9 mm, while the diameter of inhibition was (16,13.5) mm, respectively, for the concentrations used (1,0.75) mg / ml. The reason for the inhibition of gram-negative bacteria is due to the gram stain because the bacterial cells and the TiO_2 NP has opposite charges, whereby electrostatic attraction occurs between TiO_2 NP bacterial cells, which leads to disruption of the cell membrane and thus to increased permeability and cell death (**Haghi *et al.*, 2012; Bahjat *et al.*, 2021**).

CONCLUSION

The TiO_2 NP was prepared successfully by sol-gel technique at room temperature. Then, the diagnosis and characterization of the TiO_2 NP was carried out. SEM measurement confirmed that the TiO_2 NP is spherical shape, while the wavelength of the TiO_2 NP was 345 nm, which was observed by UV inspection. And through (FTIR) the effective aggregates indicating the presence of NP TiO_2 were known. While the results of the XRD analysis indicate obtaining TiO_2 (NPs) of the rutile type. It was observed in the AFM analysis The TiO_2 NPs are surface rough. The effectiveness of) TiO_2NPs) was tested on types of Gram-positive and negative bacteria, where all types of bacteria used in this research were inhibited with different diameters, and it was noted that the anti-bacterial efficiency increased by increasing the concentration of the (TiO_2NPs) solutions used.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

DESIGN, SYNTHESIS AND BIOLOGICAL EVALUATION OF MANNICH BASE SOME TRANSITION METAL COMPLEXES

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ABSTRACT

The new mannich base ligand and its metal complexes were synthesized in ethanol medium. The mannich base is derived from the condensation reaction of morpholine and Ciprofloxacin (Cp) at room temperature. This ligand and metal complexes were characterized using elemental analysis, FT-IR, UV-Vis, and NMR spectral data, molar conductivity measurements, and melting points. Elemental analysis data show that the metal complexes formed have the general formula$[Cr(L)_2ClH_2O]$ $Cl.H_2O$, $[Pt(L)_2Cl_2]$ $Cl_2.H_2O$ and $[Au(L)_2]Cl.H_2O$ where mannich base ligand (L) .Based on spectroscopic analytical, coordination with metal ions involves the 'O' donor atoms of carboxylate group, and the Cr(III) and Pt(IV) complexes are a six-coordinated octahedral structure while Au(III) complex is A four-coordinated square planer structure. Molar conductivity of these complexes showed that they were electrolytic in nature. The toxicity of the free ligand and their metal complexes as anticancer agents against MDA-Mb-231 cell lines was examined with different concentration. Anticancer testing revealed that all complexes were more effective than the ligand. The Au (III) complex exhibited the most significant toxicity effect than the other compounds.

Keyword: Metal complexes, Ciprofloxacin, Mannich base, Anticancer activity.

توصيف ، تحضير والتقييم الحيوي لبعض معقدات العناصر الانتقالية لقاعدة مانخ

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الخلاصة

تم تحضير ليكاند قاعدة مانخ الجديد من تفاعل تكثيف المورفلين وسيبروفلوكساسين عند درجة حرارة الغرفة وتم تحضير بعض المعقدات من هذا الليكاند. تم تشخيص الليكاند ومعقداته باستخدام تحليل العناصر، وبيانات طيفية FT-IR، و UV-Vis، و NMR، وقياسات الموصلية المولية، ودرجة الانصهار. اظهرت بيانات تحليل العناصر أن المركبات الفلزية المتكونة لها الصيغة العامة $Cl.H_2O$ $[Cr(L)_2ClH_2O]$، $Cl_2.H_2O$ $[Pt(L)_2Cl_2]$ و $[Au(L)_2]$ $Cl.H_2O$ حيث ليكاند قاعدة مانخ (L). بناءً على التحليل الطيفي ، يكون التناسق مع أيونات المعادن عن طريق ذرتي اوكسجين مانحة لمجموعة الكربوكسيل، ومعقدات Cr (III) و Pt (IV) سداسية التناسق ذات شكل ثماني السطوح بينما معقد Au (III) رباعي التناسق ذو شكل مربع مستوي. أظهرت الموصلية المولية لهذه المعقدات أنها ذات طبيعة إلكتروليتية. تم فحص سمية الليكاند ومعقداته كعوامل مضادة للسرطان ضد خطوط الخلايا من نوع MDA بتراكيز مختلفة. كشفت الاختبارات المضادة للسرطان أن جميع المعقدات كانت أكثر فعالية من الليكاند. أظهر معقد Au (III) تأثير سمية اعلى من بقية المركبات.

الكلمات المفتاحية : معقدات فلزية، سيبروفلوكساسين، قاعدة مانخ، نشاط مضاد للسرطان.

[*] **The research is extracted from the doctoral thesis of the first researcher.**

INTRODUCTION

Many pharmaceuticals and drugs contain metal moieties or metal-drug bonds that can coordinate or react with various metal ions, can affect biological activity, and can damage target biomolecules. increase **(Albedair, 2021).** Quinolone antibiotics **(Gellert *et al.*, 1976)** have many properties such as broad antibacterial spectrum **(Lipinski, 2000)**, high bactericidal activity, low toxicity and unique mechanism. As one of the most important antibiotics, it is widely used in the clinical treatment of various infectious diseases **(Tan *et al.*, 2012)**. Ciprofloxacin (CP) is a synthetic broad-spectrum fluoroquinolone antibiotic for oral administration **(Supuran *et al.*, 2001)**. It is active against a wide range of aerobic Gram-negative and Gram-positive bacteria **(Rawtani *et al.*, 2017)**. Mainly complicated and uncomplicated urinary tract infections and pyelonephritis, lower respiratory tract infections, skin infections, urethral and cervical gonococcal infections, bone and joint infections, infectious diarrhea, typhoid fever and acute sinusitis, it is approved for the treatment of **(Kapoor *et al.*, 2017)**. The Mannich reaction is one way to accomplish this task. In addition, many of these compounds have been synthesized and studied for their antibacterial and antitumor activity, as the Mannich bases themselves possess excellent biological activity **(Fu *et al.*, 2014)**.In the present work we are synthesis a new derivative of Ciprofloxacin with morpholine, also the metal complexes of this derivative (L) with Cr(III), Pt(IV) and Au(III) ions were synthesized. The medincinal applications as a anticancer activity was studied.

MATERIALS AND METHODS

General

Ciprofloxacin (99.5%), morpholine, formaldehyde, solvents and metal chlorides (analytical-grade) were obtained from Merck (Schnelldorf, Germany). Using an AA-6880 Shimadzu atomic absorption flame spectrophotometer (Shimadzu Corporation; Tokyo, Japan), the metal content was measured. A Bruker Avance 300 spectrometer (Bruker BioSpin GmbH, Rheinstetten, Germany) was used to record the ^{1}H and ^{13}C-NMR spectra. In order to measure the ultraviolet-visible (UV-Vis) spectra in ethanol, a Shimadzu UV-1601 spectrophotometer (Shimadzu Company; Tokyo, Japan) was used. The FT-IR 8300 Shimadzu spectrophotometer (Shimadzu Corporation; Tokyo, Japan) was used to record the Fourier transform infrared (FTIR) spectra. Direct Probe captured mass spectra. The melting points in open glass capillaries were examined. Using EA-034.mth, the elemental analyses (C.H.N.S.) were obtained. Measurements of conductivity were performed using a Corning conductivity meter 220, and they were done in an ethanol solvent with a concentration of (10^{-3} M)

Synthesis of Mannich base derived from antibiotic Ciprofloxacin 4-cyclopropyl-7-fluoro-6-(4morpholinmethyl) piperazin-1-yl)1-oxo-1,4-dihydronaphthalene-2-carboxylic acid.) (L)

General procedure for the preparation of (L) (CP, 1.65 g 0.005 mol) and morpholine (0.45 ml, 0.005 mol) in EtOH (25 mL). A solution (CH_2O) was applied and heated to reflux for 6 hours and cooled to room temperature. The precipitate was filtered and recrystallized from (ethanol in water) to give the title compound **(Feng *et al.*, 2011)**.

Preparation of metal Complexes

The desired metal ions in 1mmole (0.158g, $CrCl_3.6H_2O$, 0.22 g, 0.409g $H_2PtCl_6.6H_2O$ and 0.354 $HAuCl_4.6H_2O$) were mixed with 2mmoles (0.430 g) of mannich base (L) which was

dissolved in 10ml of absolute ethanol. The mixture was refluxed for two hours, changing the color. When the solvent was evaporated the resultant precipitates were formed and recrystallization from hot ethanol and then dried to give the metal complexes.

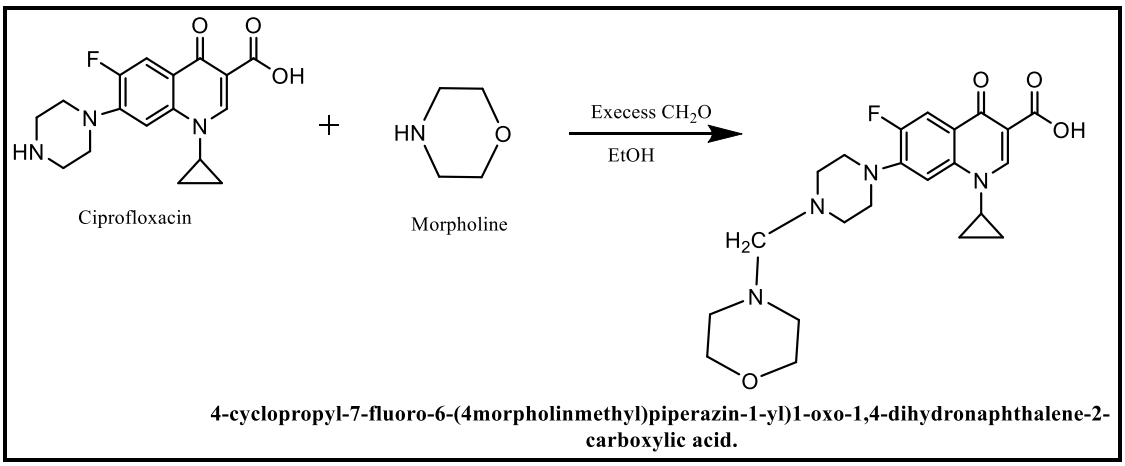
Cytotoxic studies-MTT assay

The cytotoxicity of free ligand and their metal complexes were studied against MDA cell lines by *in vitro* MTT cytotoxicity assay **(Abdulameer & Alias, 2022)**. Cell lines were evaluated 24 hours after being exposed to the compounds at various concentrations. Results from the MTT testing utilizing a desiccator were shown for ligand and their metal complexes. All the compounds produced were characterized using spectroscopic, analytical, and physical methods, as shown in Table 4. Various concentration (400,200,100,50 μg/ml) were compared to untreated negative control culture medium.

RESULTS AND DISCUSSION

Synthesis and Characterization of the ligand and Metal Complexes

The data in Table 1 suggest that (L) and its metal ion complexes are in agreement with calculated values. The suggested molecular structure is formulated and characterized by subsequent spectral and molar ratio as well as magnetic moment.



Scheme (1): Synthesis of ligand (L).

Spectral Analysis

FT-IR Analysis:

(Figure 1 and Table 2) show the assignment of the typical bands (FT-IR spectra) of the free (L). The FT-IR spectrum of the free ligand (4-cyclopropyl-7-fluoro-6-(4morpholinmethyl) piperazin-1-yl)1-oxo-1,4-dihydronaphthalene-2-carboxylicacid.) displays distinct bands between (1635 and 1620 cm^{-1}), which was ascribed to (C=O). The stretching vibration of the (OH) of the COOH group is attributed to a band in the FT-IR spectrum of the free ligand at (3531) $cm^{-1.}$ Other bands are attributed to the stretching frequency of the $\nu(CH_2$-N) and the (C=N) at (2964-2839), (1552) cm^{-1}, respectively. All complexes showed a lower frequency shift of the stretching vibration of the (OH) of the COOH group during complexation, appearing in the range (3395-3393) cm^{-1}, indicating coordination through the oxygen atom

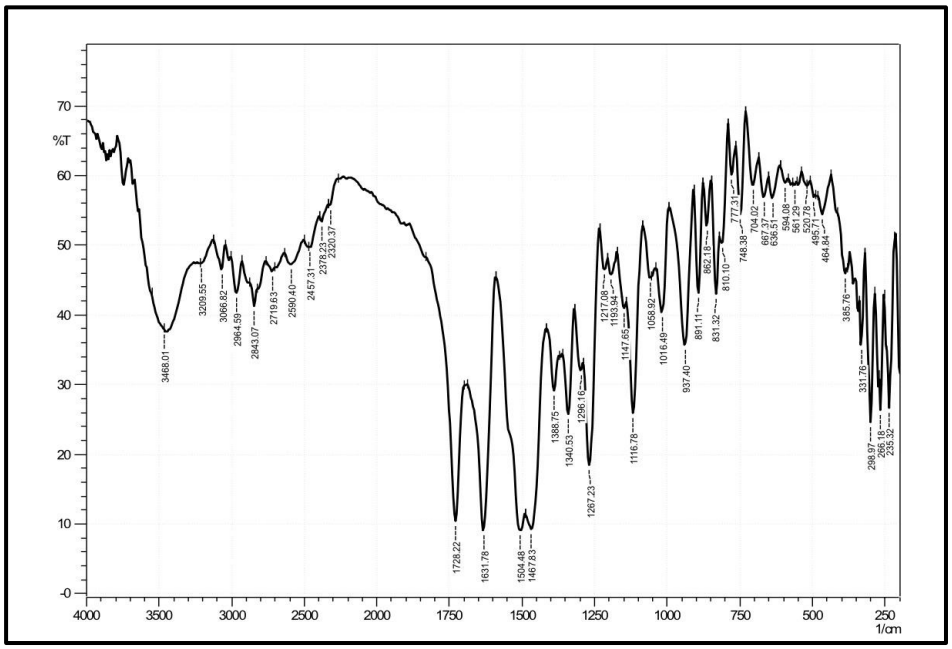
with the metal ion. The spectral data of the prepared complexes records typical frequency bands of stretching vibrations that are asymmetric $(COO-)_{asym}$ and symmetric $(COO-)_{sym}$ within the ranges (1595-1568) and (1409-1392) cm^{-1}, respectively. The low difference between the $(COO-)_{asym}$ and $(COO-)_{sym}$ values, which is less than 200 cm^{-1}, shows that the carboxylate group has bidentate binding properties **(Mahmood *et al.*, 2021)**. The FT-IR spectrum of the complexes showed the appearance of new bands at low intensities and lower wave numbers in the ranges 484-470 cm^{-1} attributed to ν (M-O) **(Taher & Mohammed, 2011)**. The complexes spectra showed new weak bands in the range (352-312) cm^{-1}, which was attributed to the stretching frequency of *ν* (M-Cl) **(Bakir, 2016)**. Table (2) displays the coordination of the δH_2O and ω H_2O band with the central metal ion (aqua) in the Cr (III) complex. Bands varied between (3452-3419) cm^{-1} appeared in all complexes referred to stretching band of H2O incoordination **(Ali & Hassan, 2022)**.

Table (1): Color, melting point, yield, and elemental composition of ligand and its metal complexes.

Compund	suggested formula	Color	melting point ℃	yield %	elemental analysis Found (cal.)				
					C	H	N	S	M
L	$C_{22}H_{27}FN_4O_4$	Light Yellow	158-160	88.5	61.45 (61.38)	5.92 (6.32)	12.92 (13.02)	---	---
L Cr(III)	$[CrL_2Cl\ H_2O].Cl.H_2O$	Green	184-186	68%	51.38 (51.42)	5.54 (5.56)	11.60 (11.66)	---	**4.89 (4.958)**
L Pt(IV)	$[PtL_2Cl_2].Cl_2.H_2O$	Dark Yellow	236-238	67.5%	43.51 (43.54)	4.45 (4.48)	9.20 (9.23)	---	**16.03 (16.06)**
L Au(III)	$[AuL_2].Cl_1.H_2O$	Orang	136-138	68%	47.64 (47.69)	4.88 (4.91)	10.08 (10.10)	---	**17.73 (17.75)**

Table (2): Selected FT-IR absorption bands of ligand and its metal complexes.

Comp.	νOH COOH	ν COOH	ν C=O	ν COO- asy.	ν COO- sy	ν Δ	ν N-H piprazin	νCH_2-N	νM-O	Others
L	**3531**	**1722**	**1629**	----	----	----		**2920 2837**	----	----
CrL	**3395**	----	**1635**	**1575**	**1394**	**181**	**2625**	**2928 2835**	**472**	ν OH(H_2O) = 3419 ω H_2O = 1039 aqua. δH_2O = 956 ν Cr-Cl = 312
PtL	**3393**	----	**1631**	**1583**	**1398**	**185**	**2630**	**2926 2830**	**470**	ν OH(H_2O) = 3452 ν Pt-Cl = 352
AuL	**3393**	----	**1620**	**1585**	**1394**	**191**	**2617**	**2924 2834**	**484**	ν OH(H_2O) = 3452



Figure(1): FT-IR spectrum of mannich base ligand

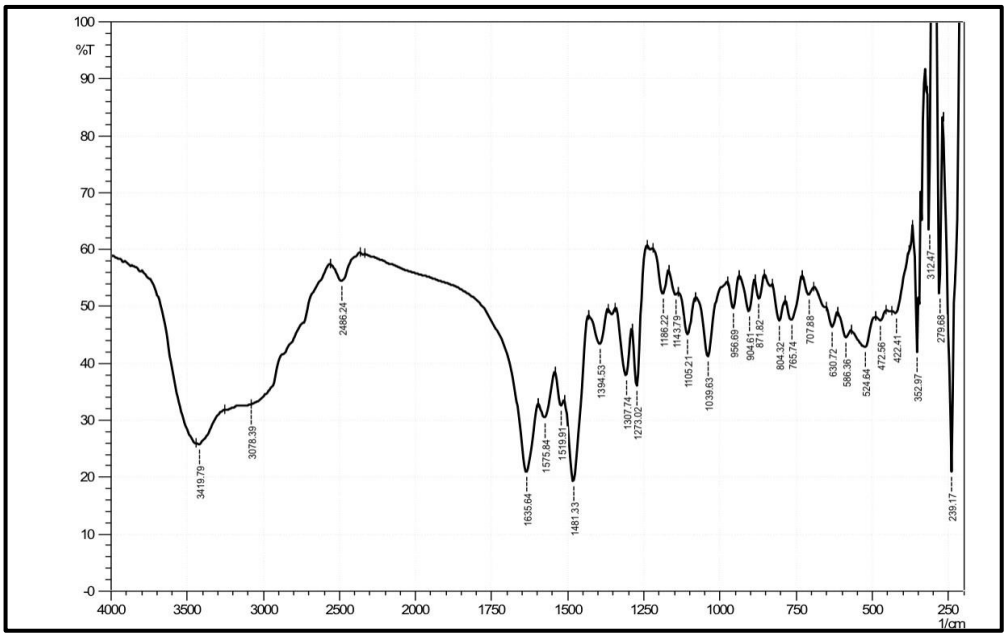


Figure (2): FT-IR spectrum of Cr(III) complex.

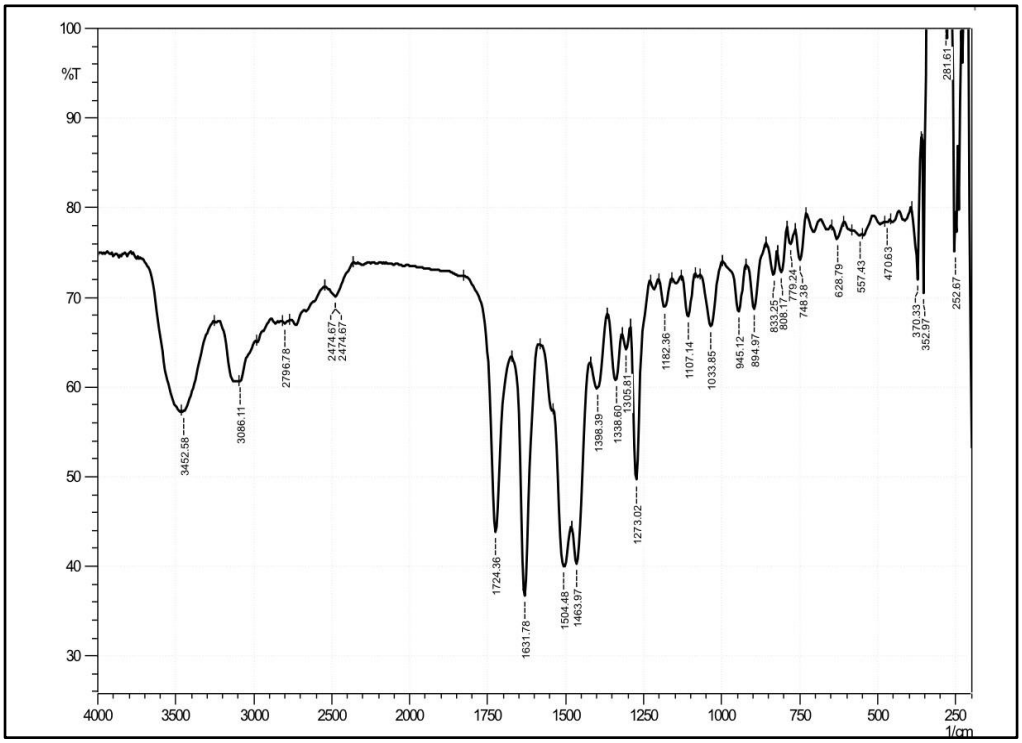


Figure (3): FT-IR spectrum of Pt(IV) complex.

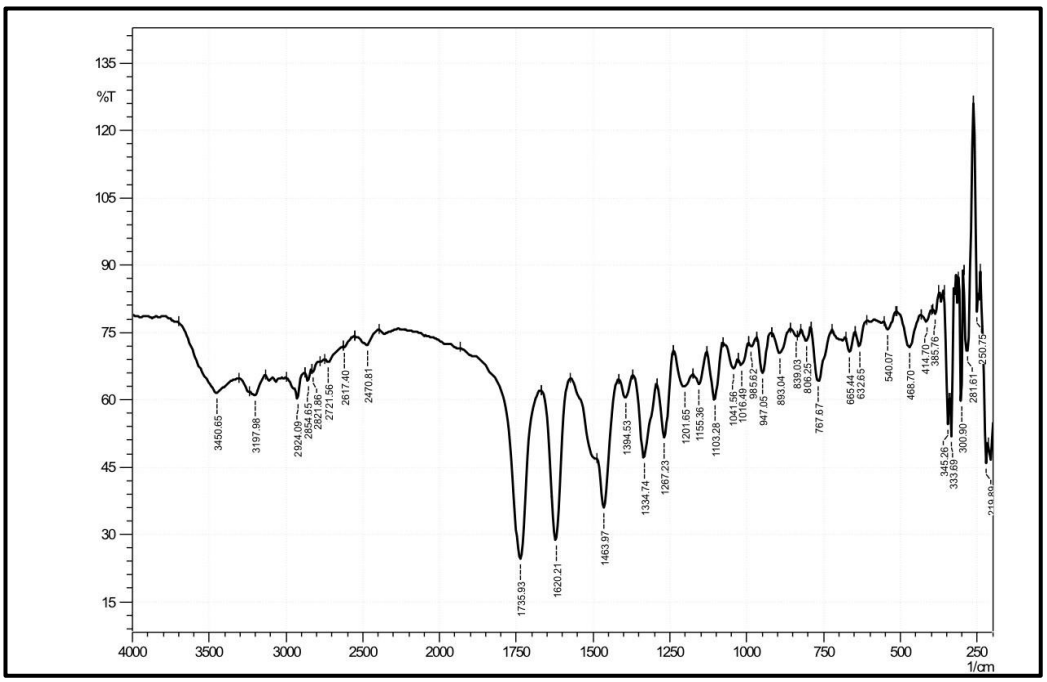


Figure (4): FT-IR spectrum of Au(III) complex**.**

Electronic Spectra

Four absorption bands are seen at (231 and 260 nm) (43290 and 38461 cm^{-1}) in the UV-visible spectra of the (L) (Figure 5) which attributed to π-π* transitions of the aromatic rings **(Mathew, *et al.*, 2017)** In addition, the absorption band at (298 and 305 nm) (33557 and 32786 cm^{-1}) that may be attributed to n–π* transition of the intraligand transitions (these transitions occur in the case of unsaturated hydrocarbons that contain ketone groups for ciprofloxacine) **(Sarwade, *et al.*, 2015)** and the N=C- groups of imidazole in addition to the pyrazine ring respectively **(Abdulghani & Hussain, 2015).**

[CrL] The spectrum in (Figure 6) shows three bands at (611,278 and 210 nm) (16366, 35971, and 47619 cm^{-1}) which assigned to the transitions $^4A_2g \rightarrow {}^3T_2g$ (F), $^4A_2g \rightarrow {}^3T_1g$ (p), and L→Cr CT, respectively **(Hasan, 2020)** The theoretical second transition calculated from the equation $15B\backslash = v_3+v_2-3v_1$ using Tanabe–Sugano diagram in d^3 configuration and found to be(404nm) (24742 cm^{-1}) ,the ligand field parameter was calculated and can been shown in (Table 3) ionic nature was observed conductivity device. The μ effect value of this complex is 3.33 BM. This indicates that the complex has an octahedral geometry around the Cr(III) ion with three parallel electrons.

[PtL] Four bands of dark yellow Pt(IV)complex at (995,350,301,and 200 nm) (10050, 28571, 33222, and 50000 cm^{-1}, which are attributed to the transitions $^1A_1g \rightarrow {}^3T_1g$, $^1A_1g \rightarrow {}^1T_1g$, $^1A_1g \rightarrow {}^1T_2g$, and (L) →Pt (C.T) respectively. The magnetic moment of the present complex, which is (0.0 B.M) of the Pt(IV) complex in its (d^6) structure, agrees with the octahedral configuration **(Abdullah *et al.*, 2016)**, indicating a diamagnetic characteristic. The complex ionic behavior revealed by the conductivity measurement in ethanol (Table 3, Figure 7), the two (Cl^-) ions are outside the coordination zone.

[AuL] The electronic spectrum of the synthesized orang-Au(III) complex (Figure 8) described three bands at (408,295 and 242 nm) (24509 , 33898 and 41322) cm^{-1} which are assigned to the transitions $^1A_1g \rightarrow {}^1B_1g$, $^1A_1g \rightarrow {}^1Eg$ and (L) → Au (C.T) respectively **(Alibrahim *et al.*, 2018).** . These results suggest diamagnetic behavior since the magnetic moment of the present complex (0.0 B.M) is consistent with the suggest square planar configuration.

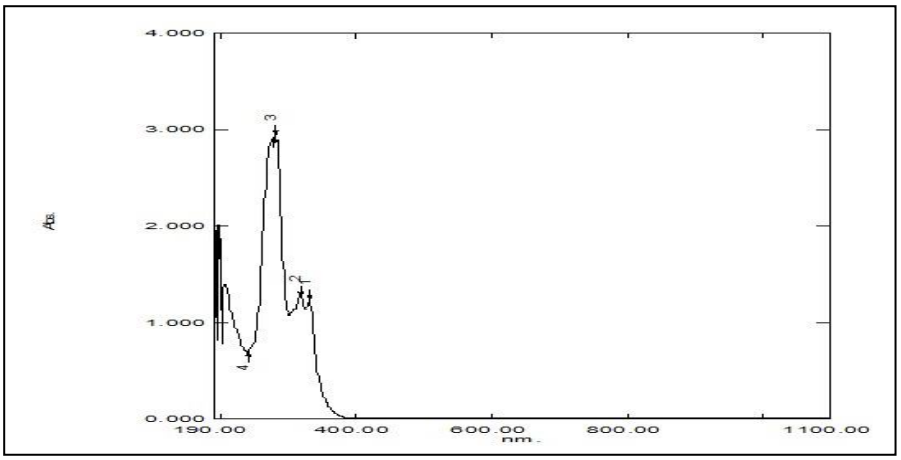


Figure (5): Electronic spectrum of (L).

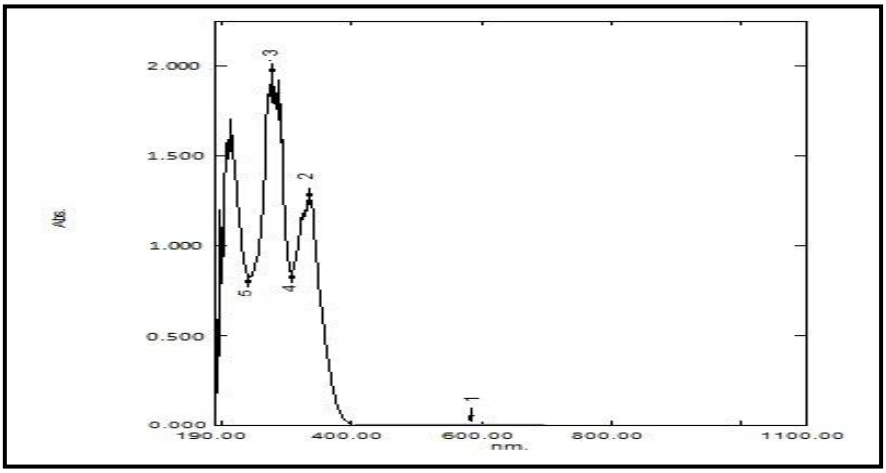


Figure (6): Electronic spectrum of Cr(III) complex.

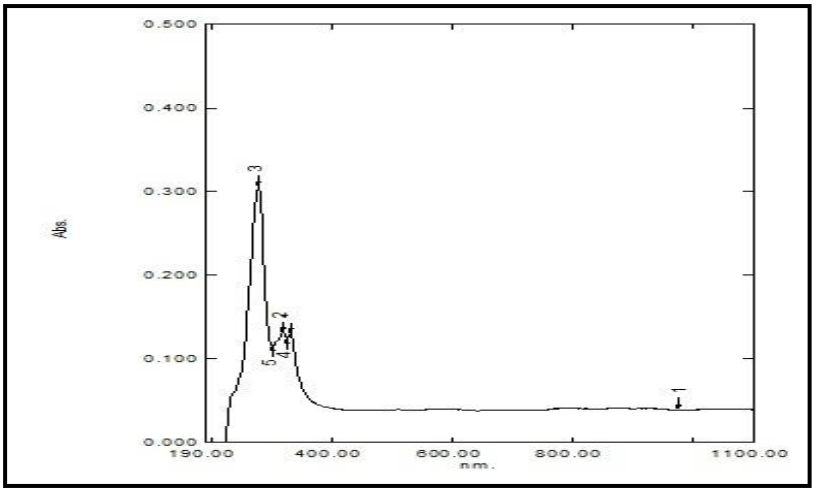


Figure (7) : Electronic spectrum of Pt(IV) complex.

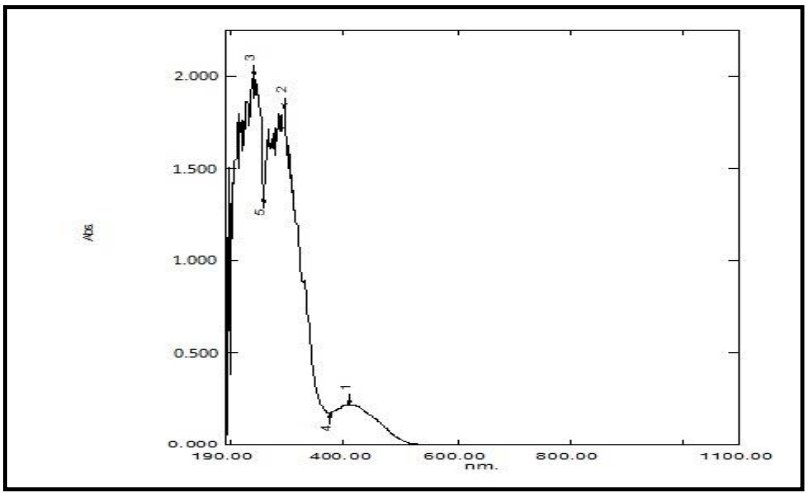
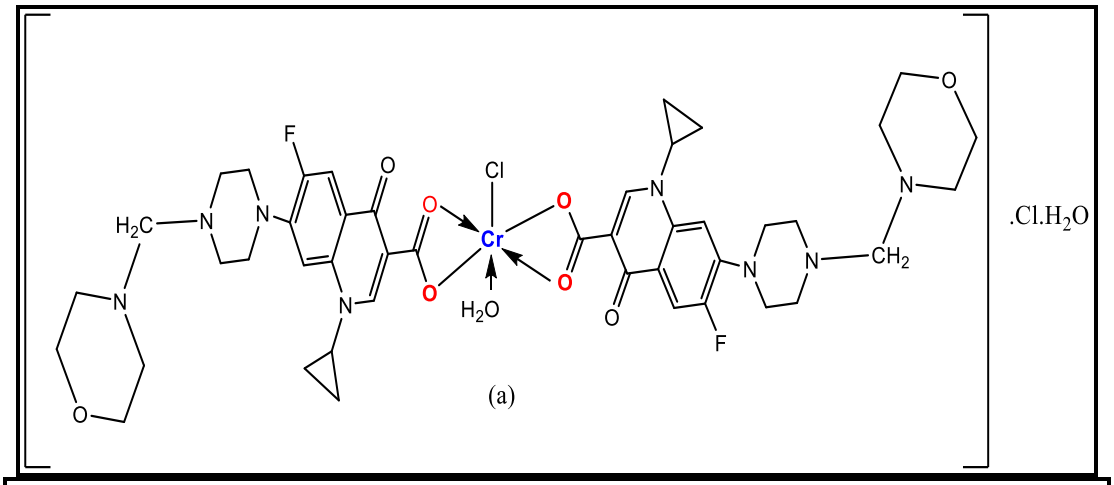
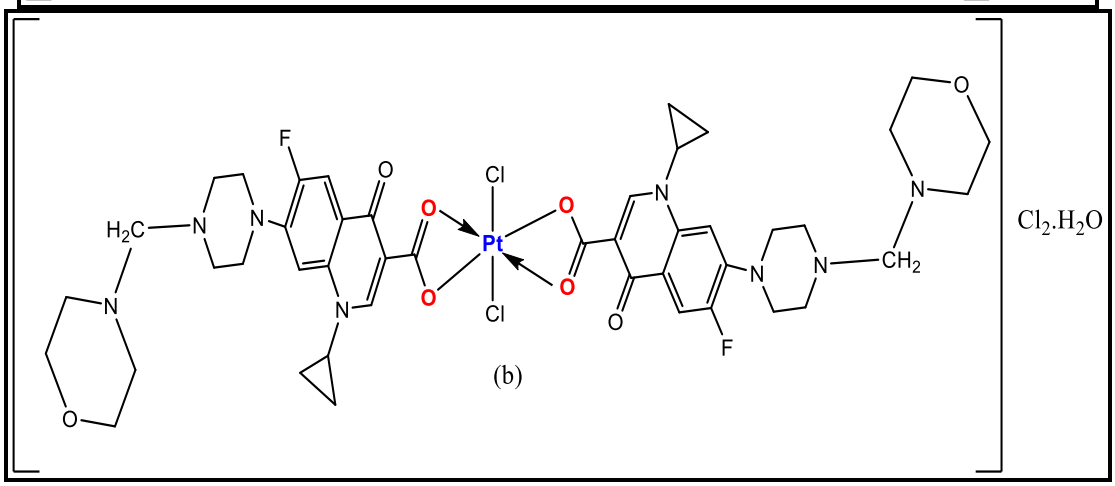


Figure (8): Electronic spectrum of Au(III) complex.

Table (3): Electronic transitions, conductivity, and suggested geometry of metal complexes

Comp.	Absorption cm^{-1}	Assignments	B°	B'	β	15 B'	10Dq	μ *eff* B.M	ϵ max $Mol^{-1}.L^{-1}$	Suggested Geometry
L	43290 38461 33557 32786	$\pi \rightarrow \pi *$ $\pi \rightarrow \pi *$ $n \rightarrow \pi *$ $n \rightarrow \pi *$	---	---	---		---	---	---	---
Cr(III)	16366 24742(Cal.) 35971 47619	$^4A_2g \rightarrow {}^4T_2g$ $^4A_2g \rightarrow {}^4T_1g$ $^4A_2g \rightarrow {}^4T_1g(p)$ L→Cr CT.	918	771	0.83	11565	16962	3.80	38	**Octahedral**
Pt(IV)	10050 28571 33222 50000	$^1A_1g \rightarrow {}^3T_1g$ $^1A_1g \rightarrow {}^1T_1g$ $^1A_1g \rightarrow {}^1T_2g$ (L) → Pt C.T	---	---	---	---	---	0.00	88	**Octahedral**
Au(III)	24509 33898 41322	$^1A_1g \rightarrow {}^1B_1g$ $^1A_1g \rightarrow {}^1Eg$ (L) → Au C.T	---	---	---	---	---	0.00	44	**square planner**





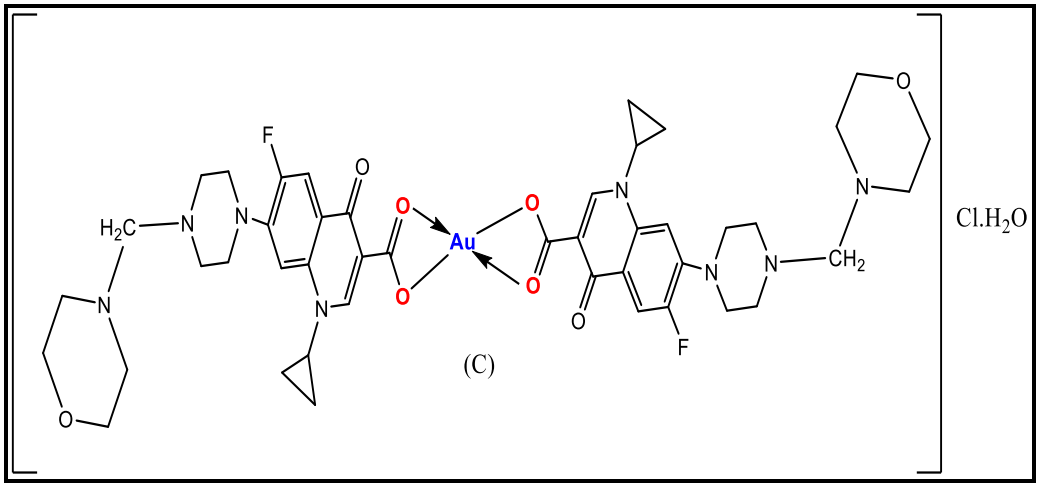


Figure (9): Proposed Structure of (a): Cr (III) complex, (b) Pt(IV) complex and (c) Au(III) complex.

Mass Spectroscopy

The molecular weight of the prepared (L) can be calculated using the mass spectrum, and it can be determined the fragmentation belongs to the compounds being studied. The mass spectrum of the synthesized ligand in (Figure 10), was compatible with the suggested structural formula $C_{22}H_{27}FN_4O_4$. One of the bands, which was found at 430.4 m/z for the ligand, was associated with the molecular ion and was recorded for the ligand in their spectra. Additional distinct peaks revealed in the mass spectra for each ligand were resulting from the successive fragmentation.

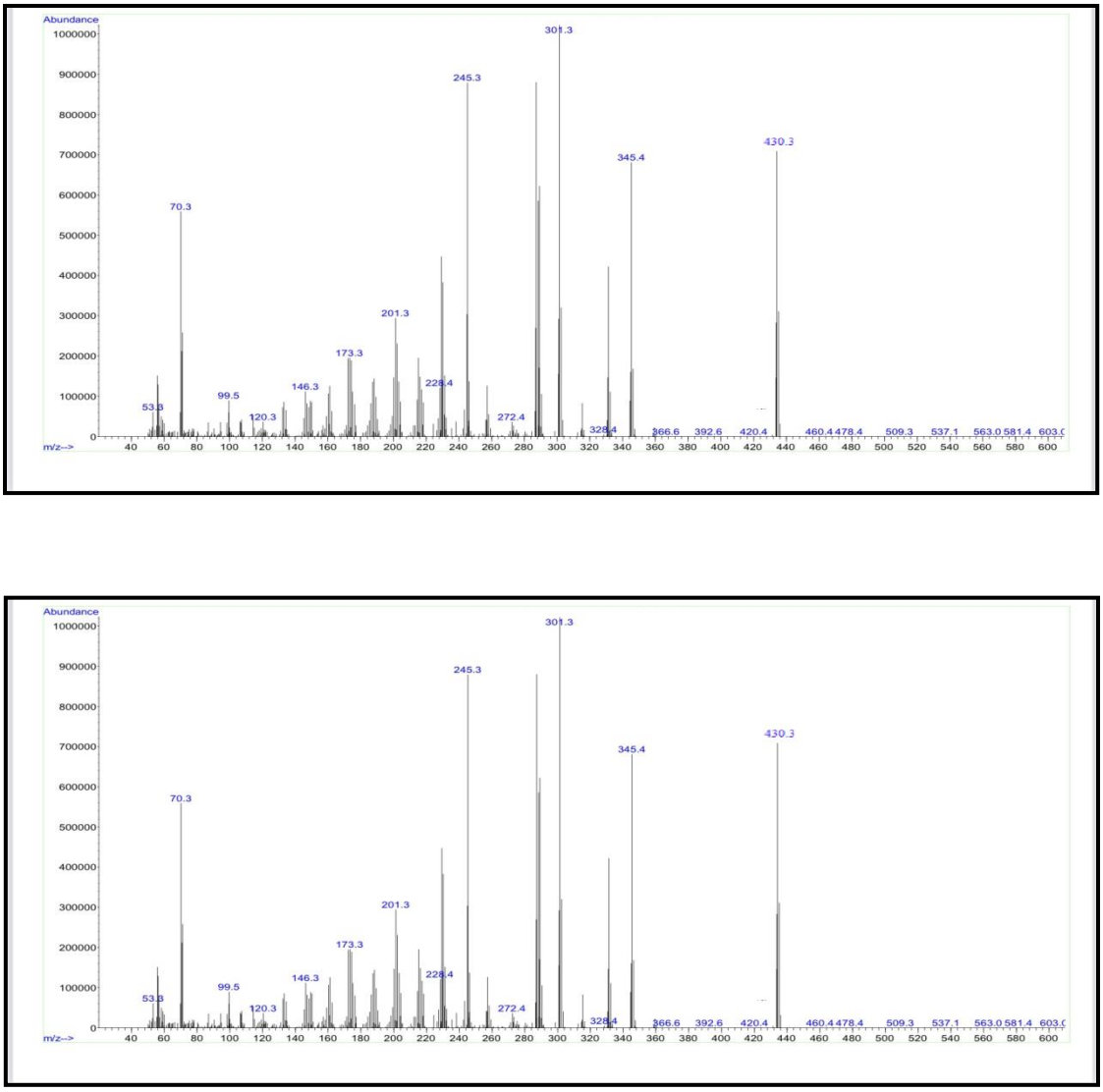


Figure (10): Mass spectrum of Ligand**.**

HNMR Spectra

The [1]HNMR spectrum of the (L) in (Figure 11) shows proton peaks at 2.5 ppm that are likely due to the solvent protons in DMSO. The spectra of the ligand also show proton bands at 1.19, 1.32–1.34, 3.64, and 7.58–8.66 ppm that are characteristic of ciprofloxacin **(Kowalczuk *et al*., 2021)**.

The 1HNMR spectrum of the ligand also display signals peak at δ 7.588 ppm (d,H,H6), 7.87ppm (d,H,H9) respectively which belong to the aromatic protons. The peak which exhibited chemical shift at (5.55) ppm (d,2H,H5 are attributed to the CH_2-N groups of Mannich bases are present in ligand **(Abdulghani& Hussain, 2015)** . The signals peak at δ 3.59 ppm (t, 2H, H12, 13) and δ 2.61 ppm (t, 2H, H11, 14) are attributed to the -O-CH_2 and N-CH_2 proton of the morpholine moiety for the ligand, respectively **(Jandourek *et al*., 2017)**. The [1]HNMR spectra of the Au (III) complex, which are examined in DMSO d6 (Figure 12).

^{13}CNMR Spectra

The^{13}CNMR spectral data for novel ligand has been recorded in DMSO. The 13C-NMR spectrum of the ligand, (Figure 13), shows the following characteristic chemical shift (DMSO-d^6,ppm) .The chemical shift which exhibited at (48. 10 and 67.20) ppm assigned to carbon atoms of morpholine (N-CH_2, and OCH_2,) respectively **(Suresh *et al*., 2013)**.

The comparison of 13CNMR spectra of Au (III) complex formed with the ligand spectrum. There is shifting in the peak of COO^- to δ 184.88 ppm group in the ligand with these metal complex this is confirming the coordination compounds, (Figures 14).

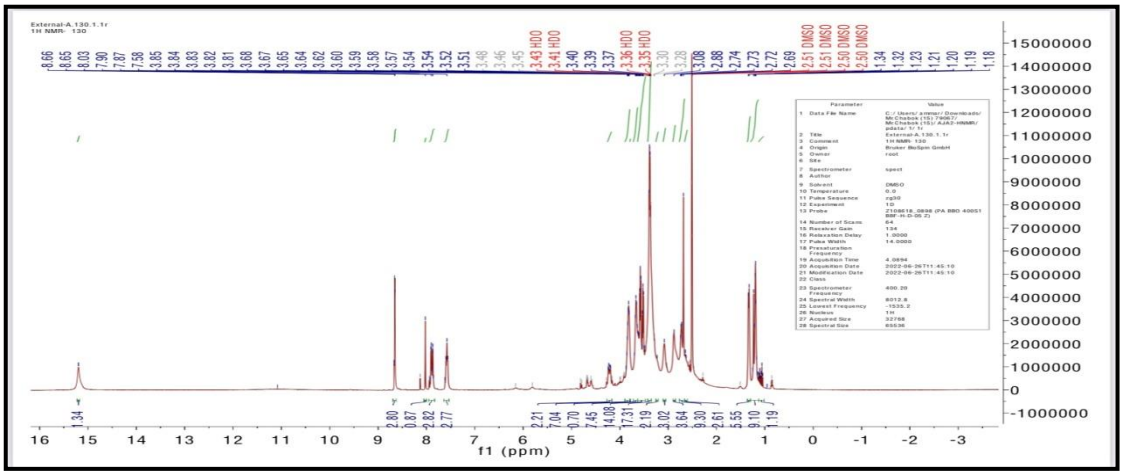


Figure (11): ^{1}HNMR spectrum of (L).

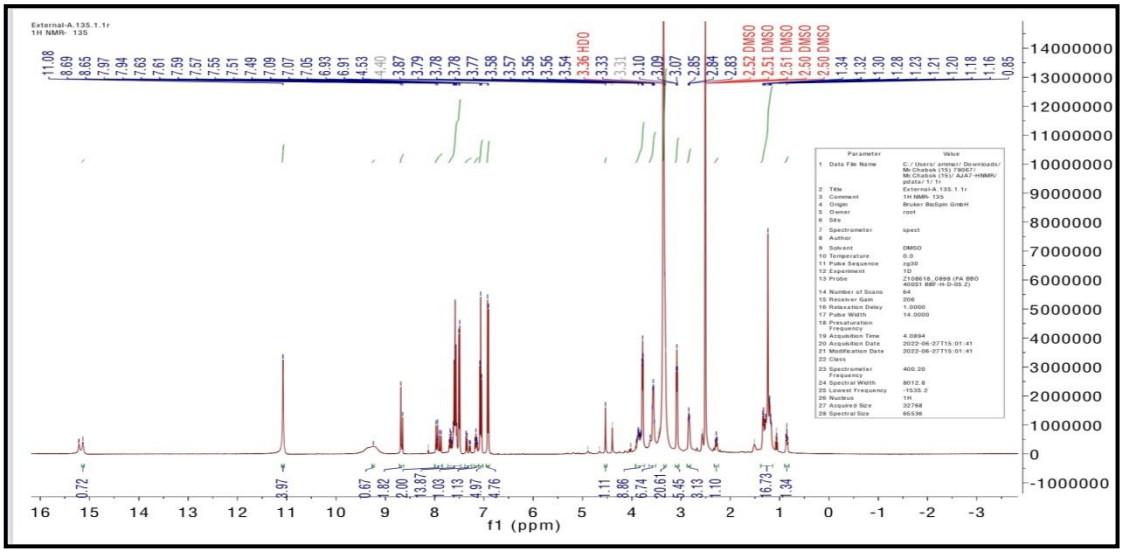


Figure (12): ^{1}HNMR spectrum of Au(III) complex.

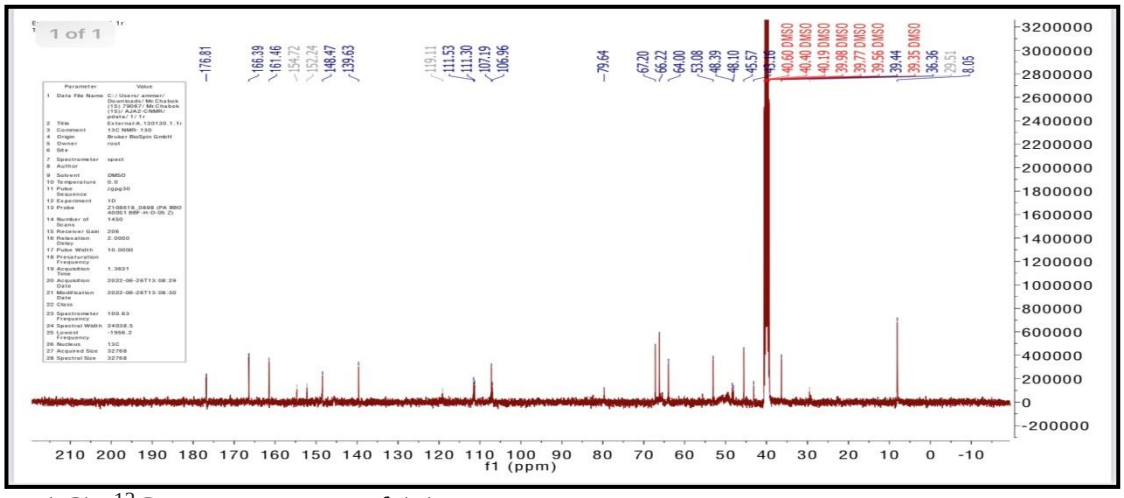


Figure (13): ^{13}CNMR spectrum of (L).

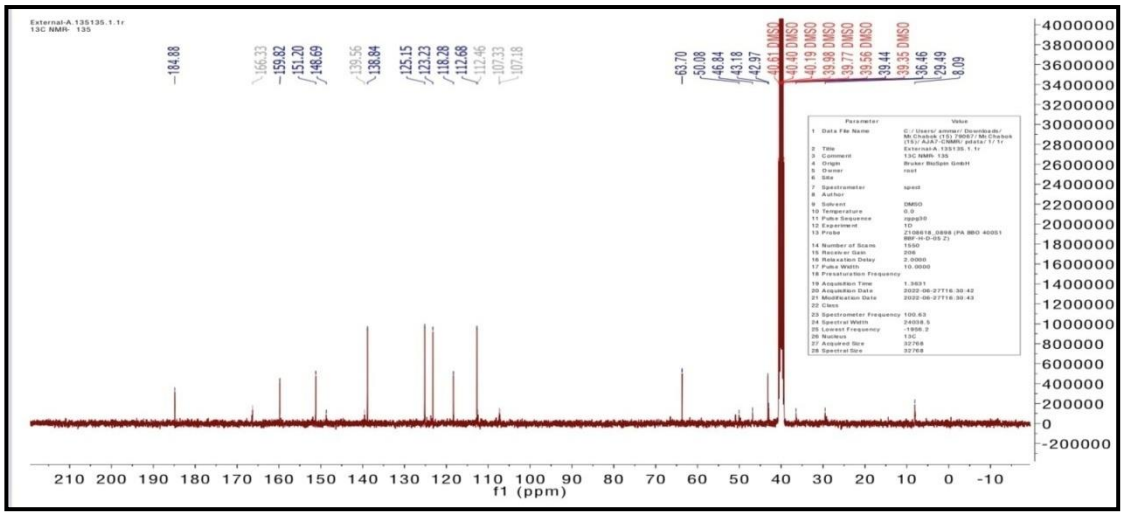


Figure (14): ^{13}CNMR spectrum of Au (III) complex.

Anticancer activity

We have examined the cytotoxic activity and mode of action for the synthesized (L) against MDA cell lines using MTT assay after incubation for 24 hours at 37C and with concentrations (50, 100, 200, and 400 μg/mL) **(Kyhoiesh *et al*., 2021)**. This was done using a cell viability assay. The chosen compounds reduced the growth of the MDA cell lines in a variety of ways, and by comparing the percentage inhibition of cell growth to the control, the level of toxicity was determined. At a 400 g/ml concentration, the ligand inhibited tumor cell death with a (65%) cytotoxic effectiveness. Contrastingly, the Complexes inhibited MDA cell to a concentration of (90.1%), (85.4%), and (80.2%) for Au (III), Cr (III), and Pt(IV), respectively. Furthermore, it can be concluded that all investigated substances were inhibited greatest when concentrations of (400 g/ml) were incubated for (24) hours, and least when concentrations of (50 g/ml). Due to the fact that many Au(I) and Au (III) compounds inhibit the proliferation of cancer cells, gold complexes have demonstrated excellent anticancer therapeutic efficacy. These complexes interact with several intracellular targets, including cysteine, nitrogen bases, glutathione reductase, thioredoxin reductase, and selenocysteine,

causing DNA damage, inhibiting mitochondrial function, and cytotoxicity **(Sun *et al*., 2012)**. The complex Au (III) showed a greater inhibition rate in comparison to the Cr(III) and Pt(IV) complexes. This could be because these complexes have square planner geometry, which is more favorable to cells than octahedral geometry. We conclude that Pt (IV) compounds are considerably more inert based on the fact that octahedral Platinum(IV) complexes can substitute ligands through a dissociative mechanism as compared to an associative mechanism for Platinum (II) **(Warad *et al*., 2013).**The overall results in this section show that coordination of the ligand with specific metal ions resulted in the increased anticancer effects. The positive charge of the metal ion increased the coordinating protonated ligand's acidity, enhancing its ability to form stronger negatively charged hydrogen bonds with the DNA of cancer cells, which resulted in the higher cytotoxicity shown with metal complexes. Design changes in the compounds geometry and coordination site tend to get the most effects on biological activity.

Table (4): Evaluation of cytotoxicity of ligand and their complexes against MDA cell lines after incubation (24 hrs) at (37 ◦C).

Comp.	%Cell Inhibition			
	Conc. $\mu g/ml$			
	400	200	100	50
L	65	50	27	15
Au L	90.1	66	42	30
Cr L	85.4	58	35	22
Pt L	80.2	58	38	18

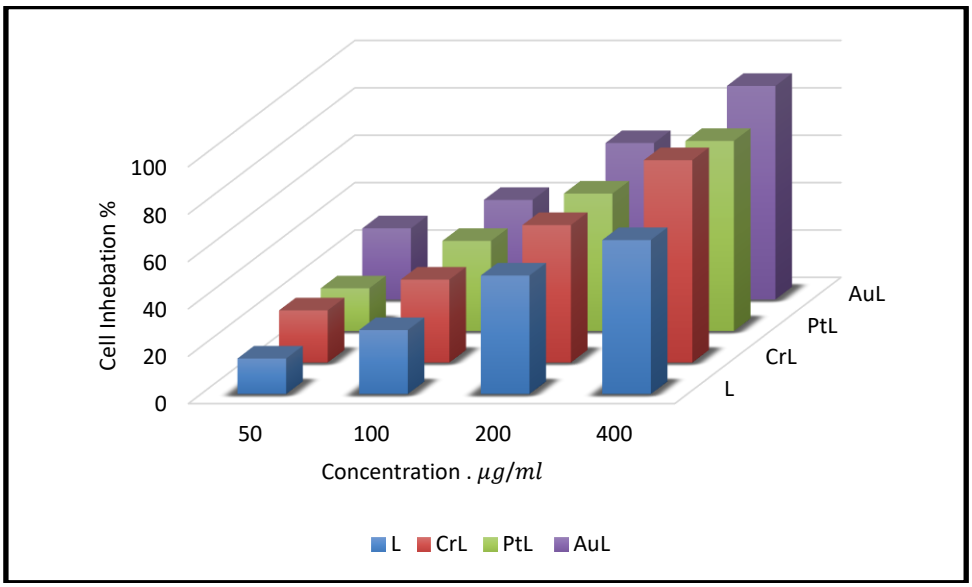


Figure (15): The percentage inhibition in (400,200,100,50 $\mu g/ml$) after exposure to ligand and its complex at 24 hrs.

CONCLUSION

This study focuses on the synthesis novel bidentate O donor mannich base (L) from morpholine and ciprofloxacin, a series of Cr (III), Pt (IV), and Au (III) complexes were synthesized. These complexes were then examined using various physicochemical methods. The physicochemical data suggested that the Au (III) complex has a four- coordinate square planner structure while the Cr (III) and Pt (IV) complexes have six-coordinate octahedral structures. The complexes molar conductance values confirmed the electrolytic nature of the compounds. Also, to evaluate the anticancer effects of the mannich base ligand (L) and its complexes on MDA cell lines. At high concentrations ($400\ \mu g/ml$), it was observed that all complexes were more active than the free ligand, and Au (III) complexes exhibited stronger anti-proliferative properties at all applied concentrations.

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وحماية المستهلك

PREPARATION OF NEW COMPLEXES OF METHYL METHACRYLAT WITH POLYVINYL ALCOHOL AND STUDY OF SOME ENVIRONMENTAL APPLICATIONS

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ABSTRACT

A new copolymer from Methyl methacrylate and polyvinyl alcohol [methyl-5-hydroxy-2-methylhexanoate] and its complexes were synthesized for some metals (Cr^{+3}, Mn^{+2}, Fe^{+3}, Co^{+2}, Ni^{+2}, Cu^{+2}, Zn^{+2}, Cd^{+2}). This ligand and its metal complexes were characterized using (FTIR) spectral, UV-Vis spectroscopy, conductivity, magnetic moment, and Thermo Gravimetric analysis. The nanoparticles for two complexes were characterized using x-ray diffraction, scanning electron microscope, and atomic force microscope. Zeolite 5A was prepared from local kaolin by hydrothermal preparation, then characterization, and used as a supporting material with prepared copolymer as a composite to remove several metals from polluted water taken from industrial water electric power stations in Dora and South of Baghdad. Trace concentrations of these metals were estimated before and after applying the prepared copolymer by atomic absorption spectroscopy. The removal using composite materials is significantly more effective, with the concentration recorded as very low and the concentrations of some metal ions like Fe^{+2}, which completely disappeared from polluted water according to a polluted water analysis before and after using the produced compounds.

Keywords: Removal of metal contaminants, Copolymer, Methyl methacrylate, Metal complexes, and Zeolite.

تحضير معقدات جديدة لمثيل ميثا اكريلت مع بولي فنيل الكحول ودراسة بعض التطبيقات البيئية .

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الخلاصة:

في هذا العمل حضرنا بوليمر مشترك جديد من ميثيل ميثا أكريلات مع بولي فنيل الكحول [ميثيل-5-هيدروكسي-2-ميثيل هكسانوات] مع بعض العناصر الثقيلة (Cr^{+3}، Mn^{+2}، Fe^{+3}، Co^{+2}، Ni^{+2}، Cu^{+2}، Zn^{+2}، Cd^{+2}) لإنتاج المعقدات المقابلة. تم تشخيص ميثيل-5- هيدروكسي-2-ميثيل هكسانوات المحضر ومعقداته باستخدام طيف الأشعة تحت الحمراء والتحليل الطيفي المرئي للأشعة فوق البنفسجية، والتوصيلية والعزم المغناطيسي، وانحراف الأشعة السينية، ومجهر المسح الإلكتروني ومجهر القوة الذرية. تم استخدام البوليمر المشترك المحضر لإزالة عدد من العناصر من المياه الملوثة المسحوبة من المياه الصناعية لمحطات توليد الطاقة الكهربائية في الدورة وجنوب بغداد وتقدير التراكيز الضئيلة لهذه العناصر قبل وبعد استخدام البوليمر المشترك المحضر باستخدام مطيافية الامتصاص الذري. تعتبر الإزالة باستخدام المواد المتراكبة أكثر فاعلية بشكل ملحوظ مع تسجيل التركيز على أنه

[*]**The research is extracted from the doctoral thesis of the first researcher.**

منخفض جدًا واختفت تمامًا تراكيز بعض أيونات المعادن مثل Fe^{+2} من المياه الملوثة وفقًا لتحليل المياه الملوثة قبل وبعد استخدام المركبات المحضرة.

الكلمات المفتاحية: إزالة ملوثات المعادن ، بوليمر مشترك ، مثيل ميثا اكريلايت، معقدات المعادن والزيولايت.

INTRODUCTION

Water contamination continues to pose a threat to people's health on a global scale **(Soubh *el al.*, 2018)**. Metal poisoning offers a severe risk because of its toxicity, non-biodegradability, and bioaccumulation in the food chain **(Ashvinder, *el al.*, 2021)**. Industrial waste is the leading cause of heavy metal contamination in water systems. The effective removal of hazardous ions from wastewater is an important and urgent issue due to its negative and direct effects on flora and fauna. As a result of heavy metals ingested by other species in the food chain, their carcinogenic effects on humans and animals were increased. These metals are absorbed by plants, which then pass them to animals and humans **(Mahmoud *el al.*, 2020)**.

A hydrophilic polymeric network forms the three-dimensional structures known as hydrogels. They are cross-linked polymer networks containing water in them. Hydrophilic functional groups connected to the polymeric backbone absorb water, whereas cross-links between network chains allow them to resist dissolution **(Balbir *el al.*, 2021)**. Alcohols, carboxylic acids, amides, and other hydrophilic groups are among the hydrophilic groups that give hydrogels their hydrophilicity **(Tain *el al.*, 2021)**. Polymer gels play an essential role in many technical fields like gene delivery and drug delivery **(Noreen *el al.*, 2022)**, scaffolds for tissue engineering **(Abdullah *el al.*, 2021)** and superabsorbent materials because of their exceptional characteristics, such as biocompatibility and smart response behavior **(Pishnamazi *el al.*,2021)**. In the past, polymers made from methyl methacrylate have been used to remove dyes in environmentally friendly ways **(Uzma *el al.*, 2022)**.

Zeolites are available in two types: natural zeolites, which are non-porous and synthetic zeolites, which are porous and have a structure. They are prepared by heating soda ash, feldspar, and china clay together. Compared to natural zeolites, these have a higher exchange capacity per unit weight. **(Karmen & Anamarija, 2022)**.

Zeolites can potentially remove various chemicals, such as heavy metals, organic compounds, dyes, pigments, reagents, and nitrogen compounds, due to their cationic exchange negative charge features and relatively low production cost **(Luciano *el al.*, 2022)**, They are used as efficient adsorbents for various environmental pollutants, especially in water treatment techniques for removing heavy metals due to the porous structure of zeolites and other special features **(Veena *el al.*, 2021)**.

MATERIALS AND METHODS

Methyl methacrylate provided by (P.D.H). Absolute Ethanol (B.D.H), PVAprovided by (SIGMA), ($CrCl_3.H_2O$, $MnCl_2.4H_2O$, $FeCl_3$, $CoCl_2.6H_2O$, $NiCl_2.6H_2O$, $CuCl_2.2H_2O$, $ZnCl_2$, and $CdCl_2.H_2O$) provided by (B.D.H).

Instruments

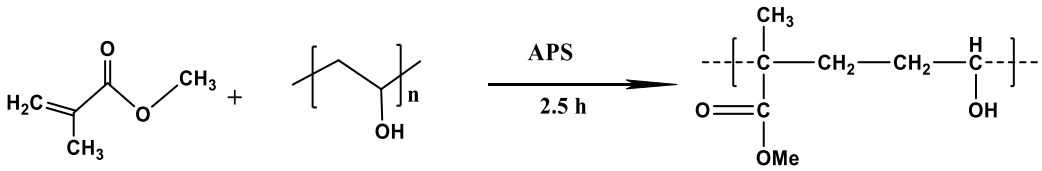
Melting points of the synthesized compounds were measured by GMMallenKampm. MF-370 devised electro-thermal at the University of Baghdad, College of Sciences for Women. Fourier Transform Infrared (FTIR) spectra were obtained using a SHIMADZUE FT-IR 8400S Fourier transform within the wavenumber region between 4000 - 400 cm^{-1} using a KBr disc

and 4000 - 200 cm^{-1} using a CsI disc. electronic spectra for compounds in the (UV-Visible) region (200-1100) nm were recorded using a SHIMADZUE 1800 Double Beam UV-Visible spectrophotometer at the University of Baghdad. ^{1}H-NMR was performed using a Bruker Ultra Shield 500 MHz at Tehran University, Iran. Thermal analyses (TGA) of samples were performed under nitrogen atmospheres at 25˚C-900˚C and a heating rate of 20˚C/min using STA500 Germany in Tehran University, Iran. Molar conductivity measurements ($\mu s.cm^{-1}$) for metal complexes (10^{-3} M) in Ethanol at room temperature were carried out using LASSCO Digital Conductivity Meter. Magnetic moments (eff. B.M) for the prepared complexes in the solid state at room temperature were measured according to Faraday's method using Bruker Magnet B.M-6.

Synthesis

Synthesis of copolymer [methyl (S)-5-hydroxy-2, 2-dimethylhexanoate] (PMMA-PVA) ligand

In around-bottomed flask with a constant oxygen stream in an inert atmosphere of nitrogen, the (PMMA-PVA)-copolymer prepared by dissolving (1g, 0.01 mol) of polyvinyl alcohol (PVA) in a mixture of (2.5ml: 12.5ml) of absolute ethanol and deionized water in the presence of (0.5 g) from ammonium persulfate (APS) as a radical initiator which dissolved in 2ml of water with continuously stirring at room temperature (25^{o}C) then adding (1g, 0.01 mol) of methyl methacrylate; the mixture was refluxed at 70-80°C for 2.5 hrs in a water bath. Then the pink product was dried at room temperature for a whole night before being washed with diethyl ether (Scheme 1).



methyl metha acrylate **poly vanyl alcohol (PVA)**

methyl (*S*)-5-hydroxy-2,2-dimethylhexanoate
Chemical Formula: $C_9H_{18}O_3$
Molecular Weight: 174.2

(PMMA-PVA)

Scheme (1): Preparation of Polymethylmetha acrylate –PVA (PMMA-PVA) ligand

Synthesis of [methyl-5-hydroxy-2, 2-dimethylhexanoate] (PMMA-PVA) ligand complexes

To prepare ligand-complexes of poly Methyl methacrylate-PVA (PMMA-PVA) at a ratio of 2:1 from ligand to metal, (0.0576 g, 0.002 mol) of the ligand was dissolved in (5 ml) of distilled water and (20 ml) of absolute ethanol with continuous stirring in a condensation flask until it dissolved. The corresponding weight of (0.001 mol) metal salt dissolved in (10 ml) of absolute ethanol was added. The mixture was refluxed with continuous stirring at 45°C for 3hrs. The product was placed in a watch glass and let dry at room temperature.

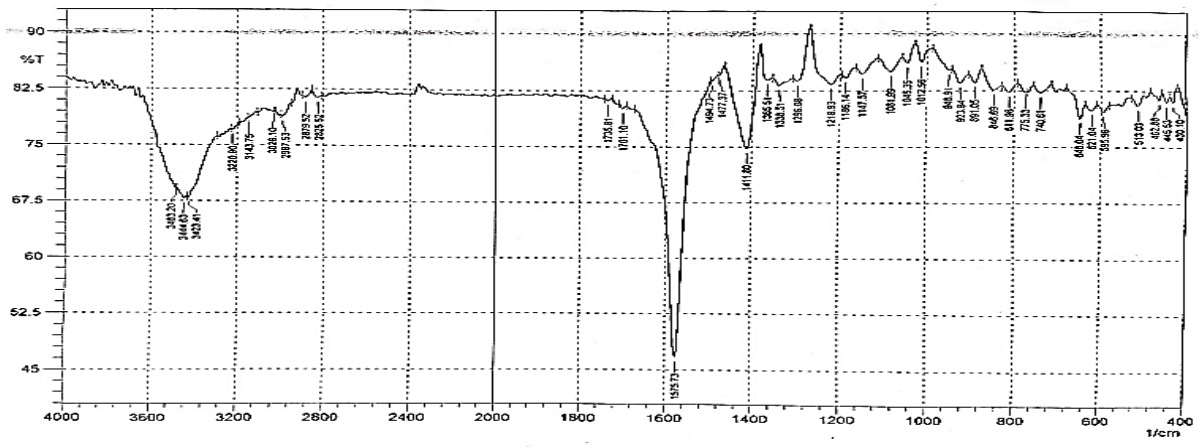
RESULTS AND DISSCUSIONS

FTIR Spectra

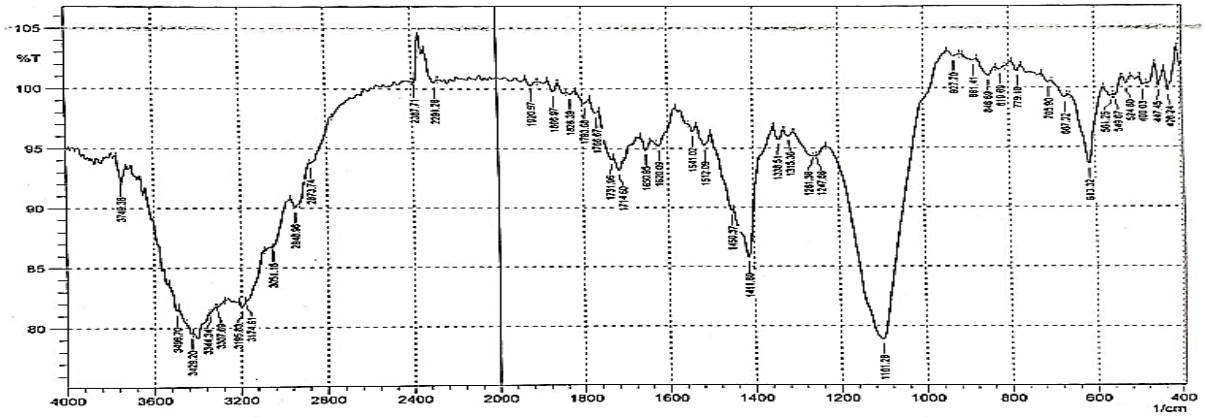
Specific vibrations of chemical bonds or functional groups within molecules were reflected as peaks in FTIR spectra **(Mathur *el al*.,2018)**. It is shown in Figure 1 that KBr FTIR spectroscopy in the range of 4000-400 cm^{-1} and CsI FTIR spectroscopy in the range of 4000-200 cm^{-1} were used to determine the experimental and theoretical structure of the (PMMA-PVA) polymer complexes. Experimental FT-IR showed a distinctive band of the -OH functional group in the range of 3446-3213 cm^{-1} in the IR spectra of the compounds, suggesting its nonparticipation in coordinate bond formation towards all metal ions. Furthermore, as shown in Figure 1. b, the peak's width expanded after coordination. This may be due to moisture in the sample or complexes containing coordinated water molecules **(Abdi *el al*., 2020; Anacona *el al*., 2021)**. Other stretching bands were found at 1701-1616 cm^{-1} for υ(C=O) carboxylic of ester **(Nabeel *et al*., 2022; Tabarek & Ahlam 2023)**. The loss of the C=O signal, originally at 1616-1650 cm^{-1}, was consistent, providing strong evidence for the coordination of Ligand (PMMA-PVA) towards the central metal ion, Figure 1. Table 1 shows that the typical peak at ~1700 cm^{-1} for compounds containing the (C=O) ester group relocated to 1600 cm^{-1}**(Muna *et al*., 2022)**. In complexes, the carbonyl group was weakened after bonding due to creating a coordination bond between the oxygen of the C=O group and the central metal ion, as indicated by the peak displacement.

Table (1): The FT-IR spectrum of the synthesized ligand and its complexes.

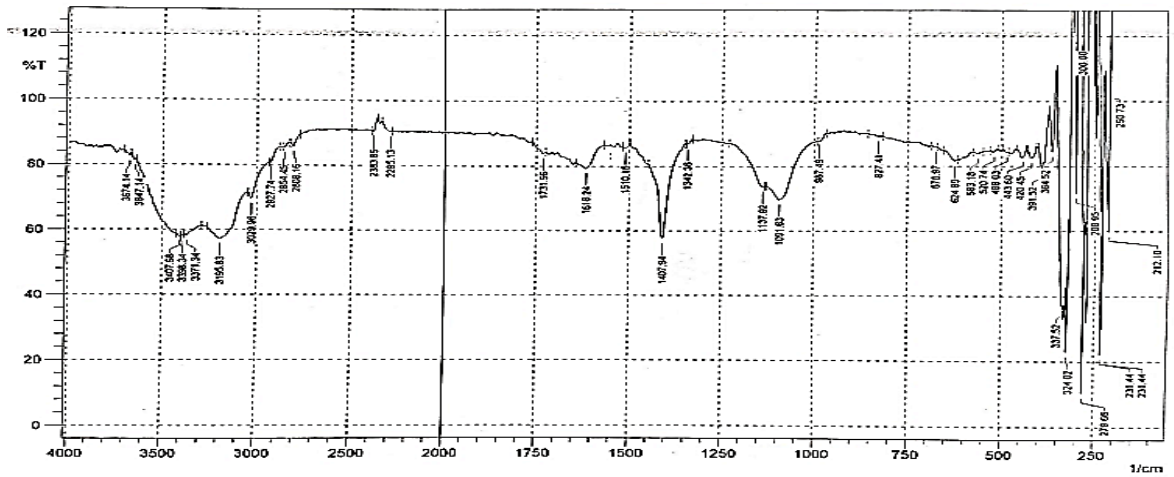
Comp.	υ(OH)	υ(COO) ester	υ(C-OCH_3)	υ(CH-CH_2)	υ(M-O)	υ(M-Cl)	Others.
L	3444	1701	1147	2987-2879	-		
CrL	3434	1647	1124	2921-2871	599	335	w H_2O = 910 ρ OH =846
MnL	3446	1622	1118	2947-2860	559	329	w H_2O = 984 ρ OH =833
FeL	3429	1623	1118	2929-2873	597	327	w H_2O = 997 ρ OH =864
CoL	3438	1649	1083	2925-2856	557	316	w H_2O = 983 ρ OH =846
NiL	3240	1616	1095	2927-2854	567	324	w H_2O = 987 ρ OH =827
CuL	3417	1714	1101	2948-2873	549	335	w H_2O = 927 ρ OH =846
ZnL	3240	1620	1095	2927-2856	567	331	w H_2O = 983 ρ OH =881
CdL	3213	1650	1101	2950-2875	520	329	w H_2O = 987 ρ OH =875



A



B



c

Figure (1): The FTIR Spectra for (a) PMMA-PVA -L (b) NiL (c) CuL

The Electronic spectra, (UV-Vis) of ligand and its complexes:

Intense absorption at (323) nm (20661) cm^{-1} in the UV-Vis spectrum of (PMMA-PVA)-ligand was ascribed to the (n→π*) transition, while intense absorption at (229) nm (43668) cm^{-1} was ascribed to the (π→π*) transition, Figure 2. Table 3 contains information about the spectra, molar conductivity, and magnetic moments of all metal complexes of the (PMMA-PVA)-ligand in ethanol for MnL, FeL, CdL complexes and in DMSO for CrL, CoL, NiL, CuL, and ZnL complexes.

Where three bands of complexes were uncultivated: Three bands, corresponding to $^6A_1g\rightarrow{}^4T_1g$ (G), $^6A_1g\rightarrow{}^4T_2g$ (G), and $^6A_1g\rightarrow{}^4A_2g$+Eg (G) were seen for the Mn (II)-PMMA-PVA complex at 12468, 20080 and 28248 cm^{-1} respectively (**Al-Issa *el al*., 2017)**.

Co (II)-PMMA-PVA complex showed three bands in the visible region with an average of 14749cm^{-1}. This value which assigned to transition $^4T_1g\rightarrow{}^4T_2g$ (F) **(Nuha&Naser, 2023)** and a value with an average of 16260 cm^{-1} for $\upsilon^4T_1g\rightarrow{}^4T_1$ (P) while $^4T_1g\rightarrow{}^4A_2g$ appeared at 17211 and it forbidden transition Scheme 2(a).

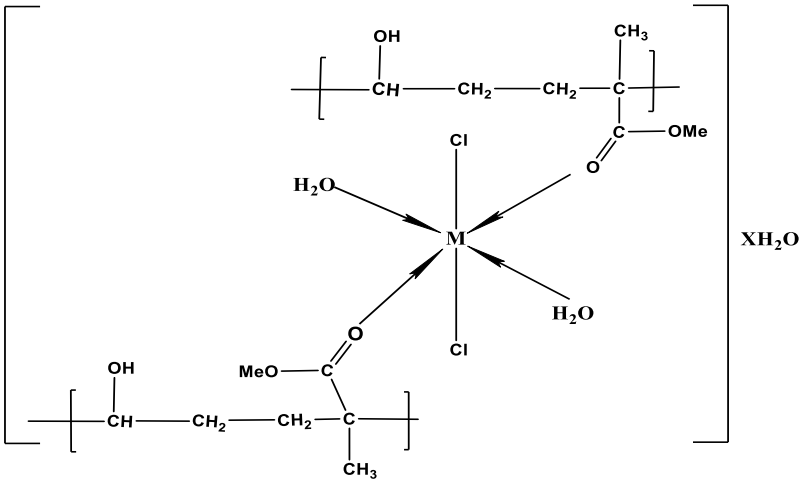
The spectrum of Cr (III) complex olive showed three absorption bands at (619, 451, and 259) nm (16155, 222172, and 38610) cm^{-1} assigned to $^4A_2g\rightarrow{}^4T_2g$, $^4A_2g_{(F)}\rightarrow{}^4T_1g$ and $^4A_2g_{(F)}\rightarrow{}^4A_2g$ transitions, suggesting an octahedral geometry **(Sahar *el al*., 2020; Rasha& Abbas, 2023).)**.

The Fe (III) complex spectrum showed three bands at 682, 590, 416 nm (14662, 16949, 24038) cm^{-1} assigned to $^6A_1g\rightarrow{}^4T_1g$,$^6A_1g\rightarrow{}^4T_2g$, and $^6A_1g\rightarrow{}^4A_1g$ +^4Eg respectively. Transition at 336 nm (29776) cm^{-1} attributed to C.T (LMCT) and that suggesting an octahedral geometry **(Anum *el al*., 2022** the magnetic moment value is 5.6 BM, Scheme 2(c).

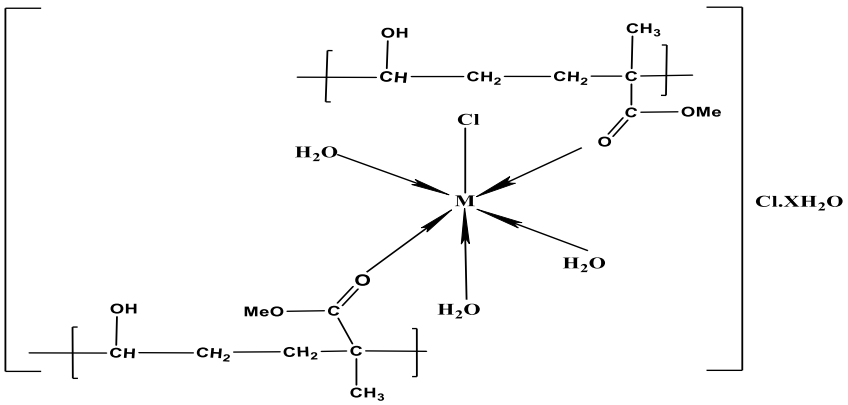
For Ni(II) complex spectrum showed three bands at (920, 769, 502, 385, and 290) nm (10809, 13003, 19920, 25974, and 34482)cm^{-1} assigned to $^3A_2g\rightarrow Eg$, $^3A_2g\rightarrow{}^3T_2g$,$^3A_2g\rightarrow{}^3T_1g_{(F)}$ and $^3A_2g\rightarrow{}^3T_1g_{(P)}$ transition, respectively; the magnetic moment value is 2.3 BM suggesting an octahedral geometry **(Sahar *el al*., 2018*;* Veyan *el al*., 2020)** , Scheme 2 (b)

Cu (II) complex spectrum showed one band at 920nm (10869) cm^{-1}, assigned to $^2Eg\rightarrow{}^2T_2g$ and C.T transition; the magnetic moment value is 1.2 BM suggesting an octahedral geometry, Scheme1 (a) **(Sahar *el al*., 2021)**.

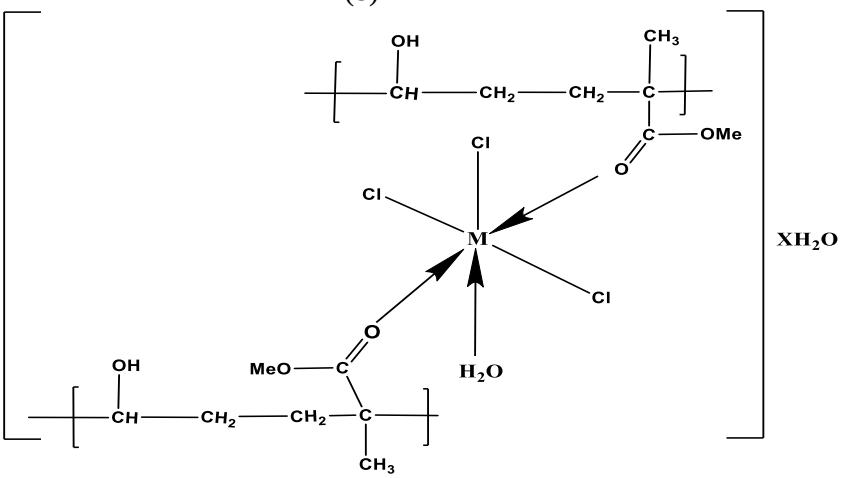
Finally, the magnetic moment value is diamagnetic for both Zn (II) and Cd (II) complexes (ZnL) and (CdL), which is attributed to metal to ligand charge transfer, but the spectra show no d-d electronic transitions in the visible region. The absorption bands are 505 and 498 nm (34364, 20080) cm^{-1} **(Haneen & Sahar, 2022)**. Scheme1(a)



(a) M= Mn, Cu, Zn, Cd, Co



(b) M= Ni



(c) M=Cr,Fe

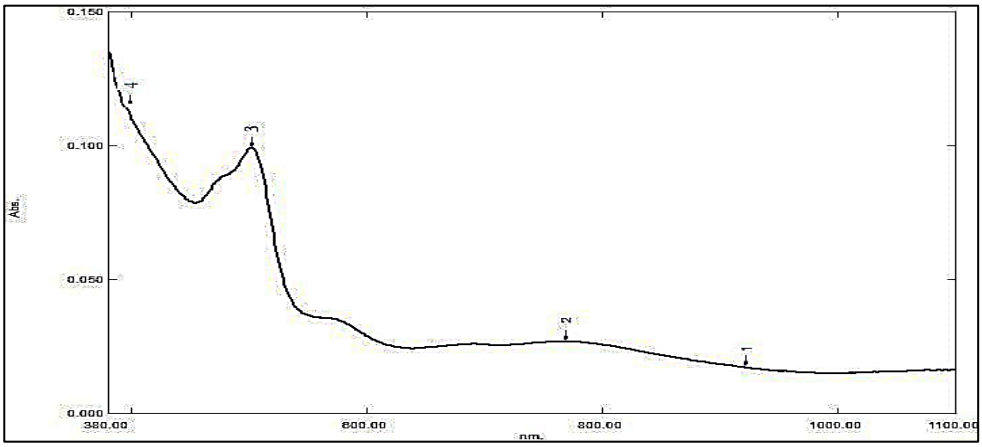
Scheme (2). The geometrical structure of a. Octahedral [ML_2 (Cl_2)]. XH_2O b. Octahedral [ML_2Cl] Cl.XH_2O c. Octahedral [ML_2Cl_3].XH_2O

Table (2): Electronic spectra, spectral parameters, molar conductivity, and μ*eff* of L- metal complexes.

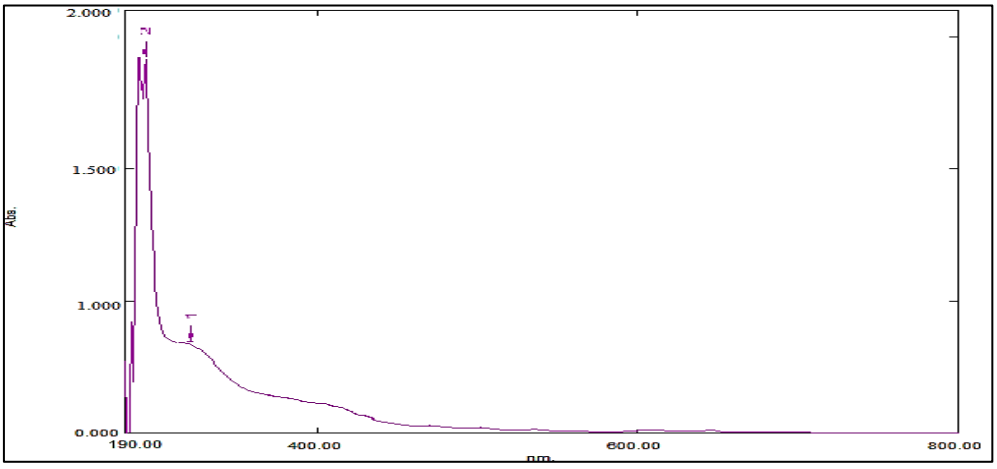
Comp.	λnm	ῡcm-	Assignments	Molarcond.	μeff(B.M)	Structure
L	323	30959	n→π*			
	229	43668	π→π*			
Cr-L	619	16155	4A2g→4T2g	6.1	3.7	Octahedral
	451	22172	4A2g→4T1g			
	397(cal.)	25188	4A2g→4T1g			
	259	38610	Intra ligand			
Mn-L	802	12468	6A1g→4T1g(G)	27	5.2	Octahedral
	498	20080	6A1g→4T2g(G)			
	354(cal.)	28248	6A1g→4A2g+4Eg(G)			
	275	36363	Intra ligand			
Fe-L	682	14662	6A1g→4T1g	17.3	5.6	Octahedral
	590	16949	6A1g→4T2g			
	416(cal.)	24038	6A1g→4A1g+4Eg			
	336	29776	C.T (LMCT)			
	266	37594	Intra ligand			
Co-L	678	14749	4T1g→ 4T2g (F)	11.5	5.3	Octahedral
	615	16260	4T1g→4T1g(P)			
	581	17211	4T1g →4A2g			
	275	36363	Intra ligand			
Ni-L	920	10869	3A2g→Eg	42	2.3	Octahedral
	769	13003	3A2g→3T2g			
	502	19920	3A2g→3T1g(F)			
	385	25974	3A2g→3T1g(p)			
	290	34482	Intra ligand			
Cu-L	920	10869	2Eg→2T2g	8.9	1.2	Octahedral
	296	33783	C.T			
	209	47846	Intra ligand			
Zn-L	505	34364	C.T	17.3	Diam.	Octahedral
	268	28571	Intra ligand			
Cd-L	498	20080	C.T	3.5	Diam.	Octahedral
	286	33223	Intra ligand			

Table (3): Physical properties of the ligand and its complexes.

Compounds	M.p°C (dec) °C	Color
L	180-182	Pink
FeL	190-192	Yellow
CuL	138-140	greenish yellow
MnL	170-172	Pink
CdL	188-190	Off-white
CrL	248-250	Olive
NiL	198-200	Yellow
CoL	240-242	turquoise
ZnL	230-232	Light Pink



(a)



(b)

Figure (2): Electron spectrum of a. (PMMA-PVA) ligand b. NiL complexe

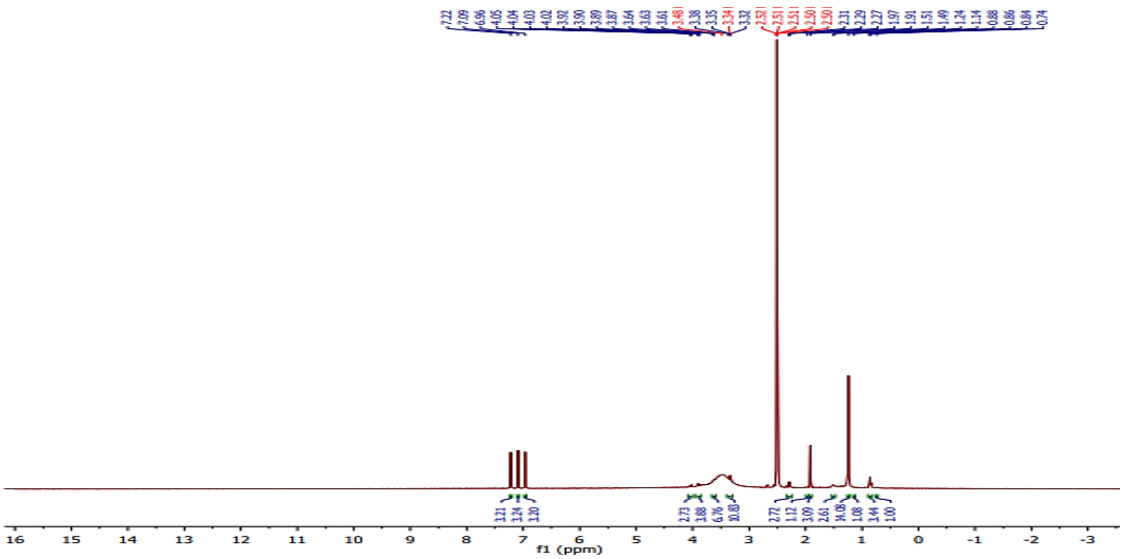
HNMR spectrum

Nuclear magnetic resonance spectroscopy (1HNMR) is essential for studying substances and their structures. The ^{1}H-NMR technique was used to characterize the synthetic polymer. Figure 3 and Table 4 showed that the methyl and methylene protons in the PMMA-PVA structure correspond to a signal at 1.16–1.8 ppm and 2.0–2.5 ppm, respectively. The peaks at δ 3.61-3.92 ppm were identified as protons for the methylene group. The Proton of the hydroxyl group was identified as the source of the peak at δ 10.83 ppm **(Haroon *el al.*, 2021)**. The presence of the Methyl methacrylate chain, and hence the occurrence of polymerization to generate the desired product, was confirmed by the ^{1}HNMR spectrum.

Table (4): ^{1}H NMR spectral data of L-PMMA-PVA and CrL complexe

Chemical shifts (ppm)	Assignments in DMSO
1.16 - 1.8 ppm and 2.0 - 2.5 ppm	methyl and methylene protons
3.61 - 3.92ppm	protons for methylene group
10.83 ppm	Hydroxyl group proton (OH)

(a)



(b)

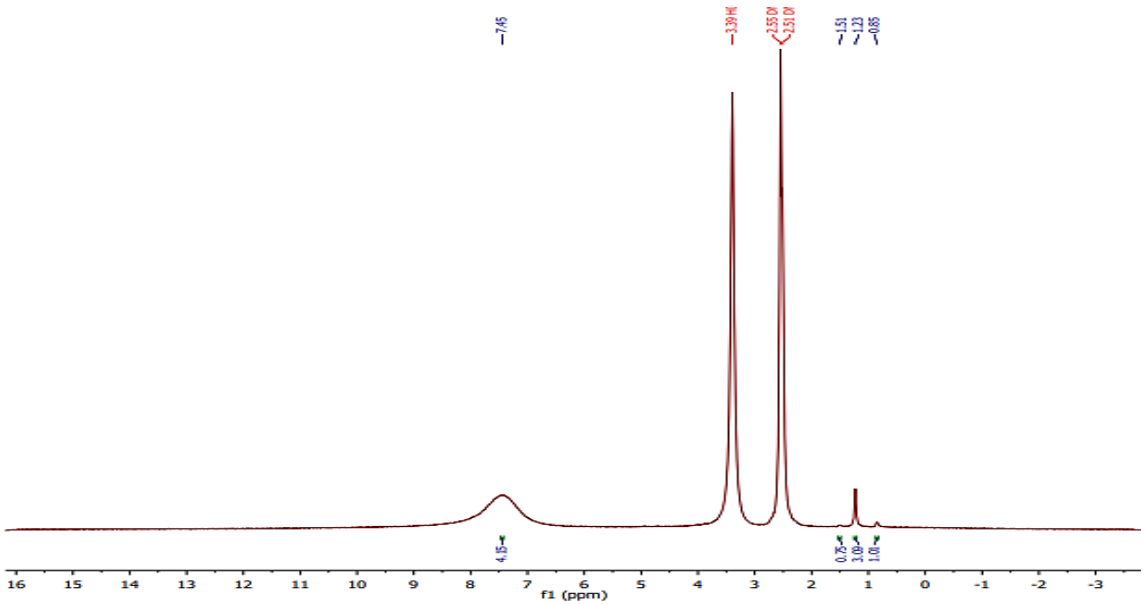


Figure (3): HNMR spectrum of (a) L (PMMA- PVA) and (b) CrL

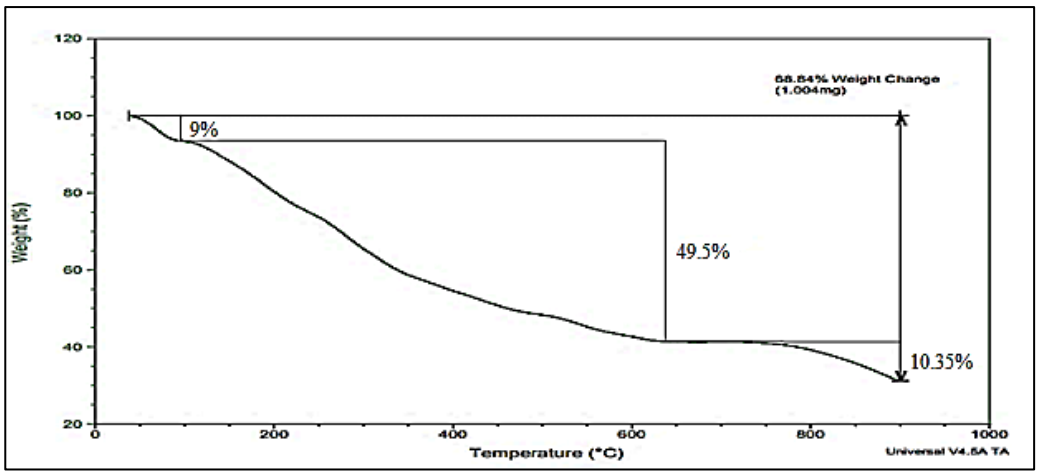
Thermal Gravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was used to understand the effects of temperature and time on the weight of polymeric materials. Polymeric materials can undergo weight changes due to decomposition, oxidation reactions, and physical processes, including sublimation, evaporation, and desorption **(Dilkes *el al.*, 2019)**. TGA-curve of two (PMMA-PVA)-complexes as illustrated in Figure 4 and Table 5. Thermal stability of Cr and Fe complexes studied by TGA. The TGA tests were performed at ~30 ºC to 900 ºC. The CrL complex was shown to be disassembled into three parts in Figure 4.a. First, a small loss of 10.31% from total mass when compared to a temperature of 110C suggests that water molecules were evaporated from the sample. The polymer (ligand) chains, CO_2, CO, and Cl fragments caused a 40.34% weight loss when heated to around 625ºC. The remaining complex lost 7.8% of its mass upon decomposing from 625-900 ºC **(Ashok *el al.*, 2020)**. The degradation of polymer residues for the coordinated ligand molecules accounted for the modest increase in degradation observed compared to the previous stage. Upon further heating, the polymer chains and metal oxide residue were left as the final residue at 41.55 %. Almost similar changes were observed in the FeL complexe TGA curve (Figure 4. b). However, the intermediate residue stability was less than the CrL complex, which gradually decomposed in three stages. The loss of water molecules results in (9%) at about 130 ◦C. The second weight loss of 49% was observed at 631 ◦C, again due to the first weight loss along with the polymer chains and CO_2, CO, and Cl as gases from the ligand (PMMA-PVA) Scheme 3. The residue of polymer (ligand) chains and the Fe^{+3} oxides contributed nearly 31.16 %. Our results were supported by previous knowledge of the stabilities of other complexes containing these transition metals **(Neha *el al.*, 2018; Wu *el al.*, 2003)**.

Table (5): Thermal analyses data for CrL and FeL.

Comp.	Dissociation stages	Temp range in °C	Weight loss found %	Weight loss (Cal.) %	Final Weight residue found %	Final Weight residue (cal.) %
CrL	StageI StageII StageIII	20-150 150-625 625-900	14.42 36.21 7.8	5 48 5	41.55	42 it represent residue of polymer and metal oxide
FeL	StageI StageII StageIII	20-126.05 126.05-631.81 631.81-900	9 49.5 10.35	8.61 49.9 10.34	31.16	31.15 it represent residue of polymer and metal oxide

(a)



(b)

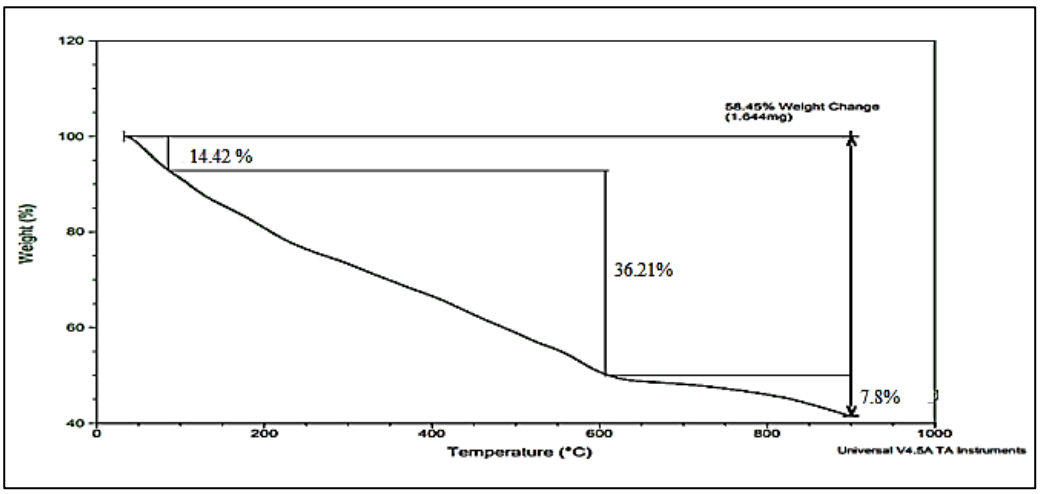
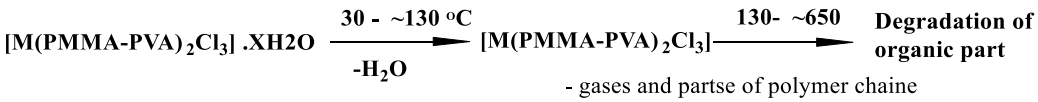


Figure (4): TGA analysis for: (a) CrL and (b)FeL



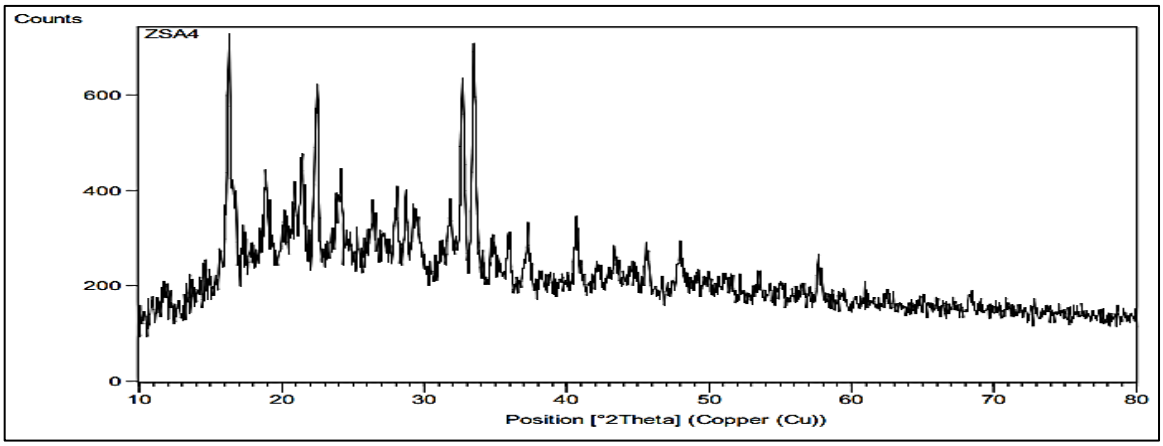
M= Cr or Fe

Scheme (3): Thermal behavior of CrL and FeL complexes.

XRD analysis

X-ray diffraction helps investigate nanoparticles **(Peighambardoust *et al.*, 2020)**. which were performed on a category of ligands. The sharp peaks demonstrated the Nanoscale feature of Cu(II) and Fe(II) complexes. Comparatively distinct peaks at 2Θ (16, 18.5, 22, 33, 34, and 41) were observed for the CuL Nano complex, but for the FeL Nano complex, the spectra were entangled, and the unique peaks vanished. Probably, X-ray spectroscopy cannot figure it out because of the shielding effect of the represented ligand-polymer molecules **(Siva *el al.*, 2019)** Figure 5.

(a)



(b)

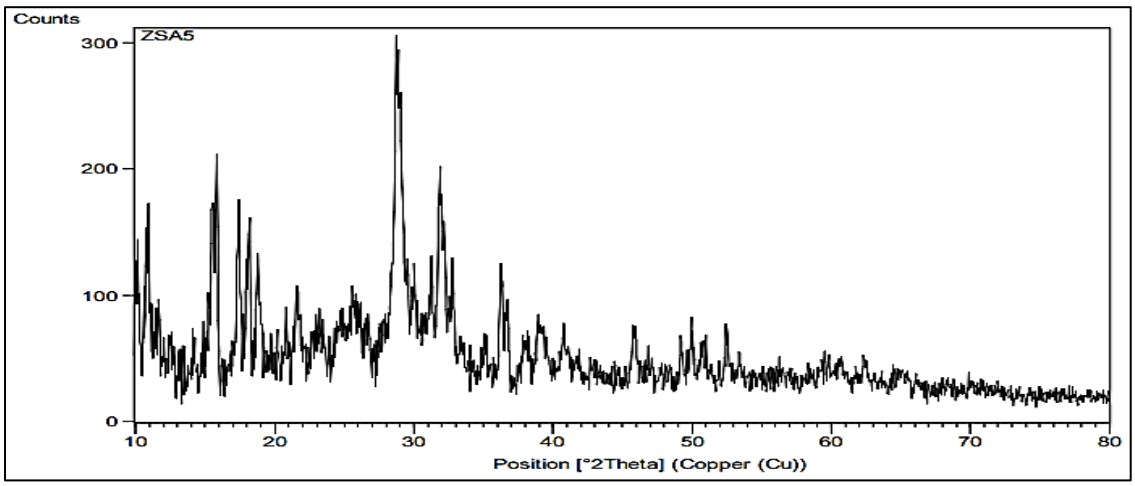


Figure (5): XRD patterns of a. CuL Nano complex b. FeL Nano complex.

Atomic Force Microscopy (AFM)

The method measures the forces exerted by a sharp cantilever tip on a surface at a very close distance, yielding two- and three-dimensional surface profiles at the nanoscale. The 3D and two-dimensional pictures of nano (CuL and FeL) complexes are shown in Figure 6. The granularity accumulation distribution charts for these complexes were displayed and shown in Figure 7. The average diameters and ten hight of CuL and FeL nano complexes particles were displayed in Table 6, with a mean diameter ranging from (4.5-9.0 nm) and an average diameter of 82.96 nm. The diameters of the particles making up FeL nano complex range between (20-45 nm), with an average diameter of 97.74 nm.

Table (6): Summary of the AFM information for CuL and FeL nano complexes

Sample	Roughness Averege (nm)	Root Mean Square (nm)	Average Hight (nm)	Average Diameter (nm)
CuL	1.31	1.73	6.89	82.9
FeL	8.3	10.3	42	97

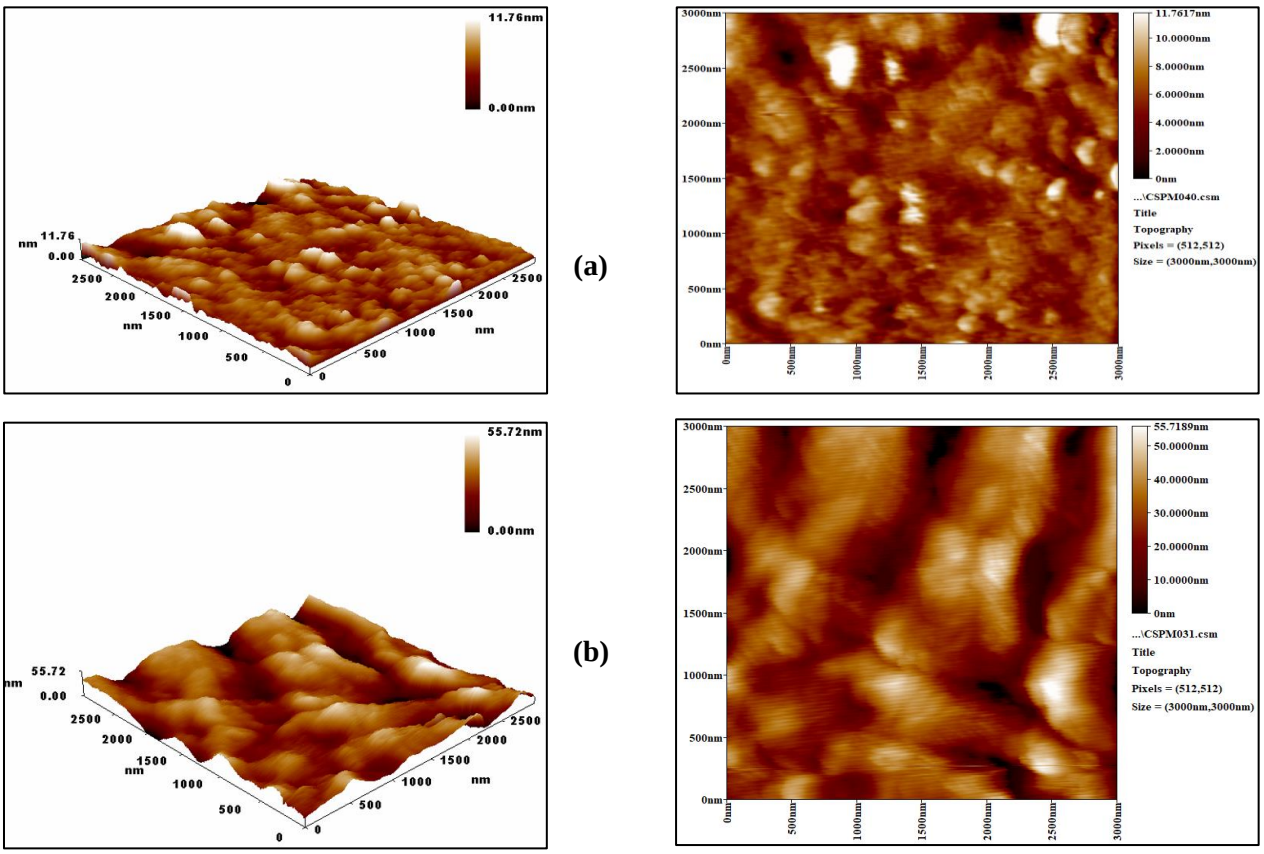
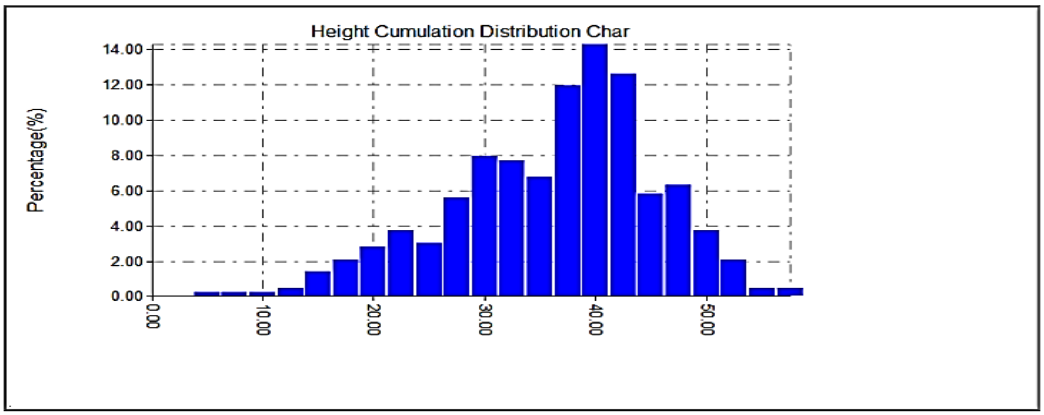


Figure (6): AFM three and two-dimensional image for (a) CuL (b) FeL nanoparticles complexes

(a)



(b)

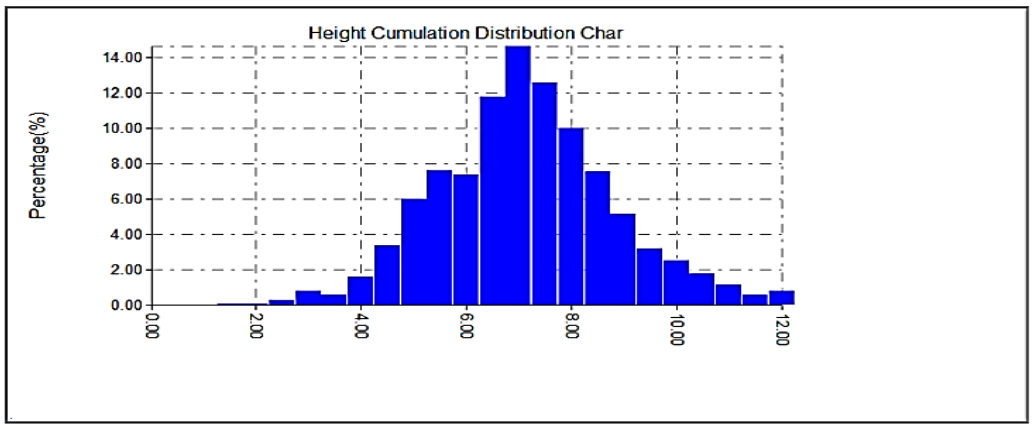
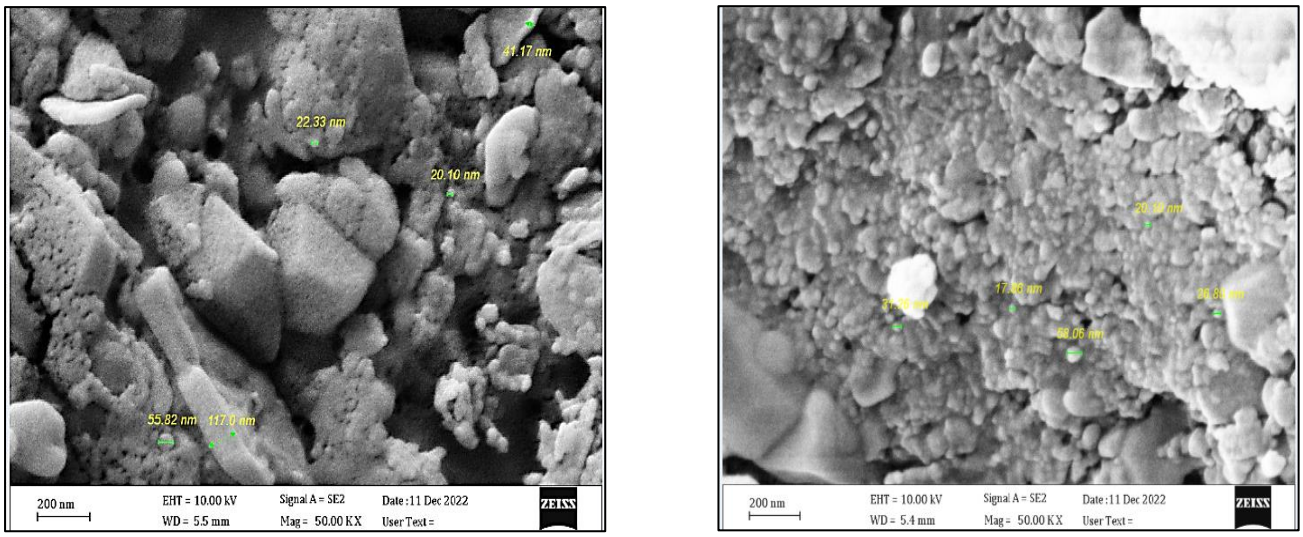


Figure (7): Granularity accumulation distribution of (a) CuL (b) FeL nanoparticles complexes.

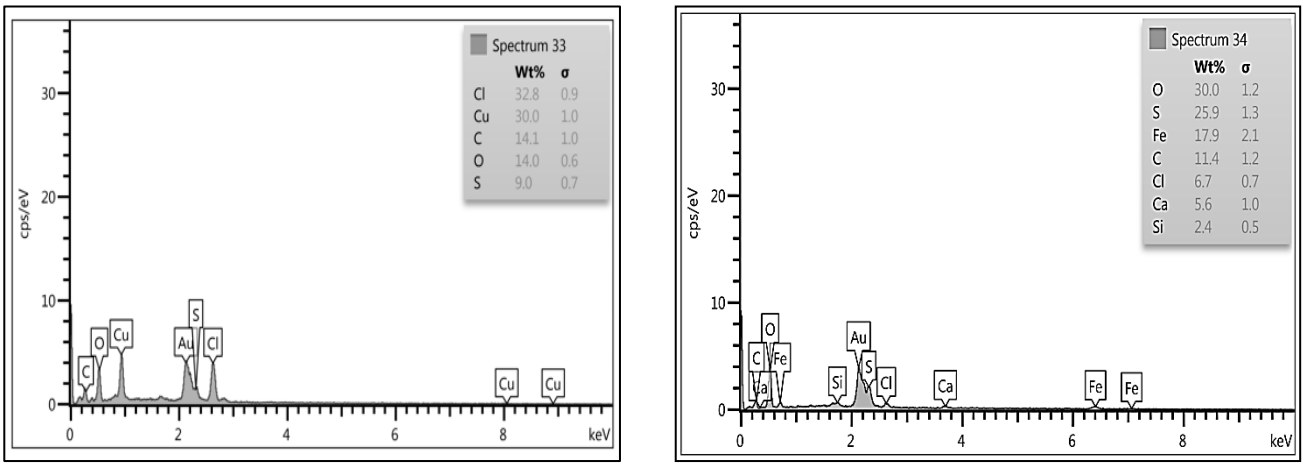
(SEM) Scanning Electron Microscopy and Energy Distributed X-Ray Spectrometry (EDS)

Pictures of the CuL Nano complex taken using a scanning electron microscope (SEM) reveal particles with sizes ranging from around (20-55nm), Figure 8. Similar results are seen for the FeL Nano complex; scanning electron microscopy (SEM) pictures of the FeL Nano complex were also displayed, revealing particle sizes of around (35-46nm).



(a) **(b)**

Figure (8): SEM of (a) CuL (b) FeL nanoparticles complexes.



(a) **(b)**

Figure (9): EDSof (a) CuL (b) FeL nanoparticles complexes.

Application of PMMA-PVA to remove elements from contaminated water.

Traditional and membrane approaches are available for removing heavy metal pollutants from wasted streams. Contaminated heavy metals must be recovered to prevent further contamination and gain economic benefit.

An internal standard method (In) and a multi-standard calibration method were used to precisely analyze metals in water samples **(Husam *el al.*, 2013; Maysoon *et al.*, 2022)**.

PMMA-PVA was applied to remove some of the elements in polluted water that were left over from the power stations, where very trace concentrations of the elements that were previously detected in the samples taken from the liquid waste left over from two power

stations were prepared, which are (20, 40, and 60) ppm, using the atomic absorption technique to determine its percentage after the removal process, which conducted through the preparation of a polymeric composite of each of the prepared PMMA-PVA and zeolite. The obtained results are listed in Table (7).

Table (7): The experimental result of removal metals (Mn(II), Fe(III), Ni(II) and Cu(II)) by designed PMMA-PVA - composite.

Metal	Initial concentration ppm.	Final concentration (after applying PMMA-PVA) ppm.	Final concentration (after applying PMMA-PVA with composite at 40 ppm from each metal)	Concentration ppm. (in the wastewater from Al Doraa power station after application PMMA-PVA with composite)	Concentration ppm. (in the wastewater from South of Baghdad power station after application PMMA-PVA with composite)
Mn(II)	20	4	0.9	0.95	0.54
	40	10			
	60	37.5			
Fe(III)	20	4.5	ND	ND	ND
	40	10			
	60	17.5			
Ni(II)	20	2.8	0.27	0.23	0.06
	40	17			
	60	38			
Cu(II)	20	15	0.088	0.16	0.06
	40	20			
	60	44			

CONCLUSION

The findings point to the potential for producing Nanocomposites by complexation of polymeric ligand methyl(S)-5-hydroxy-2-methylhexanoate (PMMA-PVA) with metals that were only partially soluble in solvents like water, ethanol, and DMSO. Because of the copolymer's fast complexation with elements, this property of the (PMMA-PVA) copolymer allows for its wide range of use.

Complexation and adsorption are two mechanisms by which it successfully purges water of harmful substances, wherein one approach involves ligand-to-metal interactions in polluted water. In contrast, in the adsorption method, composites were made by combining zeolite and poly (Methyl methacrylate-co-polyvinyl alcohol) or (PMMA-PVA). Examination of polluted water before and after using the produced compounds revealed that the removal utilizing the composite materials was significantly more effective, with the concentration being recorded as very low and the concentrations of some metals completely disappearing from polluted water.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

SYNTHESIS AND DETERMINATION OF CALCIUM ION-IMPRINTED POLYMERIC AND ITS APPLICATION IN SERUM SAMPLE

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ABSTRACT

This study was aimed to synthesis new molecular imprinted polymers from different monomers which useful for determination of calcium ion in different serum samples. Calcium ion play an important role in blood clotting and bone mineralization. In plasma, 40 percent of circulating calcium is bound to proteins, 10 percent is in the form of inorganic complexes, and 50 percent is present as free (ionized) calcium. In this study, Blood samples were taken from patients with type 2 diabetes from Kadhimiya Hospital in order to determine the concentration of calcium ions in the blood, enter it into the column, and then calculate the final concentration in order to know the amount of dose taken by the patient. To acquire the highest adsorption capacity, molar ratios of the template, monomer, and cross-linking agent, as well as solvents and multiple monomers were investigated. Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) were used to analyze the calcium ion polymer. The elution of calcium has a small effect on the surfaces of the three-dimensional network structure. Calcium (II) ions were successfully eluted using a mixture of methanol and acetic acid. The calcium absorption capacities were7.989 µmol/g and 8.250 µmol/g (Q_{max}), respectively. Solid-phase extraction (SPE) syringes packed with ionic imprinted polymers (IIPs) were used to selectively separate and preconcentration the calcium (II) ion from serum to determine the calcium ion by flame atomic absorption spectroscopy (FAAS). Through the results obtained and compared to the atomic absorption device, which is considered the most accurate and sensitive device for the elements, there is no difference in the results, and because the atomic absorption device test is expensive and needs electricity all the time, we can use the molecular printing technique to separate, assign, and concentrate the elements.

Keywords: Molecularly imprinted polymer; calcium ion, styrene, monomers

تخليق وتحديد الطور الصلب البوليمري المطبوع بايون الكالسيوم في السيرم وتطبيقها في عينات سيرم

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الخلاصة

هدفت هذه الدراسة إلى تخليق بوليمرات جزيئية جديدة مطبوعة من مونومرات مختلفة والتي تفيد في تحديد أيون الكالسيوم في عينات مصل مختلفة. يلعب أيون الكالسيوم دورًا مهمًا في تخثر الدم وتمعدن العظام. في البلازما، 40 في المائة من الكالسيوم المنتشر مرتبط بالبروتينات، و 10 في المائة في شكل معقدات غير عضوية، و 50 في المائة موجود على شكل كالسيوم مجاني (مؤين) .في هذه الدراسة، تم أخذ عينات دم من مرضى السكري من النوع 2 من

مستشفى الكاظمية بهدف تحديد تركيز أيونات الكالسيوم في الدم، وثم أدخالها في العمود، ومن ثم حساب التركيز النهائي للكالسيوم لمعرفة مقدار الجرعة التي يأخذها المريض وقد تم تحديد أيونات الكالسيوم من خلال إضافة مونومر ستايرين. للحصول على أعلى سعة امتصاص، تم فحص النسب المولية للقالب، والمونمر، وعامل الربط المتبادل، وكذلك المذيبات والمونومرات المتعددة. تم استخدام المسح المجهري الإلكتروني (SEM) و Fourier Transform Infrared Spectroscopy (FTIR)لتحليل بوليمر أيون الكالسيوم. كان لشطف الكالسيوم تأثير ضئيل على أسطح بنية الشبكة ثلاثية الأبعاد. تمت تصفية أيونات الكالسيوم (II) بنجاح باستخدام خليط من الميثانول وحمض الخليك. كانت سعات امتصاص الكالسيوم 8.250 µmol/g و7.989 µmol/g على التوالي. تم استخدام حقنة للاستخلاص بالطور الصلب (SPE) المعبأة بالبوليمرات الأيونية المطبوعة (IIPs) للفصل الانتقائي والتركيزوالتقدير للايون الكالسيوم (II) في الدم بطريقة مطيافية الامتصاص الذري باللهب (FAAS). من خلال النتائج التي تم الحصول عليها ومقارنتها بجهاز الامتصاص الذري، والذي يعتبر الجهاز الأكثر دقة وحساسية للعناصر، لا فرق في النتائج. ولأن فحوصات جهاز الامتصاص الذري غالي الثمن ويحتاج للكهرباء طوال الوقت، يمكننا استخدم تقنية الطبعة الجزيئية لفصل العناصر وتعينها وتركيزها.

الكلمات المفتاحية :الطبعة الجزيئية البوليمرية ,كالسيوم ايون, ستايرين ,مونمر

INTRODUCTION

Calcium is a metallic element that comprises more than 3% of the earth's crust, ranking fifth in abundance. It is found in leaves, bones, teeth, shells, etc. It has never been found in nature alone uncombined calcium seems to be silver. Calcium is an essential component of human and animal bodies because it protects the bone system and serves as a regulatory ion both within and outside the cell **(Aljabari & Al-Bayati, 2021)**. Bulk polymerization is the most straightforward way to create pure polymer forms **(Al-Bayati &Hadi, 2022; Andac & Denizli, 2004; Irshad Ahmad *etal*., 2018)**. The ion-imprinting process consists of three steps: (i) template (metal ions) complexation with a polymerizable ligand; (ii) polymerization of this complex; and (iii) template removal after polymerization. The specificity of the ligand, the coordination geometry, and the coordination number of the ions, as well as their charges and sizes, all have an influence on the selectivity of a polymeric adsorbent in the ion imprinting process **(Al-Bayati & Aljabari, 2016).** The template can be delivered into the system in a variety of ways, including standing alone or being bonded to a surface, resulting in 3D or 2D imprinting environments that respect polymerization. This could be covalent, non-covalent, or semi-covalent in nature **(Zaheer *eta.*, 2021*).*** Non-covalent imprinting is by far the most prevalent approach due to its ease of production and the vast range of monomers available **(Al Fatease *et al*., 2021; Pedro Melendez *etal*., 2017).** By virtue of their existence, they are required for molecular interactions. Acceptance procedures are widely employed since they are considered the most efficient and successful method for molecularly imprinted polymer (MIP) synthesis. The researchers are currently working on creating a method for selective preconcentration of sorbents utilized in solid-phase extraction (SPE) **(Al-Batatyi & Abd, 2017; Beeregowda, 2014).** Waste water or river water is examples of complicated matrices. SPE is a more straightforward, quick, and cost-effective method of extraction that is also environmentally friendly. The most significant issue is the use of standard stationery stacked in SPE columns **(Mohsen & Al-Bayati, 2021; Nose *et al*., 1988)**. The retention phase's low selectivity mechanism it's possible to achieve a desired level of selectivity **(Agarwal & Kasana, 2019)**. Here, we investigate the selective separation and preconcentration of the calcium (II) ions from aqueous solutions by the addition of an allyl chloride monomer, resulting in bulk polymerization formation, to determine the calcium ion by flame atomic absorption spectroscopy (FAAS). This study was aimed to synthesis new molecular imprinted

polymers from different monomers which useful for determination of calcium ion in different serum samples.

Chemicals and materials

Calcium chloride dihydrate (99.9%), styrene (99.9%), ethylene glycol methacrylate (EGDMA) (99.9%), benzoyl peroxide were purchased from Sigma Aldrich, methanol, nitrogen gas and acetic acid.

Preparation and Processing:

Preparation of ionic imprinted polymer: For preparation of the number one calcium ionic imprinted polymer (calcium-IIP), calcium chloride dihydrate (0.147g, 1 mmol) was dissolved in methanol (2 mL), then mixed with styrene (4 mmol) as a monomer in methanol (2 mL) and left for few seconds at room temperature. Then, ethylene glycol methacrylate (EGDMA) (3.9 g, 20 mmol) was dissolved in methanol (2 mL) (as a cross-linker), and benzoyl peroxide (300 mg) as an initiator) was dissolved in chloroform (2 mL) and then added to the solution to obtain a homogeneous solution, and the mixture was shaken for 5 minutes. After wards, nitrogen gas was passed for 30 minutes through the mixture to extract oxygen from it. The solution was then placed in a water bath at 60 °C for 6 hours. When the reaction was completed and the Ca-IIP were formed. they were left for 24 hours to dry and then they were crushed and ground by a mortar and pestle, the sieve was used to obtain the particles with a diameter of 125-150 μm and then collected. The Ca ion was extracted from polymers Ca-IIP by using soxhlet of (60:10:20) (methanol: acetic acid: acetonitrile) for weak. After that, the polymer was dried for 24 hours at room temperature and collected to be used as a substance in a solid-phase extraction syringe**.** Each plastic syringe (column) was packed with Ca-IIP (200 mg) and used 3 mL solution for solid-phase extraction by a peristaltic pump.

Sampling procedure

Serial concentrations (20, 40, 60, 80, 100ppm) were prepared from $CaCl_2.2H_2O$ (0.147g, 1 mmol) by dissolving in methanol in a 100 mL volumetric flask. Calibration curve between concentration of calcium. and its absorption A, this was achieved using at (274nm by UV-VIS instrument).as shown in figure.1.

Concentration of Ca^{2+} ion ppm	Absorption
20	.03940
40	0.0543
60	0.0748
80	0.1002
100	0.125

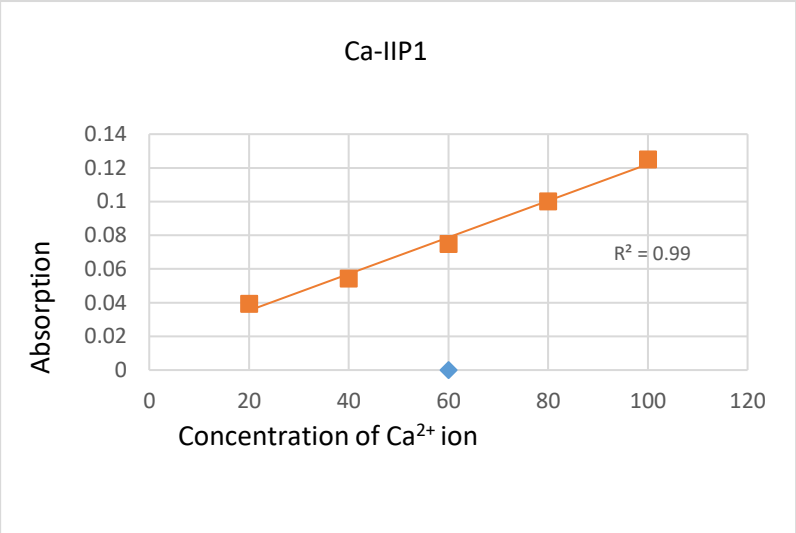


Figure. (1): Calibration curve between concentration of Calcium ion standard ppm and its absorptions in UV-VIS spectrophotometer technique.

RESULTS AND DISCUSSION

Transform Infrared Spectroscopy (FTIR) analysis:

To detect the functional groups, present in a compound, FTIR an important chemical characterization process. The FTIR method was successfully applied to the study of molecularly printed materials and has been beneficial in identifying the functional groups of polymers. FTIR spectroscopy was especially sensitive to the structural characteristics of polymer formation. The spectrum of Ca-IIP before elution where appears peaks at 1639 cm^{-1} for Ca-Cl stretching,3433cm^{-1} for O-H stretching , 1730 cm^{-1} for C=O stretching,3172 cm^{-1} for C-H stretching, 1232 cm^{-1} for C-O-C stretching, 3016 cm^{-1} for C-H aromatic stretching, 1556 cm^{-1} for C=C aromatic stretching (Agarwal & Kasana, 2019).When compare with FTIR after removal the calcium ion show disappearance the peak of Ca-Cl which indicate that calcium ion was removed and form the ionic imprint polymer are shown in Figure (4) .

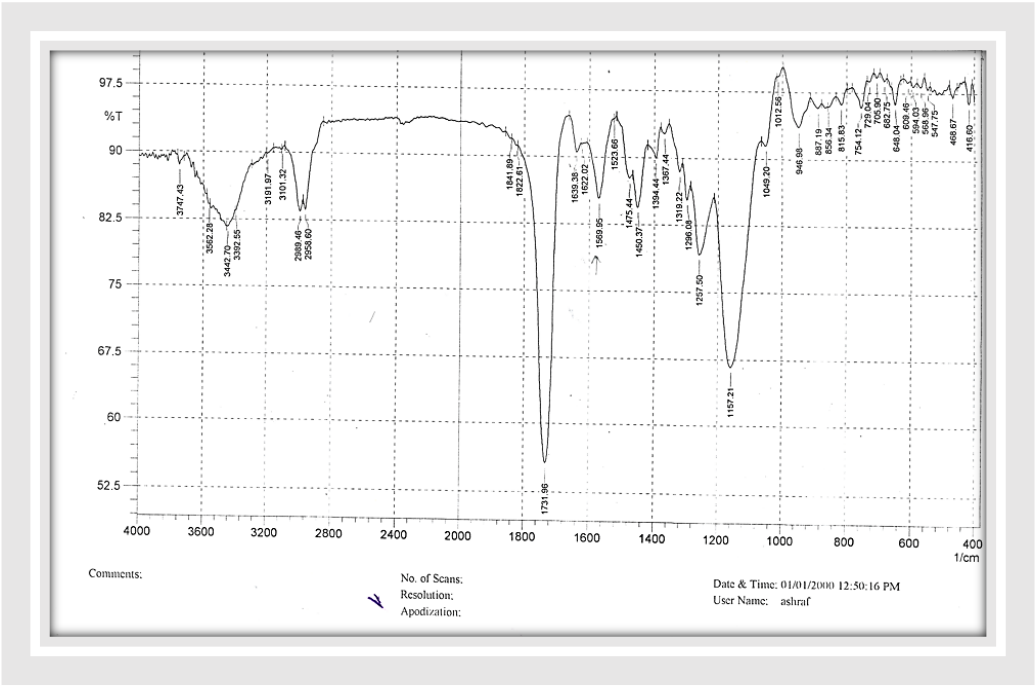


Figure (2): FTIR spectra of salt $CaCl_2 .2H_2O$.

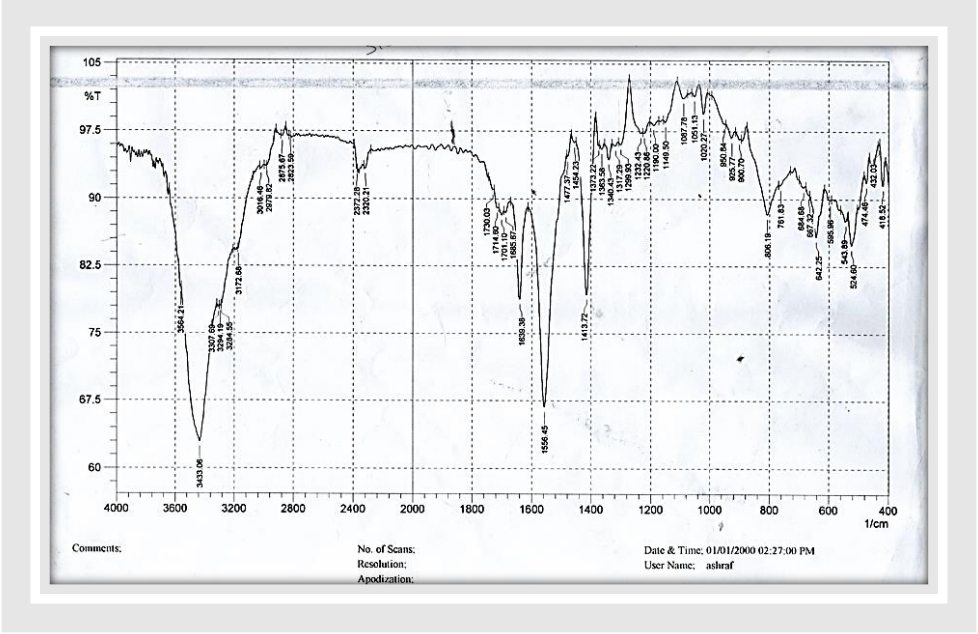


Figure (3): FTIR spectra of Ca-IIP1 (styrene) before remove the Ca^{2+}ion

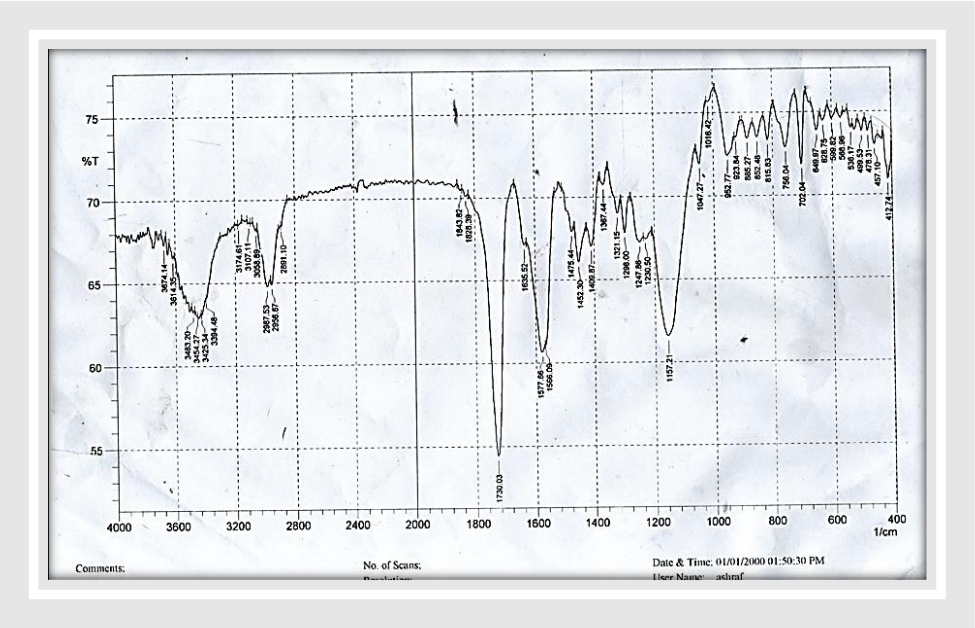


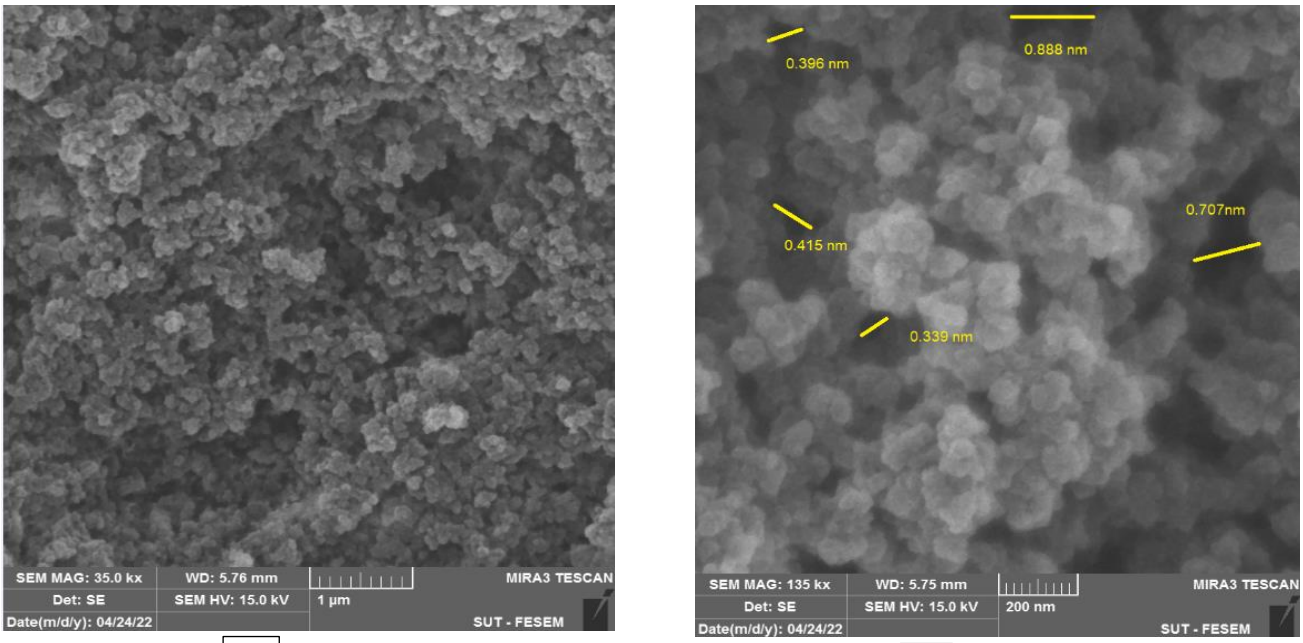
Figure (4): FTIR spectra of Ca-IIP (styrene) after remove the Ca^{2+}ion

Table (1): The most identified peaks of FTIR spectra for Ca-IIP using (styrene) as a functional monomer.

	Functional Group	CaCl2.2H2O	Ca -IIP(Styrene) before template removal	Ca –IIP (Styrene) After template removal
1-	v Ca-Cl cm-1	1639	1639	----------
2-	v O-H cm-1	3442	3433	3454
3-	v C=O cm-1	----------	1730	1730
4-	v C-H cm-1 aliphatic	----------	2979 2875	2987 2956
5-	v C-H cm-1 olf	----------	3172	3174
6-	v C-O-C cm-1 ester	----------	1232	1230
7-	v C-H cm-1 aromatic	----------	3016	3058
8-	v C=C cm-1 aromatic	----------	1556	1577

Scanning electron microscope (SEM)

SEM creates a high-resolution image by scanning the surface of a comparison surface; this figure (5) depicts the morphology of IIP for calcium before and after washing. The figure(5) reveals obvious calcium holes in the sizes eliminated by soxhlet extraction(**Zaheer *et al.*, 2021**).



A B

Figure (5): SEM photograph of the surface of Ca-IIP (styrene), (A) before calcium removal, (B) after calcium removal.

Table (2): Results obtained using different ratios of [D:M:C] and progeny for the synthesis of IPs and NIPs for Ca-IIP

NO. of IIP	Ratio%	Salt $CaCl_2.2H2O$	Monomer Styrene	Cross linker EGDMA	Initiator Benzoyl peroxide	Solvent	**Result**
IIP1	**%**	**9.302**	**18.604**	**72.093**	**0.3**	**6ml CH_3OH**	**White**
	mmole	**2**	**4**	**15.5**	**0.32**		
IIP1	**%**	**6.976**	**18.604**	**74.418**	**0.3**	**6ml CH_3OH**	**White**
	mmole	**1.5**	**4**	**16**	**0.32**		
IIP1	**%**	**4.048**	**16.177**	**79.772**	**0.3**	**6ml CH_3OH**	**White**
	mmole	**0.999**	**4**	**19.675**	**0.32**		
NIP1	**%**	**------**	**16.177**	**79.772**	**0.3**	**6ml CH_3OH**	**White**

The optimum ratios employed in the synthesis of Ca-ion-imprinted polymers (IIPs) and non-imprinted polymers (NIPs) are summarized in Table 2. After the calcium ion is removed, the control NIPs and IIPs, however, exhibit the same spectra and structural similarities. This demonstrates that removing the template molecule and leaving particular recognition binding sites in the polymer structure may be accomplished by washing the IIP particles in a (methanol:acetic acid, 60:10) solution using the soxhlet extraction method.

Table (3): The optimal synthesis conditions for the ionic imprinted polymer for Ca-IIP1 (styrene) developed in this study used UV-VIS technique.

Ca-IIP1 (styrene)					
Mass of IIP mg	Ci ppm	C_i µM	C_{free} µM	Q µMole/g	Q_{free} mL/g
0.2	ppm20	0.136	0.097	1.935	19.886
	ppm40	0.272	0.195	3.465	17.769
	ppm60	0.408	0.285	4.305	15.105
	ppm80	0.544	0.392	4.560	11.632
	ppm100	0.680	0.526	4.620	8.783

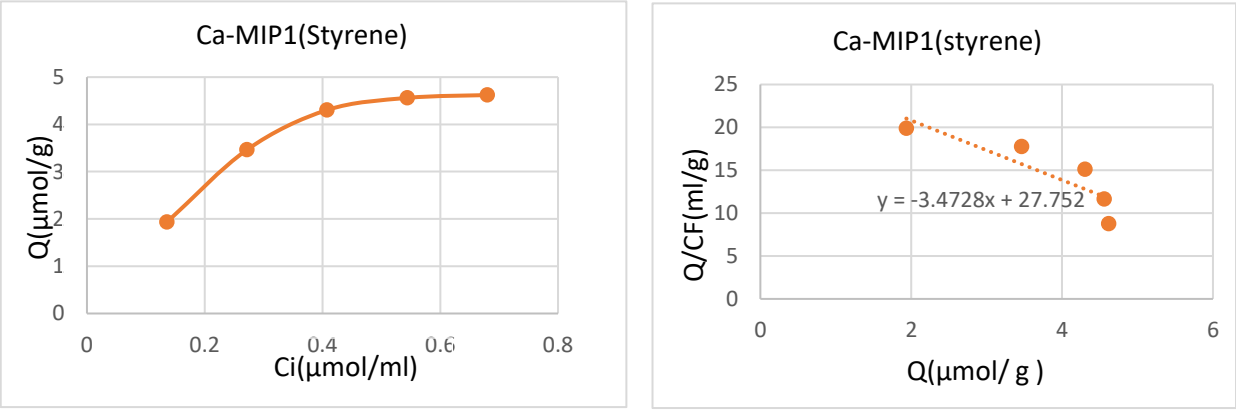


Figure (6): Illustrate Langmuir isotherm model.

Slop=-1/kd
-3.4728=-1/ kd =0.2879
Intercept=Q max/kd
27.752=Q max/0.2879
`Q max=7.989 µmol/g

Atomic absorption spectroscopy (AAS)

Standard solutions with concentrations of 20, 40, 60, 80and 100ppm were prepared and measured by atomic absorption at wavelength 422.7 nm, as shown in Figure 7

Concentration of Ca^{2+} ion ppm	Absorption
20	0.053
40	0.078
60	0.115
80	0.147
100	0.199

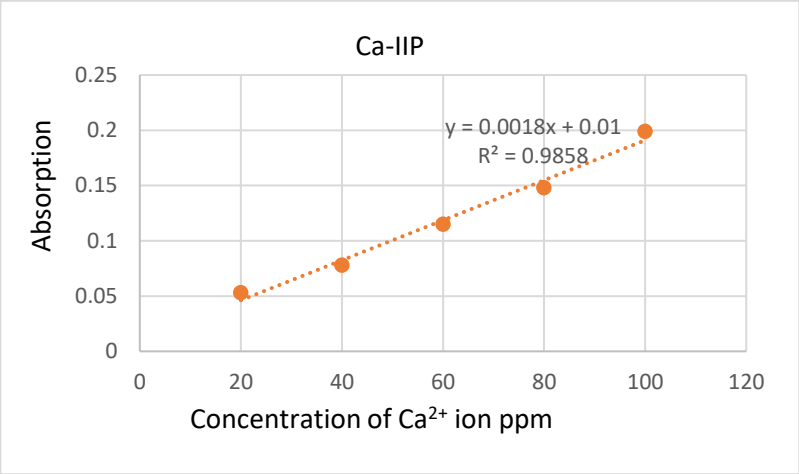
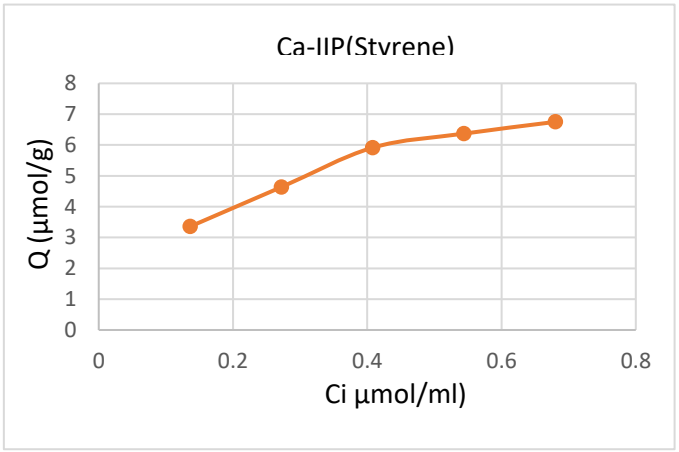


Figure (7): Calibration curve between concentration of calcium ion standard ppm and its absorptions in A.A.S technique

Table (4): The optimal synthesis conditions for the molecularly imprinted polymer for IIP1 - Ca (styrene) developed in this study used A.A.S technique.

IIP1 - Ca (styrene)					
Mass of MIP mg	Ci ppm	C_i µM	C_{free} µM	Q µMole/g	Q_{free} mL/g
0.2	ppm20	0.136	0.068	3.355	48.693
	ppm40	0.272	0.169	4.635	27.290
	ppm60	0.408	0.239	5.915	24.740
	ppm80	0.544	0.331	6.369	16.500
	ppm100	0.680	0.455	6.750	14.835



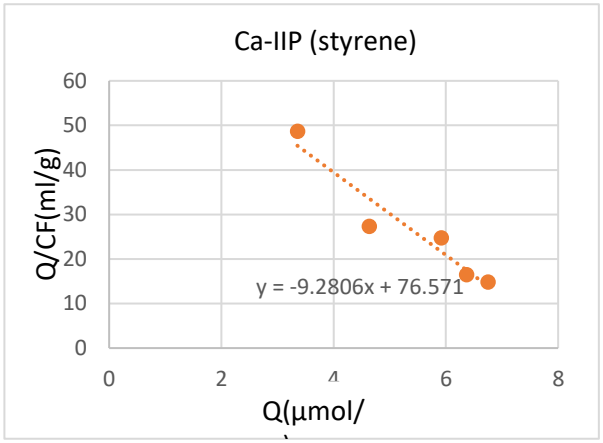


Figure (8): Illustrate Langmuir isotherm model.

Slop=-1/kd
-9.2806=-1/ kd =0.10775
Intercept=Q max/kd
76.571=Q max/0.10775
Q max= 8.250µml/g

In human serum

1- Sample collection

10 ml of blood was collected in plain tubes from each patient . Blood samples were allowed to stand for 5 minutes following centrifugation at ~ 2000 rpm. The serum was frozen at 20ºC so that it could later be employed for the estimation of the calcium in the serum of patients with type II diabetes.

2- Procedure

1 ml of serum transferred to volumetric flask (10) ml was diluted in 10 ml of deionized water., and it was examined in the atomic absorption instrument for determination the calcium ion in the serum. A concentration of a serum sample containing calcium, were taken and applied to the ion-imprinted polymers. The rang value for calcium in the serum (0.0083-0.0106) ppm.

$S_1=2.541\times10^{4-}$
$S2=2.645\times10^{4-}$
F-test = $\frac{S1^2}{S2^2}$
F-test= 1.084

Table (5): The statistical values for F test between tabular values and observed values.

	S1	S2	F-test	F- table
1	2.541×10^{-4}	2.645×10^{-4}	1.084	19.2

It found F-test calculated < F-tab at confidence level 95% therefor there is no significant difference between two methods, So Null hypothesis will be accepted.

CONCLUSION

Bulk polymerization was used to create a novel calcium-IIP. EGDMA was chosen as the cross-linker and styrene as the functional monomer. In addition, benzyl peroxide was utilized as an initiator when chloroform was the solvent. The ideal calcium (II) ion to monomer and crosslinker dosage molar ratios were investigated. Three-dimensional network structure of polymers and their unpredictable shapes were studied using SEM. The results of FT-IR demonstrated that the Ca (II) ion was successfully eluted by a solution of methanol: acetic acid: acetonitrile (60:10:20v/v). The exceptional stability and regeneration capabilities of calcium-IIP are illustrated by the fact that the elution process has little to no impact on the chemical characteristics of the polymer or the shape of the cavity. According to the previous results, between two methods analytical technique by atomic absorption and our method IIP by UV for Ca^{2+}ion, there was no significant difference between two methods evidence of the method's efficiency and reliability in the analysis and estimation of the elements. Therefore, we can dispense with the atomic absorption device, which is costly and requires continuous electricity, and use the molecular print to estimate the elements.

Future works:

- Study the selectivity of prepared ionic imprinted polymer towards the other metals.
- Study the possibility of using the prepared polymers on extraction several times and the effect of this on the structure and properties of the polymer and its adsorption capacity.
- Prepare polymer by using a new molar ratio and study the effect of that on the adsorption capacity.
- Several salts of the same metals are used as a template in preparing imprinting polymers and studying the characterization to compare them with each other.
- Prepare new polymer by using a different type of monomers and crosslinkers and also by using two monomers in the same polymer.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

SELECTIVE EXTRACTION OF GLIMEPIRIDE IN PHARMACEUTICAL PREPARATION AND IN HUMAN SERUM VIA SYNTHESIZED MIP-SPE TECHNIQUE

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ABSTRACT

This paper demonstrates that the synthesizing and storage of molecular-imprinted polymers (MIP) at room temperature using bulk polymerisation of Glimepiride (Glim.) is characterized by high sensitivity, reduced costs, increased stability, and extended life. The research used 1:15:20 mmol ratios of template, monomer and cross-linking agents for the polymerisation in order to ensure an appropriate adsorption capacity. Benzoyl peroxide BPO was employed as the initiator for the functional monomer Allyl chloride C_3H_5Cl , cross-linked with Ethylene glycol dimethacrylate EGDMA $C_{10}H_{14}O_4$, thereby creating MIP for Glimepiride (Glim-MIP) that could be characterised with UV-Visible Spectrophotometry at 274.5nm, for pharmaceutical drugs. Fourier-transform infrared spectroscopy (FTIR) and Scanning electron microscopy (SEM) was used for the human serum. The elution process that was applied to the template (Glim.) from the Glim-MIP created cavities that were caused by the porogenic mixture solvents that were created from (acetic acid, methanol) 1:9 respectively, successfully removed by repeated washing for 20 hours, the polymer was dried at room temperature. The maximum adsorption capacity was 11.7797 µmol/g using (0.1) g weight of Glim-MIP. which adhered to the Langmuir isotherm model. A solid-phase extraction (SPE) syringe packed with molecular imprinted polymers (MIPs) was employed to selectively separate and pre-concentrate the Glimepiride in multiple pharmaceutical drugs from several sources. The human serum was based on the use of deionized water to dilute the serum, followed by heating of the serum with methanol. Subsequently, few drops of 1N hydrochloric acid were applied to detect Glimepiride at UV region 274.5 nm by applying the standard addition method.

Keywords: Isotherm process, Glimepiride, (Molecular Imprinted Polymers) MIP, Serum, (Solid-Phase Extraction) SPE.

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الاستخلاص الانتقائي للكليميبيرايد في المستحضرات الصيدلانية وفي مصل الانسان عن طريق تقنية MIP-SPE المركبة

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الخلاصة

يوضح هذا البحث تحضير وتخزين البوليمرات الجزيئية المطبوعة (MIP) في درجة حرارة الغرفة عن طريق البلمرة الصلدة لـ Glimepiride (Glim) والتي تتميز بالحساسية العالية والتكلفة المنخفضة والاستقرار العالي. اذ تم أخذ نسب 1: 15: 20 ملي مول للقالب، و للمونومر ولعوامل الربط المتصالب للبلمرة من أجل ضمان قدرة امتزاز مناسبة. المونومر الوظيفي اليل كلورايد C_3H_5Cl تم ربطه مع إيثيلين جليكول ثنائي ميثاكريلات EGDMA $C_{10}H_{14}O_4$ رابط التشابك وبالتالي إنشاء MIP لـ Glimepiride كـ Glim-MIP تم تمييزه باستخدام مقياس الطيف الضوئي UV-VIS عند 274.5 نانومتر، والتحليل الطيفي بالأشعة تحت الحمراء والمسح المجهري الإلكتروني. أنشأت عملية الشطف التي تم تطبيقها على القالب اي انتزاع القالب ال Glim من Glim-MIP تجاويف ناتجة عن استخدام خليط مسامي من الميثانول وحمض الخليك (10:90) على التوالي. اذ اجريت عملية الشطف بنجاح لمدة 20 ساعة ويجفف البوليمر بدرجة حرارة الغرفة ،كانت السعة القصوى للامتزاز Glim-MIP هي 11.7797 ميكرو مول / غم عند استخدام وزن 0.1 غم من Glim-MIP. يتبع الامتزاز بواسطة Glim-MIP نموذج Langmuir isotherm. تم استخدام حقنة استخلاص ذات المرحلة الصلبة (SPE) معبأة ببوليمرات مطبوعة جزيئيًا (MIPs) للفصل الانتقائي والتركيز المسبق للكليميبيرايد في العديد من الأدوية الصيدلانية من عدة مصادر. اعتمد المصل البشري على استخدام الماء منزوع الأيونات لتخفيف المصل، يليه تسخين المصل بالميثانول. بعد ذلك، تم تطبيق بضع قطرات من حمض الهيدروكلوريك N1 للكشف عن الكليميبيرايد في منطقة الأشعة فوق البنفسجية 274.5 نانومتر من خلال تطبيق طريقة الإضافة القياسية.

الكلمات المفتاحية: عملية الايزوثرم ، كليميبيرايد ، بوليمرات الطبعة الجزيئية ، المصل، استخلاص الطور الصلب.

INTRODUCTION

Glimepiride; Amaryl a second-generation of antidiabetics sulfonylurea, it was patented in 1979 and approved for medical use in 1995**(Basit *et al*., 2012)**
Glimepiride stimulates pancreatic beta cells to secrete insulin and improves the sensitivity of peripheral tissues to insulin thereby increasing peripheral glucose uptake, and reducing plasma blood glucose levels and glycated hemoglobin (HbA1C) levels **(Zekry, *et al*. 2023; Sola *et al*., 2015)**
Molecular Formula: $C_{24}H_{34}N_4O_5S$, Molecular Weight : 490.62 , figure (1) illustrates the structure of Glimepiride

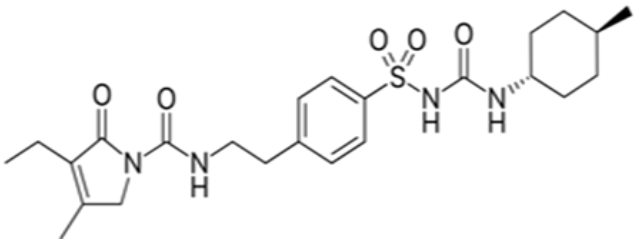


Figure (1): Structure of Glimepiride

1-[[p-[2-(3-ethyl-4-methyl-2-oxo-3-pyrroline-1carboxamido) ethyl] phenyl] sulfonyl]-3-(trans-4-methylcyclohexyl)urea) **(Basit *et al*., 2012).**

It was found to be practically insoluble in water, slightly soluble in dichloromethane and very slightly soluble in methanol. It was soluble in DMSO (>10 mg/ml) and ethanol (<1 mg/ml). In acidic and neutral aqueous solutions glimepiride exhibits very poor solubility at 37 ^oC (<0.004 mg/ml) **(Kari *et al*., 2023)**.

A molecular imprinting polymer (MIP) creates a multifaceted monomer (**Samarth *et al*, 2015).** A highly cross-linked polymer structure was used to secure functional groups in situ. Moreover, the steric patterns of these connections and the template are significant for the formation of binding sites that supply the shape, size, and flexibility required to encourage selective identification, in addition to elevated target correspondence. Consequently, the process can be deemed to be comparable to enzyme-proven mechanisms or substrata. Hence, the complex was created in a manner akin to a lock/key model **(Al-Bayati & Hadi, 2022; Aljabari & Al-Bayati, 2023; Mohsen & Al-Bayati, 2022).** Figure (2) presents the polymerization cycle.

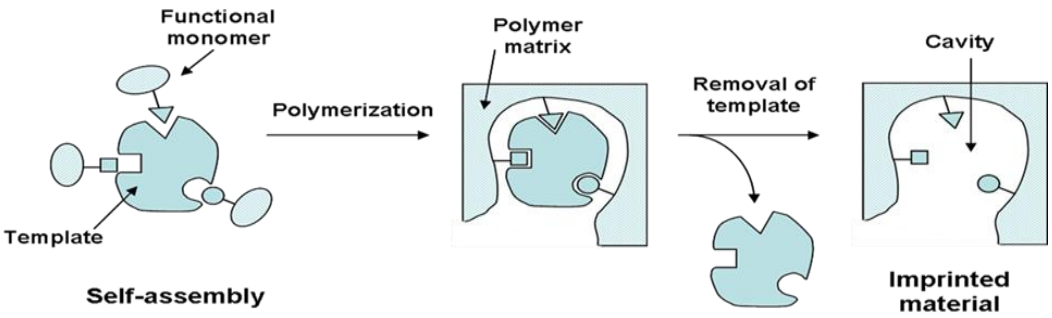


Figure (2): Molecular imprinted polymer cycle **(Yan & Row, 2006)**

After this cycle, certain MIP have been prepared using SPE **(Knoll *et al.*, 2020; Lim, Oh *et al.* 2020)**

The solute concentration in the fluid phase at a constant temperature provides an adsorption isotherm. An isotherm is the relation between the concentrations of a solid and fluid, used to describe states of the sorption process **(Yu *et al.*, 2012).**

- Solid phase extraction (SPE) is a technique designed for rapid, selective sample preparation and purification **(Mahdi Z & Al-Bayati 2020**; **Abd Jaber & Al-Bayati, 2020)** prior to the chromatographic analysis (e.g. HPLC, GC, TLC) **(Nakamura *et al.*, 2022; Qasim *et al.* 2020)**. In SPE, one or more analytes from a liquid sample are isolated by extraction, partitioning, and/or adsorption onto a solid stationary phase, washing and elution to cover the analyte under investigation as been by Figure (3).

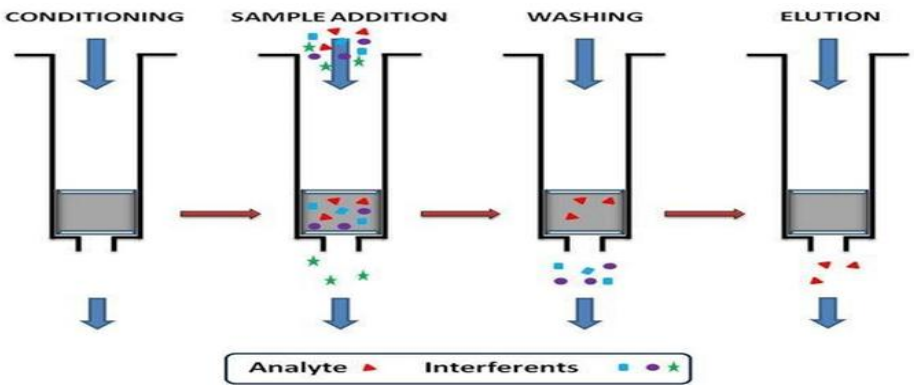


Figure (3): Illustrate the process of SPE.

In this work identify the MIP preparation was performed in conjunction with the recognition site Allyl chloride C_3H_5Cl with crosslinking Ethylene glycol dimethacrylate EGDMA $C_{10}H_{14}O_4$, whereby benzoyl peroxide BPO functioned as the target molecule (Glimepiride) initiator. Subsequently, the impact of monomer dosage on adsorption performance was observed. This study also examined the adsorption behavior of diverse functional monomers, cross-linking agents, and solvents. SEM, FTIR was employed to characterise the primed MIPs. Furthermore, this study investigated the impact of solid phase extraction and initial Glimepiride concentration on the adsorption capacity.

MATERIALS AND METHODS

MATERIALS

Glimepiride standard as template from Samarra/Iraq was provided , Allyl chloride as monomer, EGDMA as cross-linker and Benzoyl peroxide as initiator were purchased from Sigma Aldrich (St. Louis, MO, USA, www.sigma-aldrich.com) , Methanol, Nitrogen gas (99.99) supplied by Al-Watan factory (Al-Nahda street/ Baghdad/Iraq), Chloroform and Acetic acid were purchased from Merck (Darmstadt, Germany), Glimepiride/UK and Glypride/ julphar Emirate as pharmaceutical drugs of glimepiride purchased from pharmacy.

METHODOLOGY

With the recognition sites of monomer allylchloride C_3H_5Cl, crosslinking Ethylene glycol dimethacrylate EGDMA $C_{10}H_{14}O_4$ with benzoyl peroxide BPO as initiator was synthesized for the target molecule Glimepiride:

1. Glim-MIP was prepared by dissolving 1mmol of Glimepiride 0.4906g in 5-6 drops of 1N HCl after that methanol was added. The resultant solution was stirred and warmed for 10-20 seconds to obtain a transparent solution.
2. A 15 mmol of allylchloride 1.1480g with 2 ml methanol was added.
3. The mixture (step1 and 2) was allowed to stand for a few seconds at room temperature.
4. A cross-linker 20 mmol Ethylene glycol dimethacrylate EGDMA 3.9644g with 2ml methanol and 0.3 g benzoyl peroxide dissolved in chloroform as an initiator were added to the above solution.
5. The ratio 1:15:20 Glim-MIP was completed, the solution was shaken and bubbled for 20 min with pure nitrogen gas to remove the dissolved oxygen from the monomer solution immediately.
6. The tube was sealed with a rubber stopper. The solution was left overnight in a water bath at 60 ºC for 72 hours.

A white color polymer with a fluff structure was formed, Figure 4.

7. Soxhlet solid liquid phase extraction for the template was performed to remove the template glimepiride from MIP using a porogenic solvent (acetic acid, methanol) 1:9 respectively, successfully removed by repeated washing for 20 hours,
8. The MIP was dried at room temperature, after that it was crushed with mortar, sieved to particle size 125µm.
9. A plastic syringe (10 ml) of solid phase extraction vacuum (column) was used, and each syringe packed with 0.1 g of Glim-MIP.

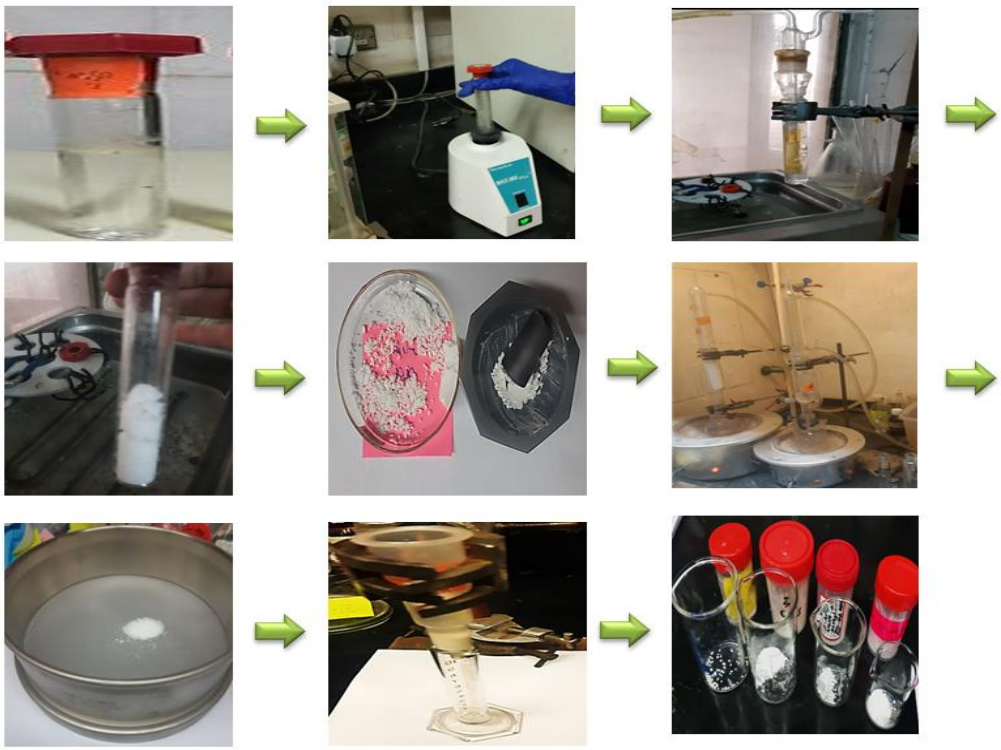


Figure (4): The preparation of imprinting polymer in the laboratory: firstly combined template, monomer, cross linker and initiator, mix well in shaker, bubbled with nitrogen gas, the polymer become solid MIP after placed in a water bath, drying and grinding, Soxhlet solid liquid extraction to separate the template, crushed and sieved the MIP to required a suitable particle size(using 125µm), packed in a cartridge to prepare a column for isotherm process, finely store MIPs in suitable containers**.**

A solutions (standard solution, pharmaceutical drugs of glimepiride and serum) was poured from the top of the column and the movement of the solution was by electric vacuum at 70 rpm. A series of standard solutions of Glimepiride (0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 1.2) µmol/ml was prepared by dissolving 0.0589g Glim. in 1-2 drops of 1N HCl to create a buffer solution, after which methanol was added. The resultant solution was stirred and heated for approximately 15-20 seconds methanol volumetric flask 100 ml as a stock solution. A calibration curve was constructed between concentration of Glim. and its absorbance A, This was achieved using at (274.5 nm by UV-VIS instrument).

Sample preparation of Glimepiride (pharmaceutical samples)

Ten tablets were weighted then crushed and grinded. Tablets containing 4 mg of Glimepiride were weigh 0.1422g, 0.1418g, (equivalent to 0.0196g& 0.0343g of active ingredient, 4×10^{-4}, 7×10^{-4}mMol/L) for Glimepiride (Glimepiride/UK, Glypride/ julphar Emirate) respectively (Table 1) and dissolved in several drops of 1M HCl in a 100 mL volumetric flask. Methanol was added agitated and warmed using a magnetic stirrer for at

least 30 minutes, the solution was filtered to get rid of undissolved materials, the residue was washed with methanol and completed the volume to 100ml with methanol.

Table (1): Pharmaceutical drugs prepared for treating with Glim-MIP polymer

No. of samples	Commercial name, Country Content 500mg	Average weight for 10 of tablets	Weight of sample equivalent to 0.0196g (4×10^{-4}) mmol/mL (0.4µmol/mL)of the active ingredient	Weight of sample equivalent to 0.0343g (7×10^{-4}) mmol/mL (0.7 µmol/mL) of the active ingredient
1	Glimepiride/UK	0.1422	0.6968	1.2194
2	Glypride/ julphar Emirate	0.1418	0.6950	1.2159

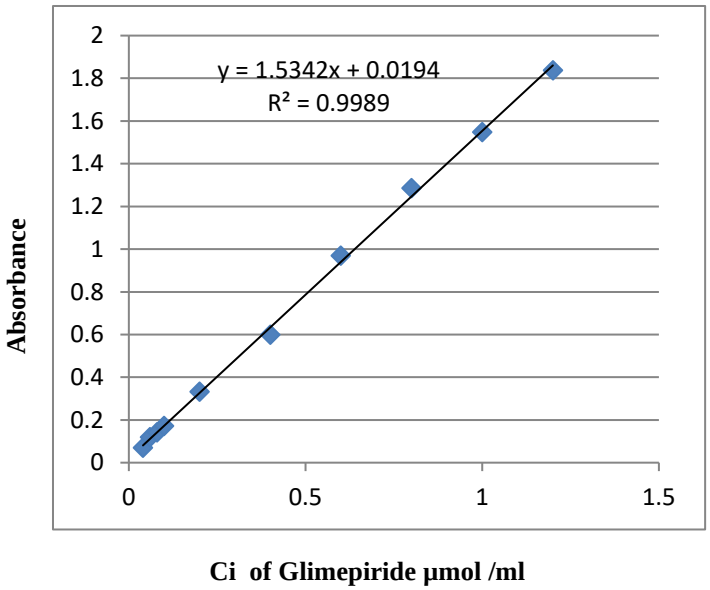
RESULTS AND DISCUSSION

RESULTS

Accuracy of the work for extraction and determination of Glimepiride

UV-VIS Spectrophotometry

A calibration curve between concentrations of standard Glimepiride (0.04-1.2) µmol /ml and their absorbance was plotted figure 5.



Concentration of Glimepirideµmol /ml	Absorbance
0.04	0.0688
0.06	0.1191
0.08	0.141
0.10	0.1712
0.20	0.3314
0.40	0.5978
0.60	0.9688
0.80	1.2856
1.00	1.5473
1.20	1.8367

Figure (5): Calibration curve of standard concentrations of Glimepiride and its absorptions.

After passing the solution of Glimepiride in syringe packed with Glim-MIP the residue which has less absorption was measured by UV-VIS that indicate to lower concentration at final process, for good expressive example of the advantages of the use of impressed polymers in SPE in the quantification of the Glimepiride, figure 6, 7 and 8.

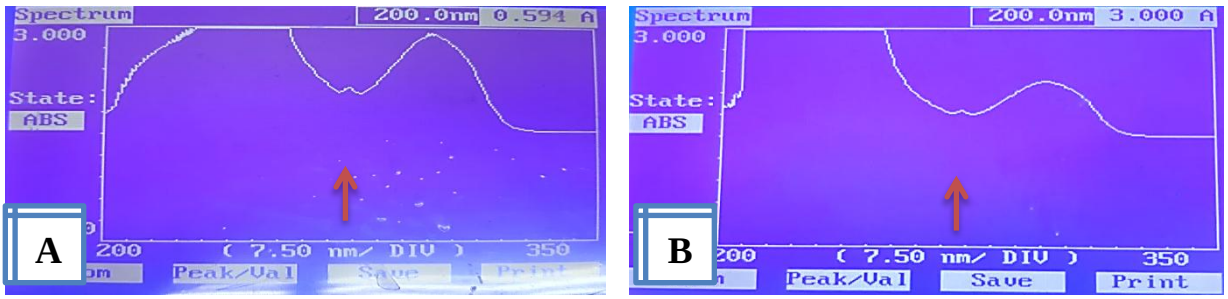


Figure (6): A, B the absorption at 274.5 nm of Glimepiride standard at 4.0×10^{-4} , 7.0×10^{-4} mmol/mL (0.4, 0.7 µmol/ml)) respectively

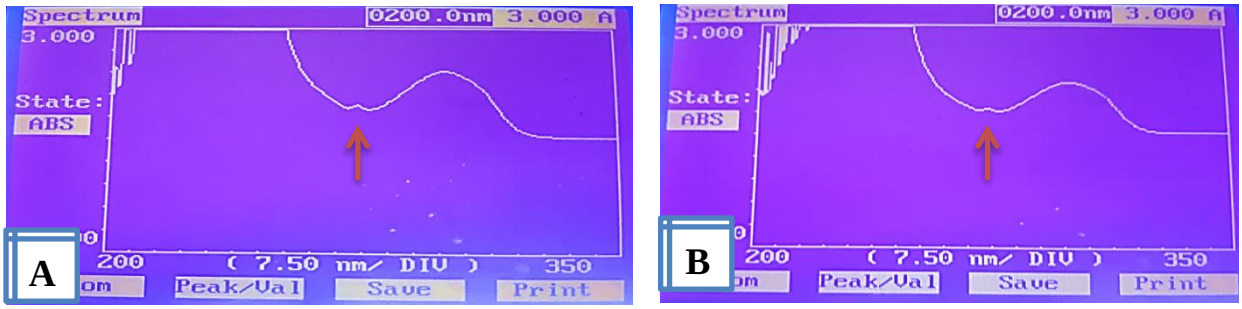


Figure (7): A,B the absorption of the concentration of Glimepiride drug (Glypride/ julphar Emirate) at 7×10^{-4} mmol/mL (0.7 µmol/ml) before& after passing through Glib-MIP column at wave length 274.5 nm

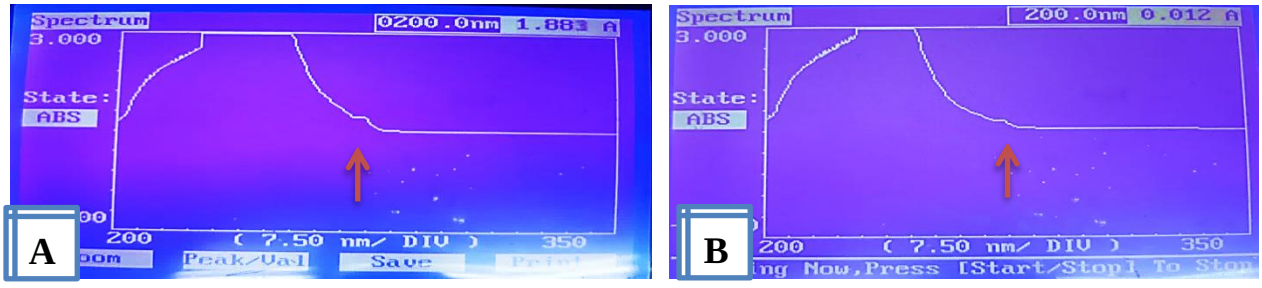


Figure (8): A,B the absorption of the concentration of Glimepiride drug (Glimepiride/UK) at 4×10^{-4} mmol/mL (0.4 µmol/ml) before& after passing through Glib-MIP column at wave length 274.5 nm.

FT-IR spectrum of standard and molecularly imprinted polymers for (Glim.)

Fourier transmission infrared spectrometer is an important chemical characterization process to detect the functional groups which have been presented in a compound. The standard of Glimepiride FT-IR spectra shows several functional groups and Glim-MIP before and after removal of template in the following Figures 9& 10A,B for Glim-MIP

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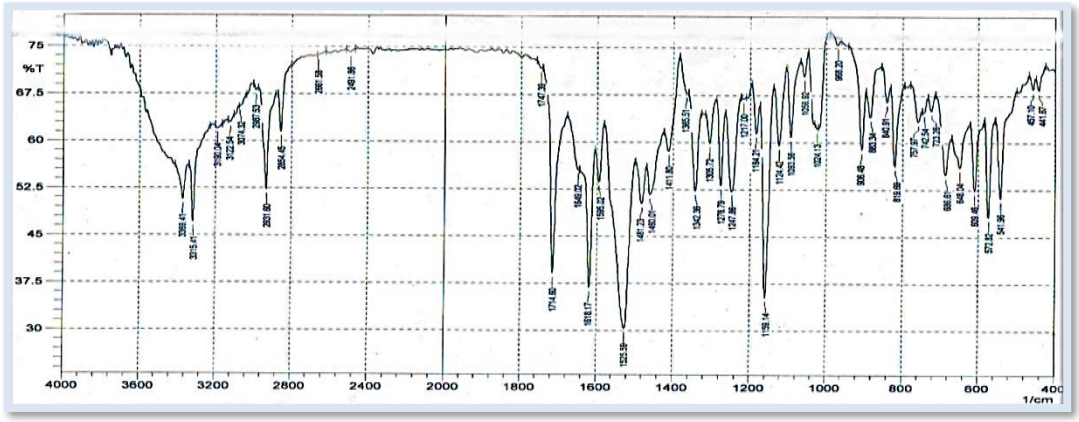


Figure (9): FTIR spectra of standard Glimepiride

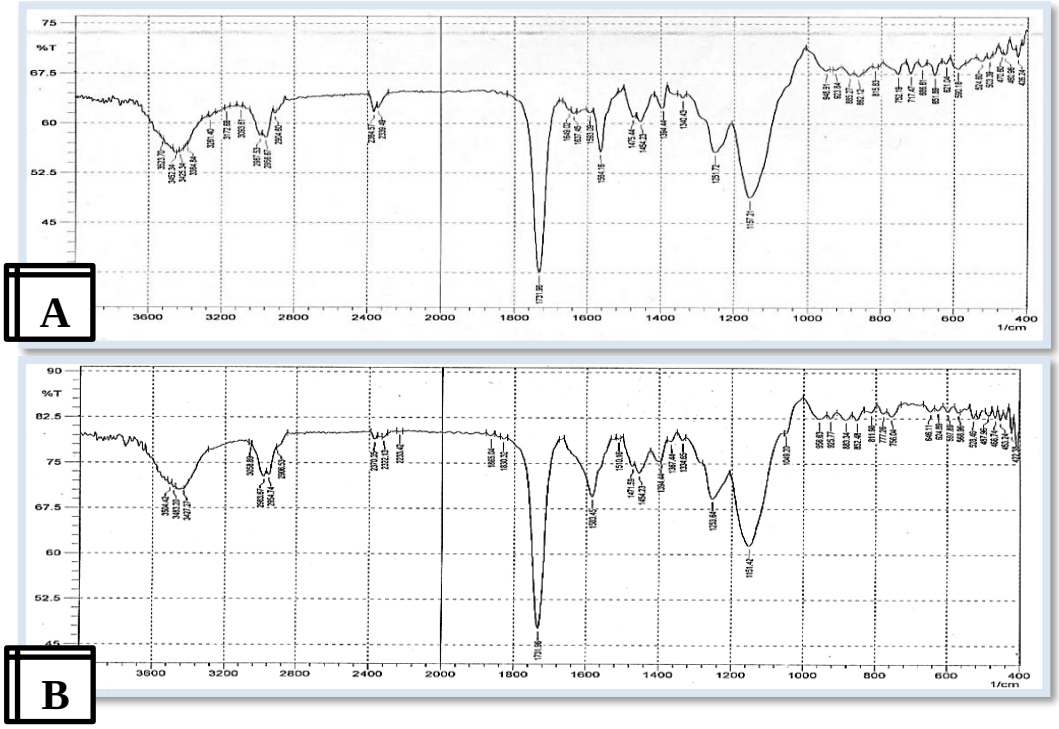
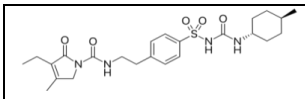
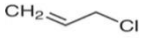
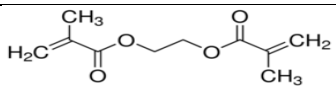


Figure (10): A,B FTIR spectrum of Glim-MIP before and after extraction (after removal the template Glimepiride.

It can be seen that the spectra for Glim- MIP before and after removal Glimepiride have approximately similar bands, that mean elution process has a slight impact on the architecture structure Table 2.

Table (2): The structures of the main three compositions of Glim-MIP and the bands indicate MIP before &after removal template

 Template Glimepiride	 Monomer Allyl chloride	 Cross linker EGDMA	
Band	**Drug(Template)**	**MIP before extraction**	**MIP after extraction**
N-H str.	**3369** **3315**	**3384** **3452**	/
Ar-H aromatic	**3074**	**3093**	/
C-H aliph.	**2931** **2854**	**2987** **2956**	**2954** **2906**
C-O est.	**1247**	**1251**	**1253**
C=O	**1714**	**1731**	**1731**
C=C ole.	**1595**	**1593**	**1583**
C=C arom.	**1618**	**1637**	/

SEM of molecularly imprinted polymers for (Glim.):

Figure 10 (A,B) shows the surface morphologies of the particles before and after elution for Glim-MIP and table 3 shows the measurements of 5 selected cavities

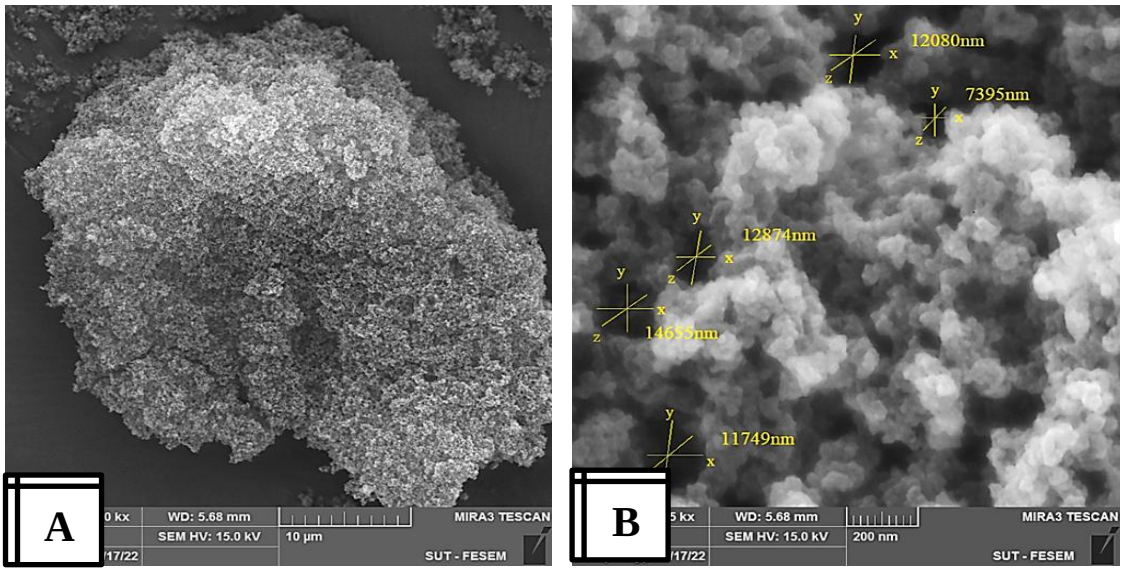


Figure (11): A, B surface morphologies of the particles before and after elution for Glim- MIP respectively, and three dimensions of cavities with their areas.

Table (3): Calculated mean, angle , lengths of some cavities (selected six of them) and their areas.

Cavities	Area	Mean	Min-Max	Angle	Length
1	305.822	11749.35	10172.19 - 15806	-0.744	152.481
2	262.694	14655.9	11989 - 20501	0	130.687
3	164.674	12874.92	11196.27 - 16132.14	0	80.524
4	176.436	7395.47	5613.045- 13208	-178.698	87.147
5	101.941	12080.53	9467.92- 18686	-177.709	49.542
Total Mean	202.313	11751.23	9687.684- 16866.63	-71.43	100.076
SD	81.426	2682.389	2472.731-2809.303	97.471	41.19
Min-Max	101.941-305.822	7395.47-14655.9	5613.045-13208 11989-20501	-178.698-0	49.542-152.481

From Figure 10 and Table 3 the 3D of Cavities between min = 7395.47nm (7.3954µm) to max = 14655.9nm (14.6559µm) we notice that the holes vary in diameter range between (7395.47-14655.9)nm and most of the holes are deep, which leads to the retention of large quantities of the drug and this is consistent with the high value of the capacity in isotherm.

Adsorption capacity and pre-concentration for Glim-MIP: A series of absorption achievement for different initial concentrations of Glim-MIP ranging from 0.04 to 1.2 µmol/ml on adsorption capacity µmol/g was studied using the following equation **(Al-Janabi, 2017; Huang *et al*, 2018)**.

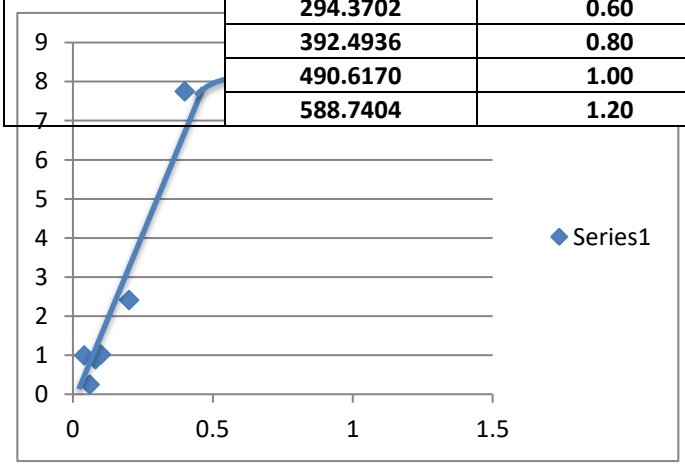
$$\mathbf{Q} = (\boldsymbol{Ci} - \boldsymbol{Cf})(\boldsymbol{\mu mol/ml}) * \frac{\boldsymbol{vol\ (ml)}}{\boldsymbol{Wof\ Mip(g)}}$$

Ci- initial concentration , Cf - final concentration (after passing through column packed with Glim-MIP)

Pre-concentration refers to the process of obtaining a high local concentration at the sensor surface, the concentrations from (0.06-1.2)µmol/ml consume (3)ml range of volumes while at concentration 0.04µmol/ml consume (4)ml, when using 0.1g weight of Glim-MIP, Table 4.

Table (4): The optimal synthesis conditions for the molecularly imprinted polymer for Glimepiride developed in this study in 0.1 g of MIP

W/ MIP (g)	Ci Conc.in (ppm)	Ci Conc.in (µmol/ml)	Cf (µmol/ml)	Vol (ml)
0.1	19.6247	0.04	0.0153	4
	29.4370	0.06	0.0517	3
	39.2494	0.08	0.0503	3
	49.0617	0.10	0.0662	3
	98.1234	0.20	0.1196	3
	196.2468	0.40	0.1417	3
	294.3702	0.60	0.3334	3
	392.4936	0.80	0.5237	3
	490.6170	1.00	0.7288	3
	588.7404	1.20	0. 9218	3

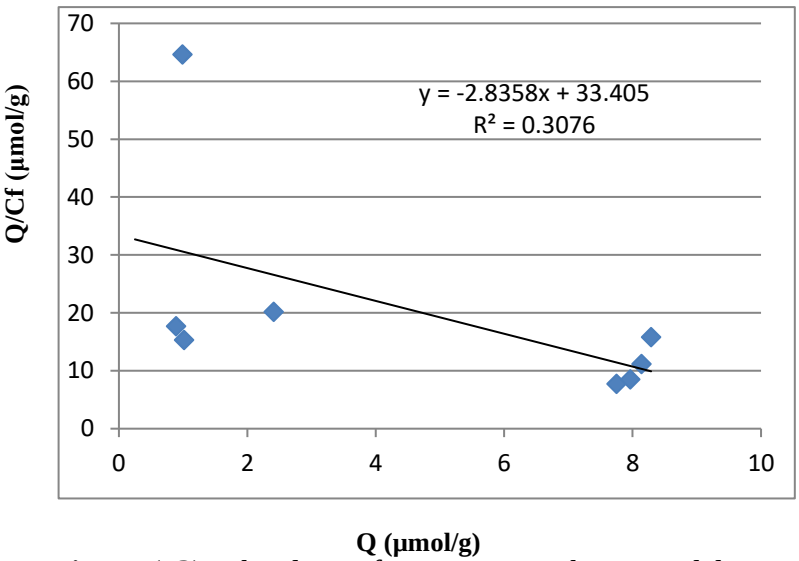


Ci (µmol/ml)

Relation between initial concentration Ci (µmol/ml) and capacityQ (µmol/g):

Ci (µmol/ml)	Q (µmol/g)
0.04	0.9884
0.06	0.2493
0.08	0.891
0.1	1.0128
0.2	2.412
0.4	7.7484
0.6	7.998
0.8	8.288
1	8.1355
1.2	7.9639

Figure (12): Illustrate Langmuir isotherm model The relation between capacity Q (µmol/g) and Q/Cf (µmol/g):



Q µmol/g	Q/Cf (ml/g)
0.9884	64.6436
0.2493	4.82293
0.891	17.7137
1.0128	15.2899
2.412	20.1672
7.7484	7.7484
7.998	23.9461
8.288	15.8278
8.1355	11.1629
7.9631	8.5192

Figure (13): The slope of Langmuir isotherm model

Table (5): Results of maximum capacity in µmol/g for Glim-MIP using 0.1g weight of MIP

Slope	Kd = -1/ slope	Intersept	Qmax= Intersept × Kd µmol/g
-2.8358	0.3526	33.405	11.7797

Qmax = 11.7797 µmol/g for Glim-MIP 0.1g weight

In human serum

1- Sample collection

In total, 5 ml of blood was gathered and placed in serum separator tubes (SST). The clot activator SST contained a gel in the form of an inert thixotropic polymer**(Schrapp *et al.*, 2019; Yasar & Konukoglu, 2020)**, which was located at the bottom, its purpose being to separate blood cells from serum through centrifugation. This was performed for each patient and healthy individual. Blood samples were allowed to stand for 5 minutes following centrifugation at ~ 2000 rpm. the serum was kept at 20ºC so that it could later be employed for the estimation of Glimepiride.

2- Procedure

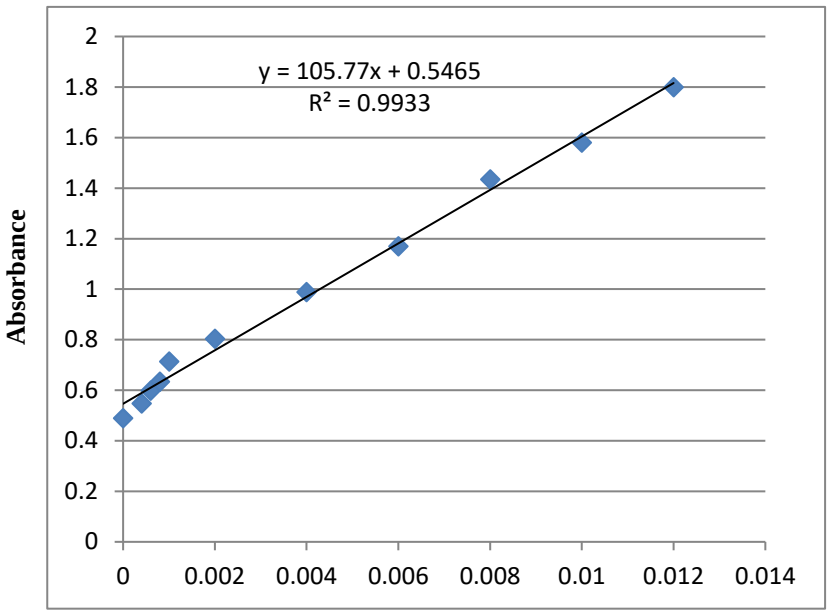
This method uses one ml of each human serum. In other words, it requires serum from the control group (healthy individuals who do not take Glimepiride) and the patient group (who take Glimepiride drug), both of which were diluted in 10 ml of deionized water. Subsequently, 1 ml of diluted serum was placed in a 10 ml volumetric flask, to which was added 2-3 drops of 1 N HCl solution, the purpose being to eliminate the viscosity of the serum **(Constable *et al.*, 2019)**. Methanol was used to make the volume up to 10 ml. The solutions were then warmed in a water bath for 10-15 minutes at a temperature not exceeding 60 ºc in order to create a transparent solution.

Several series of solutions were created for each control and patient group. This was realized through the transferal of 1 ml to each eleven volumetric flask (10 ml) (We doubled the amount of serum to get the quantity needed for 11 volumetric flask) followed by the addition of constant volumes of standard Glimepiride (0.1 ml) from different concentrations (0, 0.04, 0.06, 0.08, 0.1, 0.2, 0.4, 0.6, 0.8, 1, 1.2) µmol/ml to obtain (0, 0.0004, 0.0006, 0.0008, 0.001, 0.002, 0.004, 0.006, 0.008, 0.01, 0.012) µmol/ml. Flask No.1 is the sample (serum). The findings were subjected to mathematical evaluation ($M_1V_1=M_2V_2$ for the standard addition method) (see Table 6). Furthermore, the absorption recorded for each volumetric flask was gauged with the assistance of UV-Visible spectrophotometry, which focused on the control serum and then measured the patient serum at the maximum 274.5 nm absorption, the objective being to eradicate the majority of interferences. Subsequently, the resultant solution was scanned in the 200-350 nm range. Fig. 13 presents the calibration curve that was plotted between the concentrations and absorptions.

Table (6): Results of standard addition for the estimation of Glim in human serum.

Human	**Diluted Serum**	**1N HCl**	**Glimepiride µmol/ml**										
Control	**1 ml**	**2-3 drops**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**	**0**
Patient	**1 ml**	**2-3 drops**	**0**	**$4x10^{-4}$**	**$6x10^{-4}$**	**$8x10^{-4}$**	**$1x10^{-3}$**	**$2x10^{-3}$**	**$4x10^{-3}$**	**$6x10^{-3}$**	**$8x10^{-3}$**	**$1x10^{-2}$**	**$1.2x10^{-2}$**

Glimepiride in serum was statistically evaluated by considering the length of time the drug was in the body of the patient, the rate at which it was metabolized, and the medication dose. These variables differ between patients. In addition, Glimepiride is reported to undergo hepatic metabolism, the elimination half-life of glimepiride is approximately 5 -8 hours **(Li *et al.*, 2016)** Calibration curve between concentrations and absorptions.



Concentration of Glimepiride in serum µmol /ml by standard addition method	Absorbance of Glimepiride in serum by standard addition method
0	0.4887
0.0004	0.5467
0.0006	0.5982
0.0008	0.6331
0.001	0.7125
0.002	0.8024
0.004	0.9877
0.006	1.1695
0.008	1.4337
0.010	1.5788
0.012	1.7989

Figure (14): Calibration curve between concentrations of Glimepiride in serum using standard addition method µmol/ml and its absorbance.

When y= 0.4887 that mean the absorbance of Glimepiride in this sample of serum is 0.4887 It found that the absorption 0.4887 are nearest to the absorption 0.3314 which has concentration 0.20 µmol /ml in calibration curve (Figure 12) and substituting for y= 0.4887 the concentration is 0. 209µmol /ml. That mean the concentration of Metformin in this sample of serum is 0.2949 µmol /ml by ratio and proportion. so, a comparison for absorption of this concentration after passing through Glim-MIP column has been studied in pharmaceutical drugs solution and human serum.
*To know the concentration of drug in human serum we must multiply this concentration 0.2949µmol /ml x 10(Dilution coefficient).

DISCUSSION

This paper presents a comparison between two approaches to the drug Glimepiride The T-Test statistical evaluation**(Deckard, 2016; Haaland & Thomas, 1988)**, was designed to facilitate a comparison between the identification of Glimepiride once it had passed through the Glim-MIP syringe solid phase extraction process and the human serum at 274.5 nm:

$$/t/= \bar{X}i1 - \bar{X}i2/(S(\sqrt{1/n1 + 1/n2}))$$

If $\bar{X}i1 = \bar{X}i2$ $\longrightarrow$ **Null hypothesis when** $t_{calculated} < t_{tab}$

That mean $\bar{X}i1 - \bar{X}i2$ **= zero**

$Xi1 \neq Xi2$ ⟶ **Alternative hypothesis when** $t_{calculated} > t_{tab}$

That mean $\bar{Xi}1 - \bar{Xi}2 > < zero$

* $\bar{Xi}1 = 0.1248$ Mean for n1=3 absorption value after passing through Glim-MIP column in pharmaceutical drugs solution with S1 variance= 0.086

$\bar{Xi}2 = 0.1625$ Mean for n2=3 absorption value after passing through Glim-MIP column in human serum with S2 variance=0.075

$S^2 = (n1 -1)*S^2_1 + n2-1)*S^2_2 / n1+n2- 2$

t calculated=0.574, t tab = $t_{0.05/2, (n1+n2)-2} = 2.776$

It found $t_{calculated} < t_{tab}$ at confidence level 95% therefor there is no significant difference between two approaches, So Null hypothesis will be accepted.

CONCLUSION

New and novel bulk polymers were created by using Allyl chloride C_3H_5Cl & crosslinking Ethylene glycol dimethacrylate EGDMA $C_{10}H_{14}O_4$ as Glim-MIP, different studies and experiments were used to reach for selective molecular imprinted polymer by prepare and optimize required monomers, cross-linker using suitable solvents, porogen solvent for template removal and the optimal molar ratios of Template (Glimepiride)to monomer to cross-linker. Irregular shapes three-dimension network structure of polymers can be seen by SEM before and after removal template, FTIR, isotherm processing all improves the healthy work.

one slope gain when studied the capacity of adsorption of Glim-MIP which follow Langmuir isotherm model with uniform values (homogeneous structure), The maximum adsorption capacity was 11.7797 µmol/g for 0.1g of Glim-MIP. A standard addition method using to eliminate the interferences when detect the concentration of Glimepiride in human serum. T-Test statistical evaluation was designed to facilitate a comparison between the identification of Glimepiride once it had passed through the Glim-MIP syringe solid phase extraction process and the human serum at 274.5 nm and when it found that $t_{calculated} < t_{tab}$ at confidence level 95% by UV for Glimepiride drug therefor there is no significant difference between two methods, So Null hypothesis will be accepted.

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Al -Fahdawi & Mustafa

المجلة العراقية لبحوث السوق
وحماية المستهلك

THE COMBINED EFFECT OF BIOSTIMULANTS AND ANTIOXIDANTS ON THE VEGETATIVE, YIELD CHARACTERISTICS AND ITS COMPONENTS OF SOYBEAN

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ABSTRACT

A field experiment was carried out for the year 2022 at Experimental Station A of the College of Agricultural Engineering Sciences/ University of Baghdad/ Al-Jadriyah, located at latitude 33°N and longitude 44°E. The aim of the experiment was to determine the appropriate amount of bio-stimulants and antioxidants to produce the best chemical content of oil with a good protein ratio compared to seed and oil productivity, antioxidant compounds, and oxidative efficiency of soybean (*Glycine max* L.) seeds. The experiment was conducted according in a factorial arrangement the randomized complete block design (RCBD) for two factors and their interactions with three replication. The first factor included three levels of biostimulants: no spray, humic acid, and folic acid at a concentration of 2 g L^{-1} for each. The second factor included three levels of antioxidants: no spray, ascorbic acid, and glutathione acid at a concentration of 100 mg L^{-1} for each. Spraying was applied on vegetative system, first was one month after planting, the second after one month of the first spraying at the formation of branches stage, and the third at the 50% flowering stage. The results showed that bio stimulants had a significant effect on most growth traits, with the treatment of 2 g L^{-1} humic acid achieving the best results in growth traits, height plant (180.47cm), leaf area (104.15 dm^{-1}), number of branches(12.44 branch $plant^{-1}$) ,fresh weight (450.16 g),dry weight (308.03 g). while the treatment of 2 g L^{-1} folic acid achieved the best results in seed yield (289.10)g and total yield(3.08) g . Antioxidants also had an effect on most growth and yield traits, with the spray of 100 mg L^{-1} ascorbic acid achieving a significant increase in leaf area(98.01 dm^2), while the treatment of 100 mg L^{-1} glutathione acid outperformed significantly in fresh weight (514.46 g) , dry weight (284.56 g), plant seed yield(307.50 g $plan^{-1}$), 100 seed weight(19.47 g), total yield(3.28 t ha^{-1}), and oil yield (0.68 t ha^{-1}). The combined effect of the study factors was significant in most growth and yield traits, but the treatment of 2 g L^{-1}humic acid and 100 mg L^{-1}ascorbic acid achieved a significant increase in plant height (148.53 cm), leaf area (110.95 dm^{-1}). While the treatment of 2 g L^{-1} humic acid and 100 mg L^{-1}Glutathione acid outperformed significantly in the number of branches (13.00 branch $plant^{-}$), seed yield in the plant (337.80 g), total yield (3.60 g) , and oil yield (0.99 t h^{-1}). The combined effect of the treatment of 2 g L^{-1} folic acid and 100 mg L^{-1} Glutathione acid produced a significant increase in fresh weight (562.10 g) and 100 seed weight (22.00 g).

Keywords: Bio-Stimulants, antioxidant yield, yield components, soybean. Top of Form

[*]**Research extracted from a master's thesis by the first researcher.**

المجلة العراقية لبحوث السوق وحماية المستهلك

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التأثير المشترك للمحفزات الحيوية ومضادات الاكسدة في الصفات الخضرية والحاصل ومكوناته لفول الصويا

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الخلاصة

نفذت تجربة حقلية لعام 2022 في محطة التجارب A التابعة لكلية علوم الهندسة الزراعية/ جامعة بغداد/ الجادرية الواقعة عند خط عرض 33° شمالاً وخط طول 44° شرقاً بهدف تحديد كمية المحفزات الحيوية ومضادات الاكسدة المناسبة لأنتاج افضل محتوى كيميائي من الزيت مع نسبة جيدة من البروتين بالمقارنة مع إنتاجية البذور والزيت والمركبات المضادة للأكسدة والفعالية التأكسدية لبذور فول الصويا *Glycine max L.* طبقت التجربة وفق ترتيب التجارب العاملية وبتصميم القطاعات العشوائية الكاملة RCBD لعاملين وتداخلاتها وبثلاث مكررات ، شمل العامل الأول ثلاث مستويات من المحفزات الحيوية هي بدون رش و Humic acid و Folic acid بتركيز 2 غم لتر$^{-1}$ لكل منهما، والعامل الثاني ثلاث مستويات من مضادات الاكسدة هي بدون رش و Ascorbic acid و Glutathion acid بتركيز 100 ملغم لتر$^{-1}$ لكل منهما استخدمت رشاً على المجموع الخضري الأولى بعد شهر من الزراعة والثانية بعد شهر من الرشة الأولى مرحلة نشوء وتكون الافرع والثالثة مرحلة 50 % تزهير. أظهرت النتائج ان المحفزات الحيوية اثرت معنوياً في اغلب صفات النمو وقد حققت المعاملة 2 غم لتر$^{-1}$ Humic acid افضل النتائج في مؤشرات النمو، ارتفاع النبات (180.47 سم) المساحة الورقية (104.15 دسم^2) ،عدد الافرع (12.44 فرع نبات$^{-1}$)، الوزن الرطب (450.16غم)، والوزن الجاف (208.03 غم)الا ان 2 غم لتر$^{-1}$ Folic acid سجل افضل النتائج في حاصل البذور في النبات(289.10غم) والحاصل الكلي(3.08 طن هـ$^{-1}$) ، كما اثرت مضادات الاكسدة في معظم صفات النمو والحاصل فقد حقق الرش 100 ملغم لتر$^{-1}$Ascorbic acid زيادة معنوية في صفة المساحة الورقية (98.01 دسم^2) بينما تفوقت المعاملة 100 ملغم لتر$^{-1}$ Glutathion acid معنوياً في صفة الوزن الرطب (514.46 غم)، الوزن الجاف (284.56 غم) ، حاصل البذور في النبات(307.50 غم، وزن 100 بذرة (19.47 غم) ،الحاصل الكلي (3.28 طن هـ$^{-1}$)وحاصل الزيت (0.86 طن هـ$^{-1}$).كان التأثير المشترك لعوامل الدراسة معنوياً في اغلب صفات النمو والحاصل، إلا ان المعاملة 2 غم لتر$^{-1}$ Humic acid و100 ملغم لتر$^{-1}$ Ascorbic acid زيادة معنوية في صفة ارتفاع النبات (184.53 سم) والمساحة الورقية (110.95 دسم^2). بينما تفوقت معنوياً المعاملة 2 غم لتر$^{-1}$ Humic acid و100 ملغم لتر$^{-1}$ Glutathion acid وسجلت اعلى النتائج في عدد الافرع (13.00 فرع نبات$^{-1}$)،حاصل البذور في النبات(337.80 غم)، الحاصل الكلي (3.28 طن هـ$^{-1}$) وحاصل الزيت (0.99 طن هـ$^{-1}$).وأنتجت معاملة التأثير المشترك 2 غم لتر$^{-1}$ Folic acid و100 ملغم لتر$^{-1}$ Glutathion acidزيادة معنوية في صفة الوزن الرطب (562.10 غم) ووزن 100 بذرة(22.00 غم).

الكلمات المفتاحية: المحفزات الحيوية، مضادات الاكسدة، الحاصل، مكونات الحاصل، فول الصويا.

INTRODUCTION

Soybean (*Glycine max* L.) is considered one of the most important oil crops used in the food and pharmaceutical industries worldwide, as well as in the dye industry **(Al-Karawi, 2022).** It belongs to the fabaceae family and is distinguished from other legume species by containing all eight essential amino acids necessary for the human body to produce protein and oil. This makes it an excellent source of complete plant protein, with a content of no less than 37%. Soybean seeds also contain oil with a value that exceeds 27%, as well as sugars, saponins, and sterols. The fatty acids are the active essence of the plant, as crude soybean oil contains oleic acid, linoleic acid, and lenolenic acid, which give soybean oil greater stability, making it an antioxidant. It also contains palmitic acid, stearic acid, myristic acid, arachidonic acid, tocopherol, delta-tocopherol, and alpha-tocopherol, which work alone or together to reduce triglyceride levels in the body and lower blood sugar levels. It is considered a primary building block for muscles, bones, and nerves, and is a powerful stimulant and restorative for the body, achieving balance between cells **(Arab Organization for Agricultural Development, 2014)**.

All of these vital compounds for plants and humans can be produced by this medicinal oil plant in economical quantities. However, their concentrations in the soybean crop can be affected by the levels of organic acids and their different types, in addition to vitamins, which represent the basic structures in the secondary plant metabolism, energy compounds, enzymes and their accompaniments, and their companions. Therefore, it is a major and determinant factor in the growth and development of the crop, its transition from one stage to another until completing its life cycle and producing the seed yield. It is important to determine the appropriate amount of bio-stimulants and antioxidants for optimal production. For example, bio-stimulants are encouraged in plant growth because they are carbon-based substances or compounds that build plant tissues, and they work within mechanisms that reducing the harmful biotic and abiotic stress to which the plant is exposed to during its growth period **(Saheed & Darwesh, 2021 & Mustafa, 2022).** Spraying antioxidants on plants plays a major role in stimulating physiological and vital processes, producing oil and proteins, and manufacturing carbohydrates by building chlorophyll and stimulating the process of photosynthetic and amino acid metabolism, which contributes to the formation of proteins and other components, such as the aforementioned compounds that this crop is famous for. In combination with bio-stimulants, the production of antioxidant compounds is encouraged, including glutathione **(Conklin &Barth, 2004).** Ascorbic acid is known for its ability to revive its production, and a group of vitamins including thiamine (vit. B1), biotin (vit. H), lipoic acid, and the enzymatic co-factors coenzyme A **(Barth *et al.*, 2006),** as well as compounds like thioredoxins and sulfolipids that play an important role in the plant's resistance to pests and diseases **(Suleiman, 2017),** These factors, with their individual or interacting effects, reflect on the effectiveness of the plant's biological defense system (as they work within antioxidant mechanisms), which depends entirely on the metabolism of active compounds and their production levels. At the same time, the level of antioxidant effectiveness is determined to resist biotic stress (pest infestation) and abiotic stress (unfavorable environmental conditions) that can cause damage or disturbances in the plant cell, such as damage to DNA and RNA nucleic acids or ribosomes, resulting in the production of ineffective proteins or enzymes or damage to the cell membrane, which loses its selectivity and eventually dies.

MATERIALS AND METHODS

The field experiment was conducted at the research station A of the College of Agricultural Engineering Sciences / University of Baghdad in a sandy soil with the aim of (knowing the joint effect of bio stimulants and antioxidants on the vegetative traits, yield and its components of soybean plant) Shimaa cultivar. The first factor included biostimulants: no spray, humic acid, and folic acid at a concentration of 2 g L^{-1} for each. The second factor included three levels of antioxidants without spraying, Ascorbic acid and Glutathion acid at a concentration of 100 mg L-1 each. Field experiment was conducted at Research Station A, of the College of Agricultural Engineering Sciences / University of Baghdad, in sandy loam soil with the aim of determining the combined effect of bio-stimulants and antioxidants on the vegetative traits, yield, and yield components of Shimaa soybean variety.

The experimental field plowed twice perpendicular to each other and divided it into 27 experimental units, each consisting of 4 rows with a spacing of 0.75 cm between rows and 0.25 cm between plants, resulting in a plant density of 53,333 plants ha^{-1}. Superphosphate fertilizer was added at a rate of 80 kg ha^{-1} of triple superphosphate P_2O_5 in one application before

planting, while urea fertilizer at a rate of 160 kg ha^{-1} of 46% N was added in two applications, the first at the vegetative stage and the second at the beginning of the flowering stage **(Ali, 2012)**. Crop management practices including irrigation and weeding were carried out as needed, and the crop was harvested at maturity. Statistical analysis was performed using the Genstat software, and the least significant difference (LSD) test was used to compare means at a probability level of 0.05 **Steel & Torri (1980)**

The studied traits are:

1- Plant height (cm): The average of five plants was calculated using the metric tape from the soil surface to the top of the plant
2- Number of branches per plant.: According to the average number of branches on the main stem and the bearer of the pods
3- Leaf area (dm^2 $plant^{-1}$):.LA = 0.624 + (0.723) (L .W) (**Wiersma & Bailey, 1975**)
4- Fresh weight of the plant (g) :Using the sensitive scale for five plants and calculating their average
5- Dry weight of the plant (g). :Using the sensitive scale of five dried plants and calculating their average
6- Seed yield per plant (g).: The seeds of five plants were weighed and averaged
7- 100-seed weight (g).: After mixing the seeds, I weighed and weighed 100 seeds using the sensitive scale and calculated their average
8- Total seed yield (t ha^{-1}).: Five plants were randomly harvested from the two central markers of the experimental unit, weighed, their average extracted, and converted from $plant^{-1}$ gm to ton hectare by multiplying them by the plant density and dividing the result by 10^6.
9- Plant oil yield (t ha^{-1}): According to the oil yield × total seed yield

RESULTS AND DISCUSSION

1. Plant height

The results indicate that plants treated with biological stimulants significantly outperformed untreated plants in terms of plant height, specifically with 2 g L^{-1} Humic acid treatment mean at 180.47 cm compared to untreated plants averaging of 164.46 cm, while 2 g L^{-1} of Folic acid treatment mean at 167.78 cm (Table 1). The results showed that the antioxidant concentrations of 100 mg L^{-1} Glutathione acid and 100 mg L^{-1} Ascorbic acid did not differ significantly from untreated plants, as the latter achieved the highest mean plant height at 173.91 cm. Regarding the combined effect of the study factors, the results revealed that plant height was significantly affected by the 2 g L^{-1} Humic acid treatment with both untreated and treated plants, as well as by 100 mg L^{-1} Ascorbic acid with an increase of 11.47% and 10.27%, respectively, compared to untreated plants. The latter also achieved an increase of 7.60% with 100 mg L^{-1}Ascorbic acid. This is attributed to the effective role of Humic acid in increasing membrane permeability and nutrient transfer, which helps activate the serine with indole ring to form tryptophan, which is the source of the hormone auxin (IAA) that leads to cell division and elongation, thus increasing plant height (**Abid Al-Ameen ,2010).** This is consistent with the findings of **Abdul Aziz *et al.*, (2018)** & **(Mahmood and Zeboon(2019),** &**Abdul Qadir *et al.*, (2022)** on the role of Ascorbic acid in activating photosynthesis and increasing cell division and expansion, as concluded by **Al-Aboudi *et al.*, (2016).**

Table (1): The combined effect of biostimulants and antioxidants and the interaction between them on plant height (cm) of Soybean (2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	167.33	155.50	171.11	164.64
Humic acid 2gm	186.53	184.53	170.37	180.47
Folic acid 2gm	167.87	168.40	167.00	167.78
$LSD_{0.05}$		8.11		4.68
Mean antioxidants	173.91	169.5	169.49	
$LSD_{0.05}$		N.S		

2. Leaf area (dm^2)

The results indicate that plants treated with 2 g L^{-1} Humic acid achieved the highest mean leaf area of 104.15dm^2 (Table 2), while 2 g L^{-1} of Folic acid treatment mean at 88.44 dm^2, compared to untreated plants which had the lowest mean of 86.97 dm^2. The same table shows that plants treated with 100 mg L^{-1}Ascorbic acid had a significant difference in mean leaf area of 98.01 dm^2, followed by plants treated with 100 mg L^{-1}Glutathione acid with mean of 92.24 dm^2, compared to untreated plants with mean of 89.31 dm^2. Regarding the combined effect of the study factors, Table 2 revealed that plants sprayed with 2 g L^{-1} Humic acid and 100 mg L^{-1} Ascorbic acid achieved a significant increase of 30.18% in leaf area compared to untreated plants. Plants treated with 2 g L^{-1}Humic acid with distilled water also achieved a significant increase of 27.16% compared to untreated plants. This is attributed to the effective role of Humic acid in the biological processes involved in photosynthesis, respiration, and the plant's ability to utilize solar energy, which positively influenced the increase in green biomass, including leaf area **Danta (2007),** consistent with the findings of **Abdul Qader *et al.* (2022).** Ascorbic acid also works to stimulate cell division and expansion, as well as protect chloroplasts from oxidation **Al-Alaf (2017).** This is consistent with what was found by **Alak and Al-Sabagh, (2020) .**

Table (2): The combined effect of bio stimulants and antioxidants and the interaction between them on leaf area (dm^2) of Soybean (2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	85.23	85.49	90.21	86.97
Humic acid 2gm	108.38	110.95	93.13	104.15
Folic acid 2gm	74.34	97.59	93.39	88.44
$LSD_{0.05}$		5.94		3.43
Mean antioxidants	89.31	98.01	92.24	
$LSD_{0.05}$		3.433		

3. Number of branches (branch $plant^{-1}$)

Results show that plants treated with 2 g L^{-1} Humic acid achieved the highest mean number of branches with 12.44 branch $plant^{-1}$ (Table 3), while the 2 g L^{-1} of Folic acid treatment mean at 10.67 branch/$plant^{-1}$, compared to untreated plants which had the lowest

mean of 9.78 branch plant^{-1}. The results indicates that the general behavior of antioxidant-treated plants differed significantly. Plants treated with 100 mg L^{-1} Glutathione acid had the highest mean of 11.22 branch plant^{-1} compared to untreated plants with an mean of 11.34 branch plant^{-1}, while plants treated with 100 mg L^{-1}Ascorbic acid had the lowest mean of 10.33 branch plant^{-1}. Regarding the combined effect of the study factors, the same table revealed that treatment with 2 g L^{-1}Humic acid with distilled water and 2 g L^{-1}Humic acid with 100 mg L^{-1}Glutathione acid with distilled water achieved an increase of 62.36% and 59.90%, respectively, compared to untreated plants. This is attributed to the efficient distribution of products of photosynthesis between different plant parts, which plays a major role in increasing cytokinin's that counteract auxins, leading to the differentiation of the vascular connection area between lateral buds and stem and the growth of more vegetative branches. Additionally, Glutathione acid plays a role in the process of cell division and differentiation in flowers **Noctor *et al.*, (2011**) This is consistent with what was found by **Al-Hasani (2018) .**

Table (3): The combined effect of bio stimulants and antioxidants and the interaction between them on Number of branches (branch plant^{-1}) of Soybean(2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	**8.13**	**10.11**	**11.10**	**9.78**
Humic acid 2gm	**13.20**	**11.13**	**13.00**	**12.44**
Folic acid 2gm	**12.33**	**9.76**	**9.93**	**10.67**
LSD$_{0.05}$	1.361			0.78
Mean antioxidants	11.22	10.33	11.34	
LSD$_{0.05}$	0.78			

4. Plant fresh weight (g)

The results indicate that the bio-stimulants achieved significant differences in the fresh weight of the plant (Table 4). The treatment with 2 g L^{-1} Humic acid and the treatment with 2 g L^{-1} Folic acid recorded the highest mean for the trait, reaching 450.16 g and 446.50 g, respectively, while the untreated plants recorded the lowest mean of 366.93 g. As for the effect of antioxidants, Table 4 shows that the treatment with 100 mg L^{-1} Glutathione acid achieved the highest mean of 514.46 g, followed by the treatment with 100 mg L^{-1} Ascorbic acid with an mean of 417.43 g, compared to the untreated plants which recorded the lowest mean of 331.70 g. The combined treatment showed significant differences, especially the treatment with 2 g L^{-1} Folic acid with 100 mg L-1 Glutathione acid, and the treatment with 2 g L^{-1} Humic acid with 100 mg L^{-1} Glutathione acid, with an increase of 81.91% and 80. 58%, respectively compared to the untreated plants . The reason for the increase is attributed to the role of Humic acid as a biological enhancer, which increased the plant's ability to efficiently carry out carbon metabolism and absorb water and nutrients, which reflected an increase in plant size and thus an increase in fresh weight (**Ferrara & Brunetli, 2010).** The positive role of Glutathione acid and its cycle in overall biological processes within the plant, including increasing the rate of cell division, also contributed to the increase in fresh weight **Noctor ,2011).**

Table (4): The combined effect of bio stimulants and antioxidants and the interaction between them on plant fresh weight (g) of Soybean(2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	309.00	368.60	423.20	366.93
Humic acid 2gm	385.80	406.60	558.10	450.16
Folic acid 2gm	300.30	477.10	562.10	446.50
$LSD_{0.05}$		30.86		0.78
Mean antioxidants	331.70	417.43	514.46	
$LSD_{0.05}$		17.82		

5- Dry weight (g)

The results shows that the biostimulants have significantly increased the dry weight of the plants (Table, 5), particularly the treatment with 2 g L^{-1} Humic acid which recorded the highest mean of 308.03 g compared to the untreated plants which had the lowest mean of 234.63 g. The plants treated with 2 g L^{-1} Folic acid also recorded a significantly higher mean of 249.30g compared to the untreated plants. The results also indicate that the antioxidants have significantly increased the dry weight, with the treatment of 100 mg L^{-1} Glutathione acid recording the highest mean of 284.56 g, followed by the treatment of 100 mg L^{-1} Ascorbic acid with an mean of 262.36 g, while the untreated plants recorded the lowest mean of 245.03 g.

Regarding the combined effect of the study factors, it was observed from the same table that the plants treated with 2 g L^{-1} Humic acid with distilled water and the plants treated with 2 g L^{-1} Humic acid with 100 mg L^{-1} Ascorbic acid achieved an increase percentage of 61.26% and 59.95%, respectively, compared to the untreated plants. The increase in dry weight can be attributed to the positive effect of the biostimulants on the growth indicators, including plant height, leaf area, and number of branches as shown in Tables (1), (2), and (3), which positively affected the dry weight of the plants. This is consistent with the findings of **Baqer & Zboun, (2019)** in their study on the response of wheat to foliar spraying with humic acid.

Table (5): The combined effect of bio stimulants and antioxidants and the interaction between them on plant dry weight (g) of Soybean(2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	199.00	234.00	270.90	234.63
Humic acid 2gm	320.90	318.30	284.90	308.03
Folic acid 2gm	215.20	234.80	297.90	249.30
$LSD_{0.05}$		49.96		28.84
Mean antioxidants	245.03	262.36	284.56	
$LSD_{0.05}$		28.84		

6- Seed yield per plant (g).

The results indicate a significant effect of the biological stimulants on the seed yield of the plants (Table 6). Treatment with 2 g L^{-1} Folic acid produced the highest mean seed yield of

289.10 g plant^{-1}, while treatment with 2 g L^{-1} Humic acid resulted in an mean of 272.03 g plant^{-1}. The control treatment had the lowest mean seed yield of 196.33 g plant^{-1}. The same table also shows that treatment with antioxidants, especially 100 mg L^{-1} Glutathion acid, significantly outperformed the control group with an mean seed yield of 307.50 g plant^{-1}. Treatment with 100 mg L^{-1} 1 Ascorbic acid resulted in an average seed yield of 257.90 g plant^{-1}, which was also significantly higher than the control group with an mean seed yield of 192.06 g plant^{-1}.

Regarding the combined effect of the factors, treatment with 2 g L^{-1} Humic acid and 100 mg L^{-1} Glutathion acid, as well as treatment with 2 g L^{-1} Folic acid and 100 mg L-1 Glutathion acid, both showed a significant increase in seed yield compared to the control treatment, with percentage increases of 150.22% and 145.48%, respectively. The reason for the increase can be explained by the interaction of factors with each other, which led to an increase in plant yield.

Table (6): The combined effect of bio stimulants and antioxidants and the interaction between them on plant seed yield (g plant^{-1}) of Soybean(2022)

Bio stimulants g L^{-1}	**Antioxidants mg L^{-1}**			**Mean Bio stimulants**
	Control	**Ascorbic acid 100 mg**	**Glutathion acid 100 mg**	
Con.	135.00	200.70	253.30	196.33
Humic acid 2gm	189.50	288.80	337.80	272.03
Folic acid 2gm	251.70	284.20	331.40	289.10
LSD$_{0.05}$	30.53			17.62
Mean antioxidants	192.06	257.90	307.50	
LSD$_{0.05}$	17.62			

7- The weight of 100 seeds (g)

The results indicate non-significance effect between treatments of biostimulants. The same table shows a significant effect when plants are treated with antioxidants, as the treatment of 100 mg L^{-1} Glutathione acid achieved the highest mean of 19.47 g compared to the treatment of 100 mg L^{-1} Ascorbic acid, which recorded an mean of 15.63 g, while non-treated plants recorded the lowest mean at 13.23 g. The combined effect of the study factors was significant, and the plants treated with 2 g L^{-1} Folic acid and 100 mg L^{-1} Glutathione acid exceeded non-treated plants by a percentage increase of 69.62%, followed by plants treated with 100 mg L^{-1} Glutathione acid with distilled water, which recorded a significant increase over non-treated plants of 43.95%. This trait is related to the efficiency of the photosynthetic process and the interrelated relationship between the source and the sink and the activation of physiological activities within the plant, as the weight of the seeds is determined according to the activity of the plant and the quantity and quality of the primary and secondary metabolic substances formed for it. This is consistent with what was reached by **Mahmoud (2019)** on the maize plant.

Table (7): The combined effect of bio stimulants and antioxidants and the interaction between them on weight of 100 seeds (g) of Soybean(2022)

Bio stimulants	**Antioxidants mg L^{-1}**	**Mean**

g L^{-1}	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	Bio stimulants
Con.	12.97	16.57	18.67	16.07
Humic acid 2gm	13.33	16.67	17.75	15.91
Folic acid 2gm	13.40	13.66	22.00	16.35
$LSD_{0.05}$	1.24			N.S
Mean antioxidants	13.23	15.63	19.47	
$LSD_{0.05}$	0.71			

8- Total grain yield (t ha^{-1})

The results show a significant effect of biostimulants (Table 8). Plants treated with 2 g L^{-1} Folic acid achieved mean of 3.08 t ha^{-1}, while plants treated with 2 g L^{-1} Humic acid recorded mean of 2.90 t ha^{-1} compared to non-treated plants that gave the lowest mean at 2.09 t $ha^{-1.}$ The results also showed that plants treated with 100 mg L^{-1} Glutathione acid achieved mean of 3.28 t ha^{-1} compared to plants treated with 100 mg L^{-1} Ascorbic acid, which recorded mean of 2.75 t ha^{-1}, while non-treated plants recorded the lowest mean at 2.04 t ha^{-1}. The combined effect of the study factors was significant, as plants treated with 2 g L^{-1} Humic acid and 100 mg L^{-1} Glutathione acid and plants treated with 2 g L-1 Folic acid and 100 mg L-1 Glutathione acid showed a significant increase of 151.75% and 146.85%, respectively, compared to non-treated plants for the two treatments respectively. Total yield is the final result of the biological activities carried out by the plant, and the effective role of biostimulants and Glutathione acid in improving physiological traits has contributed to increasing plant efficiency in carrying out the photosynthetic process and increasing its metabolic products, which effectively contributed to increasing the total yield. This is consistent with what was reached by **Al-Saeedi (2018**) when spraying Humic acid on fenugreek plants and spraying Folic acid on maize plants (**Yassin, 2020).**

Table (8): The combined effect of bio stimulants and antioxidants and the interaction between them on total grain yield (g) of Soybean(2022)

Bio stimulants g L^{-1}	Antioxidants mg L^{-1}			Mean Bio stimulants
	Control	Ascorbic acid 100 mg	Glutathion acid 100 mg	
Con.	1.43	2.14	2.70	2.09
Humic acid 2gm	2.02	3.08	3.60	2.90
Folic acid 2gm	2.68	3.03	3.53	3.08
$LSD_{0.05}$	0.32			0.18
Mean antioxidants	2.04	2.75	3.28	
$LSD_{0.05}$	0.18			

9- oil yield (t ha^{-1})

The results showed that the biostimulants achieved a significant increase, and the treatment of 2 gm L^{-1} Humic acid with the highest yield of oil (0.81 ton ha^{-1}), which outperformed the untreated plants with mean of 0.61 ton ha^{-1}. Antioxidants also had a

significant effect, as Treatment with 100 milligrams per liter of glutathione acid recorded the highest mean of 0.86 tons ha^{-1}, while untreated plants recorded the lowest mean at 0.57%. There was a significant interaction between the study factors, as Treatment with 2 grams per liter of humic acid and 100 milligrams per liter of glutathione acid achieved the highest value of 0.99 tons ha^{-1}, followed by Treatment with 2 grams per liter of humic acid and 100 milligrams per liter of ascorbic acid with a value of 0.87 tons ha^{-1}, while untreated plants recorded the lowest value for the trait at 0.43 tons ha^{-1}. The oil yield is a final result of the percentage of oil and the total yield of the plant, so the increase is the result of the positive relationship between them.

Table (9): The combined effect of bio stimulants and antioxidants and the interaction between them on oil yield (t ha^{-1}) of Soybean(2022)

Bio stimulants g L^{-1}	**Antioxidants mg L^{-1}**			**Mean Bio stimulants**
	Control	**Ascorbic acid 100 mg**	**glutathione acid 100 mg**	
Con.	0.43	0.63	0.76	0.61
Humic acid 2gm	0.57	0.87	0.99	0.81
Folic acid 2gm	0.71	0.76	0.82	0.76
$LSD_{0.05}$	0.09			0.18
Mean antioxidants	0.57	0.75	0.86	
$LSD_{0.05}$	0.01			

CONCLUSIONS

1- Treatment with 2 g L^{-1} of humic acid outperformed in most vegetative growth traits. However, Treatment with 2 g L^{-1} of folic acid achieved the highest mean for seed yield in the plant and total yield.

2- Treatment with 100 mg $L^{-1}$of glutathione acid resulted in an increase in most growth traits, including fresh weight, dry weight, seed yield, 100-seed weight, total yield, and oil yield, while Treatment with 100 mg L^{-1} of ascorbic acid recorded a significant increase only in the leaf area.

3- The interaction between the study factors had a significant effect on most study traits in the desired direction. Treatment with 2 g $L^{-1}$of humic acid and 100 mg $L^{-1}$of glutathione acid and Treatment with 2 g $L^{-1}$of folic acid and 100 mg $L^{-1}$of glutathione acid resulted in a significant increase in most vegetative growth traits and yield.

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Mahmoud & Essa

المجلة العراقية لبحوث السوق
وحماية المستهلك

USING A SCANNING ELECTRON MICROSCOPE IN DIAGNOSING OF CLAY MINERALS IN SOME IRAQI RICE SOILS

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ABSTRACT

The Middle Euphrates region, represented by the governorates of Diwaniyah and Al-Najaf, was chosen to conduct the current study. As the two governorates are famous for cultivating various varieties of rice crop. Two methods using to irrigated these soils during the growing season, and called locally wet and dry methods. The morphological features of clay minerals were studied using scanning electron microscope (SEM), to describe the changes that occurred to the mineralogical features of these minerals due to the influence of the irrigation patterns used during irrigation of these soils. The scanning electron microscope (SEM) results show that were many changes in morphological features occurs such as in size and shape of clay minerals, in particular to smectite minerals. The scanning electron microscope (SEM) figure of clays in Al-Najaf$_{(dry)}$ soil showed some of particles appeared as well-formed imperfect hexagonal shape, which revealed that these particles belong to the chlorite mineral. While the mica minerals were appeared in lath-shaped, and rounded flakes in clays of all studied soils. Whereas the montmorillonite particles appear as a thin, webby crust, and have a flat, perming morphology. The variation in the size of the montmorillonite particles was adopted as a basis for the occurrence of the Mg-hydroxide layer within the interlayers of montmorillonite.

Keywords: Clay Minerals, Formal appearances, Rice Soils.

استخدام المجهر الألكتروني الماسح في تشخيص المعادن الطينية في بعض ترب الرز العراقية

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الخلاصة

تم اختيار منطقة الفرات الأوسط ممثلة بمحافظتي الديوانية والنجف لإجراء الدراسة الحالية. حيث تشتهر المحافظتان بزراعة أصناف مختلفة من محصول الرز. هناك طريقتان تستخدمان لري هذه التربة خلال موسم النمو، وتسمى بالطرق الرطبة والجافة محليًا. تمت دراسة المظاهر الشكلية للمعادن الطينية باستخدام، المجهر الإلكتروني الماسح، لوصف التغيرات التي حدثت في المظاهر المعدنية لهذه المعادن بسبب تأثير أنماط الري المستخدمة أثناء ري هذه التربة. تظهر نتائج المجهر الإلكتروني الماسح أن العديد من التغييرات في المظاهر الشكلية تحدث مثل حجم وشكل معادن الطين، ولا سيما معادن السمكتايت . أظهرت صور المجهر الإلكتروني الماسح (SEM) للطين في تربة النجف أن بعض

[*]**The research is taken from a master's thesis by the first researcher.**

المعادن ظهرت بشكل جيد سداسي الشكل غير كامل، مما أظهر أن هذه الجزيئات تنتمي إلى معدن الكلوريت. بينما ظهرت معادن الميكا في شكل رقائق دائرية الشكل في الطين من جميع الترب المدروسة. في حين أن دقائق معدن المونتموريلونايت تظهر على شكل قشرة رفيعة متشابكة، ولها شكل مسطح ومتعرج . تم اعتماد التباين في حجم دقائق معدن المونتموريلونيت كأساس لحدوث طبقة Mg-hydroxide داخل الطبقات البينية للمونتموريلونيت.

الكلمات المفتاحية : معادن الطين ، المظاهر الشكلية ، تربة الرز.

INTRODUCTION

The formation and transformation of clay minerals and their various characteristics are among the important processes in most soils in general, including the soils of rice farms in particular. As the nature of their presence, content and different characteristics they show, reflect the states of oxidation and reduction in those soils, just as their degree of crystallization, surface area and charge density affect their surfaces in the soil of rice farms (Churchman **&** Velde, 2019). Clay mineralogical studies using other methods such as X-ray, DTA, and IR are still scarce **(Al-Jibury & Essa; 2016, Yang *et al*; 2018; Al-Khalil & Essa, 2020; Al-Shamary & Essa, 2020).**

There are many studies have been done on Iraqi soils, which were used both types of electron microscope, scanning electron microscope (SEM) and transmission electron microscope (TEM) for diagnosis of clay minerals. In a study conducted by (**Al-Shamary, & Essa, S.K. 2020)** she used SEM to diagnose some of clay minerals and found that the montmorillonite particles appeared in cloudy-shape with indistinct edges, while the mica particles were appeared as lathe elongated-shape. Also, (**Shahad, 2021)** during their study on Iraqi rice soils, were used a SEM to study the morphological features of clay minerals, and they found that the edges of mica particles appeared in a pale color that distinguishes them from the surface of lath-shaped particles.

In the present study, an attempt has been taken to characterize the clays in the rice soils of Middle Euphrates region using SEM technique, and due to the lack of adequate studies dealing with the study of the effect of variation in irrigation patterns within the soil of rice farms on the nature of the presence and properties of clay minerals, the importance of the current study came to diagnose the characteristics and type of clay minerals by using SEM .

MATERIALS AND METHODS

The Middle Euphrates region represented by the governorates of Al-Diwaniyah and Al-Najaf was chosen to conduct the current study (Table-1). The two governorates are famous for cultivating multiple varieties of the rice crop. The study soils are exploited by rice crop and irrigated by two methods (Flooded and Dry) during the growing season.

Table (1): some of chemical properties of the study soil

Organic matter	Active lime	Carbonate minerals	$EC_{1:1}$ Desi siemens $^{1-}$m	$pH_{1:1}$	Depth cm	irrigation method	location
$^{1-}$g kg							
8.1	50.0	175.82	3.3	7.0	30 - 0	dry 1	Diwaniyah
9.6	57.5	87.91	6.3	7.3	60 - 31		
6.0	50.5	219.78	2.8	7.5	30 - 0	dry 2	Diwaniyah
6.5	60.0	197.80	2.9	7.3	60 - 31		
8.1	47.5	246.15	1.8	7.6	30 - 0	wets 1	Diwaniyah
7.0	50.5	250.55	1.7	7.4	60 - 31		
5.0	50.0	261.54	1.2	7.5	30 - 0	wets 2	Diwaniyah
4.0	80.0	221.98	2.2	7.6	60 - 31		
16.6	70.0	248.35	7.1	7.0	30 - 0	control	Diwaniyah
13.6	85.0	259.34	6.9	7.1	60 - 31		
8.3	60.0	217.58	1.0	7.2	30 - 0	dry 1	Najaf
9.6	70.0	237.36	1.8	7.2	60 - 31		
11.6	60.0	224.18	1.9	7.6	30 - 0	dry 2	Najaf
5.0	85.0	204.40	2.7	7.5	60 - 31		
4.0	55.5	123.08	1.5	7.5	30 - 0	wets 1	Najaf
3.0	75.0	228.57	1.4	7.6	60 - 31		
5.0	70.0	175.82	1.4	7.6	30 - 0	wets 2	Najaf
4.0	85.0	197.80	2.0	7.5	60 - 31		
6.11	55.0	224.18	20	7.1	30 - 0	control	Najaf
5.8	90.0	263.74	18.30	7.1	60 - 31		

The flooded or (wet) method is the traditional method used to grow rice crop, in which the soil is flooded throughout the growing season of the rice crop, while the (dry) method has been applied recently in Iraq to irrigate the rice crop, in order to water consumption, so that the soil is flooded for three days, and followed by three days drying till mid of season, and then soil flooded till end of season. Two sites were chosen in each governorate, as the first site represents: soil grown with the rice crop and irrigated by wet method. Whereas the second site

represents a soil grown with rice crop and using the dry method to irrigate it. An additional site was chosen in each governorate for uncultivated soils, which were considered as comparative soils. Soil samples were taken from all sites at depths of 0-30 and 31-60 cm, and air dried, crashed and passed through 2 mm sieve. The air-dried soil samples <2mm were dispersed in distilled water, and then the binding materials were removed, using NaOAc- at pH 5 for removing $CaCO_3$, while the organic matter was removed by treating the soil samples with NaOCl- 14%, and the free oxides were removed with citrate bicarbonate dithionate (C.B.D). The sand fraction was separated from the clay and silt fractions using a 50 µm sieve. While the clay fraction < 2 µm was separated from the silt by repeated sedimentation and syphoning based on the Stocks Law, taking into account the separation conditions of temperature and the size of the particles as stated in (**Jackson, 1979**). After the completion of the separation processes, the clay samples was taken and air-dried, and scanned under a scanning electron microscope SEM of the type Inspect S50. SEM imaging was carried out using Inspect S50 scanning electron microscope. The SEM was operated at 20 KV.

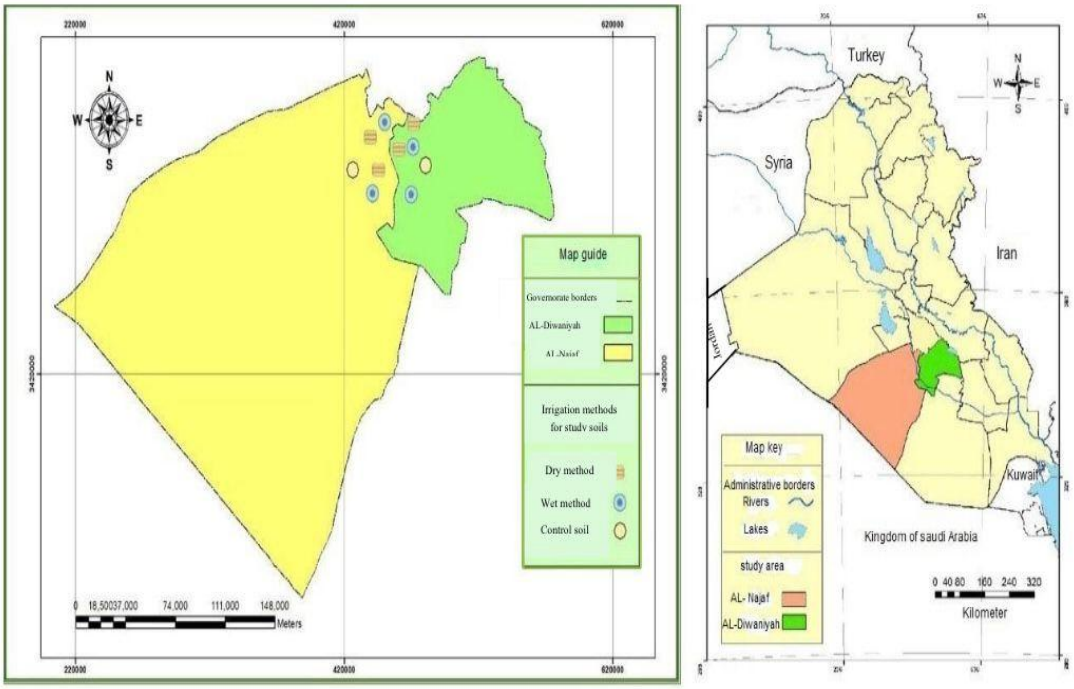


Figure (1): The locations of study area.

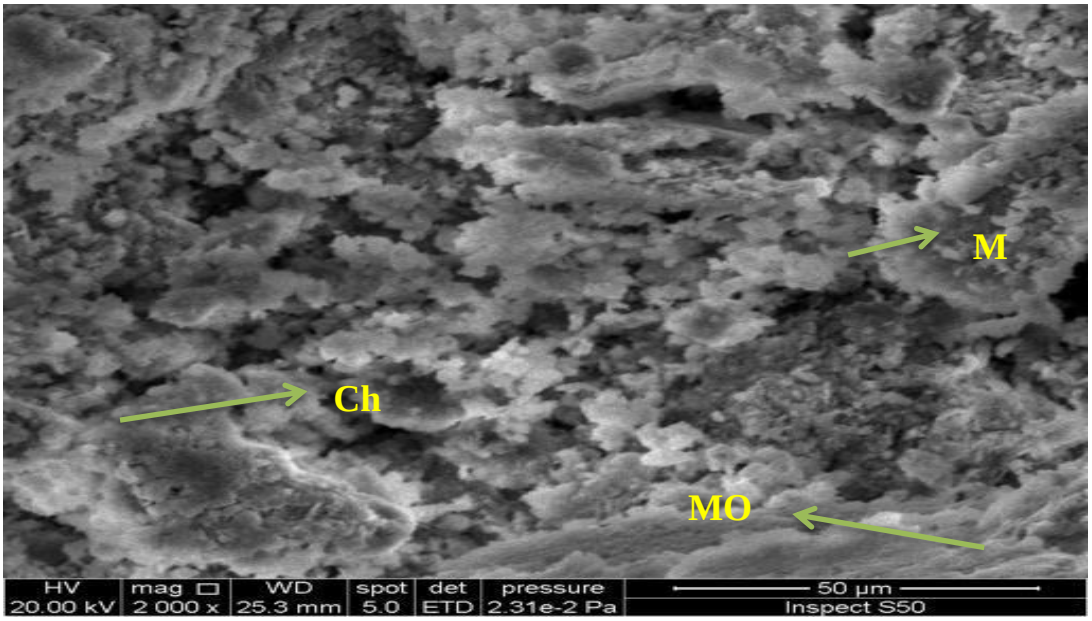
RESULTS AND DISCUSSION

SEM Inspection

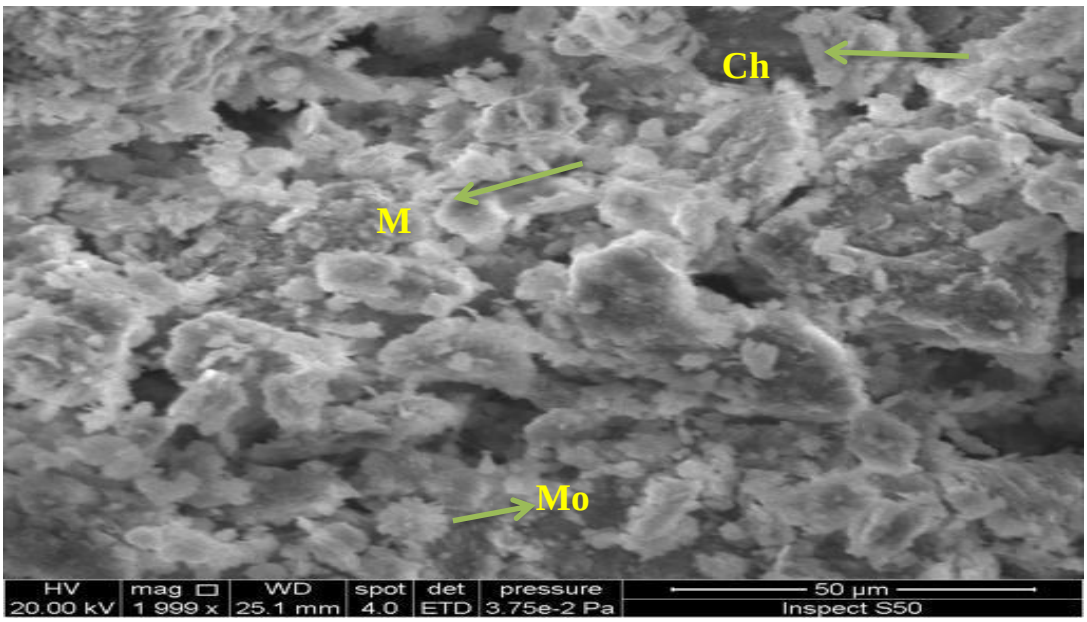
The SEM was used to studying the morphological features of clay minerals in the studied soils, to investigate the changes that occurred to the mineralogical structure of these minerals, due to the impact of the irrigation patterns used in the irrigation of the study soils.

The SEM of the clays sample at depth of 0-30 cm in Al-Najaf$_{(dry)}$ soil (Plate 1A) revealed that some of particles appeared as well-formed imperfect hexagonal shape, which confirms that these particles belong to the chlorite mineral. The scanning results in (plate 1A) showed that the chlorite particles appeared in different sizes, and some of their edges were exposed to weathering processes. These results are in agreement with finding of many studies **(Alam *et al*, 1999; Rajkumar *et al*, 2014; Perri, 2020 & Laird, 2001)**. Also, the lath-shaped, rounded, and semi defined flake edges that have been diagnosed in (plate 1A) were belong to the mica minerals, as diagnosed in many previous studies (**Bohor & Hughes, 1971; Dixon et al, 1977; Essa & Al-Sheikhly, 2001**). In addition, the results of scanning in (plate 1A) showed that some of mica edges appeared in pale color, which was completely surrounds the particle or part of it. These morphological features of mica edge particles can interpret into two possibilities. First, the pale color is result of exposing these particles to the weathering processes, perhaps within the locations of their sources, or during transport and sedimentation (**Meunier & Velde, 2004; Almashhadani & Al-Hasanay, 2023**). These results came in agreement with several studies (**Al-Dahi, 2009; Majeed, 2017**) which were conducted on Iraqi soils. While the second possibility is exposure of these particles to the weathering in current sites, as a result of the effect of alternating wet-dry cycles, these finding consistent with (**Shahad, 2021**) during his study of rice soils in some governorates of the Middle Euphrates in Iraq.

Results of (plate 1A) also, showed the presence of montmorillonite particles, which appeared as cloudy shaped fluffy mass flaks. While some of the montmorillonite particles appears as a thin, webby crust, and have a flat, perming morphology **(Islam *et al*, 2022).** Most of these particles were interstratified with the mica particles, which may indicate a stage of shifting the mica minerals towards the expanded 2:1 minerals group. In general, through the results of the (plate 1A), it can be observed that the presence of montmorillonite and mica minerals simultaneously has more abundance of montmorillonite than mica. Moreover, montmorillonite particles were found individually, and have large size compared to the size of other mineral particles in the sample. Results of scanning in (Figure 1A) also, reveled some particles have well-formed six-sided flakes (Hexagonal shape), with a prominent elongation in one direction. From our point of view these six-sided flake particles are 1:1 type antigenic (well-crystallized) kaolinite minerals, according to the diagnosis given by **(Dixon *et al*, 1977).**



A



B

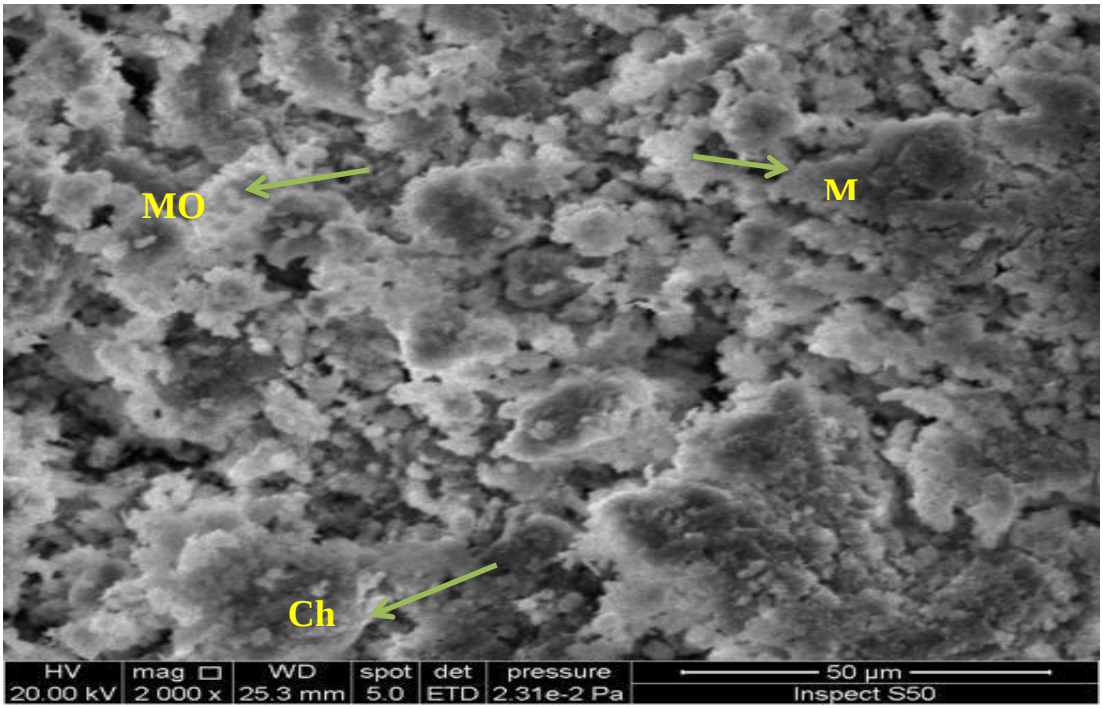
Figure (1): Scanning electron microscopy (SEM) images at magnification (X2000) of Najaf soil at depth of 0-30 cm, irrigated by, A. Dry method, and B. Wet method.
M = Myca, Mo = Montmorillonite, Ch = Chlorite

In (Figure 1B), which represent Al-Najaf$_{(wet)}$ soil, results of scanning showed, almost the same morphological features of clay minerals with some differences, the most important of which is the size of the mineral particles, especially the size of montmorillonite particles, which

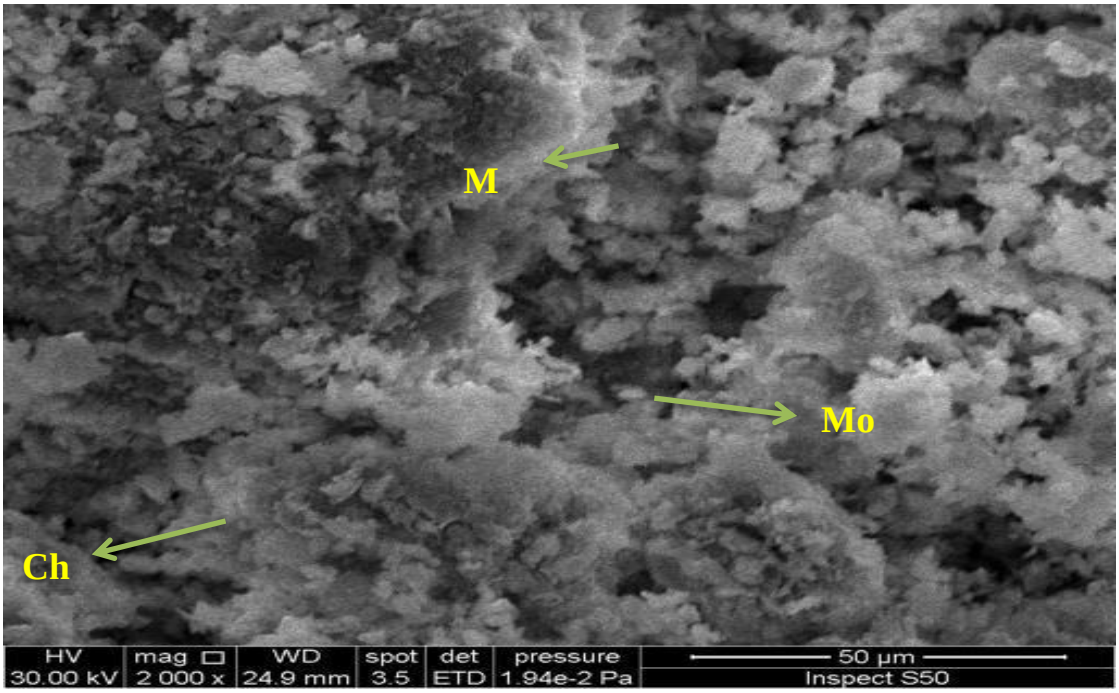
seemed smaller in size than its counterparts in rice soil irrigated by dry method. It appears that the dry irrigation method, in which the soil is exposed to continuous cycles of wet-dry process, which encouraging the precipitation of Mg-hydroxide into Montmorillonite interlayers, which causes an increasing of surface area of mineral particles, and this increase depends on degree of filling for Mg-hydroxide **(Al-Watifi &Abbas Sabr Sarwan, 2012).**

The results of scanning in (Figure 2A, B) for the clay fraction of the Al-Diwaniyah$_{(dry)}$ soil, showed the presence of montmorillonite mineral particles, which appeared as cloudy shaped fluffy mass flaks. (Figure 2 A), and were found in two typs, the first overlapping with mica minerals, or in the type of individual particles secondly. (Figure 2A) also, shows the presence of mica minerals with weathered edges, which appeared in a pale color that distinguishes them from the surface of lath-shaped, rounded mica minerals. Further, the results of (Figure 2A) shows the presence of the chlorite mineral, which has an well-formed imperfect hexagonal shape.

The results of the scanning in (Figure 2B) of the clay sample in Al-Diwaniya$_{(wet)}$ soil, show the predominance of the cloudy-shaped montmorillonite mineral particles with indefinite edges, followed by the predominance of the mica minerals with lath-shaped, and the weathered edges of some of them, as these edges appeared in a pale color that distinguishes them from the surfaces of the mica minerals in the examined sample. In addition the montmorillonite particles in this sample, seemed smaller in size than its counterparts in rice soil irrigated by dry method.



A

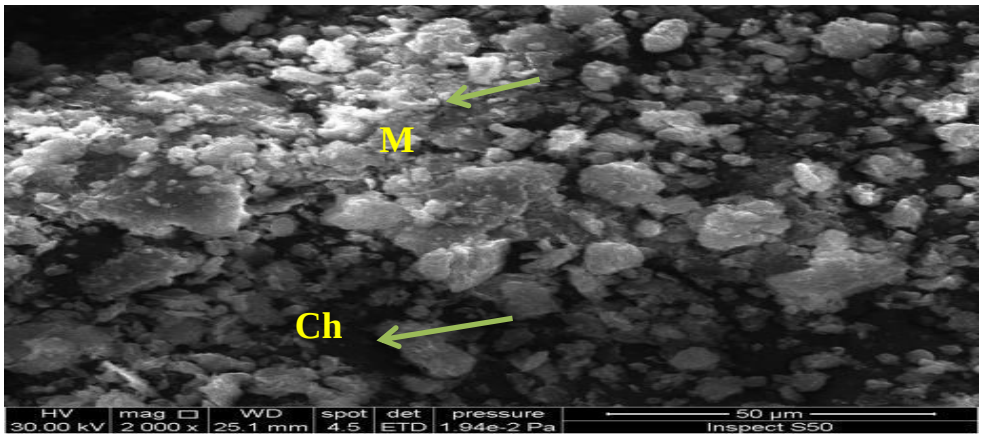


B

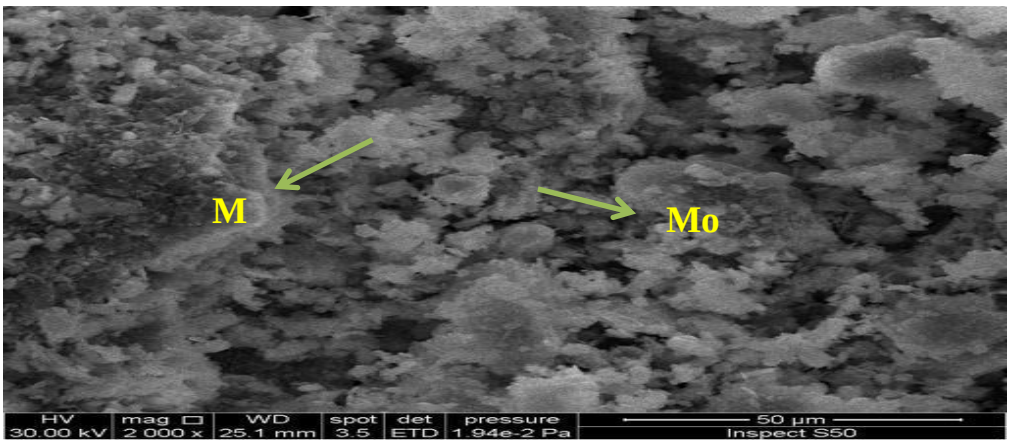
Figure (2): Scanning electron microscopy (SEM) images at magnification (X2000) of Al-Diwaniyah soil at depth of 0-30 cm, irrigated by, A. Dry method, and B. Wet method.
M = Myca , Mo = Montmorillonite , Ch = Chlorite

In order to improve that presumption, three samples of clay were chosen from depth of 0-30 cm, which is the depth as a most affected by the fluctuation of the irrigation patterns used to irrigate the study soils. Also, the selected samples were represented each of the control unexploited Al-Najaf soil, and the second sample was representative of the Al-Najaf$_{(wet)}$ soil. While the third sample, was for Al-Najaf$_{(dry)}$ soil. All selected clay samples were scanned by SEM under one magnification (1990-2000X). The aim of that selection was to study the effect of variation in irrigation patterns on the morphological features of the montmorillonite, because of the deposition of Mg-hydroxide within the interlayer of the mineral. The variation in the size of the montmorillonite particles according to our point of view was adopted as a basis for the occurrence of the deposition process of Mg-hydroxide layer within the interlayers of montmorillonite, based on what was reported by many studies (**Dubbin, 1995., Goldbery *et al,* 2000**). which were conducted in this regard. They showed that because of the deposition of the Mg-hydroxide layer within the interlayers of montmorillonite, the d-spacing of the mineral increases due to the expansion of the particles size. Accordingly, the results of scanning in (Figure 3 A, B, C) showed a clear variation in the size of montmorillonite particles within the soil clay samples that were examined. Where the clay particles of montmorillonite in control soil were recorded the smallest size (Figure 3A), followed by the size of clay particles in Al-Najaf$_{(wet)}$ soil (Figure 3B), while the montmorillonite particles in clay sample of Al-Najaf$_{(dry)}$ (Figure 3C), were exhibited in the largest sizes.These results were consistent with what the current study obtained from the results of the X-ray examination for the clay fraction of the

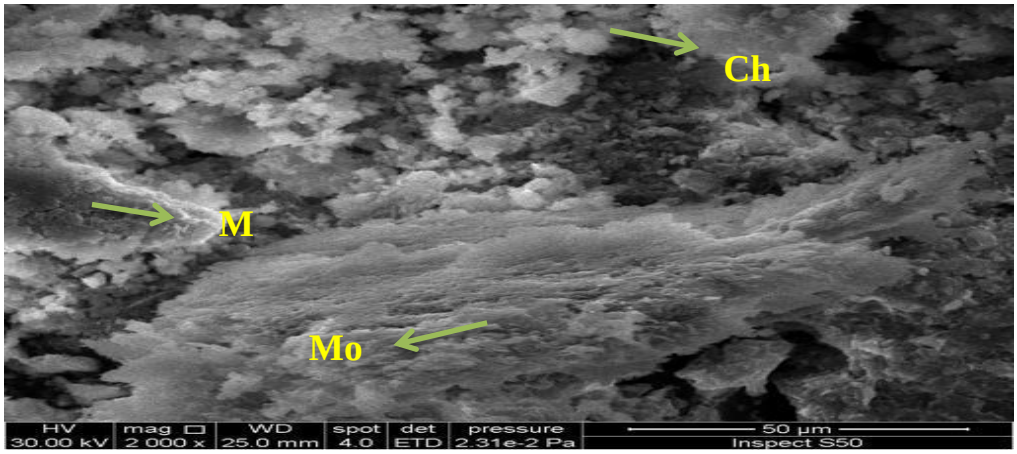
study soils , which showed that the d-spacing of the montmorillonite varied according to the degree of filling of the Mg-hydroxide layer deposited within the interlayers of the montmorillonite, and the highest value of the d-spacing of montmorillonite containing an interlayer of Mg-hydroxide was recorded in Al-Najaf$_{(dry)}$ soil, accompanied by the retention of the montmorillonite diffraction at its d-spacing of 15.05 A^0 in the treatment of k-saturated and heated to the 350 and 550 ^{0}C, which reflects the effect soil subjected to successive of wet-dry cycles, which create suitable conditions for the Mg-hydroxide layer to prispetat within the interlayers of montmorillonite in the soils that are irrigated by the dry.



A



B



C

Figure (3): Scanning electron microscopy (SEM) at magnification (X2000) of Al-Najaf soil, A. control soil, B. irrigated by wet method, C. irrigated by dry method.
M = Myca, Mo = Montmorillonite, Ch = Chlorite

These results were identical to what was consisted with finding of (**Al-Watifi, 2012**) in his study of the phenomenon of chloritization within some of the Iraqi soils.

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AI-Saadi & AL-Hilfy

المجلة العراقية لبحوث السوق
وحماية المستهلك

EFFECT OF SOME PLANT AND ALGAE EXTRACTS ON GROWTH AND BIOLOGICAL YIELD OF SUNFLOWER

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ABSTRACT

A field experiment was conducted at the fields of Dubana Company for Modern Agricultural Equipment in the Yusufiyah area, Al-Rashid district, Baghdad Governorate, located on a line between 44°E longitude and 33°N latitude during the spring season of 2022. The objective was to study the effect of using some plant extracts and seaweed on the growth of three genotypes of sunflower and their reflection on the biological yield. The experiment was conducted using randomized complete block design (RCBD) with split-plot arrangement with three replications, including five bio nutrition treatments (spraying the biological stimulant with seaweed extract BMstart, spraying seaweed extract Alga600, adding bamboo extract Seek, spraying Moringa leaf extract, and a control treatment spraying with distill water only) allocated to the main plots, and three genotypes (Lilo, Ishaqi-2, and Flamy) allocated to the sub plots. The results showed the superiority of the treatment of spraying seaweed extract Alga600 in stem diameter (2.87 cm), plant height (207.50 cm), leaf area (2414.3 cm^2), and biological yield (22.28 tons ha^{-1}), while the treatment of spraying Moringa leaves extract showed superiority in the number of leaves (30.28 leaf $plant^{-1}$) and harvest index (22.49%). As for the genotypes, Ishaqi-2 showed superiority in stem diameter (2.72 cm), leaf area (2325.6 cm^2), biological yield (24.11 tons ha^{-1}), and harvest index (21.92%), while Lilo was superior in number of leaves (29.31 leaf $plant^{-1}$), and Flamy was superior in plant height (201.98 cm).

Keywords: Sunflower, Biological yield, Growth enhancement, Bio stimulants.

تأثير بعض مستخلصات الطحالب والنباتات على النمو والحاصل البيولوجي لزهرة الشمس

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الخلاصة

نفذت تجربة حقلية في احد الحقول التابعة لشركة دبانة للتجهيزات الزراعية الحديثة في منطقة اليوسفية ناحية الرشيد التابعة الى محافظة بغداد والواقعة على خط بين خطي طول 44° شرقا و دائرتي عرض 33° شمالا في خلال الموسم الربيعي لعام 2022، بهدف دراسة تاثير استعمال بعض المستخلصات النباتية والاعشاب البحرية في النمو لثلاثة تراكيب وراثية من زهرة الشمس وانعكاسها على الحاصل البايلوجي، استعمل تصميم القطاعات العشوائية الكاملة RCBD بترتيب الالواح المنشقة Split-plot بثلاثة مكررات، شملت الالواح الرئيسة Main plot على خمسة معاملات تغذية (رش المحفز الحيوي مع مستخلص الاعشاب البحرية BMstart، ورش مستخلص الاعشاب البحرية Alga600 ، واضافة مستخلص قصب الخيزران Seek ورش مستخلص اوراق المورنكا، ومعاملة المقارنة رش بالماء فقط)، وتضمنت الالواح الثانوية ثلاثة تراكيب وراثية (ليلو، واسحاقي2، وفلامي)، اظهرت النتائج تفوق معاملة رش

[*] **The research is taken from a master's thesis by the first researcher.**

مستخلص الاعشاب البحرية Alga600 في قطر الساق(2.87 سم)، وارتفاع النبات (207.50 سم) والمساحة الورقية (2414.3سم^2) والحاصل البايلوجي (22.28 طن هـ $^{-1}$)، بينما تفوقت معاملة رش مستخلص اوراق المورنكا في عدد الاوراق (30.28 ورقة نبات$^{-1}$)، ودليل الحصاد (22.49%).اما بالنسبة للاصناف فقد تفوق التركيب الوراثي اسحاقي2 في قطر الساق (2.72 سم) والمساحة الورقية (2325.6 سم^2) والحاصل البايلوجي (24.11 طن هـ$^{-1}$) ودليل الحصاد،(21.92%) وتفوق التركيب الوراثي ليلو بعدد الاوراق (29.31 ورقة نبات$^{-1}$)، بينما تفوق التركيب الوراثي فلامي في ارتفاع النبات (201.98 سم) .

الكلمات المفتاحية: زهرة الشمس ,الحاصل البايلوجي, النمو، تعزيز النمو، المنشطات الحيوية.

INTRODUCTION

Sunflower is an oilseed crop belonging to the composite family, with seeds containing an oil content of 40-50% **(Nasralla *et al.*, 2014).** rich in unsaturated fatty acids (90%), which contributes to reducing cholesterol levels in the blood and is also rich in protein. It occupies an important place among oil crops due to its short season and ability to adapt to a wide range of environmental conditions (**Bajehb, 2010)**. The low productivity of this crop in Iraq may be due to the non-use of suitable and promising genotypes, as well as poor crop management and the lack of adoption of modern agricultural methods, especially nutrient management, which is one of the most important areas of management for farmers. The intensive use of mineral fertilizers leads to the deterioration of the physical and chemical soil properties, exacerbating pollution problems, in addition to contaminating water and food with the residues of these fertilizers, which have harmful effects on human and animal health **(Al-Hilfy & Al-Temimi, 2017)** . Each crop has potential energy for production, which is rarely accessible in the field due to one or more factors that determine production, some of which can be distinguished and diagnosed in the field and controlled, such as nutrient deficiency and the balance of nutrients that the plant needs for growth and development during its different stages of growth, and the method of fertilization **(Al-Temimi 2021)**. Many studies have indicated that genotypes generally differ from each other due to the dominance of genetic actions that affect growth stages and differences in the physiological performance of the genotypes and their response to growth conditions. The response of genotypes (Ishaqi-1, Ishaqi-2, Ekmars, and Tarsan) to the study conditions varied in growth traits, biological yield, and harvest index, ekmars excelled in plant height (200.03 cm), stem diameter (3.45 cm), leaf area (1.033 m^2), and biological yield (14.917 micrograms ha^{-1}) **(Elawi & Zeboon2020; Hassan, 2019).** Although chemical fertilizers are efficient in increasing production and improving quality, they have harmful effects on human health, in addition to their high economic costs, to reduce the amounts of mineral fertilizers added, complementary organic compounds can be added that are harmless to the environment, improve soil properties, provide plants with nutrients, and increase their tolerance to harsh environmental conditions **(khashan *et al*, 2021).** Researchers focus on using environmentally friendly and safe alternatives that lead to increased production and fertility rates for crops and reduce losses, such as biological stimulants like plant extracts and seaweed that stimulate physiological processes in plants and reduce stress during different growth stages, characterized by ease of application and low cost **(Shukla *et al.*, 2019; Jaafar & Alnaimi, 2022; Al-Omairi & Al-Hilfy, 2021).**

Studies have shown that the use of seaweed extracts, in their various types, as organic and biologically active fertilizers is an important source that is used in many applications on economic crops due to their natural, environmentally friendly, biodegradable and cost-effective properties **(Khan *et al.*, 2009; Saudi, 2017; Al-Hilfy *et al.*, 2018).** Marine algae extracts

contain macro and micro elements, amino acids, and some basic plant growth hormones such as auxins, gibberellins, and cytokinins, which, when used on plants, lead to a significant increase in yield **(Lotze & Hoffman, 2016).** The growth indicators can be improved and increased by spraying seaweed extracts at concentrations of 10% on maize **(Al-Temimi & Al-Hilfy, 2022; Al-Omairi & Al-Hilfy, 2024)**. The number of leaves can be increased to 40.39 leaf Plant^{-1} and stem diameter (22.82 mm) when spraying seaweed extract Alga600 at a concentration of 20% on sunflowers **(Al-Naimi, 2018).**

Studies have shown that using extracts of Moringa leaves as an organic and cheap stimulant is a similar source to the effect of industrial growth regulators, as it contains macro and micronutrients, antioxidants, purine adenine, and zeatin, which work to enhance the properties of a number of antioxidant enzymes and protect cells from the effects of aging caused by various types of reactive oxygen species **(Nita *et al*., 2022).** Spraying a 6% concentration of Moringa leaf extract on maize resulted in an increase in growth and biological yield by 8.42% and 2.55% for two consecutive seasons **(Al-Temimi, 2021).** And wheat spraying a 20% concentration of Moringa leaves extract (Sura **& Al-Hilfy, 2022).** Spraying a 20% concentration of Moringa leaf extract on sunflowers increased the leaf area by 14% and the plant height by 10%, due to the nutrients necessary to meet the plant's needs and activate several enzymes for good growth **(Iqbal, 2014).**

The use of organic material for fertilizing is the foundation that should be established to reduce environmental pollution resulting from the excessive use of chemical fertilizers and to increase the productivity of agricultural lands **(Al-Hilfy, 2014).** Various sources of organic waste, such as animal and plant waste, can be used to improve soil and plant growth **(Ibrahim *et al*., 2008).** However, there are also less well-known and unused materials such as Seek, an organic material derived from bamboo cane waste, rich in vitamins and amino acids that enhance soil water retention and nutrient retention in the root zone, improve fertilizer availability, reduce leaching and fertilizer waste in the soil, and help increase plant nutrient absorption capacity **(Nasralla *et al*.,2014)**.

Based on the aforementioned, the study aims to investigate the effect of the Moringa leaf extract, marine algae extract Alga600, bamboo cane waste Seek, and the bio stimulant B Mstart on some growth traits of three genotypes of sunflowers and their reflection on the biological yield and harvest index.

MATERIALS AND METHODS

To study the effect of using some plant extracts and seaweed on the growth of three genotypes of sunflower and its reflection on the biological yield, a field experiment was conducted during the spring season of 2022 in the Yousufiya area, Al-Rashid district, in the fields of Dubaneh Company for Modern Agricultural Equipment, which is located 29 km from the center of Baghdad province. The experiment was conducted using randomized complete block design (RCBD) with split-plot arrangement with three replications The study included two factors: the main factor included four treatments of bio nutrition spraying in addition to the control treatment (spraying with distill water only) which included spraying with the biological stimulant (BMstart), spraying with marine algae extract (Alga600), adding bamboo waste (Seek), and spraying with Moringa leaves extract as described in Table 1.

Table (1): Components of the nutrition treatments used in the experiment.

Moringa Leaves Extract	Seek	Alga600	BMstart
Macro-elements: (nitrogen, phosphorous, potassium, calcium, magnesium) Micro-elements: (Manganese, Boron, Iron, Zinc, Copper) Enzymatic and non-enzymatic antioxidants: (GA3, IAA, POD, SOD, CAT) Protein	Organic substances derived from bamboo cane debris, such as fulvic acid, amino acids, humic acid, and NPK	Organic material derived from seaweed, natural nitrogen, phosphorus, and potassium, as well as a comprehensive assortment of microelements, humic acids, amino acids, proteins, enzymes, and natural vitamins.	GA142 Seaweed Emulsion, Boron, Magnesium

The sub-plot factor included three genotypes for sunflower crops (Lilo, Ishaqi-2, and Flamy). The field was prepared by plowing, smoothing, and dividing into 45 experimental units of 9 m² (3*3) area, containing four rows with a distance of 75 cm between each row and a distance of 20 cm between plants. The seeds were planted on 21/2/2022 And harvest 4/6/2022, and DAP fertilizer was added at a rate of 220 kg ha^{-1} (48% P_2O_5 and 18-21% N) during planting. The plants were also supplemented with urea (46% N) at a rate of 360 kg ha^{-1} to complete their nitrogen needs **(Al-Rawi, 2001)** with the first dose added at the rosette stage (4-3 true leaves) and the second dose at the beginning of the appearance of floral buds **(Jenkins & Leitch, 1986**). Other managements such as irrigation, and weed control were done as needed.

At flowering, ten plants were randomly selected from the middle two rows of each experimental unit to studying the following characteristics:

1- Plant height (cm): measured using a measuring tape from the soil surface to the base of the disc.
2- Stem diameter (cm): measured from the middle area using a Verniea.
3- Number of leaves: counted from the first green leaf above the soil surface to the last leaf on the plant **(Hunt, 1982).**
4- Leaf area (m²): by using the following equation: The sum of the squares of the maximum width of the sixth Roll x 4.31 **(Hardan & Elsahooki, 2014).**
5- Biological yield (ton ha^{-1}): calculated at harvest by averaging the dry weight of ten plants from each experimental unit. The plants were cut (stems, leaves, heads) from the area of contact with the soil, air-dried, and then the weight was converted to ton after multiplying it by the plant density (66666 plants ha^{-1}).
6- Harvest Index: calculated after harvest using the following equation:
Harvest Index = (seed yield / biological yield) x 100

The data were statistically analyzed using the Genstat v.7 according to the randomized complete block design (RCBD) with split plot arrangement. The mean comparisons were performed using the least significant difference (LSD) test at a significance level of 5% **(Steel & Torrie, 1980).**

RESULTS AND DISCUSSION

RESULTS

Number of leaves

The results showed significant differences between the different bio nutrition treatments in the number of leaves (Table 2). The treatment of spraying the Moringa leaves extract gave the highest number of leaf at 30.28, leaf plant^{-1}, followed by the treatment of spraying the seaweed extract Alga600 at 29.32 leaf plant^{-1}, and there was no significant difference between the treatment of adding bamboo waste (Seek) and the control treatment with 28.54 and 28.52 leaf plant^{-1}, respectively. While the treatment of the biological stimulant BMstart gave the lowest number of leaves at 27.60 leaf per plant^{-1}.

The genotypes also differed significant in this trait, Lilo hybridgave the highest number of leaves at 29.31 leaf plant^{-1} with no significantly different from cultivar Ishaqi-2 with 28.78 leaf plant^{-1}, while the Flamy genotype recorded the lowest number of leaves (28.47 leaf plant^{-1}).

The results indicate a significant interaction between the two factors, the combination of the Ishaqi-2 cultivar with the Moringa leaves extract gave the highest number of leaves, at 31.13 leaf plant^{-1}, It did not differ significantly from Lilo hybrid with the Moringa leaves extract, which reached 30.63 leaf plant^{-1}. The lowest mean was 26.87 leaf plant^{-1} for the combination of the Lilo hybridwith the BMstart biological stimulant.

Table (2): The number of sunflower leaves as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	26.87	28.60	27.33	27.60
Alga600 seaweed extract	30.47	29.00	28.50	29.32
Seek (bamboo plant residue)	29.43	27.27	28.93	28.54
Moringa leaves extract	30.63	29.07	31.13	30.28
Distill water (control)	29.13	28.43	28.00	28.52
L.S.D. 5%	1.27			0.93
Mean	29.31	28.47	28.78	
L.S.D. 5%	0.58			

Stem diameter (cm)

The results indicate significant differences between the different bio nutrition treatments, genotypes, as well as their interaction, in the stem diameter (Table 3). The bio nutrition treatments led to an increase in stem diameter compared to the control treatment, and the treatment of spraying the seaweed extract Alga600 had the highest stem diameter, reaching 2.74 cm, which did differ significantly from the treatment of Moringa leaves extract (2.67 cm). while the control treatment recorded the lowest stem diameter of 2.53 cm.

As for the genotypes, the Ishaqi-2 cultivar gave the highest mean for stem diameter, which reached 2.72 cm, and did differ significantly from the Flamy cultivar (2.68 cm), while the Lilo genotypes recorded the lowest mean of 2.67 cm.

The results indicate a significant interaction between the two factors , the response of the genotypes to the bio nutrition treatments differed, and the highest value was for the combination of the Ishaqi-2 cultivar and the seaweed extract Alga600, reaching 2.93 cm, which

did not differ significantly from Lilo hybrid and the seaweed extract Alga600, which reached 2.89 cm, and the combination of the Ishaqi-2 cultivar and Moringa leaves extract (2.89 cm). The lowest value was 2.45 cm, which was for the combination of the Lilo hybrid and the control treatment.

Table (3): Stem diameter of sunflower as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	2.65	2.60	2.65	2.63
Alga600 seaweed extract	2.89	2.79	2.93	2.87
Seek (bamboo plant residue)	2.66	2.70	2.64	2.67
Moringa leaf extract	2.67	2.66	2.89	2.74
Distill water (control)	2.45	2.65	2.49	2.53
L.S.D. 5%	0.10			0.08
Mean	2.67	2.68	2.72	
L.S.D. 5%	0.04			

Plant height (cm)

The results indicate a significant effect of the different nutrition treatments, genotypes, and their interaction on plant height (Table 4). All bio nutrition treatments led to a significant increase in plant height compared to the control treatment, with the treatment of spraying the seaweed extract Alga600 showing the highest increase percentage of 20.76%. It did not differ significantly from the treatment of adding bamboo waste (Seek), which reached 201.30 cm, and the treatment of spraying Moringa leaves extract, which showed an increase percentage of 12.57%. while the control treatment recorded the lowest plant height of 171.97 cm.

As for the genotypes, the Flamy genotypes outperformed and recorded the highest plant height, which reached 201.98 cm, followed by the Ishaqi 2 cultivar (194.60 cm). The Lilo variety had the lowest plant height of 178.04 cm.

Regarding the interaction between genotypes and bio nutrition treatments, the response of the genotypes to the treatments differed, with the highest plant height of 221.00 cm being recorded for the combination of the cultivar Flamy and the seaweed extract Alga600. It did not differ significantly from the combination of the cultivar Flamy and bamboo waste (Seek), which reached 214.30 cm, and the combination of the cultivar Flamy and the control treatment. The lowest value was recorded for the combination of the hybrid Lilo and the control treatment, which reached 128.50 cm.

Table (4): Plant height (cm) of sunflower as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	187.30	172.50	190.20	183.33
Alga600 seaweed extract	195.20	221.00	206.30	207.50
Seek (bamboo plant residue)	185.60	214.30	204.00	201.30
Moringa leaf extract	193.60	191.40	195.80	193.60
Distill water (control)	128.50	210.70	176.70	171.97
L.S.D. 5%	14.01			11.02
Mean	178.04	201.98	194.60	
L.S.D. 5%	5.48			

Leaf area (cm^2)

The bio nutrition treatments led to an increase in leaf area by a percentage of 32.09%, 31.71%, 27.55%, and 13.84% for the treatment of spraying the seaweed extract Alga600, spraying Moringa leaves extract, spraying the biological stimulant BM Start, and adding bamboo waste (Seek), respectively, compared to the control treatment, which gave the lowest leaf area of 1827.67 cm^2 (Table 5).

The data also indicate significant differences between genotypes, with the Ishaqi 2 cultivar outperforming and recording the highest value of 2325.6 cm^2, while the Flamy cultivar recorded the lowest value (2116.6 cm^2) and did not differ significantly from the Lilo hybrid (2194.6 cm^2).

Similarly, the interaction between the two factors was significant, with the highest leaf area recorded for the combination of the Ishaqi 2 cultivar and Moringa leaves extract, which reached 2767.0 cm^2. The combination of the Ishaqi 2 cultivar and the control treatment recorded the lowest value of 1662.0 cm^2.

Table (5): Leaf area (cm^2) of sunflower as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	2644.0	2161.0	2189.0	2331.3
Alga600 seaweed extract	2222.0	2398.0	2623.0	2414.3
Seek (bamboo plant residue)	1856.0	1999.0	2387.0	2080.7
Moringa leaf extract	2313.0	2142.0	2767.0	2407.3
Distill water (control)	1938.0	1883.0	1662.0	1827.7
L.S.D. 5%	257.50			173.80
Mean	2194.6	2116.6	2325.6	
L.S.D. 5%	114.70			

Biological yield (ton ha^{-1})

The results indicate significant differences between the bio nutrition treatments, genotypes, and their interaction on the biological yield (Table, 6). The seaweed extract treatment Alga600 had the highest biological yield, reaching 22.28-ton ha^{-1}, with no significant difference with the from spraying the Moringa leaves extract, which recorded 22.27-ton ha^{-1}. While the control treatment recorded the lowest value of 19.86-ton ha^{-1}.

The cultivar Ishaqi 2 had the highest biological yield value at 24.11-ton ha^{-1}, while the Lilo hybrid recorded the lowest value of 18.19-ton ha^{-1}.

As for the interaction, the interaction between the Ishaqi2 cultivar and the Moringa leaves extract spray had the highest value for the biological yield, reaching 26.97-ton ha^{-1}, with no significant difference from the Ishaki2 cultivar and the Alga600 seaweed extract spray treatment, which reached 26.08-ton ha^{-1}, while the lowest value was for the interaction between the Lilo hybrid and the control treatment was 16.41 ton ha^{-1}.

Table (6): Biological yield (ton ha^{-1}) of sunflower as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	18.10	20.06	22.36	20.17
Alga600 seaweed extract	19.05	21.71	26.08	22.28
Seek (bamboo plant residue)	18.72	21.75	23.16	21.21
Moringa leaf extract	18.71	21.14	26.97	22.27
Distill water (control)	16.41	21.15	22.02	19.86
L.S.D. 5%	1.44			0.969
Mean	18.19	21.16	24.11	
L.S.D. 5%	0.64			

Harvest index (%)

The results indicate significant differences between the bio nutrition treatments, genotypes and their interactions on the harvest index (Table 7). Spraying Moringa leaves extract had the highest harvest index reach at 22.49%, while adding bamboo extract waste (Seek) had the lowest value of 19.63%.

Regarding the genotypes, the Ishaqi2 cultivar had the highest harvest index value of 21.92%, with no significant difference from Flamy cultivar of 21.82%. While the Lilo hybrid recorded the lowest mean (19.58%).

As for the interaction, the combination of the Flamy cultivar and the Moringa leaves extract spray had the highest value (24.34%), with no significant difference the combination of the Flamy cultivar and the control treatment (23.54%) As well as the interactions of cultivar Ishaqi 2 with each of seaweed extract Alga600 and bamboo extract waste Seek (23.73 and 23.54%, respectively)., respectively. However, the combination of the Lilo hybrid and the bamboo extract waste (Seek) had the lowest value of 15.38%.

Table (7): Harvest index (%) of sunflower as affected by some bio nutrition treatments and genotypes and their interaction for the spring season 2022.

Treatment of Bio Nutrition	Cultivar			Mean
	Lilo	Flamy	Ishaqi-2	
BMstart bio stimulants	21.44	21.54	20.21	21.06
Alga600 seaweed extract	19.37	20.20	23.73	21.10
Seek (bamboo plant residue)	15.38	19.96	23.54	19.63
Moringa leaf extract	22.13	24.34	20.99	22.49
Distill water (control)	19.59	23.04	21.15	21.26
L.S.D. 5%	1.59			1.06
Mean	19.58	21.82	21.92	
L.S.D. 5%	0.71			

DISCUSSION

Spraying the Moringa leaves extract resulted in an increase in the number of leaves for the sunflower (Table, 2), which may be attributed to containing antioxidants and hormones that protect the cell from the damage of free radicals during the plant's metabolism, such as carbon assimilation and respiration. This led to improve the cell's condition to carry out these processes, and therefore, reflecting on plant growth and an increase in the number of leaves, as shown in its components in (Table, 1). In addition, the extracts contain major nutrients, including nitrogen, which plays an important role in increasing cell division, elongation, and resulting in an increase in plant vegetative growth and height (Table, 4). This is reflected in an increase in the number of leaves in Table 2 **(Hassanin, 2020).** This is consistent with **Hamza (2003),** who indicated differences in genotypes in the number of leaves when using foliar nutrition.

The increase in stem diameter (Table, 3) when spraying the Alga600 seaweed extract may be attributed to its role in increasing vascular bundles, which reflects on the stem diameter. In addition, its role in preserving the largest number of leaves and increasing the chlorophyll content, which reflected in the improvement of the efficiency of carbon assimilation and an increase into its products, especially carbohydrates, and this is reflected in the increase in stem diameter **(Al-Naimi, 2018).**

The reason for the superiority of the Alga600 bio stimulant in plant height (Table, 4) is due to the role of seaweed extract that contains a high percentage of nitrogen (Table, 1), which has a major role in increasing plant growth as it is involved in building chlorophyll., which increases the efficiency of photosynthesis by increasing the leaf area (Table, 5) and producing a high percentage of carbohydrate and protein products, thereby increasing vegetative growth. Additionally, the role of plant growth regulators present in the extract affects cell growth and division, leading to an increase in stem elongation, which is reflected in an increase in plant height **(Hassanin, 2020).** The role of calcium, which plays an important role in the division of plant cells and the growth of plant tissue, also contributes to increasing the elongation of the growing tip of sunflower plants. This is consistent with **Al-Naimi (2018),** who indicated an increase in leaf area when using the Alga600 seaweed extract. The extract also contains amino acids and major nutrients such as phosphorus, nitrogen, and potassium (Table, 1), all of which contribute to increased vegetative growth by expanding cells and stimulating their division, which is reflected in an increase in leaf area. This result is consistent with **Abdul-jabar *et al.***

(2012); **Saudi (2017),** who found in their study that spraying seaweed extracts on plants led to significant differences in leaf area. In addition, the seaweed extract contains auxins and cytokinins, which stimulate physiological activities and increases the content of chlorophyll in the leaves, which positively affects the effectiveness of photosynthesis and manufactured materials, and then reflects positively on the vegetative growth characteristics of the plant **(Zodape, 2001; Al-Jubouri, 2017).** Based on the above mentioned, the biological yield increased due to the increase in its components represented in the plant height (Table, 4) and leaf area (Table, 5). These results are consistent with (Al-**Hilfy & Al-Omairi, 2023)** who reported an increase in biological yield when using growth-promoting substances.

Regarding the harvest index (Table, 7), which indicates the efficiency of converting the products of photosynthesis into economic and biological yield, it increased when spraying with Moringa leaves extract due to the increase in both economic and biological yield. The reason for the difference in growth and yield among cultivar is attributed to their different responses to growth conditions and the genetic makeup that controls the studied traits. This is consistent with the findings of **Nasralla *et al*. (2014)**; **Elawi & Zeboon (2020),** who noted the variation in genotypes and their responses to management.

Also, the difference in genotypes is due to the nature of their genetic material and their response to growth factors. This result is consistent with **Hassan (2016); Al-Nuaimi (2018),** who found significant differences between genotypes in the number of leaves.

Similarly, the difference in stem diameter between genotypes of sunflower may be due to differences in the physiological performance of the gene controlling growth. This result is consistent with **Khan *et al*. (2015)**, who found significant differences in stem diameter between genotypes. This may be due to the different genetic material of the varieties and their response to growth conditions (Table, 4). These results are consistent with **Abed & Zeboon (2020)**, who reported differences between genotypes.

CONCLUSION

In conclusion, the results of the study showed the superiority of spraying seaweed extract treatment in leaf area and yield of biology, while the treatment of spraying Moringa leaves extract was superior in number of leaves and harvest index. As for the genotypes, Ishaqi 2 gave the highest leaf area, biological yield and harvest index, while the Lilo recorded the highest number of leaves and the genotypes Flami genotypes the highest height of the sunflower plant.

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وحماية المستهلك

PROBLEMS FACING AGRICULTURAL EXTENSION SERVICE PROVIDERS TO FACE THE EFFECTS OF CLIMATE CHANGE IN BAGHDAD GOVERNORATE

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ABSTRACT

The research aimed to determine the problems facing agricultural extension service providers to face the effects of climate change, the research community included all workers in the provision of agricultural extension service in the province of Baghdad and distributed to the Department of Agriculture of Baghdad Karkh and agricultural divisions affiliated to it and the Department of Agricultural Extension and Training in the province of Baghdad, the number of (110) individuals. All members of the community were taken as a sample for research due to their small number. To achieve the objectives of the research, a questionnaire was prepared to collect data from the respondents, consisting of (28) items distributed in three areas (problems concerned to farmers, problems concerned to agricultural extension service providers themselves, problems concerned to coordination and government support). The results of the research showed that there are several problems that hinder the workflow of service providers' agricultural extension services in providing their extension services to farmers in the field of facing the effects of climate changes in varying degrees, as the problem of the lack of specialists in agricultural extension in the field of climate changes came in the first place in terms of importance, which got a weighted mean of (3.68) degrees and a weight percentile of (92%), While the problem of extension service providers not having the skills and knowledge in determining the best agricultural practices to face climate change came in the last place in terms of importance, which got a weighted mean of (3.13) degrees with a weight percentile of (78.25%). The researcher recommended the necessity of adopting the research findings regarding the problems faced by agricultural counseling service providers and working on resolving them through the development of a training and educational plan to enhance their skills and capacities in dealing with agricultural challenges associated with climate change. Additionally, it emphasized the importance of governments and financial institutions providing financial and moral support to enhance their ability to deliver effective agricultural counseling services and assist them in tackling the challenges and problems they encounter.

Keywords: Agricultural extension service, Climate changes, Agricultural extension.

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المشكلات التي تواجه مقدمي الخدمة الارشادية الزراعية لمواجهة اثار التغيرات المناخية في محافظة بغداد

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الخلاصة

هدف البحث الى التعرف على المشكلات التي تواجه مقدمي الخدمة الارشادية الزراعية لمواجهة اثار التغيرات المناخية , شمل مجتمع البحث جميع العاملين في تقديم الخدمة الارشادية الزراعية في محافظة بغداد والموزعين على قسم زراعة بغداد الكرخ والشعب الزراعية التابعة لها ودائرة الارشاد والتدريب الزراعي في محافظة بغداد والبالغ عددهم (110) فرداً ، اخذت جميع افراد المجتمع كعينة للبحث لقلة عددهم . لتحقيق اهداف البحث اعدت استبانة لجمع البيانات من المبحوثين متكونة من (28) فقرة موزعة على ثلاث مجالات (مشكلات متعلقة بالمزارعين ، مشكلات متعلقة بمقدمي الخدمة الارشادية الزراعية انفسهم ، مشكلات متعلقة بالتنسيق والدعم الحكومي). اظهرت نتائج البحث وجود عدة مشكلات تعرقل سير عمل مقدمي الخدمة الارشادية الزراعية في تقديم خدماتهم الارشادية الى المزارعين في مجال مواجهة اثار التغيرات المناخية وبدرجات متفاوتة ، اذ جاءت مشكلة (عدم وجود مختصين بالإرشاد الزراعي في مجال التغيرات المناخية) في المرتبة الاولى من حيث الاهمية والتي حصلت على وسط مرجح قدره (3.68) درجة ووزن مئوي قدره (92%) ، بينما جاءت مشكلة (عدم امتلاك مقدمي الخدمة الارشادية للمهارات والمعارف في تحديد افضل الممارسات الزراعية لمواجهة التغيرات المناخية) في المرتبة الاخيرة من حيث الاهمية والتي حصلت على وسط مرجح مقداره (3.13) درجة بوزن مئوي قدره (78.25) واوصت الباحثة بضرورة اعتماد ما توصل اليه البحث من نتائج تخص المشكلات التي يعاني منها مقدمي الخدمة الارشادية الزراعية والعمل على حلها من خلال وضع خطة تطويرية لتدريب وتعليم المقدمين على هذه المهارات وتعزيز قدراتهم للتعامل مع التحديات الزراعية المرتبطة بالتغيرات المناخية فضلاً عن ضرورة تقديم الحكومات والمؤسسات المالية دعمًا ماليًا ومعنوياً لتعزيز قدرتهم على تقديم خدمات إرشادية زراعية فعالة ومساعدتهم على التعامل مع التحديات والمشكلات التي يواجهونها.

الكلمات المفتاحية: الخدمة الارشادية الزراعية، التغيرات المناخية ، الارشاد الزراعي.

INTRODUCTION

The agricultural sector is one of the most important economic sectors in most countries of the world, as it contributes to providing food needs as well as commodities and raw materials as inputs to a number of manufacturing industries and thus contributes to achieving the gross domestic product of any country **(Arab Monetary Fund, 2019)**. In general, the agricultural food sector provides available food energy to consumers, accounting for approximately 30% or more of the total **(Al-Saffar, 2017)** ,Climate change is one of the most important and dangerous environmental issues facing the planet today. Its effects are being felt globally, but the impact varies in different regions due to the diverse nature of the environment **(Salih, 2009)**. In Iraq, the agricultural sector plays a significant role in providing local income and securing the food needs of the population. However, the rapid increase in human activities without considering their environmental impact has led to major shifts in the Earth's climate, resulting in climate change **(Abdulkareem *et al*.,2011)** Climate change poses significant challenges to the agricultural sector, affecting the growing seasons of crops and ultimately impacting agricultural food production. Rising temperatures and changing rainfall patterns have led to a sharp decrease in the level of drinking and irrigation water, further exacerbating the situation **(Saqar, 2014)**. Moreover, the severity of climatic changes, such as desertification, dust storms, and soil salinity, has added additional pressures on farmers and their economic conditions **(Towij, 2021; Mohammed,2016).** The consequences of climate change and its potential future impact are alarming. Without effective strategies to mitigate these changes, it is

projected that the air temperature will continue to rise, further threatening agriculture and global food security **(Al-Taye *et al.*, 2021)**. Recognizing the urgency of the situation, Iraq, along with other nations worldwide, is actively seeking appropriate agricultural methods and practices to address the effects of climate change. The focus is on developing sustainable solutions while preparing and training workers in institutions responsible for the agricultural sector's development **(Al-Taye *et al.*, 2020) .**In this context, the role of the Agricultural Extension Agency is crucial. As an essential element in achieving sustainable agricultural development and food security, the agency is responsible for addressing the various effects of climate change on agriculture **(Al-Taye *et al.*, 2020)**. It has transformed into an institutional network that supports knowledge and provides vital support beyond the conventional definition of the public sector **(Al-Saaedy &Al-Badri, 2022)**. It seeks to advance the agricultural sector and develop the skills and knowledge of farmers **(Naji &Hamza, 2019).** Through education and training, the agency plays a pivotal role in empowering rural communities to make optimal use of natural resources and adopt modern agricultural techniques **(Moawad &Amer, 2007; Abdulrazzaq &Salman, 2018)** To ensure the success of the extension institution and its ability to overcome the challenges posed by climate change, it is essential to invest in the capabilities, skills, and knowledge of its employees **(Habib &Abdulmaseeh, 1989)**. Agricultural extension workers, being the backbone of the institution, are responsible for effectively delivering solutions and practices to farmers, persuading them to adopt these approaches **(Durra 2003; Salman & Karim, 2016).** The performance of agricultural extension work is influenced by various external and internal factors, underscoring the need for specialized expertise and a comprehensive understanding of the field **(Taha &Naji, 2020).**

Recognizing the problems and difficulties faced by extension service providers is crucial for the institution's officials to overcome these obstacles and improve performance **(Nofan &Youssef,2021).** By addressing these challenges and focusing on the continuous improvement of services, the extension institution can fulfill its role effectively, support farmers in adapting to climate change, and contribute to sustainable agricultural development in Iraq **(Al-Hafiz &Al-Taye, 2022),** the current research came to answer the following questions: What are the problems facing the agricultural extension service providers in the field of facing climate change? What is the degree of importance of these problems from their point of view?

RESEARCH AIMS:

The aim of the research is to determine the problems facing the agricultural extension service providers to face the effects of climate change according to its importance, which includes the following sub- objectives:

1.1- Determine the problems concerned to farmers.

1.2- Determine the problems concerned to agricultural extension service providers.

1.3- Determine problems concerned to government coordination and support.

METERIALS AND METHODS OF THE RESEARCH:

Research community and sample: The province of Baghdad was chosen to conduct the current research, due to the presence of large numbers of employees who provide agricultural extension services. The research community included all workers in providing the agricultural extension service in Baghdad Governorate and distributed to the Baghdad Karkh Agriculture

Department and its affiliated agricultural divisions and the Agricultural Extension and Training Department in Baghdad Governorate, whose number is (110) individuals for the year 2022.

Research tool: A questionnaire was prepared to collect data by examining scientific sources, studies and research concerned to the subject, and consulting specialists in the subject from researchers and university professors. Accordingly, (28) problems were identified, divided into three areas: the problem of farmers with (10) problems, the problem of extension service providers with (12) problems, and the problem of government support with (6) problems.

Research scale : To measure these problems, a five-level scale consisting of five levels was developed according to importance, which are (a very large problem, a large problem, a medium problem, a small problem, and no problem) and weights were given to it (0,1,2,3,4) respectively, and to determine which of the problems were more important to the extension service providers, they were arranged in descending order of importance and depending on the weighted mean for each problem.

Validity and reliability procedures: The initial questionnaire was presented to 10 experts specialized in the current research topic to assess face validity and content validity. Validity refers to the extent to which a measurement tool is suitable for measuring the intended characteristic or phenomenon **(Atiya, 2009)**. The experts' feedback was sought regarding the research areas and dimensions, as well as the scientific accuracy of the statements, and their suggestions for modification, deletion, or addition, to reach the optimal formulation before presenting it to the respondents. To achieve this, a three-point scale was used, consisting of the expressions "Agree," "Agree with modification," and "Disagree," with corresponding weights o (3,2,1), respectively. A cutoff threshold of 75% was set for the areas and statements included in the questionnaire. Agreement among reviewers at a rate of 75% or higher indicates the tool's validity **(Drouza, 2005)**, and necessary adjustments were made accordingly.
Based on that, a preliminary pre-test was conducted on a sample of 18 respondents from the Directorate of Agriculture in Baghdad/Al-Rusafa (outside the research population) in November to assess the reliability of the scales included in the questionnaire. To measure reliability statistically, the data from the preliminary test were analyzed using the Cronbach's alpha equation. The test results showed high reliability coefficients, with a total reliability coefficient of 0.93 for the Development of Agricultural Extension Service Providers' Capabilities questionnaire. Thus, the tool is considered suitable for field application.

Data collection: Data was collected from the (110) respondents during the period from 5/12/2022 to 20/1/2023.

Data analysis: After collecting the data, it was coded and tabulated, then those data were analyzed using statistical (and manual analysis and the SPSS program), and statistical methods (weighted mean and percentile weight) were used to analyze that data.

RESULTS AND DISCUSSION

Based on the answers of respondents who are agricultural extension service providers concerned to the problems they face in the field of coping with the effects of climate change, (28) problems have been identified distributed in three fields: problems concerned to farmers, problems concerned to agricultural extension service providers, and problems of coordination and government support). The results of the research showed that the weighted mean of the problem fields according to their importance for the respondents ranged from (3.33 - 3.55) degrees and percentage weights ranged from (83.4 88.91%) with a total weighted mean rate of (3.46) degrees and a weight percentile of (86.66%), which is much higher than the mean score the theoretical mean of the scale of (2) degrees, which indicates the presence of various and many problems that extension service providers suffer from in the field of confronting climate changes that prevent them from performing their extension work in the correct scientific manner, and this gives an indication that the studied problems are really realistic problems and of great importance, which requires Standing on them and addressing them by the competent authorities concerned to these problems, as shown in Tables (1):

Table (1): Arrangement of problem fields according to the weighted mean of their importance according to the answers of agricultural extension service providers.

Seq.	Problems Fields	Weighted mean of the axis	Weight percentile of the axis	Order
1	Problems concerned farmers	3.50	87.5	2
2	Problems concerned to agricultural extension service provider	3.33	83.25	3
3	Government coordination and support problems	3.55	88.75	1
Average of the total weighted mean		**3.46**	**86.5**	

It is clear from Table (1) that most of the respondents emphasized that the problems experienced by the agricultural extension service providers are many and varied, some of which are concerned to farmers and others concerned to the extension service providers themselves as well as problems concerned to government coordination and support. The results also showed that the problems concerned to coordination and government support came in the first place in terms of the degree of importance for the respondents, as it got a weighted mean of (3.55) degrees and a percentage weight of (88.91%) the reason for this may be due to the importance of government support and its role in providing the necessary supplies to confront climate changes, which help agricultural extension service providers to perform their tasks successfully.

While the problems concerned to the extension service providers themselves came in the last rank in terms of the degree of importance for the extension service providers, as it got a weighted mean of (3.33) degree and weight percentage of (83.4%), The reason for this may be that agricultural counseling service providers face significant pressures in their daily work, such as visiting a large number of farmers, dealing with various agricultural problems, and coping with climate change and environmental risks. The workload pressure and field challenges may affect the counselors' well-being and their ability to provide quality services.

1. Problems Concerned Farmers:

The problems concerned to farmers that face the agricultural extension service providers in the field of coping with climate change, which are (10) problems, got weighted mean according to the degree of their importance to the respondents. It ranged from (3.32 - 3.65) degrees and percentage weights ranged from (83-91.25%), with an average of the value of (3.50) degrees with a weight percentage of (87.67%), which is higher than the theoretical mean of (2) degrees. Accordingly, all problems concerned to farmers impede the work of agricultural extension service providers in the field of confronting the effects of climate changes, despite the slight discrepancy between their weighted means. As shown in the table (2):

Table (2): Arrangement of problems concerned to farmers according to the weighted mean of their importance according to the answers of agricultural extension service providers.

Seq.	Problems Fields	Weighted mean of the axis	Weight percentile of the axis	Order
5	The poor financial and material capabilities of some farmers reduces their ability to apply some agricultural practices to reduce the effects of climate change.	3.65	91.2	1
4	Poor farmers' management of the irrigation process to reduce water scarcity.	3.58	89.5	2
6	Lack of farmers' review of extension units to benefit from available information about climate change.	3.57	89.2	3
3	Farmers' reluctance to participate in training and educational courses concerned to climate change.	3.54	88.5	4.5
8	Farmers' adherence to wrong and traditional agricultural practices and their unwillingness to apply modern practices to limit the effects of climate change.	3.54	88.5	4.5
10	Lack of use of the agricultural cycle system to improve systems farm by farmers.	3.51	87.7	6
9	Lack of use of organic fertilizers to improve soil fertility and moisture retention	3.50	87.5	7
2	Lack of awareness of farmers of the dangers and damages of climate change on their crops	3.44	86	8
1	Lack of knowledge of farmers about the effects of climate change on their daily agricultural practices	3.42	85.5	9
7	Lack of sources of information related to the phenomenon of climate change	3.32	83	10
Average of the total weighted mean		**3.50**	**87.5**	

It is clear from table (2) that the problem (weak financial and material abilities of some farmers reduces their ability to apply some agricultural practices to limit the effects of climate change) came in the first place in terms of the degree of importance for the respondents, as it obtained a weighted mean of (3.65) degrees. With a percentage weight of (91.25%). The reason for this may be due to the fact that the financial and material abilities, if they exist among farmers, means increasing their ability to purchase and provide some agricultural technologies and practices that contribute to limiting the effects of climate changes when applied, and thus the respondents realize the importance of the availability of financial and material abilities among farmers. to help them perform their work successfully. While the problem (lack of

sources of obtaining information related to the phenomenon of climate change) came last in terms of the degree of importance for the respondents, as it obtained a weighted mean of (3.32) degrees and a percentage weight of (83%). The reason for this may be due Agricultural counseling service providers may believe that information related to climate change is widely available and easily accessible from various sources, such as the Internet or agricultural counseling agencies. Alternatively, they may have the belief that there is already a sufficient amount of information readily available, and therefore, this problem does not pose a significant issue for them.

2. Problems concerned to Agricultural Extension Service Providers:

The (12) problems concerned to the extension service providers themselves, which they face in the field of confronting climate change, got weighted mean according to the degree of their importance to the respondents, as they ranged from (3.13-3.68) degrees and percentage weights ranged from (78.25-92%) with a median rate of (3.33) degrees, with a percentage weight of (83.4%), which is higher than the theoretical mean of (2) degrees. Accordingly, all problems concerned to agricultural extension service providers impede their work in the field of confronting the effects of climate changes despite the discrepancy between their weighted means, as shown in Table (3):

Table (3): Arranging the problems concerned to the agricultural extension service providers themselves according to the weighted mean of their importance according to their answers.

Seq.	Problems Fields	Weighted mean of the axis	Weight percentile of the axis	Order
8	The lack of specialists in extension in the field of climate change	3.68	92	1
7	Relying on traditional methods and techniques in communicating agricultural information and practices related to the issue of climate change.	3.41	85.2	2.5
9	Some technical recommendations related to reduction the effects of climate change are characterized by the difficulty of implementation by extension service providers.	3.41	85.2	2.5
3	Lack of radio and television programs on the subject of climate change.	3.37	84.2	4
4	The lack of extension apparatus and necessary supplies and tools to deal with climate change.	3.35	83.7	5
6	There is a weakness at the level of field agricultural extension units in real guidance in the field of reducing the phenomenon of climate change.	3.34	83.5	6.5
10	Weakness of the extension service provider in preparing and planning programs and activities related to reducing the effects of providing climate change services.	3.34	83.5	6.5
11	The technical recommendations provided by extension service providers on the	3.31	82.7	8

	issue of climate change are not suitable for application to farmers.			
12	Lack of publications and pamphlets related to climate change.	3.26	81.5	9
5	Lack of sources of information related to climate change in the extension service providers.	3.24	81	10
1	The extension service providers lack methods of persuasion and effective communication with farmers.	3.2	80	11
2	The extension service providers do not have the skills and knowledge in determining the best agricultural practices to confront climate change.	3.13	78.2	12
Average of the total weighted mean		**3.33**	**83.2**	

It is clear from Table (3) that the problem (lack of specialists in extension in the field of climate change) came in the first place, as it obtained a weighted mean of (3.68) degrees and a weight percentage of (92%). The reason for this may be due to not taking the issue of climate change Considered by the Extension Institution, through the employment of a number of specialists in the field of climate change to benefit from their services in the implementation of agricultural Extension activities related to the subject, while the problem (the Extension service providers not having the skills and knowledge in determining the best agricultural practices to face climate changes) came in The last rank in terms of the level of importance, as it got a weighted mean of (3.13) degrees and a weight percentage of (78.25%). The reason for this may be due to respondents' belief that information related to the phenomenon of climate change has the potential to Determine appropriate agricultural best practices to reduce the effects of climate change, so they were the least interested in their belief.

3. Problems of Government Coordination and Support:

There were (6) problems concerned to the field of Coordination and Government Support facing agricultural extension service providers in the field of coping with climate change got weighted mean according to their importance to the respondents, as they ranged from (3.53-3.6) degrees and percentage weights ranged from (88.3-90%). With a median rate of (3.55) degrees, with a weight percentile of (88.87%), which is higher than the theoretical mean of (2) degrees. Therefore, all problems concerned to the field of coordination and government support impede the work of agricultural extension service providers in the field of confronting the effects of climate change, despite the slight discrepancy between Their weighted mean, as shown in Table (4):

Table (4): Arrangement of problems concerned to government coordination and support according to the weighted mean of their importance according to the answers of the agricultural extension service providers.

Seq.	Problems Fields	Weighted mean of the axis	Weight percentile of the axis	Order
4	Weak government awareness of the importance of the role of agricultural extension in facing climate change	3.60	90	1
3	Lack of government support for agricultural institutions and organizations in the field of reduction the effects of climate change	3.58	89.5	2
1	Lack of services provided by the state to rural areas affected by climate change	3.57	89.2	3
2	Lack of early warning stations for the effects of extreme and sudden climatic changes on farmers	3.53	88.2	5
5	The lack of a clear financing policy by the government to help farmers obtain modern agricultural practices such as modern irrigation systems.	3.53	88.2	5
6	Poor coordination between the extension departments responsible for providing services to rural people and other parties interested in the issue of climate change	3.53	88.2	5
Average of the total weighted mean		**3.55**	**88.7**	

It is clear from Table (4) that the problem (the government's weak awareness of the importance of the role of agricultural extension in facing climate change) came in the first place, as it obtained a weighted mean of (3.60) degrees with a weight percentage of (90%). The reason for this may be due to the lack of clarity The government's policy towards the agricultural sector, and therefore the absence of a clear agricultural policy to limit the effects of climate change on the agricultural sector, which negatively affected the level of knowledge and awareness of the direct role of the agricultural extension institution in providing educational extension service to farmers in the field of facing the effects of climate changes on their crops, while The problem (weak coordination between the extension departments responsible for providing services to rural people and other parties interested in the issue of climate change) came in the last place in terms of the level of importance, as it got a mean of (3.52) degrees with a percentage weight of (88%), despite its final arrangement, however, was likely a problem of great importance suffered by the agricultural extension service providers. The reason for this may be due to the absence of an administrative unit with clear functions within the organizational structure of the Agricultural Extension and Training Department concerned with the coordination process between it and other parties concerned with limiting the effects of climate change.

CONCLUSION

1- Agricultural extension service providers face multiple problems hindering their ability to effectively address the impacts of climate change.
2- These problems include issues related to farmers, extension service providers themselves, coordination, and government support.
3- The most significant problem identified was the lack of specialists in agricultural extension who specialize in climate change.
4- On the other hand, the least important problem was the lack of skills and knowledge among extension service providers in determining the best agricultural practices to confront climate changes.

RECOMMENDATIONS

1- The importance of addressing the problems faced by agricultural extension service providers and addressing them by the relevant authorities, especially the General Directorate of Agricultural Extension and Training.
2- Integrating agricultural extension service providers working in climate change adaptation into intensive and well-designed training programs that enable them to successfully perform their duties in this field.
3- Enhancing cooperation and coordination among different stakeholders with the aim of addressing the challenges associated with climate change.
4- Providing financial and moral support by the government and financial institutions to enhance the capacity of agricultural extension service providers to deliver effective services in the agricultural sector and address the challenges and problems they face.

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المجلة العراقيه لبحوث السوق
وحماية المستهلك

THE MODERN FOOD INDUSTRY: TRENDS, CHALLENGES, AND INNOVATIONS

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ABSTRACT

New product development and improvement of existing ones, design as the creation of new food formulations, and the production process of foodstuffs are a major challenge for modern food companies in a competitive environment, in which the ability to identify consumer preferences more accurately becomes more urgent. It is an integral part of the technological activities of the modern food industry. Creating a new kind of food product is not only a scientific but also a commercial component, in which the development of the commercial aspect is extremely important for food companies in terms of the cost of creating a technological market environment. But in planning, studying, market analysis, defining, and targeting the audience, fear is considered one of the main driving forces of economic activity in both the market and the real economy. Its role is to encourage potential buyers of a product or service from a practical point of view to avoid market failure, every company has a detailed market study that helps determine the precise market limits and specify the most important activity to achieve commercial success.

Keywords: new food products, company success, development, consumer satisfaction.

صناعة الأغذية الحديثة: الاتجاهات، التحديات والابتكارات

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الخلاصة

إن تطوير المنتوجات الغذائية الجديدة وتحسين المنتوجات الحالية، والتصميم مثل إنشاء تركيبات غذائية جديدة، وكذلك إنشاء عملية إنتاج المواد الغذائية يمثل تحديًا كبيرًا لشركات الأغذية الحديثة في بيئة تنافسية، تصبح القدرة على تحديد تفضيلات المستهلك بدقة أكثر إلحاحًا. إن هذا الامر أصبح جزء لا يتجزأ من الأنشطة التكنولوجية لصناعة الأغذية الحديثة في الشركات الغذائية. إن إنشاء نوع جديد من المنتوجات الغذائية ليس مكونًا علميًا فحسب، بل هو مكون تجاري أيضًا، اذ إن تطوير الجانب التجاري أمر في غاية الأهمية بالنسبة لشركات الأغذية من حيث تكلفة خلق بيئة السوق التنافسية. ولكن في عملية التخطيط والدراسة وتحليل السوق وتحديد الجمهور المستهدف لها، يعد الخوف أحد القوى الدافعة الرئيسية للنشاط الاقتصادي في كل من السوق والاقتصاد الحقيقي. ويتمثل دورها في تشجيع المشترين المحتملين لمنتج او خدمة معينة من تجنب فشل السوق، ويكون لدى كل شركة دراسة سوقية تفصيلية تساعد على تحديد حدود السوق بدقة وتحديد النشاط الأكثر أهمية لتحقيق النجاح التجاري.

الكلمات المفتاحية: منتوجات غذائية جديدة، نجاح الشركة، تطوير، إرضاء المستهلك.

INTRODUCTION

The modern food industry is a broad and diverse subject encompassing many topics relevant to how food is produced, processed, distributed, marketed, sold, prepared, and eaten, and the factors affecting all of these. The modern food industry is growing in complexity due

to new developments. These developments include shifts in how food is produced (for example, vertical farming, smart agriculture, animal-less food production, alternative proteins, bioengineering food products), processed (for example, automation), distributed (for example, online grocery stores, drone deliveries), marketed (for example, social media marketing), and eaten (for example, meal kits, food delivery apps). These developments also include factors such as population growth, distributed and urbanized populations, growing concern for food safety and quality, environmental impacts of food production, processing, and distribution, and a growing desire for convenience and novel experiences (**Aguilar *et al.*, 2019**). The modern food industry is also a constantly changing subject, and as new developments are adopted and new factors emerge, innovations will be created in response to these developments and factors.
The food industry has witnessed rapid and dramatic changes due to the food industry revolution in recent years (**Ding *et al.*, 2023**). These changes involve the introduction of new technologies that revolutionize food processing and distribution to improve food quality and safety, provide personalized nutrition and diet, support sustainability, and conserve energy and resources (**Alkhafaji M. & Herrara R., 2021**). In this part of the article, the processing, distribution, and consumption of food products are examined and advancements in machine learning, artificial intelligence, big data, blockchain, the internet of things, and biotechnology, all of which address challenges in the food industry and help create a new generation of food industry equipment are explored.

Historical Overview of the Food Industry

The history of the food industry dates back to ancient times when mankind began to cultivate crops. While early agricultural activities were largely associated with nature, civilizations grew and food was processed within cities. At the same time, food processing techniques were improved, and methods such as curing, drying, and smoking were developed to extend the shelf life of food and facilitate storage and transport. Before the 18th century, the food industry was limited in scale and mostly operated in the form of small workshops within cities, producing raw and simple food items such as flour and salt. The lack of close collaboration and information sharing between raw material producers, processors, and retailers led to inefficiencies in supply chain management, generating excessive inventory and food waste. The food industry originated in ancient times when mankind first began to cultivate crops. While early agricultural activities were largely associated with nature, civilizations grew and food was processed within cities. Simple food processing took place, such as roasting grains and corn, boiling tubers, filtering oil from seeds, etc. The food industry then went through several development stages, from food processing being determined by nature to food processing being determined by communities and cities. During the nascent stages in certain regions, food processing was thus initiated in the form of small-scale processing. As agricultural production evolved, machinery and equipment such as stone mills and presses were gradually introduced to make processing more efficient and to maintain the freshness and quality of food. As agricultural techniques improved, the production and processing of food, as the most important commodity, gradually expanded into a larger scale. Food processing technology was gradually developed with industrial characteristics, including salting, curing, pickling, etc. Meanwhile, to ensure the quality of food, brewing and food preservation technology was innovated, including fermentation, cooling, smoking, and carbonating. (**Pilcher, 2023**)

Key Players in the Modern Food Industry

The modern food industry is a complex and vast network that involves multiple players and stakeholders, each having a distinct role in various operations. Each of these entities contributes significantly to the overall functioning and dynamics of this network, directly or indirectly influencing its constituents and operation. These players are primarily associated with food production, processing, preservation, packaging, distribution, marketing, and selling of food items, including a range of services endowed to them (**Aguilar *et al.*, 2019**).

Food producers, food processors, food distributors, food retailers, and food service establishments are among the major contributors to the modern food industry. Other influential actors include importers, exporters, wholesalers, brokers, resellers, and logistics service providers, such as trucking companies, railroads, shipping companies, and airlines. Some companies undertake a combination of these roles. Moreover, companies in the modern food industry are also in competition with producers, quality assurance technicians, grading and sorting facility managers, food quality assurance managers, restaurant managers, food service managers, food marketing managers, and food advertising personnel (**Green *et al.*, 2018**). Various government regulators oversee food safety and security, food trade, and food service guidelines, such as the Food and Drug Administration (FDA), US Department of Agriculture (USDA), Federal Trade Commission (FTC), and Environmental Protection Agency (EPA).

Globalization and the Food Industry

The profound impact of globalization on the modern food industry is thoroughly examined. The food industry, which provides food for humans and animals, is dedicated to collection, processing, preserving, packaging, marketing, and distribution. The food industry is a major pillar in any society or region; the globalization of the food industry is now equally evident. The globalization of the food industry is concerned with the production, processing, storage, and distribution of food, which changes the food supply, consumption, and demand patterns in a country through international trade (**Aguilar *et al.*, 2019**).

The early globalization of the food industry can be traced to the new world discovery by Christopher Columbus and the later establishment of the Columbian Exchange. Still, the food available in a country would mainly depend on the climate, agricultural products, and technology. The industrial revolution in the 1820s and consequently the advancement of agricultural technology narrowed the gap in food availability, supply, and consumption patterns among countries. Nevertheless, land provided equal agricultural products in a region. Land use was maximized by transportation technology in distant land, and eventually, comparative advantages in the agricultural products were created among different regions and countries. The agricultural product trade based on the comparative advantage is the earliest form of food industry globalization, which later expanded to raw materials and processed food. The modern globalization of the food industry resulted from advances in transportation and communication technologies, which radical changes were made to the distribution, supply, demand, and consumption of food. The Internet and satellite technologies enhanced food advertisement, marketing, and retailing. In addition to the McDonaldization world, the food tradition and culture of other countries have also gained acceptance and recognition, such as the tea culture and sushi culture. The food preference, custom, and behavior are dramatically reshaped by the modern world food culture in addition to changing the food supply and consumption pattern (**Ding *et al.*, 2023**).

The food industry is an indispensable component of the modern economy, providing basic human requirements for survival. The food industry also employs many people and makes substantial contributions to gross income, especially for developing countries. The food industry is one of the results of industrialization, which supplies processed food to wholesalers or retailers to meet the demand for huge quantities and variety of food products in urbanization. The modern food industry is based on an interdependent and tightly coupled system spanning from farm production to consumer consumption with increasingly shrinking food supply chains. (**Crippa *et al.*, 2021**)

Sustainability and Environmental Impact in Food Production

Canada's food production and distribution systems are under scrutiny. Safety precautions are required to ensure wholesomeness in foods and beverages offered for sale. The American, European, and to a lesser extent Canadian food production systems are being evaluated and re-evaluated to ensure environmental sustainability. The criticisms include growing foods with artificial fertilizers and pesticides, implementing intensive livestock operations and applying hormones and antibiotics, and concentric venture to food package transactions, all with proposed negative contributions to the ecological footprint (**López-Gálvez *et al.*, 2021**). It is a deliberate universal effort to overcome environmental degradation by curtailing or putting limits to industrial emissions, using the fear of global warming, or climate change. The chemical nitrous oxide is about 300 times more potent than carbon dioxide for the greenhouse effect; the excessive use of nitrogen fertilizers is its principal anthropogenic source. Out of the dimension, current agriculture is not sustainable, at least with present uses and rates of resources (N and P fertilizers, fossil fuels, and various pesticides). Therefore, it is understandable that better use of agricultural resources can reduce "waste" with environmental impacts and sustainability labels (**Kusch-Brandt, 2020**).

Food Safety Regulations and Standards

Food safety regulations and standards are necessary to protect the consumer's health and ensure the quality of food products. Food safety regulations refer to the set of laws, regulations, and government-mandated guidelines that help control the production and distribution of food products. Standards, on the other hand, refer to industry-mandated guidelines, which are also important but are not necessarily enforced by regulations (**Henson & Caswell, 1999**). To ensure compliance with regulations and standards, food processors are expected to monitor the production and distribution of their products through quality assurance programs. The consequences of violations can range from warnings and fines to cessation of production and criminal prosecution. Often, packaging for products includes disclaimers and cautionary messages indicating that the product is not responsible for certain risks (**Abu Bakar, 2012**).

Food safety regulations and standards are extremely complex. In the food industry, the distribution of products is often coordinated through just-in-time delivery systems where many ingredients are outsourced and transported over long distances. This requires food processors to maintain rigorous records of the food product's history throughout the production process, including where ingredients were received, how the product was processed, storage conditions, and shipping information. These records can be very extensive, especially for large firms that process many different products and are sometimes several inches thick for a single batch of a

product. In the event of a food safety violation or perceived violation, state or federal authorities may review the records in an attempt to trace the problem back to the source. Because processors have a legal obligation to maintain these records, companies that do not comply with this requirement can face serious penalties including the loss of business licenses and criminal prosecution.

Technology and Innovation in Food Production

The food industry includes five distinct sectors: crop cultivation, animal husbandry, fishery, food processing, and food consumption (**Aguilar *et al.*, 2019**). At the same time, contemporary food production processes have been revolutionized by novel types of machinery and production techniques based on new technologies within all sectors of the food industry. The food industry along the entire food production chain from farm to table is experiencing gradual digitalization and smarter operation with control, monitoring, data acquisition, optimization, and even designation processes carried out by computer software, digital, and interconnected devices (**Hassoun *et al.*, 2023**).

It has a significant impact on product development, food safety, food quality, and energy/chemical usage during food production and is influencing global competition across contemporary food industries worldwide. States of the art food industry innovations, particularly in production machinery and food processing, flavor, functionality, and healthy enhancement of foods, as well as innovative packaging and storage techniques, digitalized food industry equipment, big data usage for food monitoring, and control, optimization of food production will be discussed in this research.

Supply Chain Management in the Food Industry

Supply Chain Management (SCM) is a complex network of various activities that provide the flow of goods. The food industry is considered one of the most complex systems in that respect. It includes such activities as sourcing, purchasing, transportation, production, processing, packaging, and distribution. The food industry should consider the specific characteristics of food products in the implementation of Supply Chain Management. The agricultural production is highly dependent on factors like climate and weather conditions. The vegetables harvested at a particular place cannot be substituted by the other products. Operations are performed by agricultural cooperatives or by producing and processing companies in combination. The food supply chains are widely exposed to shocks, e.g. climatic shocks, industrial accidents, or terrorist attacks. The strength of impact largely depends on the food product type and on the country level (**Gusarova *et al.*, 2019**). The aim is to provide a segmentation based on vulnerability against various hazards so as to support the decision making in counter-measure design.

Reliable information flows are critical for effective Supply Chain Management. Food products have a great number of intrinsic characteristics influencing the selected distribution channels, e.g. storage and transport temperatures, shelf life, volatility, perishability, quality specifications, size, tariff classification codes, value, etc. Food safety and quality are the top priority of consumers that require validation of safety and quality specifications of food products in each link of the food supply chain. Knowledge about food supply chains is often inadequate or asymmetrical at some supply chain levels causing a lack of transparency (**Soysal et al., 2012**). The reviewed experience of managing food supply chains demonstrates that the

following problems in managing food supply chains can occur: absence of methodology ensuring uninterrupted supplies and durable relations with suppliers; insufficient quality of information flow along the supply chain; high costs, underutilization of storage facilities, excess stocks and chronic shortages of raw products by product types, suppliers, delivery periods, etc. Problems concerning transportation both during raw material delivery from suppliers to processing enterprises and during product distribution from processing enterprises to consumers, including the execution of contracts on the food products delivery are also revealed. The introduction of the proposed methodology in enterprises will create a methodological basis for the selection and effective implementation of decision-making tools in Supply Chain Management (SCM).

Marketing and Consumer Behavior in the Food Industry

The food industry is not only vast and complex, but it is also an economic powerhouse with a monumental impact on food policy. As mentioned in the introduction, nowhere is the relationship between the human population and its survival more apparent than in food issues, most especially in the modern food system. Food issues constitute a fascinating and bewildering array of topics and questions about various aspects of how to survive, and many of the complexities of the modern food industry are illustrated in these questions. The food industry involves more than the people, firms, and farms involved in the production and processing of food, the industry also includes everyone involved with feeding people, from people who visit restaurants to those who put ready-to-eat meals in microwaves. This tremendously diverse group of people, firms, and farms are brought together by one thing: they transform food, for example, take it from a field or container and prep it in some way so that it is more desirable. Because there are so many different aspects of food, food trends, and people involved with food, an equally diverse set of questions can be asked about food topics that touch the modern food industry. Examining these questions sheds light on how food and the food industry shape how we view, engage with, perceive, and think about the world, and food issues highlight the importance of food in many aspects of life, from politics to geography, sociology, economics, and culture. Since the way that somebody eats, what types of food they eat, and how these divorces and purchases are made reflect one's life experiences, upbringings, beliefs, relationships, and social class, this foodiness. (**Sovacool et al., 2021**)

Food Waste and Loss Management

Food waste and loss is an issues that is receiving increased attention and action from governments, organizations, and companies worldwide. According to estimates from the United Nations, Food and Agriculture Organization (FAO), about one-third of global food production is wasted or lost each year. This amounts to about 1.3 billion tonnes of food. Food waste and loss have economic, environmental, and social consequences. On the economic side, food wastage costs the global economy about US$940 billion annually. For developing countries, it amounts to losses of approximately US$310 billion, whereas industrialized countries incur losses of about US$680 billion. Food waste and loss also contribute to environmental degradation. Reducing food waste and loss could mitigate pressures on land, water, and biodiversity (**Centre for Agricultural & Rural Cooperation, 2016**).

Food waste and loss occur throughout the supply chain from primary production to consumption. To decrease waste and loss in the food supply chain, it is essential to identify and

understand the reasons for the waste and losses. For farm and harvest practices, these reasons can be poor agronomic practices, resulting in low productivity and quality. Poor field selection, incorrect seeding, inadequate irrigation, and insufficient fertilizer application can also lead to crop damage and wastage (**Papargyropoulou *et al.*, 2014**). Careless handling of crops can result in crop damage during harvesting and transport. Distributional losses can occur due to poor infrastructure and the ability to transport only for a limited distance. During the processing stage, losses may occur due to process inefficiencies. Packaging and storage losses may occur due to inappropriate or inadequate packaging. Transportation losses may occur due to damage during transport or vehicles not meeting food safety requirements.

Emerging Food Trends and Future Directions

Consumers desire healthy, high-quality food with minimal additives, preservatives, and processing (**Aguilar *et al.*, 2019**). Active edible food packaging has recently become a promising food preservation technology. This packaging includes biopolymers, antimicrobial agents, and antioxidants that maximize food shelf life. The herbal field's evolution has sparked significant interest in edible packaging development using natural plant components. Essential oils, due to their antimicrobial activity, can inhibit gram-positive and gram-negative bacteria and fungi. However, incorporating the oil in edible films can lead to biopolymer solubility reduction. The objective of these innovations is to maximize processing efficiency, preservation times, and food safety to minimize food waste (**Ding *et al*., 2023**).

Consumer profiles have evolved in line with these new processing and packaging technologies. New food platforms for ready-to-eat meals and on-the-go food demand the fast preparation of nutritious, tasty foods with minimal cooking time. Other consumer needs, such as health knowledge, personalized service, food security, and access to groceries, have emerged. The industry has control over food composition and quality to meet these challenges through nutrient bioengineering tools, agricultural techniques, and precision farming technologies.

Challenges and Opportunities in the Food Industry

More than twenty years have passed since the food industry entered the 21st century, setting the stage for another series of changes to meet the new demands of a growing global population with changing economic and social characteristics. With the emergence of a new generation of technologies, a new model of economic development, the rapid integration into the world economy, and the seemingly endless pursuit of benefits and convenience, the global food industry has faced a wide array of challenges, including alternative food networks, the twisting of food trade regimes, and a mismatch between regional policy architecture and food value chains. Borders are becoming more porous; the boundaries between agriculture, food processing, food retailing, and consumer life are increasingly blurred and converged. Farm production is no longer just farm production; it is redefined as a part of a global value chain driven by food processors, retailers, and consumers, while food processors are transforming from manufacturing-centric firms into lifestyle purveyors and brand securities.

Globalization presents challenges, but it also opens up opportunities for innovation. The convergence of economic, social, and technological factors is inspiring a redefinition of the busyness of the food industry and generating a new series of mega technological trends in food processing, storage, distribution, transportation, trading, hazard prevention, and control. The

food industry is in the midst of an inevitable transformation, with a scope of technological trends in and after the 2020s expected to change the industry in a way comparable to the global Green Revolution and Food Chemistry Revolution in the past half century (**Aguilar *et al.*, 2019**). It is no longer possible to fulfill the usage of stable and affordable food for the population over decades because of rising food prices, jarring public grievances on food safety, security hazards, food scares, and the suffering of over 950 million undernourished individuals.

Food Industry and Social Responsibility

The food and agriculture industry, comprising agribusiness, food/beverage processing, retailing and marketing, restaurants, catering services and catering food service industries, is an important part neither only in economic growth but also in social change. In addition to the economic dimension of semi-globalisation, there are social, political, cultural, environmental and demographic dimensions which have important implications for the food production-consumption continuum. The food and agriculture industry has been a key driver of social change as countries become more urbanized and engage in the global economy. This is the case in emerging economies and developing countries but also in developed countries. This paper highlights five key social change issues which are considered important future research opportunities. These key issues pertaining to food safety, health and nutrition, food price inflation, food quality and convenience, and food waste (**Dellios *et al.*, 2009**). To tackle the above issues, the food and agriculture industry has an important role to play in terms of contributing to a more sustainable and equitable food production-consumption continuum, adapting to and mitigating climate change, addressing global poverty and inequitable food price inflation, enhancing food safety and compliance, and ensuring food quality, affordability and accessibility (**Jae Morgan, 2016**).

The social responsibility of the food industry pertains to all business activities that concern ethical conduct at social, environmental, and related issues. Corporations recognize that they are responsible before their stakeholders (employees, consumers, suppliers, investors, and the wider community); and the community where their enterprise has been established. To be socially responsible, organizations must assume a commitment to conduct their business following laws, as well as with ethical and social expectations. Corporate social responsibility is a business strategy with growing acceptance in the food industry. Many food and agribusiness corporations developed and implemented strategies of corporate social responsibility. In some cases, companies consider social responsibility as a task of corporate image marketing.

The Role of Artificial Intelligence in Food Production

In enhancing various aspects of food production/distribution, artificial intelligence (AI) is growing in influence. With the powerful capabilities of machine learning, the food industry can become more efficient, and the issues of food safety and quality can also be resolved. Aspects dominated by AI include food safety and quality control. The aim is to apply AI in the food industry that guarantees food safety and quality and discuss the challenges involved in it, including the ethical aspects. In recent years, with the boost from the pandemic, agriculture and the food industry have turned to automation and robotics technology. Within this new model, there are also great demands for using AI in the abovementioned aspects (**Ding *et al.*, 2023**). AI can be used in agriculture to predict the emergence of crop pests through image analysis,

and management systems can be developed to optimize resource use and climate control, hence enhancing the yield of crop production (**Min *et al*., 2023**). Moreover, in food production, it can be used to support automatic defect inspection and analysis via different sensing methods, hence ensuring product safety and quality. The food packaging industry is also being dominated by AI because of the advent of intelligent robots capable of automatically packing food according to the object detected in an image by an artificial vision system. Processed food production is another aspect where AI can be used in understanding the various mechanistic models in food processing. In the quality control of processed food, smart sensors and data analysis techniques can be explored to develop online automatic systems for quality inspection in food processing and packaging machines.

CONCLUSION

The food industry has undergone fast-paced changes in recent years due to influences from the overall food industry revolution. The development of artificial intelligence, big data, the internet of things, and other information and communication technologies gives more chances and opportunities to the food industry. The futures of the food industry will be based on technologies such as smart agriculture, robotic agriculture, drones, 3D printing, and digital twin technologies. With the considerate development of industrial AI and big data, food manufacturing now shifts from traditional manual production to an automated production phase. Packaging, warehousing, distribution, marketing, and consumer service will also move toward an automated phase. The emergence of these new production and service models brings more innovations and opportunities for the food industry but also higher requirements for the workforce of the food industry (**Ding *et al*., 2023**). Regarding the food industry, food safety issues have become a serious global concern.

Technologies for food processing have gained much attention in recent years. These new technologies have transformed various aspects of the food industry, such as food processing and inspection. The integration of intelligent systems into traditional industries can enhance the safety and quality of processed food, standardize food production processes and formulation, lower the production cost and time, conserve energy and resources, and minimize food loss and waste. Inspired by the successful application of AI in the other sectors, and the fact that food industries are mostly traditional manual-based industries, more researchers started to conduct in-depth research in the food sector, leading to a startup of a large number of AI applications in the food industry (**Aguilar *et al*., 2019**). The latest AI technologies in the food industry include, but are not limited to, synthetic food, food production & energy efficiency management, supply chain management, sales forecasting, assisted cooking, and personalized nutrition. Besides, applying biotechnology along with AI creates food with new attributes either by a new processing technique or combination resource that has not been combined before using technologies similar to 3D printing.

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Ali & et al.,

المجلة العراقية لبحوث السوق
وحماية المستهلك

SYNTHESIS, CHARACTERIZATION AND STUDY THE BIOLOGICAL ACTIVITIES OF NEW HETEOCYCLIC COMPOUNDS CONTAINIG CREATININE

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ABSTRACT

This work included the synthesis of new heterocyclic compounds containing creatinine moiety (A4-A9). These compounds were synthesized from reaction of Schiff bases (A2, A3) with glycine, thioglycolic acid, and glycolic acid to produce imidazolidine-4-one (A4, A5), thiazolidine-4-one A6, A7, and oxazolidine-4-one (A8, A9) respectively. FTIR and ^{1}HNMR were used to identify these compounds. *In vitro* experiments, the antimicrobial properties of compounds (A1, A2, and A4) were evaluated and showed good results.

Keywords: Schiff bases, imidazolidine-4-one, thiazolidine-4-one, oxazolidine-4-one, anti-microbial activity.

تحضير، تشخيص و دراسة الفعالية البيولوجية لمركبات حلقية غير متجانسة جديدة حاوية على الكرياتينين

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الخلاصة

تضمن هذا العمل تحضير مركبات حلقية غير متجانسة جديدة حاوية على جزء الكرياتينين (A4-A9). تم تحضير هذه المركبات من مفاعلة قواعد شيف (A3, A2) مع الكلايسين ،حامض ثايوكلايكولك وحامض الكلايكولك للحصول على ايميدازوليدين-٤-اون (A5, A4)، ثايازوليدين-٤-اون (A7, A6) و اوكسازوليدين-٤-اون (A9, A8)على التوالي. تم استخدام مطيافية FTIR و ^{1}HNMR لتشخيص هذه المركبات. في تجارب مختبرية، تم تقييم الخصائص المضادة للبكتريا للمركبات (A2, A1, A4) واظهرت نتائج جيدة.

الكلمات المفتاحية : قواعد شيف; ايميدازوليدين-٤-اون; ثايازوليدين-٤-اون; اوكسازوليدين-٤-اون; الفعالية المضادة للمايكروبات.

INTRODUCTION

Schiff base molecules containing (carbon-nitrogen) double bonds have attracted an abundance of interest because of their simplicity in synthesis and wide range of uses **(Yassen & Al-Azzawi, 2023; Gatea &Al-Tamimi, 2022)**. Schiff bases additionally have applications in a wide range of other fields, such as chemical analysis, anti-corrosion, ligands for metal complexes, and dyes **(Mahmood, 2021)**. However, Sulfur and nitrogen-containing heterocyclic molecules are essential in therapeutic chemistry applications **(Etivand *et al.*, 2019)**.

One of the heterocyclic compounds with a carbonyl group, nitrogen atoms, and carbon atoms in its structure is imidazolidine-4-one. These compounds have many uses in pharmacology and therapy (**Aftan *et al.*, 2021; Dalaf *et al.*, 2021)**.

Thiazolidin-4-one derivatives are a major class of heterocyclic compounds due to the possibility of medical uses, including antimicrobial, anticancer antifungal, cardiovascular effect s, antitubercular, and anticonvulsant activities **(Trotsko, 2021; Sachin, 2021)**.

Oxazolidinones are efficient bioactive molecules, versatile optical subordinates, and essential synthesis intermediates for natural and bioorganic pigments, and agricultural pesticides **(Abid & Abbass, 2017; Bhat *et al.*, 2011)**. The aim of this research is to synthesize new imidazolidine-4-one, thiazolidine-4-one, and oxazolidine-4-one derivatives from creatinine. In addition, the antimicrobial effects for some of them have been studied.

MATERIALS AND METHODS

Materials and instrumentation

Merck, Sigma/Aldrich, and CDH provided all of the chemicals and solvents that were used in this work. The Merck Company provided the thin layer chromatography and the spots were recognized by iodine vapors. The melting points were recorded using Gallen Kamp equipment. The Fourier transform infrared (FT-IR) spectra of the compounds in a KBr disc have been recorded using a Shimadzu FTIR-8400s Fourier transform infrared spectrophotometer. On a Bruker spectrophotometer (400 MHz), spectrum data for several of the produced substances were measured.

Synthesis of acid hydrazide A1 (Ismail & Al-Tamimi, 2020)

Creatinine derivative was prepared according to the literature procedure **(Ali *et al.*, 2022).** A 1.5 ml of hydrazine hydrate in absolute ethanol (0.03 mol, 99 %), and creatinine derivative (0.01 mol, 2.89 g) were mixed followed by reflux for 6 h, then the precipitate was washed with water. After evaporation of the solvent a recrystallization with ethanol was done. The physical properties of compound A1 is listed in Table (1).

Synthesis of Schiff bases A2, A3 (Ayyash, 2020)

Compound A1 and benzaldehyde/ *p*-hydroxybenzaldehyde/ (0.005 mole) were dissolved in 20 mL of ethanol absolute. Glacial acetic acid was introduced in very small amounts. After that, the reaction mixture was refluxed for six hours. The reaction was then cooled; the formed precipitate after cooling was filtered and recrystallized by ethanol. The physical properties of compounds A2 and A3 are listed in Table (1).

Synthesis of Imidazolidine-4-one derivatives A4, A5 (Muhiebes & Al-Tamimi, 2021)

In a round-bottomed flask, 10 ml of 1,4-dioxane, (0.001 mol, 0.075 g) of glycine, and 0.001 mol of Schiff bases A2/A3 were added. After that, the reaction mixture was heated at 80 ^{0}C for (14–16) hours. The precipitate was then filtered and recrystallized using ethanol. The physical properties of compounds A4 and A5 are listed in Table (1).

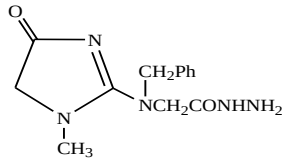
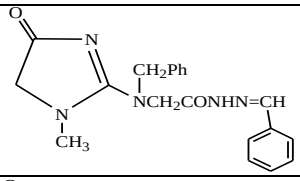
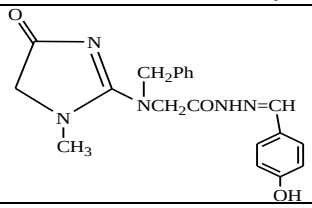
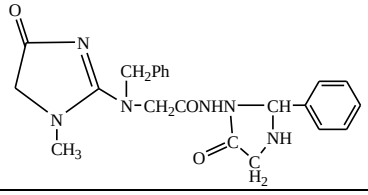
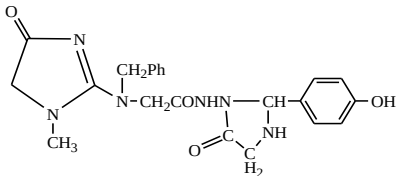
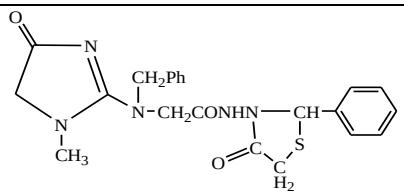
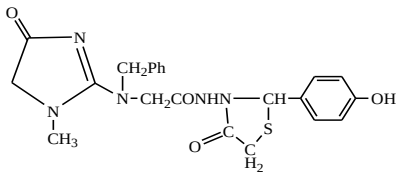
Synthesis of thiazolidine-4-one derivatives A6, A7 (Gupta *et al.*, 2016)

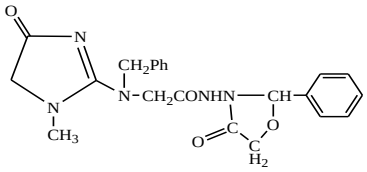
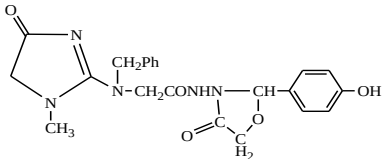
Schiff bases A2/A3 (0.002 mol), thioglycolic acid (0.002 mol, 0.184 ml), and anhydrous zinc chloride (0.0016 mol, 0.21 g) were dissolved in (10) ml of dry 1,4-dioxane. The mixture was heated at 80 ^{0}C for (8–10) hours. The precipitate that resulted from pouring the reaction liquid over crushed ice was filtered, dried, and then recrystallized from ethanol. The physical properties of compounds A6 and A7 are listed in Table (1).

Synthesis of oxazolidine-4-one derivatives A8, A9 (Vivek & Pandurangan, 2014)

As glycolic acid (0.002 mol, 0.152 ml) and Schiff bases A2/A3 (0.002 mol) had been thoroughly mixed in dry 1,4-dioxane (10 ml), then anhydrous zinc chloride (0.0016 mol, 0.21g) was added. After that, the mixture was heated at 80 ^{0}C for (7-9) hours. It took some time for the combination to cool to room temperature. From ethanol, the solid products were recrystallized which their physical properties are presented in Table (1).

Table (1): Physical properties of compounds A1-A9.

Compound No.	Compound structure	Molecular formula	M.wt	Yield %	M.P	Color
A1		$C_{13}H_{17}O_2N_5$	275.31	80	185-187	Pale Yellow
A2		$C_{20}H_{21}N_5O_2$	363.41	94	99-100	Yellow
A3		$C_{20}H_{21}N_5O_3$	379.41	75	190-192	Yellow
A4		$C_{22}H_{24}N_6O_3$	420.46	75	195-197	Yellow
A5		$C_{22}H_{24}N_6O_4$	436.46	80	200-202	Orange
A6		$C_{22}H_{23}N_5O_3S$	437.15	77	175-177	Orange
A7		$C_{22}H_{23}N_5O_4S$	453.51	85	199-200	Yellow

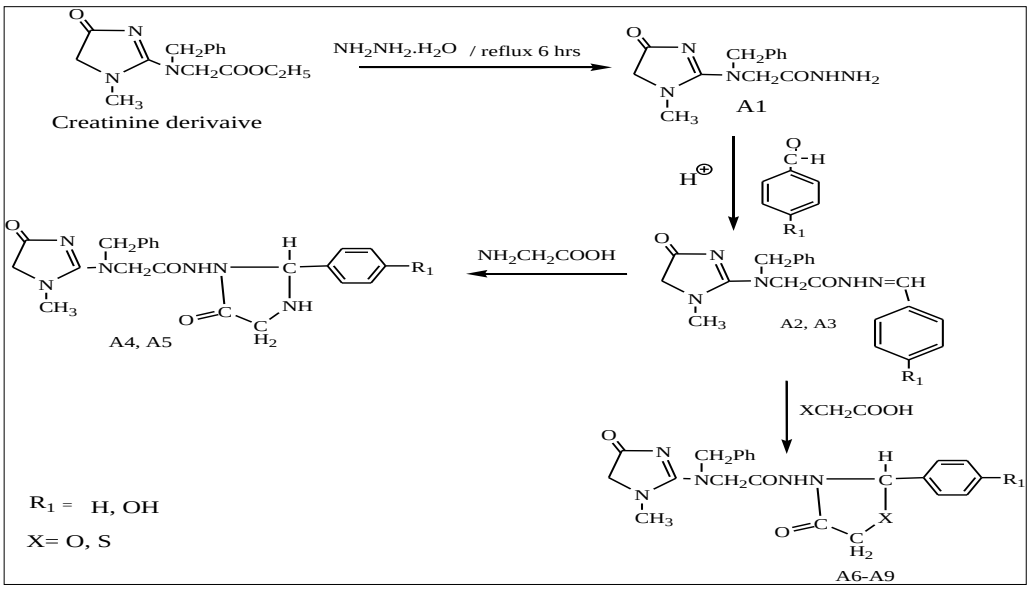
A8		$C_{22}H_{23}N_5O_4$	421.45	75	218-220	Red
A9		$C_{22}H_{23}N_5O_5$	437.45	73	205-207	Green

Biological evaluation (Khorsheed *et al.*, 2020)

Some of the synthesized compounds (A1, A2, A4, and A9) were examined for their antimicrobial effects using the agar diffusion method on two kinds of bacteria (*Staph. aureus* and *Escherichia coli*) and two types of fungi (*Candida albicans* and *Aspergillus flavus*). These sterile agar media were poured over Petri dishes, allowed to set, and then, by using the tidy triangular loop, microbe suspensions were applied to the surface. The synthesized compounds were applied serially using a micropipette and allowed to diffuse for an hour. These plates underwent a 24-hour incubation period at 37 °C. The zone of inhibition in the cup was studied and quantified in mm.

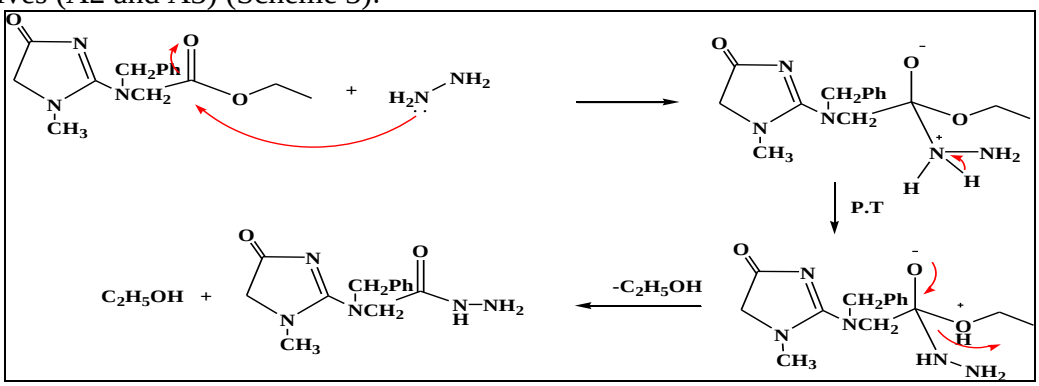
RESULTS AND DISCUSSION

Scheme (1) illustrates the synthetic routes of compounds A1-A9.

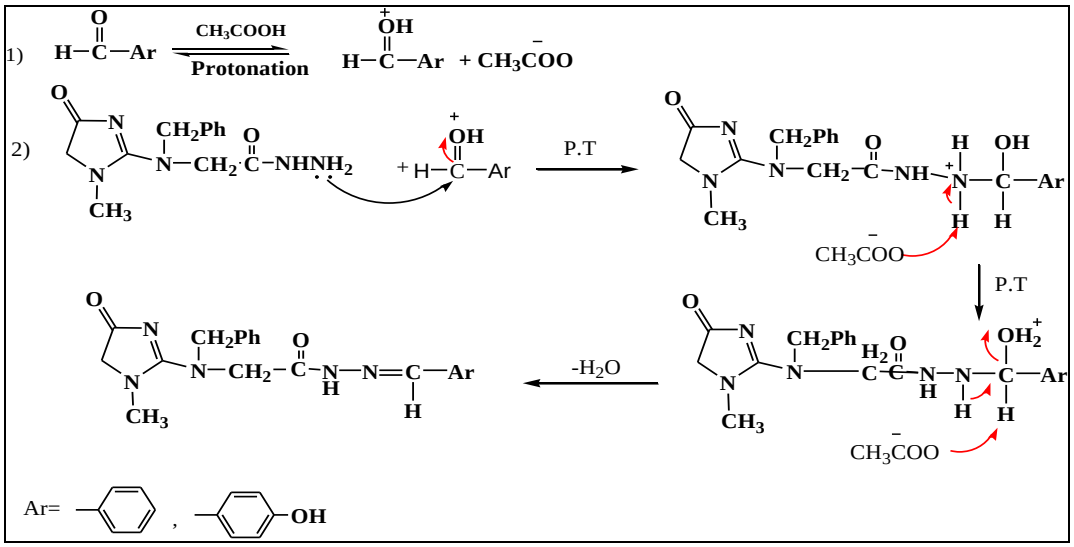


Scheme (1): Routs synthesis of compounds A1-A9.

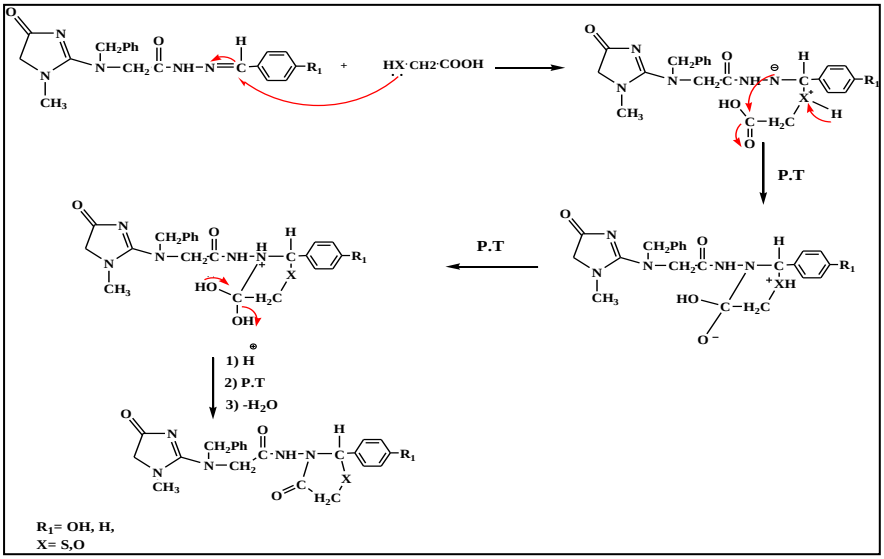
The derivative of creatinine was reacted with hydrazine hydrate to form acid hydrazide derivative A1(Scheme 2) which then reacted with aromatic aldehydes to form Schiff bases derivatives (A2 and A3) (Scheme 3).



Scheme (2): The mechanism for synthesis of hydrazide derivative A1.



Scheme (3): The chemical steps for the synthesis of Schiff bases A2 and A3 Then, the resulting Schiff bases were given a cyclization with glycine, thioglycolic acid, and glycolic acid to produce imidazolidine-4-one (A4, A5), thiazolidine-4-one (A6, A7), and oxazolidine-4-one (A8, A9) compounds respectively (Scheme 4).



Scheme (4): The mechanism of reaction for compounds A6-A9

The FITR of the compound revealed the appearance of the N-H band, NH_2 band, and (C=O) band of hydrazide. While, in Schiff bases the existence of C=N bands were revealed, as shown in Table (2) which includes these bands as well as additional bands.

Table (2): The FT-IR Spectral data of compounds A1-A3 cm^{-1}

Compound number	C-H υ aliphatic	C=O υ cyclic amide C=O amide	υC=N C-N υ	C-H υ aromatic	C=C υ aromatic	(N-H) υ	Others
A1	Asym 2983 sym 2812	1718 1668	1643 1334	3018	1575 1502	3257	(NH_2) Asym. 3419 Sym 3375
A2	Asym 2979 Sym 2820	1700 1680	1640 1340	3050	1571 1550	3274	------
A3	Asym 2979 Sym 2937	1720 1699	1640 1336	3050	1573 1514	3353	(O-H) 3427

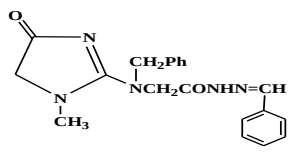
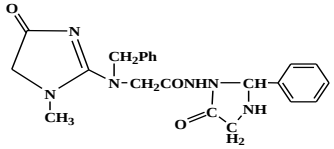
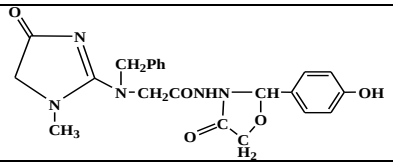
The FTIR data for the synthesized compounds (A4-A9) revealed the creation of C-S bands in thiazolidine-4-one as well as the presence of a distinctive band that was brought on by the C=O cyclic amide of the imidazolidine-4-one, thiazolidine-4-one, and oxazolidine-4-one rings. These bands are listed in Table (3) along with some others.

Table (3): The FT-IR Spectral data of the synthesized compounds A4-A9 in cm^{-1}.

Compound number	C-H υ aliphatic	C=O υ cyclic amide C=O amide	ring υC=N C-N υ	C-H υ aromatic	C=C υ aromatic	(N-H) υ	υ C-S υ C-O	Others
A4	Asym 2975 sym 2997	1710 1680	1637 1334	3050	1573 1502	3294	----- -----	----
A5	Asym 2968 Sym 2880	1699 1679	1639 1336	3033	1602 1556	3286	------ ------	(O-H) 3433 **υ**
A6	Asym 2948 Sym 2887	1710 1689	1643 1330	3050	1623 1575	3280	649 -----	---
A7	Asym 2941 Sym 2881	1710 1670	1641 1338	3002	1606 1575	3220	648 -------	(O-H) 3340 **υ**
A8	Asym 2937 Sym 2885	1710 1690	1647 1332	3050	1622 1573	3220	----- 1253	------
A9	Asym 2939 Sym 2850	1700 1679	1666 1336	3050	1606 1591	3253	----- 1228	**υ** (O-H) 3332

The ^{1}H-NMR spectra of compounds A2, A4, and A9 are listed in Table (4).

Table (4): The ^{1}H-NMR of compounds A2, A4, and A9

Compound number	Compound structure	^{1}H-NMR spectral data
A2		1.2 (s, 3H, N-$\underline{CH_3}$); 2.5 (t, 2H, $\underline{CH_2}$C=O); 3.3 (s, 2H, CH_3-N-$\underline{CH_2}$); 3.8 (t, 2H, $\underline{CH_2}$-Ph); 6.3 (s, 1H, N=$\underline{CH}$); (7.5-7.9) (m, 10 H aromatic); 8.7 (s, 1H,NH)
A4		1.2 (s, 3H, N-$\underline{CH_3}$); 3 (d, 1H, N-$\underline{CH}$-Ar); 3.3 (t, 2H ,$\underline{CH_2}$C=O); 3.5 (s, 2H, $\underline{CH_2}$-C=O of imidazolidine ring); 3.5 (s, 2H, CH_3-N-$\underline{CH_2}$); 3.9 (t, 2H, $\underline{CH_2}$-Ar); (7.4-8.1) (m, 10H aromatic); 8.6 (s, 1H, C-NH proton of imidazolidine ring); 9.2 (s, 1H, NH)
A9		1.1 (s, 3H, N-$\underline{CH_3}$); 2.5(s, 3H, Ar-C$\underline{CH_3}$); 3.2 (t, 2H, $\underline{CH_2}$C=O); 3.3 (s, 2H, $\underline{CH_2}$-C=O of oxadiazolidine ring); 3.5 (s, 2H, CH_3-N-$\underline{CH_2}$); 3.9 (t, 2H, $\underline{CH_2}$-Ph); (6.8-7.7) (m, 11H, aromatic); 8.5 (s, 1H, NH); 10.1 (s, 1H, OH)

Biological activity

The synthetic compounds (A1, A2, and A4) demonstrated different biological effects against the gram-positive and gram-negative bacteria *staphylococcus aureus* and *Escherichia coli* with amoxicillin as standard as well as the two types of fungi *Aspergillus flavus* and *Candida albicans* in comparison to the standard fluconazole drug. As seen in the results, compound A1 has the highest activity against *staphylococcus aureus*, *Escherichia coli*, and *Aspergillus flavus*, *Candida albicans* **(Hussein *et al.*, 2022).** While, compounds A2, and A4 showed moderate activity against *Staphylococcus aureu*s and inactive against *Escherichia coli* and fungi, as shown in Table (5).

Table (5): Antimicrobial activity of compounds A1, A2, and A4.

Compound Number	*Staphylococcus*	*Escherichia coli*	*C.albicans*	*Aspergillus flavus*
	Conc. (0.02 g/ml) Inhibition zone diameter (mm)	**Conc. (0.02 g/ml) Inhibition zone diameter (mm)**	**Conc. (0.02 g/ml) Inhibition zone diameter (mm)**	**Conc. (0.02 g/ml) Inhibition zone diameter (mm)**
A1	15	30	25	20
A2	11	-	-	-
A4	11	-	-	-
Amoxicillin	32	-	-	-
Fluconazole	-	-	25	27

CONCLUSIONS

In the present research, new hydrazide derivative A1 and Schiff bases (A2, A3) were used to synthesize new imidazolidine-4-one (A4, A5), thiazolidine-4-one derivatives (A6, A7), and oxazolidine-4-one compounds (A8, A9). The identification of these new compounds was based on spectrum data (FT-IR and 1H-NMR). Additionally, the antibacterial activities of several of the produced compounds A1, A2, and A4 were assessed. The results revealed that compound A1 has activity against *Escherichia coli* which the slandered Amoxicillin do not have, as well as an antifungal activity.

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Hadi & Sabet

المجلة العراقية لبحوث السوق
وحماية المستهلك

EVALUATE THE EFFECTIVENESS OF OLEIC ACID AND LINOLEIC ACID IN CONTROLLING THE *TROGODERMA GRANARIUM* EVERTS (COLEOPTERA: DERMESTIDAE)

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ABSTRACT

This study aimed to evaluate the effectiveness and efficiency of the two fatty acids, Oleic acid, and Linoleic acid, by contact method as a natural and safe alternative to chemical pesticides in controlling the motile stages (adults, larvae) of grain beetle *Trogoderma granarium* Everts. This laboratory study included four concentrations (50, 100, 150, 200) ppm with three exposure times (8, 16, 24) h. The results showed that the treatment of insect adults with oleic and linoleic acid gave the highest killing rates at a rate of 96.66% and 93.33%, respectively, while the killing rates for larvae after treatment with the two acids were 75.55% and 74.44%, respectively, at a concentration of 200 ppm and a 24-h exposure period of both transactions. The results showed, in general, significant effects of the concentrations and durations of exposure and the interaction between both acids on insect-killing rates. It was also noted that oleic acid was superior to linoleic acid in causing killing rates in the two cycles at most concentrations and exposure times used for its ability to penetrate the cuticle layer in insects from their flexible regions and respiratory openings when they moved on it and causing many deformations of their tissues because of its double bonds that make it occupy a larger cross-section. It increases kinetic freedom in the membrane of the target organism as soon as it enters its body and induces cellular toxicity, causing its death.

Keywords: Oleic, Linoleic acid, Contact, The mobile stage of Khapra.

[*] **The research is taken from a master's thesis by the first researcher.**

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تقييم فعالية حامض الاوليك واللينوليك في السيطرة على*Trogoderma granarium* (Everts) (Dermestidae: Coleoptera)

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الخلاصة

أجريت هذه الدراسة بهدف تقييم فعالية وكفاءة الحامضين الدهنيين Oleic acid وLinoleic acid بطريقة الملامسة كبديل طبيعي وآمن عن المبيدات الكيميائية في مكافحة الأدوار المتحركة (البالغات، اليرقات) لحشرة خنفساء الحبوب الشعرية *Trogoderma granarium* Everts . تضمنت هذه الدراسة المختبرية استعمال أربع تراكيز هي (50 ،100 ،150 ،200) جزء بالمليون مع ثلاث فترات تعريض(8، 16، 24) ساعة. أظهرت النتائج إن معاملة بالغات الحشرة بحامض الاوليك واللينوليك أعطت أعلى نسب القتل فيها بمعدل 96.66% و93.33% على التوالي، في حين كانت معدلات القتل لليرقات بعد معاملتها بالحامضين هي 75.55% و74.44 % على التوالي عند التركيز 200 جزء بالمليون وبمدة تعريض 24 ساعة في كلتا المعاملتين. وبينت النتائج بصورة عامة الى معنوية كل من تأثيرات التراكيز ومدد التعريض والتداخل بينهما في كلا الحامضين في نسب قتل دوري الحشرة. كما لوحظ تفوق حامض الاوليك على حامض اللينوليك في إحداثهِ لنسب القتل في الدورين عند أغلب التراكيز ومدد التعريض المستخدمة لقدرتهِ على اختراق طبقة الكيوتكل في الحشرات من مناطقها المرنة وفتحاتها التنفسية عند حركتها عليه وتسببهِ في تشوهات عديدة لأنسجتها لما يمتلكهُ من أواصر مزدوجة تجعلهُ يشغل مقطعاً عرضياً أكبر يعطيهُ زيادة في الحرية الحركية في غشاء الكائن المستهدف بمجرد دخولهِ لجسمها وتحريضهِ على السمية الخلوية فيها مسبباً موتها.

الكلمات المفتاحية : حامض الاوليك ، اللينوليك ، ملامسة ، الأدوار المتحركة للخابرا

INTRODUCTION

It is known that grain crops, including wheat, contain high levels of carbohydrates, proteins and fats. Therefore, the world's population considers it one of their basic foods and demands that it be stored for long periods or for export **(Pugazhvendan *et al*., 2009).** One of the most important issues facing wheat storage is its infection with insect pests widely spread worldwide. In Iraq, 31 species belonging to 16 genus were found, falling under eight families and two orders spread in most places where grain is stored, At the forefront of these insects is the *Trogoderma granarium* (Everts) which represents a significant threat to it and is also considered one of the worst species due to its difficulty controlling **(Sabit& Saadi, 2015; Hanaa & Razzaq ,2022).** The chemical control of pests, such as fumigation with aluminum phosphide gas, contributed greatly to controlling the pest and reducing its numbers **(Khaled & Nawal, 2020**). but the insect larvae showed resistance to the action of chemical pesticides due to their entering into voluntarily dormancy **(Burges, 2008).** Chemical control is also not without drawbacks, It is represented by leaving its residues, as it is one of the important sources of pollution for the terrestrial and aquatic environments and harms the health of humans and animals and causes acute and chronic fish poisoning. This is what prompted specialists in the field of pest control to search for alternative methods that contribute to protecting the environment and the safety of workers and consumers **(Bakhroini *et al*, 2023).** One of the alternative methods for chemical pesticides in pest control is using powders such as silica powder **(Falah & Azhar, 2021**). *Eucalyptus camaldulensis* leaf powder in controlling them **(Falah, 2020).** as well as plant extracts such as the ethyl extract of the leaves and seeds of *Sesbania sesban* in controlling their population densities **(Shaimma & Falah, 2020).** Also, fatty acids have been used recently in the control of insect pests, as most of them are widespread in most plant and animal sources (**Abbas & AL-Kareem, 2015**). They have been

widely used, especially oleic and linoleic acids, as they have proven to have the desired insecticidal properties and in light of the previous, the study aimed to know the effect of contact of two stearic acids on the motile stages (adults and larvae) of the hairy grain beetle with different concentrations and exposure times **(Justin *et al*., 2019; Hamad ,2021).**

MATERIALS AND METHODS

Insect culture

Some different stage of the *Trogoderma granarium* were obtained from the infected grains from the insect laboratories in the Plant Protection Department, College of Agricultural Engineering Sciences, University of Baghdad. In July 2022, to rear them, the insect was placed in its various stage in clean plastic pots and sterilized by incubator heat, capacity of 700 ml containing wheat grains of Ibaa 99, which are most sensitive to insect infestation, to ensure rapid reproduction after making sure that they are free of other insect pests by subjecting them to freezing at a temperature of (-20) degrees Celsius for (20) d**.** The pots were covered from the top with a organza fabric, tied with a rubber band, and placed in a binder incubator equipped with a hygrometer. The incubator was installed at 35 ± 1 ° C, a lighting period (hour) of 1 light: 23 dark, and a humidity of 65 ± 5. To stabilize the humidity, 3g of KOH in 100 ml water in a sealed glass container. The insect culture has also been maintained by adding wheat grains to it continuously to ensure obtaining an abundance of insects that extends the experiment in different stages, and the flour and molting skins resulting from insect activity are also disposed of by sieving from time to time which include development until pupation **(Abdullah *et al*, 2005; Al-Hayali, 2018).**

Adults

After continuous monitoring of the isolated pupae from the laboratory culture, newly hatched adults at the age of 24 hours were obtained, as 10 adults (males + females) in the rate of 1:1 were isolated in each petri dish (replication), with 3 replications for each treatment in addition to the control treatment, and they were introduced to the incubator under the same conditions of temperature and relative humidity referred to in the previous paragraph.

Larvae

Insect eggs were obtained by isolating 10 pairs of adult males and females, with a sex ratio of 1:1, at the age of 1-2 d, in a sterile plastic petri dish with a diameter of 9 cm and a depth of 1.5 cm containing 3 g of wheat grains and placing the dish in the incubator under the previously mentioned conditions. Through continuous monitoring of dishes containing insect eggs, newly hatched larvae were transferred to petri dishes using a soft brush moistened with water at the rate of 10 larvae per dish (replicated) and 3 replicates for each treatment in addition to the control treatment. They were introduced to the incubator under the previously mentioned temperature and humidity relativity conditions.

Preparation of concentration of each oleic and linoleic acid

The oleic acid concentrate prepared for control was prepared by placing 500 ml of concentrated ethanol 99% in a container with a capacity of 1000 ml, and 50 microliters of oleic acid with a concentration of 95% were dissolved in it, which quantity was moved by the

microliter device. and then the rest was supplemented with distilled water to finally reach a volume of 1000 ml. The ratio of the fatty acid to the solvent solution became (50) ppm. Thus, the rest of the concentrations were prepared 100, 150, 200 ppm, in addition to the comparison treatment, which was only 50% ethanol. As for the concentrations of linoleic acid, they were prepared in the same way, and the concentration of linoleic acid was 98% **(Shaba, 2011).**

Biological assessment of fatty acids

Petri dishes containing filter papers were prepared and sprayed with the concentrations 50,100,150,200 ppm a small hand sprayer with a capacity of 20 ml and at a distance of 15 cm from the dish at a rate of 1 ml to ensure the homogeneity of the solution. For all dishes (replicates) and for the various treatments and stages as well as the control treatment, after which the stages are transferred, the different forms of the insect from the breeding dishes to the treated dishes at the rate of 10 individuals from each stage, to transfer the treated dishes to the incubator under the conditions referred to previously, with exposure periods of (8, 16, 24) h, and at the end of the exposure periods, the stages are removed from the treated dishes to other dishes so as not to be exposed to the concentration of the pesticide more than the time prescribed for its treatment and return it to the incubator again.

Adults at the age of 24 h and larvae (15) d old were tested by contact treatment and with both acids (oleic and linoleic) and each separately, by isolating (10) individuals in each replicate and by 3 replications for each treatment, as the rotation was treated with four concentrations (50, 100, 150, 200) ppm and for three periods for each concentration (8, 16, 24) h, then the killing rates were calculated according to the corrected death rate in the equation Schneider and Orel **(Al-Jassani ,2015; Al-Ghazali *et al* ,2018).**

Statistical analysis

The Statistical analysis of the experimental data was computed using analysis of variance procedure described in the SAS (2018) mean differences were compared by using the Least Significant Difference (LSD) based on the Completely Randomized Design (CRD).

RESULTS AND DISCUSSION

1-The effect of oleic acid on *Trogoderma geanarium* adults

The results of Table (1) of the study of the contact effect of oleic acid on the adults of the beetle of the hairy grain beetle *Trogoderma granarium* show the effect of oleic acid on the killing percentages of the adults of the insect at concentrations 50, 100, 150, 200 ppm and exposure periods of 8, 16, 24 An h, the effect of concentrations on the rates of killing rates reached 28.88, 53.33, 81.11, 96.66%, respectively. In contrast, the effect of exposure periods on killing rates reached 51.66, 66.66, 76.66%, respectively.

As for the highest results of the interaction between the two factors, it was 100% at a concentration of 200 ppm and an exposure time of 24 h, while the least was 20% at a concentration of 50 ppm and an exposure time of 8 h. The statistical analysis results showed, in general, significant effects of both concentrations and durations of exposure and the interaction between them in the killing rates of adult insects, which indicates an increase in killing rates with increasing concentrations and exposure times.

Table (1) Effect of oleic acid on *Trogoderma geanarium* adults

Concentration (in µL) PPM	killing rates (%) Time/ h			Average
	8	16	24	
50	20.00	30.00	36.66	**28.88**
100	33.33	50.00	76.66	**53.33**
150	60.00	90.00	93.33	**81.11**
200	93.33	96.66	100	**96.66**
Average	**51.66**	**66.66**	**76.66**	---
LSD 0.05	**Concentration = 7.94 * , Time= 6.22* , ،Interaction= 11.73 ***			

2. The effect of linoleic acid on *Trogoderma geanarium* adults

The results of Table (2) of the study of the contact effect of linoleic acid on the adults of the beetle of the hairy grain beetle *Trogoderma granarium* show the effect of linoleic acid on the killing percentages of the insect adults at concentrations 50, 100, 150, 200 ppm and exposure periods of 8, 16 and 24 h. The effect of concentrations on the killing rates reached 19.99, 37.77, 69.99, and 93.33%, respectively, while the effect of exposure periods on the killing rates reached 44.16, 56.66, and 64.99%, respectively.

As for the highest results of the interaction between the two factors, it was 100% at a concentration of 200 ppm and an exposure time of 24 h, while the lowest was 13.33% at a concentration of 50 ppm and an exposure time of 8 h. The statistical analysis results showed, in general, that the effects of concentrations and durations of exposure, and the interaction between them, were significant in killing rates of adult insects.

Table (2) Effect of linoleic acid on *Trogoderma geanarium* adults

Concentration (in µL) PPM	killing rates (%) Time/ h			Average
	8	16	24	
50	13.33	20.00	26.66	**19.99**
100	30.00	33.33	50.00	**37.77**
150	53.33	73.33	83.33	**69.99**
200	80.00	100	100	**93.33**
Average	**44.16**	**56.66**	**64.99**	---
LSD 0.05	**Concentration = 8.59* , Time= 7.44* , ،Interaction= 7.44 ***			

It was clear from the results of tables (1, 2) that the contact effect of oleic acid exceeded the contact effect of linoleic acid in causing killing rates for adults of the hairy grain beetle *Trogoderma granarium* at all concentrations and exposure times used, thus reducing their numerical density, as it was found that there is a direct relationship between concentrations, exposure periods, and killing rates, The higher the concentration or the duration of exposure, the higher the killing rates, The reason for killing adult with oleic acid may be attributed to its ability to penetrate the cuticle layer from the flexible areas of the body or the respiratory openings **(Shaaban & Al-Mallah, 1993)**. This ability to penetrate may be due to the presence of double bonds in it, which makes it occupy a larger cross-section, which gives it an increase in kinetic freedom in the membrane of the target organism (**Aline *et al.*, 2018).** It is also believed that one of the characteristics of the fatty acid affecting the organism's tissues is its structure, shape, and length of its carbon chain, the number of double bonds in it, and the position and direction of target organisms **(Desbois & Smith, 2010)**. In addition, oleic acid induces cytotoxicity within the bodies of adults through a change in their size and granularity, disruption of the integrity of their membranes, and fragmentation of their nucleic acid, which leads to the excretion of phosphatidylserine through flow cytometry in it. Changes also occur in the capabilities of the mitochondrial membrane. Fat inside cells lead to programmed death **(Suha, 2011; Thais *et al.*, 2006)**. The results showed that the killing rates increase with increasing concentrations and exposure times, and this is consistent with what was mentioned by **(Mousa *et al.*, 2011)** when using oleic acid in the control of adults of the insect *Sitophilus oryzae* (L.) as the killing rates increased by increasing the concentration from 2-10 µl / g of rice and increasing the exposure time from 24-72 h. It is also consistent with the results of the researcher **(Kerbel *et al.*, 2021**) when oleic acid was used in the control of adults of the falciparum insect. *Rhyzopertha dominica* showed that the contact toxicity of oleic acid increased the killing rates of adult insects by increasing doses and durations of exposure.

3. The effect of oleic acid on *Trogoderma geanarium* larvae

The results of Table (3) related to the study of the contact effect of oleic acid on the larvae of the hairy grain beetle *Trogoderma granarium* show the effect of oleic acid on the killing percentage of its larvae at concentrations of 50, 100, 150, 200 ppm and exposure periods of 8, 16 and 24 h. The effect of concentrations on the killing rates reached 16.66, 39.99, 64.44, and 75.55%, respectively, while the effect of exposure periods on the killing rates reached 43.33, 47.49, and 56.66%, respectively.

As for the highest results of the interaction between the two factors, it was 80% at a concentration of 200 ppm and an exposure time of 24 h, while the lowest was 13.33% at a concentration of 50 ppm and an exposure time of 8 h. The statistical analysis results showed, in general, significant effects of concentrations and durations of exposure, except for rates between 8 and 16 hours and the overlap between them in killing rates of insect larvae.

Table (3) Effect of oleic acid on *Trogoderma geanarium* larvae

Concentration (in µL) PPM	killing rates (%) Time/ h 8	16	24	Average
50	13.33	16.66	20.00	**16.66**
100	33.33	36.66	50.00	**39.99**
150	56.66	60.00	76.66	**64.44**
200	70.00	76.66	80.00	**75.55**
Average	**43.33**	**47.49**	**56.66**	---
LSD 0.05	**Concentration = 7.59* , Time= 6.82* , ،Interaction= 12.63 ***			

4- The effect of linoleic acid on *Trogoderma geanarium* larvae

The results of Table (4) of the study of the contact effect of linoleic acid on the larvae of the hairy grain beetle *Trogoderma granarium* show the effect of linoleic acid on the killing percentage of its larvae at concentrations of 50, 100, 150, 200 ppm and exposure periods of 8, 16 and 24 h. The effect of concentrations on the killing rates reached 16.66, 38.88, 57.77, and 74.44%, respectively, while the effect of exposure periods on the killing rates reached 38.33, 46.66, and 55.83%, respectively.

The highest results of the interaction between the two factors were 83.33% at a concentration of 200 ppm and an exposure time of 24 h, while the lowest was 13.33% at a concentration of 50 ppm and an exposure time of 8 h. The statistical analysis results showed significant effects of concentrations and exposure times and the interaction between them on the killing rates of insect larvae.

Table (4) Effect of linoleic acid on *Trogoderma geanarium* larvae

Concentration (in µL) PPM	killing rates (%) Time/ h 8	16	24	Average
50	13.33	16.66	20.00	**16.66**
100	30.00	36.66	50.00	**38.88**
150	43.33	60.00	70.00	**57.77**
200	66.66	73.33	83.33	**74.44**
Average	**38.33**	**46.66**	**55.83**	---
LSD 0.05	**Concentration = 9.13* , Time= 7.85* , ،Interaction= 15.71 ***			

It was clear from the results of tables (3, 4) that the contact effect of oleic acid was superior to the contact effect of linoleic acid in killing rates of larvae of the hairy grain beetle *Trogoderma granarium* at all concentrations and exposure times and the reason for this may be attributed to the effectiveness of oleic acid. In removing the plate located in the larval body wall, as its chitinous wall is thin in its early larval stages, the cutaneous protein decomposes sufficiently, which means that it has a fundamental stage in the analysis of the cutaneous region (epidermis) of the larvae. It also works to inhibit the action of Ecdysone 20 monooxygenase, which helps in promoting The growth of the cell membrane in insects, as it was noted that there is a clear decrease in the levels of the enzyme B-N-acetyl glucosaminidase and in the work of the per trophic membrane, which is a protective covering for the back of the middle intestine and is associated with the intestinal region**(Usharani & Kummankottil, 2012;Mahmoud & Sarah, 2013)**. It was also found that oleic acid causes swelling of the mitochondria and the endoplasmic reticulum and has a stage in vacuole enlargement followed by epithelial cell lysis and perforation of the middle intestine. An increase in the number of vesicles in the fat body and the cells of the middle intestine also has an effect on the metabolism process and the formation of the middle intestine and the fat body **(Aline *et al.*, 2018)** The results showed that the killing rates increase with increasing concentration and duration of exposure, and this is consistent with what was stated by **(Heba & Hemat, 2013**) when using oleic acid to control the first larval stages of the clove boll cycle *Pectinophora gossypiella* (Saunders) (Lepidoptera: Gelechiidae). The results also showed that using oleic acid by contact method was toxic to the insect larvae This is consistent with what was mentioned by **Santhana *et al.* (2020)** when oleic acid was used by contact method to control the larvae of two insects, *Eligma narcissus* cramer (Lepidoptera: Nolidae) and *Hyblaea Puera* cramer (Lepidoptera: Hyblaeidaea). It is also consistent with what was stated by **Imad & Tabark (2016)** when the crude alcoholic extract of *Cordia myxa* leaves was used at a concentration of 8% against the fourth larval stage of *Rhyzopertha dominica*, as the killing rate reached 93.3%.

CONCLUSIONS AND RECOMMENDATIONS

The fatty acids can be used as insecticides against the pest, and the concentrations used in the experiment and the periods of exposure to it contributed significantly to determining the killing rates in the different stages of the insect, which enables the introduction of fatty acids in integrated pest management programs, and alternative and safe methods must be developed For chemical pesticides in the control of stored pests, especially steam ones, for their safety and not leaving chemical residues**.**

The use of Oleic acid and Linoleic acid by contact method in controlling the moving stages of the hairy grain beetle *Trogoderma granarium* Everts (Coleoptera; Dermestidae) was very effective as it achieved high death rates in it.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

STUDYING THE EFFECT OF USING SUPER PROTEIN, VITAMIN C AND E AS A FOOD SUBSTITUTE IN HONEYBEE COLONY ACTIVITY

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ABSTRACT

The research was conducted in a native apiary in Baghdad Governorate- Al-Buaitha, to evaluate the effect of adding super protein, vitamin C and vitamin E to the diet of honeybee colonies, and to study their effect on the activity, growth, development and productivity of the colonies of honey.

The results of the research showed that super protein nutrition achieved the best percentages, and according to the results of the statistical analysis, it significantly outperformed the sugar solution feeding and control, in terms of measuring the honey area 2.631 cm^2, the brood area 1.622 cm^2 and the pollen area 0.378 cm^2.

The results of the use of vitamins also showed that the best treatments were using vitamin C nutrition, and according to the results of the statistical analysis, it significantly outperformed all other treatments in terms of measuring the honey area 3.820 cm^2 and the brood area 1.821 cm^2 and pollen area 0.608 cm^2. The results showed that vitamin E nutrition outperformed the sugar solution feeding and control, as the average area of honey, brood and pollen grains were (2.960 cm^2, 1.624 cm^2 and 0.473 cm^2) respectively.

Keywords: Apiary, Honeybees, Vitamins, Proteins.

* **The research is derived from the master's thesis of the first researcher.**

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دراسة تأثير استخدام السوبر بروتين، فيتامين C و E كبديل غذائي في نشاط طوائف نحل العسل

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الخلاصة

أجري البحث في منحل اهلي في محافظة بغداد - البوعيثة لتقييم تأثير إضافة السوبر بروتين وفيتاميني C و E الى غذاء طوائف نحل العسل، ودراسة تأثيرها على نشاط ونمو وتطور وانتاجية الطوائف للعسل. بينت نتائج البحث أن التغذية بالسوبر بروتين حققت افضل النسب وبحسب نتائج التحليل الاحصائي فقد تفوقت معنوياً على التغذية بالمحلول السكري والمقارنة من حيث قياس مساحة العسل 2.631 سم^2 ومساحة الحضنة 1.622 سم^2 ومساحة حبوب اللقاح 0.378 سم^2. كما بينت نتائج استخدام الفيتامينات أن افضل المعاملات كانت باستخدام التغذية بفيتامين C وبحسب نتائج التحليل الاحصائي فقد تفوقت معنوياً على جميع المعاملات الاخرى من حيث قياس مساحة العسل 3.820 سم^2 ومساحة الحضنة 1.821 سم^2 ومساحة حبوب اللقاح 0.608 سم^2. اظهرت النتائج ان التغذية بفيتامين E تفوقت على التغذية بالمحلول السكري و المقارنة اذ بلغ متوسط مساحة العسل والحضنة وحبوب اللقاح (2.960 سم2، 1.624 سم2 و 0.473 سم2) على التوالي.

الكلمات المفتاحية: المنحل، نحل العسل، الفيتامينات، البروتينات.

INTRODUCTION

honeybees *Apis mellifera* live in colonies, it is a social insect, The colony lives cooperatively, and the queen controls all members of the colony through her pheromone secretions. and the colony consists of several hundred males and thousands of workers, as its numbers depend on the surrounding environmental conditions of temperature, relative humidity, and vegetation cover **(Al-Sayegh & Mustafa, 2003; Ramal, 2005).**

There are also different types of bees in Iraq **(Augul, 2018)**. Bees and their products have many nutritional and medical benefits, as the royal jelly of honeybees is used to increase sperm activity in artificial insemination in field animals **(Hussin, 2015).**

Pollen grains are a source of vitamins, proteins, fats and minerals necessary to build body tissues, as the colony consumes some of the flowers it collects from pollen and nectar to sustain its various vital activities, and more than it needs it stores in the hexagonal eyes to benefit from it when needed **(White, 1993; Taha, 2015).**

Pollen alternatives mean any nutrient medium provided to bees and contains a protein source alternative to pollen, pollen supplements mean the nutrient medium for bees and contains a protein source added to it (5-25%) pollen **(Standifer, 1980).**

The importance of vitamins is evident during brood breeding, as the larva grows fully in the communities that fed their adults on industrial food containing vitamins, and the absence of vitamins leads to the death of the larva on the third or fourth day of its life, as well as the workers are able to raise the brood when fed on Inositol sugar in industrial food **(Nation & Robinson, 1968; Dadd, 1973).**The bee density of the colonies increases when fed with vitamins, proteins and sugar solution in November and December, which increases the amount of sealed and open honey, pollen area, eggs and larvae, as they pass the winter in good condition and give the highest weights **(Mansor *et al*., 2021).** Pollen substitutes and brood pheromone Super boost stimulate brood growth, honey space, and build wax foundations **(Shaher & Nasrallah, 2018).**

Honeybees are exposed to many insect and disease pests, such as *Nosemia serana*, which is the most common pest and most influential on members of the colony **(Abdulhay &**

Yonius, 2020). Fructose-loving lactic acid bacteria are a mechanism of protection or vital enhancers against diseases **(Saleh, 2020)**. Lactic acid bacteria are found in the stomach of honeybees **(Khaled & Ward Shaher, 2021).**

The greater wax worm is one of the important insect pests that infect beehives, as a study was conducted in which the alcoholic extract of the leaves of the dodonia plant was used against this pest due to its availability in the Iraqi environment and ease of extraction **(Mohammed & Nawar, 2020)**. There is a type of bacteria used to protect the waxy frames of honeybee hives from infection with the greater wax worm **(Al-Jassani & Dawi, 2013)**.

The eastern hornet is one of the pests to which bees are exposed, causing their death or the migration of members of the beehive's colony **(Glaiim, 2009).** Several studies have been conducted to determine the type of varroa that infects beehives in Iraq **(Awwad & Shaher, 2023).** Environmental pollution is one of the most important factors that have an impact on honeybee populations directly and indirectly **(Ward & Manjy, 2020).**

In view of the importance of nutrition in the life of honeybee colonies, the study aimed to use the super protein food alternative and add vitamins (E and C) to the diet of the bee colonies, with the aim of increasing the efficiency of queen bees in laying eggs, worker activity and increasing production, and studying its effect on the area of honey, brood and pollen.

MATERIALS AND METHODS

Prepare honeybee colonies

The study was conducted in the province of Baghdad - Al-Buaitha in a special apiary, as (15) homogeneous cells were selected in terms of the activity of the colony (the number of frames on which the bees work) product, the study was conducted on two seasons, the autumn season for the period from 1/9/2022 - 1/12/2022 and the spring season for the period from 1/1/2023 - 1/4/2023 by three replicates (cells) for each treatment (Super Protein, vitamin C, vitamin E, control treatment and sugar solution).

Preparing bee food

Use the super protein supplement from the Lebanese company Eiffel at the rate of (1 ml/ liter of water), vitamin C and vitamin E, using (vitamin E 1 ml/ liter of water) for each treatment, (vitamin C 10 g / liter of water) for each treatment, where feeding was done twice a week for all cells, and then measurements were taken for honey area, brood area and pollen area every 14 days.

Statistical design and analysis:

The experiments were designed according to the randomized complete block design (RCBD), and the results were analyzed statistically according to the analysis of variance (ANOVA) method, Analysis Of Variance, and the significant differences between the means were compared by the Least Significant Difference (LSD) test at the level of 0.05 **(Al-Sahoki & Wahib, 1990).**

RESULTS AND DISCUSSION

Effect of treatments on honey area.

The results of Table (1) showed that feeding the colonies with super protein and vitamins had an effect on increasing the area of honey, with the treatment of vitamin C superior to the rest of the treatments with significant differences between the treatments according to the statistical analysis, as the average area of honey was 2.631 cm^2, 3.820 cm^2, 2.960 cm^2, 2.529 cm^2 and 1.997 cm^2 for the treatment of super protein, vitamin C, vitamin E, sugar solution and control respectively. The best feeding period was during the months (November and January) with an average of 3.628 cm^2 and 4.069 cm^2 respectively.

Feeding honeybee populations with protein supplements increased honey area compared to sugar solution feeding **(Mattila & Seeley, 2010; Nabors, 2000).** Use a food substitute (super protein) The results of the study showed a good improvement in the performance of members of the honeybee colonies, as a positive effect was observed in terms of honey area and brood, and the colonies produced more honey than the control treatment, so it is recommended to use it to improve their strength **(Nabors, 2000).** This is consistent with the results of the research.

It was found in a study that the addition of sugar solution when feeding honeybee colonies in a ratio of 1:1 during the spring season and a ratio of 1:2 during the winter season has an effect on the growth and activity of bees **(Abou-Shaara *et al*, 2017).** Feeding dates should also coincide with the colony's need for food to obtain positive results **(Noordyke *et al*., 2021)**.

Table (1): Effect of adding super protein and vitamin C and E on honey area.

Treatments	Honey area / cm^2				Average Treatments
	27/10/2022	10/11/2022	24/01/2023	07/02/2023	
Super protein	2.148	3.233	3.834	1.308	2.631
V.C.	3.370	4.840	5.274	1.797	3.820
V.E.	1.744	3.840	4.273	1.982	2.960
sugar solution	1.667	3.393	3.537	1.517	2.529
control	1.500	2.833	3.429	0.225	1.997
Lsd0.05	0.238**				0.119**
Average Date	2.086	3.628	4.069	1.366	
Lsd0.05	0.107**				

Effect of treatments on brood area

The results of Table (2) showed outperformed of the treatment of super protein, vitamin C and vitamin E over the two treatments of sugar solution and control when measuring the brood area, and the statistical analysis showed significant differences between the treatments, as the average brood area was 1.622 cm^2, 1.821 cm^2, 1.624 cm^2, 0.987 cm^2 and 0.766 cm^2 for the treatment of super protein, vitamin C, vitamin E, sugar solution and control respectively.

The best feeding period was recorded during the months (November and February) with an average brood area of 1.339 cm^2 and 2.042 cm^2 respectively.

When supplemental alternative food is the only food available, it leads to increased brood area and adult population **(Degrandi-Hoffman *et al*., 2008).** Feeding with pollen substitutes motivated queens to lay more eggs, and encouraged workers to raise more broods, the increase in brood breeding has positive results on the number of colonies, pollen area and honey area (**Ghazala *et al*., 2006; Nabors *et al*., 2018).**

Herbert & Shimanuki (1978) showed that feeding honey beehives with vitamin C gave the highest average brood area, as bees raised more brood. Also, adding vitamins to the diet of bees led to a significant improvement in the amount of brood and thus increased its area **(Beck & Strand, 2007)**. Eggs raised by bees fed with vitamin C are heavier than those that have not been fed, and these results are consistent with those of other research **(Herbert *et al*., 1976).**

Table (2): Effect of adding super protein and vitamin C and E on brood area.

Treatments	Brood area / cm^2				Average Treatments
	27/10/2022	10/11/2022	24/01/2023	07/02/2023	
Super protein	1.140	1.577	1.589	2.183	1.622
V.C.	1.388	1.700	1.442	2.755	1.821
V.E.	0.512	1.642	1.488	2.855	1.624
sugar solution	0.700	0.943	0.955	1.350	0.987
control	0.467	0.833	0.696	1.067	0.766
Lsd0.05	0.259**				0.130**
Average Date	0.841	1.339	1.234	2.042	
Lsd0.05	0.116**				

Effect of treatments on pollen area

The results of Table (3) and according to the statistical analysis showed that there are significant differences between the treatments, as the super protein treatment gave the least area for pollen and the average area was 0.378 cm^2. While the sugar solution treatment and control outperformed the super protein treatment, the average pollen area was 0.453 cm^2 and 0.458 cm^2 for the two treatments, respectively. The reason for this is that pollen is mainly a source of protein, so the hives need for protein is low. The statistical analysis showed that feeding the colonies with vitamin C and vitamin E outweighs the nutrition with vitamin C over all other treatments when measuring the area of pollen, The average pollen area was 0.608 cm^2, while the pollen area for vitamin E treatment was 0.473 cm^2, outperforming the treatment with sugar solution and control.

Through our results, we notice that there is a positive relationship between food consumption and brood breeding and storing honey and pollen during the winter, compared to the control treatment and this is consistent with the results mentioned by the researchers **(Nabors, 2000; Mattila & Seeley, 2010)**. The importance of protein increases only at the time of scarcity, and the consumption of the supplement decreases when natural food is available **(Nabors, 2000).**

Mansor *et al*. (2021) found that feeding bee colonies with proteins, vitamins and sugar solution in November and December outperformed the control treatment in terms of bee density, sealed and open honey area, pollen area and egg and larval area, as it passed the winter in good condition and gave the highest weights.

Table (3): Effect of adding super protein and vitamin C and E on pollen area.

Treatments	Pollen area / cm^2				Average Treatments
	27/10/2022	10/11/2022	24/01/2023	07/02/2023	
Super protein	0.733	0.478	0.000	0.300	0.378
V.C.	0.563	0.185	1.442	0.242	0.608
V.E.	0.695	0.535	0.275	0.386	0.473
sugar solution	0.417	0.715	0.295	0.383	0.453
control	0.333	0.167	0.696	0.637	0.458
0.05Lsd	0.182**				0.091**
Average Date	0.548	0.416	0.542	0.390	
0.05Lsd	0.081**				

CONCLUSIONS

It is preferable to add super protein, vitamin C and E in feeding honeybee colonies because of its importance in increasing production.

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ANTIBIOTICS SUSCEPTIBILITY PATTERN OF COMMON PATHOGENIC BACTERIAL STRAINS ISOLATED FROM PATIENTS WITH LOWER RESPIRATORY TRACT INFECTION (LRTI)

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ABSTRACT

Lower respiratory tract infections LRTIs are quickly becoming the most prevalent infectious disorders affecting people. This investigation focused on characterizing the current shift in the bacterial strains causing the infections of respiratory tract among patients and their antimicrobial sensitivity pattern since antibiotic resistance has developed in all major pathogens.

In the microbiology lab of the Department of Biology Sciences / University of Baghdad, a cross- sectional research using non-probability sequential sampling method was carried out. Respiratory sputum samples taken from individuals with lower respiratory tract infections and collected 149 bacterial isolates. *M. catarrhalis*, *S. pneumonia* and *H. influenzae* were the bacterial pathogens diagnosed by VITEK 2 system and then gathered for further investigation. Frequencies along with percentages were calculated for categorical parameters including microorganism, age and gender. Continuous data like age was presented as mean ± standard deviation SD. We used chi-square test for data analysis the value of Chi Square was deemed significant when the p value was P<0.05 . The pathogen that was identified most often was *M. catarrhalis* 60.4 percent, or 90 patients, followed by *H. influenza* 28.1 %, or 42 patients and *S. pneumonia* 11.4 %, or 17 patients. Levofloxacin had a greater sensitivity to *S. pneumoniae* n=17, 11.4%. Meropenem 42 28.2% was more effective against *H. influenza*. The *M. catarrhalis* pattern showed a 57% sensitivity to Co-amoxiclav, a 49% sensitivity to Ceftriaxone, and a 34.9% sensitivity to Erythromycin susceptibility.

***M. catarrhalis*, *H. influenzae* and *S. pneumoniae* were the most frequently isolated bacteria from LRTIs. We found that the *M. catarrhalis* strain was highly resistant to moxifloxacin 49.7% and co-trimaxazole 53.7%. Co-amoxiclav resistance in *S. pneumoniae* was 8.1%, while Moxifloxacin resistance in *H. influenzae* was 5.4%.**

Keywords: Lower Respiratory Tract Infection, Antibiotics Sensitivity, *Streptococcus pneumoniae* and *Moraxella catarrhalis*.

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نمط الحساسية للمضادات الحيوية من السلالات البكتيرية الممرضة الشائعة المعزولة من المرضى المصابين بعدوى الجهاز التنفسي السفلي(LRTI)

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الخلاصة

بتسارع مضطرد أصبحت التهابات الجهاز التنفسي السفلي LRTIs من أكثر الاضطرابات المعدية التي تصيب الناس انتشارًا. ركزت هذه الدراسة على توصيف التحول الحالي في السلالات البكتيرية المسببة لالتهابات الجهاز التنفسي بين المرضى ونمط الحساسية لمضادات الميكروبات منذ أن تطورت مقاومة المضادات الحيوية في جميع مسببات الأمراض الرئيسية.

في مختبر الأحياء الدقيقة التابع لقسم علوم الأحياء/ جامعة بغداد، تم إجراء بحث مقطعي باستخدام طريقة أخذ العينات المتسلسلة غير الاحتمالية. عينات البلغم التنفسي المأخوذة من الأفراد المصابين بعدوى الجهاز التنفسي السفلي وتم جمع 149 عزلة بكتيرية. *S. pneumoniae* ,*H. influenzae* ,*M. catarrhalis* كانت من اهم العوامل الممرضة التي تم جمعها عند الاستقصاء عنها وتشخيصها باستخدام جهاز VITEK 2. تم حساب التكرارات مع النسب المئوية لمؤشرات الفئات بما في ذلك الكائنات الحية الدقيقة والعمر والجنس. تم تقديم البيانات المستمرة مثل العمر بحساب المتوسط الحسابي للقيم مع + انحرافها المعياري SD± عن المتوسط. استخدمنا اختبار chi-square لتحليل البيانات، واعتبرت قيمة Chi Square مهمة عندما كانت قيمة P<0.05 .

اظهرت نتائج الدراسة وجود بكتريا *M.catarrhalis* بنسبة 60.4% من اصل 90 مريضا عند تشخيصها، تلتها بكتريا *H. influenzae* 28,1٪، 42 مريضاً و *S. pneumoniae* 11,4٪، بعدد 17 مريضاً. كان لدى Levofloxacin حساسية أكبر *S. pneumoniae* مريض17 ، 11,4٪ . كان42 Meropenem 28,2% أكثر فعالية ضد *H. influenzae*. أظهر نمط *M. catarrhalis*حساسية 57٪ تجاه Co-amoxiclav، وحساسية 49٪ لـ Ceftriaxone، وحساسية 34.9٪ لقابليةErythromycin .

وجدنا أن *M.catarrhalis* كانت شديدة المقاومة لـ moxifloxacin 49.7٪ وco-trimaxazole 53.7٪ . كانت مقاومة Co-amoxiclav في *S.pneumoniae* هي8,1٪، بينما كانت مقاومة Moxifloxacin في *H.influenza* 5,4٪.

الكلمات المفتاحية: عدوى الجهاز التنفسي السفلي ، حساسية المضادات الحيوية ، *S.pneumoniae* ، *M.catarrhalis*.

INTRODUCTION

Primary examples of Lower Respiratory Tract Infection (LRTIs) include acute exacerbations of preexisting pneumonia and chronic bronchitis. Geographical location, age distribution and other risk variables like hospitalization all contribute to the etiology of LRTIs. Incidences of community acquired LRTIs and nosocomial have increased, mirroring the growth of other incapacitating illnesses such as respiratory system impairment (COPD and asthma), chronic kidney disease and diabetes (**Sharma & Singh., 2012**) (**Chang *et al.*, 2009**). Community-acquired LRTIs are caused by *Streptococcus pneumoniae*, *Moraxella catarrhalis*, *Haemophilus influenzae*, and *Staphylococcus aureus*, whereas nosocomial LRTIs are caused by Gram-negative organisms (*Klebsiella* spp., *Acinetobacter* spp., *Pseudomonas* and *Escherichia coli*,). Atypical pneumonias may also be caused by other pathogens, such as *Mycoplasma pneumoniae* and *Chlamydia*. Although antibiotics are often used to treat LRTIs, they are not effective against viruses. New infections and forms of resistance to standard treatments need a shift in both the antibiotics used and the approach used to treat them. *M. catarrhalis*, *H. influenzae*, and several Gram-negative bacilli are among the LRTI pathogens that have become resistant to first-line treatment because they produce β-lactamase (**Guthrie., 2021**).

The clinical features of these infections demand the use of empirical antibiotic therapy prior to learning the etiology and susceptibility patterns of the causative bacteria (**Metersky *et al.*, 2012**). Clinical efficacy of -lactam medicines has been hampered significantly, by the widely usage of extended-spectrum - carbapenems and β-lactamases. The use of antibiotics on an impromptu basis is likely to blame for this pattern of resistance (**Aslan & Akova., 2019**) (**Paterson & Bonomo., 2005**). Emergence of multi-resistant bacteria such carbapenemase, *Klebsiella pneumoniae* and *H. influenzae* β--lactamase further complicates the issue (**Aslan & Akova., 2019**). To prevent the spread of antimicrobial resistance and cut down on overall treatment expenses, it is important to choose the antimicrobial therapy for bacterial LRTIs based on an understanding of their etiology and the antimicrobial susceptibility pattern. The purpose of this research was to determine the frequency of common LRTI-causing microbes are and how they respond to different classes of antibiotics (**Llor & Bjerrum., 2014**). The rise of multidrug-resistant pneumococcal bacteria and resistance to penicillin has become a worldwide health crisis. Antibiotic-resistant pneumococci have been on the rise since the late 1980s and are now considered a pandemic threat. Pneumococci that are resistant to the antibiotic penicillin are widely distributed different regions of the world (**Appelbaum.,1987**). Resistance to penicillin in the United States rose from 5 percent to 6.6 percent between 1991 and 1992, with only 1.3% of isolates showing Minimum inhibitory concentrations (MICs) of ≥2.0 ug/ml (**Appelbaum., 1992**). Widespread and intensive monitoring is required everywhere, even in countries where resistance is relatively rare, since resistant pneumococci may move from country to country (**Dowson *et al.*, 1994**).

Pneumococcal infections are often treated using data from the past and research conducted on completely susceptible strains of the bacteria. However, significant consequences for therapy are at stake due to the rise of penicillin resistance and resistance to other antimicrobials. Serious systemic infections such as meningitis caused by pneumococcal strains with decreased penicillin sensitivity are commonly treated with ceftriaxone and cefotaxime alone or in combination with vancomycin (**Dowson *et al.*,1994**). Critically sick patients in underdeveloped nations are particularly vulnerable to (RTIs), which are spread via the air or by personal contact and are considered one of the world's most serious infectious diseases (**Mannur *et al.*, 2015**). Coughing, dyspnea, wheezing, expectoration, and/or chest discomfort often lasting 1–3 weeks are all signs of a lower respiratory tract infection (LRTI). Acute form of LRTIs may nor may involve lungs, including but not limited to: bronchiolitis, acute bronchitis, community-acquired pneumonia (CAP) either with radiological examinations or without radiological evidence and acute exacerbation of chronic obstructive pulmonary disease (**Mannur *et al.*, 2015**). In both first- world and third-world countries, LRTIs are a leading cause of death and disability. After ischemic heart disease and cerebrovascular illness, it is the third greatest cause of mortality worldwide (**Regasa, 2014**).

Several factors, such as characteristics of the high-risk population, inappropriate antibiotic therapy, immunosuppressive drugs, quality of the available health care facilities, distribution of causative agents as well as prevalence of antimicrobial resistance (**Paterson & Bonomo., 2005**), can affect the incidence and associated mortality due to LRTI. One of the current public health challenges of the 21st century is antimicrobial resistance, which makes it harder to treat and prevent a wide variety of bacterial infections. Over the course of many decades, antibiotic usage has increased drastically, and the extensive use of these medications has greatly aided the emergence of antibiotic-resistant bacteria (**Okesola & Ige., 2008**). The aims of the study were to assess the frequency of different pathogen strains of lower respiratory tract infections and to determine the frequency of Moraxella catarrhalis in the pathogenicity of lower respiratory tract infections.

MATERIALS AND METHODS

Laboratory culture data from tertiary care institutions in Baghdad, Iraq, was used in a descriptive cross-sectional analysis to estimate antibiotic susceptibility patterns in lower respiratory infection. Data from the labs of 79 male and 70 female patients diagnosed with LRTIs at tertiary care facilities in Baghdad, Iraq, were analyzed. The 6-months' time frame, from September 2021 to February 2022, was used for retrieval of information, classification of data according to research inclusion and exclusion criteria, and documentation. All medical records of patients who were potentially eligible and who had been diagnosed with a lower respiratory tract infection (LRTI) were reviewed in order to obtain laboratory results.

The culture sensitivity reports were included using a sampling approach that was convenient at the time and the laboratory data were accessed. The demographic information gathered included age, gender, and antibiotic susceptibility patterns. Gram's staining was utilized to divide bacteria into gram-negative and gram-positive strains (**Rodloff *et al*., 2008**) .

Bacterial isolates were cultured from sputum taken from individuals with LRTI. Patients between the ages of 32 and 82 years were isolated after 48 h. Ventilator-associated pneumonia and other cases of healthcare-associated pneumonia were disqualified. Bacterial identification and antibiotic susceptibility testing were conducted according to protocols developed by the Clinical Laboratory Standard Institute (CLSI). Exclusion requirements were strappingly satisfied. The institution's ethics board provided written approval. Patients' informed consent was also collected.

Information such as patient age, gender, specimen collection location, and visit type was recorded. Sheep blood agar (SBA), Mac Conkey agar and chocolate agar were used to inoculate the samples. These media plates underwent normal process streaking using a sterile wire loop. Colonies formed on the culture plates after being cultured at 35^{o}C in ambient atmosphere" for 24 h. Standard methods were used to identify the organisms produced and assess their antibiotic susceptibility depending on Clinical Laboratory Standard Institute (CLSI) (**Shen &Sergi., 2023**) (**Weinstein & Lewis., 2020**).

Colonies of *H. influenzae* were identified as "Gram negative moist, rods, tiny, smooth gray colonies with absence of hemolysis, positive oxidase and catalase test. The results of oxidase test may vary in some conditions.

Satellite growth surrounding streaks of *Staphylococcus aureus* in the presence of growth factors V and X may provide a false-negative result in an oxidase test. "Gram negative cocci on gram staining, colony morphology, oxidase test, hockey puck sign, catalase test, and butyrate esterase production, and their inability to ferment sugars" are diagnostic of *M.catarrhalis*. The presence of "tiny, round, flat, and transparent colonies, with central depression (checker piece and nail head colonies), hemolysis, catalase negative and oxidase negative, absence of bile-esculin hydrolysis, lysis by bile-salts, susceptibility to optochin, and other biochemical characters" are all characteristics used to identify *S. pneumoniae*. Whereas the diagnosis of all bacterial isolates mentioned above was confirmed by using the VITEK 2 system.

Isolated bacteria and yeast were put through Kirby-Bauer disc diffusion tests for resistance on Mueller Hinton agar plates. After 24 h in the incubator, the isolated colonies were suspended in 0.5 McFarland turbidity normal saline. Mueller Hinton agar plates were prepared by streaking them with sterile swabs. The MHA plates had antimicrobial discs of varying intensities. Oxoid (UK) and Bioanalyse (Turkey) supplied the antimicrobial discs used in this study. Antimicrobials including "ampicillin (10 g), co-amoxiclav (amoxicillin/clavulanic acid 20/10 g), meropenem (10 g), levofloxacin (5 g), co-trimoxaz- ole (trimethoprim/ sulfamethoxazole 1, ceftriaxone (30 g), cefadroxil (30 g), ciprofloxacin (5 g). After inverting the plates, they were kept for 16-18 h in 37° C incubator. The sizes of the inhibitory zones were recorded after incubation.

Table (1): CLSI antibiotic breakpoints for bacterial strains susceptibility

Antibiotic concentrations in vitro	Bacterial strains	Sensitive (S)	Intermediate (I)	Resistant (R)
ampicillin (10g)	*M.catarrhalis*	-	-	–
	S.pneumoniae	–	-	–
	H.influenzae	≤ 1	2	≥ 4
co-amoxiclav (amoxicillin/clavulanicacid20/10g)	*M.catarrhalis*	≤4	-	>1
	S.pneumoniae	≤2	4	≥8
	H.influenzae	≤ 4/2	–	≥ 8/4
levofloxacin (5g)	*M.catarrhalis*	–	-	-
	S.pneumoniae	≤ 2	4	≥ 8
	H.influenzae	≤ 2	-	-
meropenem (10g)	*M.catarrhalis*	–	-	>2

	S.pneumoniae	≤ 0.25	0.5	≥ 1
	H.influenzae	≤ 0.5	–	–
co-trimoxaz- ole (trimethoprim/ sulfamethoxazole 1, ceftriaxone (30g)	*M.catarrhalis*	≥13	11–12	<15
	S.pneumoniae	≤ 0.5/9.5	1/19–2/38	≥ 4/76
	H.influenzae	≤ 0.5/9.5	1/19–2/38	≥ 4/76
cefadroxil (30g)	*M.catarrhalis*	-	-	-
	S.pneumoniae	–	-	-
	H.influenzae	≤ 0.5	–	–
ciprofloxacin (5g)	*M.catarrhalis*	≤ 1	-	>0.5
	S.pneumoniae	–	-	-
	H.influenzae	≤ 1	-	-

STATISTICAL ANALYSIS

The data of this research study was analysed using SPSS 21 (Statistical Package for the Social Sciences). Frequencies and percentages were computed for factors including microbial strain, gender, and antibiotic resistance. Age, and other quantitative characteristics were analysed using mean and standard deviation. A chi-square test was performed, and a significance level of p value $P<0.05$ was set.

RESULTS AND DISCUSSION

The demographic information of the individuals who took part in the research is shown in table 2. There were a total of 79 males, comprising 53% of the group, and 10 females, comprising 47%. With the average age of 52.99 ± 12.35 years. The total number of smokers in the male population were 53 (67.1%), whereas only 5 (7.1%) of the women in the study population smoked cigarettes. It was reported that only 10 males (12.7%) were alcoholics. There were 13 (16.5%) males and 8 (11.4%) females who had a history of TB. The number of males was higher than the number of females , as shown in the table (2).

Table (2): Demographic Characteristics of Study Participants

Gender		Frequency	Percent
Male		79	53.0
Female		70	47.0
Smoking			
Male	**Non-Smoker**	26	32.9
	Smoker	53	67.1
Female	**Non-Smoker**	65	92.9
	Smoker	5	7.1
	Alcohol Consumption		
Male	**No**	69	87.3
	Yes	10	12.7
Female	**Yes**	0	0
	No	70	100.0
	Tuberculosis History		
Male	**No**	66	83.5
	Yes	13	16.5
Female	**No**	62	88.6
	Yes	8	11.4

Our results agree with the fact that infectious illnesses affecting the lower respiratory system (LRTIs) are now among the most prevalent worldwide. Since the causative bacteria for LRTIs vary by region, the susceptibility profile will also change depending on geographical location (**Safiri *et al.*, 2023**). Therefore, our cross-sectional study confirmed that it is crucial for the selection of suitable therapy to have updated information on the microorganisms responsible for LRTIs and their sensitivity profile.

Antimicrobial resistance patterns among microorganisms isolated from patients were investigated in this study. The most often identified organism was *M. catarrhalis* (60.4 percent, or 90 patients), followed by *H. influenzae* (28.1 percent, or 42 patients), and *S. pneumoniae* (11.4 percent, or 17 patients). The most prevalent organism found to cause community-acquired lower respiratory tract infections was *M. catarrhalis*, with a frequency of 60.4% (90). The antibiotic resistance patterns shown by *H. influenzae*, *S. pneumoniae*, and *M. catarrhalis* are described in (figure 1).

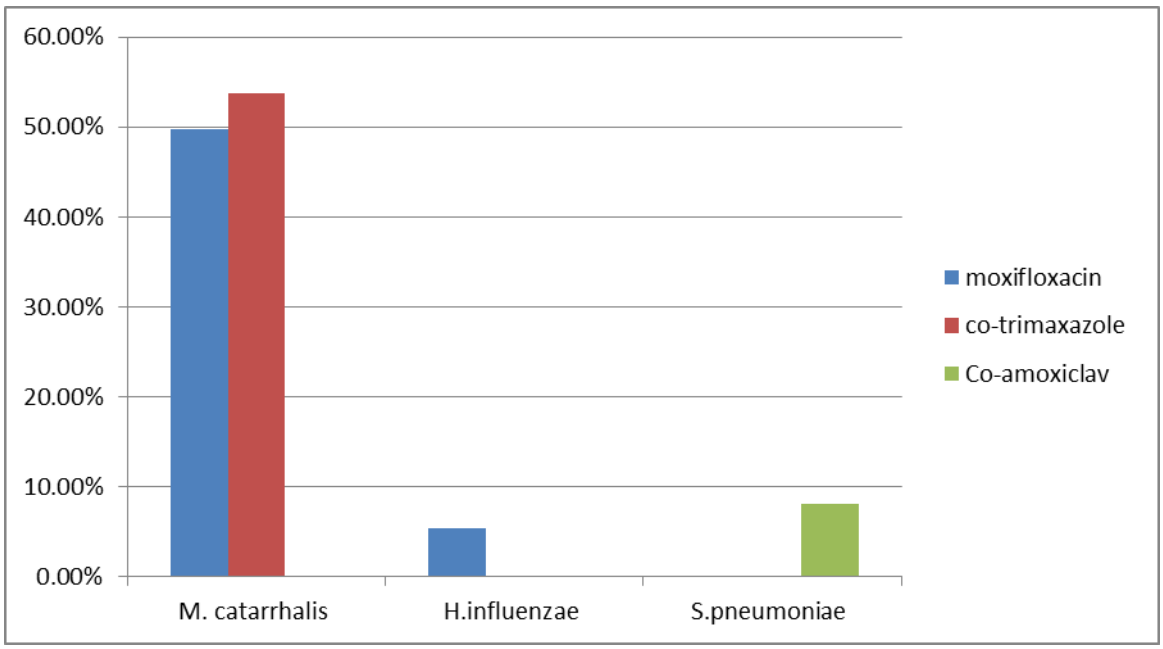

Figure (1): Antibiotic resistance patterns by the most common pathogenic strains in lower respiratory tract infections.

Both moxifloxacin (49.7% resistance) and co-trimaxazole (53.7% resistance) had higher resistance on the *M. catarrhalis* strain that we tested. The percentage of *S. pneumoniae* resistant to co-amoxiclav was 8.1%, whereas the percentage of *H.influenzae* resistant to moxifloxacin was 5.4%.

It was shown that the male and female groups that were affected by LRTI had distinct differences in the frequency pattern of pathogen strains. *M. catarrhalis* was found at a higher frequency in both groups, reaching a maximum of 50 and 40 frequencies in males and females respectively. On the other hand, the frequency of *H. influenza* was 16 in the male population and 26 in the female population correspondingly. In comparison to the other two strains, *S. pneumoniae* was the one that appeared less often. We identified a statistically significant difference between the male and female groups when we used the chi-square test to compare the frequency of these pathogen strains. The p-value for this finding was 0.021 as shown in table 3.

Table (3): Comparison of LRTI Pathogen Strains in Male and Female Participants

	Pathogen Strain			Total	p-value
	Moraxella catarrhalis	Haemophilus influenza	Streptococcus pneumoniae		
Male	50	16	13	79	0.021
Female	40	26	4	70	
	90	42	17	149	

After classifying the ages of the people who participated in our research into the various age groups, we analyzed the variance in the frequency pattern of the three types of strains that were seen among our study population. It was shown that individuals between the ages of 46 and 60 had a significantly greater incidence of both *M. catarrhalis* (n=55) and *H. influenzae* (n=23). *S .pneumoniae* was shown to be more prevalent in individuals between the ages of 60 and 80. When we compared the prevalence of LRTI pathogens using the chi-square test, we discovered that there was a significant difference between them. The p-value that was computed was P<0.01 as shown in the table (4).

Table (4): Comparison of Age Distribution and Frequency of LRTI Pathogen Strains in Recruited Samples

Age Category (Years)	Pathogen Strain			Total	p-value
	Moraxella catarrhalis	Haemophilus influenzae	Streptococcus pneumoniae		
30-40	4	19	0	23	<0.01
40-60	55	23	6	84	
60-80	29	0	11	40	
>80	2	0	0	2	

As per our study findings, patients with LRTIs were reportedly had *M. catarrhalis* isolated from their respiratory tracts. Nonetheless, *S. aureus* was shown to be the most common bacterium in Pakistan, with *S. pneumoniae* coming in second (**Khawaja *et al*. ,2013**). Another research done in Pakistan found that *H. influenza* and *S. pneumoniae* were the leading causes of LRTI, whereas *M. catarrhalis* categorized in sixth position. *M. catarrhalis* has emerged as a pathogen in the past 20-30 years and is currently thought to be a root cause of upper respiratory tract infections in adults .and even in healthy children (**Abdullah *et al.*, 2013**).

All of the *S. pneumoniae* strains that were isolated in our study exhibited variable susceptibility to vancomycin and it was confirmed by multiple studies which reported similar

findings regarding the inconsistent sensitivity of *S. pneumoniae* strains to vancomycin. Multiple studies from different parts of the world confirmed similar findings (**Rao *et al.*, 2013**) (**Knobbe *et al.*, 2020**). Resistance to penicillin is especially major concern in *S. pneumoniae*. In one study, researchers found out that only 1% of *S. pneumoniae* were resistant to penicillin (**Knobbe *et al.*, 2020**). Antimicrobial susceptibility pattern of S. pneumonia isolated from CAP patients were assessed in a Greek study from the early 1990s. While only 14% of participants in that study showed resistance to penicillin (**Maraki & Papadakis., 2014**). Nevertheless, our study findings reported 47% (12) resistance to penicillin.

S. pneumoniae drug resistance profiles differ greatly across nations. Ceftriaxone resistance in *S. pneumoniae* ranged from 0% in Greece (**Maraki & Papadakis.,2014**) whereas it was reportedly 30% in south India in a study conducted in 2013 (**Rao *et al.*, 2013**). We found widespread resistance to Co-amoxiclav (8.1%), ampicillin (6.7%, n=10) and ceftriaxone (6.7%, n=10), whereas only low levels of resistance to these drugs were reported in multiple studies. Based on the results of the current investigation, it is believed that *H. influenzae* is surprisingly sensitive to a wide variety of antibiotics. Meropenem (28.2%), ampicillin (27.5%), ceftriaxone (27.5%), cefixime (26.8%), moxifloxacin (24.2%) and co-trimaxazole showed (20.1%), higher sensitivities against *H. influenzae*. The results are consistent with those of previous research done in various regions of the world (**Maraki & Papadakis., 2014**) (**Santella *et al.*, 2021**). However, a study done in Kuala Lumpur found that cotrimoxazole resistance was present in 26% of the cases. When comparing our findings to those from Ethiopia, where sixty-six percent of the isolates were reportedly resistant to cotrimoxazole antibiotic, but then dramatic decline in cotrimoxazole resistance among *H. influenzae*. Resistance to fluoroquinolones in *H. influenzae* has been discovered less frequently in South Asian countries (**Ansarie & Kasmani.,2015**). However, we have found that 24.2 percent of *H. influenzae* isolates are resistant to the fluoroquinolone drug i.e. moxifloxacin.

In recent years, *M. catarrhalis* has become increasingly recognized as a major pathogen responsible for LRTI. 90% of *M. catarrhalis* isolates are ampicillin-resistant because they produce β-beta lactamase enzyme. *M. catarrhalis* strain was highly resistant to Moxifloxacin (49.7%) and co-trimaxazole (53.7%). Co-amoxiclav resistance in *S. pneumoniae* was 8.1%, while Moxifloxacin resistance in *H. influenzae* was 5.4%.

As a result, amoxicillin-clavulanate and other beta-lactam/beta-lactamase inhibitor combinations have been considered as first-line therapy for *M. catarrhalis* infections (**Bandet *et al.*, 2014**). Similar to previous investigations (**Tamang *et al.*, 2005**), all *M.catarrhalis* isolates showed a 100% sensitivity to amoxicillin/clavulanate and ceftriaxone. Ramana and colleagues 30 found the same degree of sensitivity to amoxicillin/clavulanate. The current study found that moxifloxacin resistance was highest (49.7%), followed by co-trimaxazole (53.7%).

Over the last two to three decades, *M. catarrhalis* has become an influential pathogen. In fact, this seems to be a rise in the prevalence of strains that produce β-lactamase, which might have major implications for the treatment of infections. The British and American Thoracic Societies recommend amoxicillin or a macrolide as the first line of antibiotic therapy for outpatients. Respiratory fluoroquinolones or β-lactam with macrolide should be used to treat individuals who do not require critical care (**Knobbe *et al.*, 2020**).

Patients with critical health conditions should take clarithromycin or another respiratory fluoroquinolone in addition to a β-lactam (**Knobbe *et al.*, 2020**). The local resistance pattern

should also be documented, as this data might help in guiding the choice of a suitable first course of antibiotic treatment. Continuous surveillance at both the local and national levels is still necessary to detect any ancillary changes in the frequency of pathogens and to observe drift in their sensitivity pattern, despite the fact that antibiotic susceptibility pattern and prevalence of these bacteria vary from nations to nations. This information could aid medical professionals in making informed decisions about antimicrobial therapy for the treatment of LRTI. In addition, it has the potential to reduce antimicrobial resistance in both natural environments and clinical settings.

Both the Council for Appropriate and Rational Antibiotic Therapy and the World Health Organization criteria stress the importance of selecting the most effective drug but within shorter period of time. Less drug exposure, fewer side effects, more time saved, better compliance, and lower healthcare costs are all possible advantages of a shorter course with higher dose therapy (**Spellberg & Rice., 2019**).

CONCLUSION

The trend of susceptibility of lower respiratory tract pathogenic strains shows that *M.catarrhalis* is the most common bacterial agent of LRTIs, followed by *H.influenzae* and *S.pneumoniae*. The most often isolated bacteria from lower respiratory tract infections were *M.catarrhalis*, *H. influenzae*, and *S.pneumoniae*. Both moxifloxacin and co-trimaxazole had higher resistance on the *M. catarrhalis* strain that we tested. The percentage of *S.pneumoniae* resistant to co-amoxiclav was higher than the percentage of *H. influenzae* resistant to moxifloxacin. Therefore, careful administration of antimicrobial medicines will lessen the burden of antibiotic resistance, allowing for appropriate patient management and reducing the morbidity as well as mortality caused by LRTIs.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

FUNCTIONAL PROPERTIES OF ISOLATED AND HYDROLYZED PROTEIN POWDER OF MORINGA LEAVES (*MORINGA OLEIFERA* LAM.)

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ABSTRACT

The study aimed to estimate the functional properties of *Moringa oleifera* Lam. leaves powder protein isolate and its hydrolysates using pepsin. Water Holding Capacity (WHC) and Oil Holding Capacity (OHC) of protein isolate were 3.02 mL /g protein and 2.62 mL /g protein respectively, superior to the oil reducing capacity of the pepsin enzyme hydrolysates. at times 30 and 60 min. They were characterized by high emulsifying activity, reaching 48.08 m^2/g and 53.06 m^2/g, respectively, and outperforming the protein isolate, which reached 41.45 m^2/g. The least emulsifying activity was for pepsin after 120 min of hydrolysis (A_4) and reached 31.50 m^2/g, and the highest emulsion stability was 63.77% for protein hydrolysates using pepsin enzyme at (37 °C) after 60 min of hydrolysis (A_2), while the least emulsifying stability was for protein hydrolysates using pepsin enzyme pepsin after 120 min (A_4) it amounted to 25.10%. The foaming ability of protein hydrolysates using pepsin after 60 min (A_2) was 41.17%, foaming stability at the beginning of hydrolysates was 39.02%, then it gradually decreased with the passage of time.

Keywords: Enzyme hydrolysates, Functional properties, Moringa protein isolate, Pepsin,.

الخصائص الوظيفية للمعزول والمتحلل البروتيني لمسحوق اوراق (*Moringa oleifera* Lam.)

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الخلاصة

هدفت الدراسة الى تقدير الخصائص الوظيفية للمعزول البروتيني لمسحوق اوراق نبات البان (المورينجا) *Moringa oleifera* Lam. ومتحللاته باستعمال انزيم الببسين, اذ بلغت قدرة الاحتفاظ بالماء والاحتفاظ بالزيت للمعزول البروتيني 3.02 مل/غم و 2.62 مل /غم على التوالي متفوقة على قدرة الاحتفاظ بالزيت لمتحللات انزيم الببسين, وتميزت المتحللات البروتينية لانزيم الببسين في الاوقات 30 و 60 دقيقة بنشاط استحلابي مرتفع وبلغت 48.08 غم/$م^2$ و 53.06 غم/$م^2$على التوالي وتفوقت على المعزول البروتيني الذي بلغ 41.45 غم/$م^2$. اما اقل نشاط استحلابي كان لمتحلل الببسين بعد 120 دقيقة من التحلل (A_4) وبلغ 31.50 غم/$م^2$. وبلغت اعلى ثباتية للمستحلب63.77% للمتحلل البروتيني باستعمال انزيم الببسين على درجة حرارة (37) مْ بعد 60 دقيقة من التحلل (A_2), اما اقل ثباتية للاستحلاب كانت لمتحلل الببسين بعد 120 دقيقة (A_4) وكانت 25.10%, وكانت قابلية تكوين الرغوة للمتحلل البروتيني بوساطة انزيم الببسين بعد 60 دقيقة من التحلل (A_2) وكانت 41.17%، وكانت ثباتية الرغوة في بداية التحلل 39.02%، بعدها انخفضت

* **The research is extracted from a doctoral thesis of the first researcher.**

تدريجيا مع زيادة فترة التحلل, وبينت الدراسة اهمية استعمال المعزول البروتيني لمسحوق اوراق المورينجا وبعض متحللاته كعامل استحلاب وعامل رغوة في تحسين جودة المنتجات الغذائية.

الكلمات المفتاحية: متحللات انزيمية، الخواص الوظيفية، معزول بروتين المورينجا، البيبسين.

INTRODUCTION

Moringa leaves have a multipurpose which used as natural medicine, food, feed, natural stimulants for fertilizers, forage and migration of bees **(Al-Taweel & Al-Anbari, 2019)**. Moringa leaves contain all of the essential amino acids in a good proportion, which are the building blocks of proteins **(Al-jubouri *et al*., 2022; Mishra *et al*., 2012)**. Proteins are considered important nutrients for the development of the human body and maintaining its health, as a person needs a sufficient amount of protein to maintain the vital functions of the body, growth, maturity, pregnancy, breastfeeding, and recovery from diseases **(Aziz, 2023; Abdul Rahman *et al*., 2023; Hamdia & Ahamed, 2023)**. Moringa oleifera is a type of fast-growing perennial plant native to India, where it is currently grown in many regions of the world and is considered one of the most useful plants in the world because almost all of its parts can be used as food and in traditional medicines **(Chalob & Abdul-Rahman, 2018; Alwan & Jawad, 2015)** as all parts of Moringa have long been used to treat diseases it is also used in water purification and in the manufacture of supplementary food for children to increase its protein content, as the use of vegetable proteins is a good source of amino acids because of its good functional properties such as solubility, emulsification, foaming, and oil-water binding **(Nasser & Hammood, 2019; Gorissen *et al*., 2018 ; Karim & Shaker, 2016).** The use of vegetable proteins is a good source of amino acids, as it possesses good properties of Moringa protein isolate, which indicates its ability to work as functional components in diets, as Moringa leaf protein isolate can be incorporated into diets for the manufacture of functional foods and the treatment of malnutrition **(Famuwagun *et al*., 2020 ; Khalaf, 2014).** The study aimed to estimate the functional properties of Moringa oleifera leaves powder protein isolate and its hydrolysates using pepsin.

MATERIALS AND METHODS

Preparing research samples

Moringa leaves under study were obtained from the University of Baghdad / College of Agricultural Engineering Sciences/ Medicinal Plants Unit for the season (2021-2022).

Preparing the forms for the study

Moringa leaves (*Moringa oleifera* Lam.) were cleaned and isolated, then dried in an electric vacuum oven at a temperature of 50°C, then ground and sifted with an 80 mash sieve, and kept in polyethylene bags at a refrigerator temperature of 4 ˚C until use.

Preparing of defatted leaf powder

The defatted leaf powder was prepared according to **(Fontanari *et al*., 2012)** by mixing the powder with hexane in a ratio of 1/5 (w/v), then placed on the magnetic stirrer for 3 h, after that the filtrate was separated and then dried at room temperature 25 ˚C for 24 h, grinding the powder and keeping it by freezing at -18 ˚C until use.

Preparing of the protein isolate

The protein isolate of the defatted Moringa leaf powder was prepared according to **(Estela, 2014)** where the defatted leaf powder was mixed with water in a ratio of 1:20 (w/v) with stirring for 2 h, after that the pH was adjusted to 9 and mixed for 2 h, followed by a refrigerated centrifugation process At a speed of 10,000 rpm for 30 min, then the leachate was separated from the precipitate, then the leachate was taken and the pH was adjusted to 4 then a refrigerated centrifugation process was carried out at a speed of 10,000 rpm for 30 min, then the precipitate was separated from the leachate, the precipitate was taken, washed with distilled water several times, and then dissolved the precipitate in a small amount of distilled water, then the pH was adjusted to 7, then the sample was dried and stored by freezing at -18°C until use.

Preparation of protein isolate hydrolysates

Enzymatic hydrolysis by pepsin

The reaction mixture was prepared according to the method of **(Popovic *et al*., 2013)** by mixing 5 g of protein isolate with 100 mL of Glycin-HCl buffer solution (0.1 M) at pH 3, then the enzyme pepsin was added at a concentration of 1%, and the mixture was placed in a shaking incubator At a speed of 200 rpm min at 37 °C, samples were drawn at different times after (30, 60, 90 and 120) min and their code (A_1, A_2, A_3, A_4), respectively. After the expiration of each time, the reaction was stopped by boiling at 100 °C for 5 min, then the samples were centrifuged at a speed of 14500 rpm/ for 5 min, then the clear liquid was separated from the precipitate, the clear liquid was taken and kept in refrigeration until use.

Study of the functional properties of protein isolate and its hydrolysates

Water Holding Capacity

Following a method previosnly described **(Mao & Hua ,2012)** was adopted, where 1 g of the sample was taken and placed in a 15 mL test tube, then 10 mL of distilled water was gradually added to it with stirring by means of the electric mixer and left for 30 min at room temperature, then a procedure was carried out. Centrifugation at a speed of 2000 rpm for 20 min, the filtrate was removed, the tube was weighed with the sample, and the percentage of bound water was calculated as follows.

$$\mathbf{WHC} = \frac{W_2 - W_1}{W_0}$$

Where W_0 = dry weight of the sample. W_1 = tube weight + dry sample before adding water.
W_2 = tube weight + weight of sediment after adding water.

Oil Holding Capacity

The method of **(Mao & Hua ,2012)** was adopted, where 1 g of the sample was taken in a 15 mL tube, the sample was mixed with 10 mL of sunflower oil, and the mixture was left at room temperature 25°C for 30 min, then a cooled centrifugation was carried out at 5000 rpm for 30 min at 25°C, after which the filtrate was carefully removed, then the tube was weighed with the sample, and the fat absorption capacity of the samples was estimated according to the following equation.

$$\mathbf{OHC} = \frac{F_2 - F_1}{F_0}$$

Where F_0 = sample dry weight. F_1 = tube weight + dry sample weight before adding oil.
F_2 = tube weight + sediment weight after adding oil

Emulsifying Activity and Stability Index (EAI , ESI)

The emulsifying properties of all samples were estimated according to the method of **(Popovic *et al*., 2013)** where 90 µL of the sample were mixed with 90 mL of sodium phosphate buffer (M 0.01) at pH 7 Then 30 mL of commercial sunflower oil was added to the mixture and mixed using an electromixer at a speed of 6500 rpm for 5 min, then 50 µL were taken from the bottom of the mixture at a time (0 and 10) min and placed in test tubes, then diluted with 5 mL of sodium dodecyl sulfate solution 0.1% prepared by dissolving it in sodium phosphate buffer (M 0.01) At a pH of 7, then mixed with a slight stirring of the shift, and then read the absorbance of the diluted solution at a wavelength of 500 nm in time (0, 10) min, then estimated the capacity and stability of the emulsion based on the following equation

EAI (m^2/g) = (2.303 ×2×100×A0)/ (C×0.25×10.000)

Where EAI(m^2/g) = emulsifying activity, A = absorbance at 500 nm, C = protein concentration (g/mL) 0.001.

ESI = $A_0 \times t/(A_0 - A_{10})$

Where ESI = stability of the emulsion. A_0 = absorbance at time zero.

t = time after naturalization. A_{10} = absorbance after 10 min.

Foaming Capacity and Stability (FC, FS)

The volume and foam constant of the samples were estimated according to the method of **(Popovic *et al*., 2013)** by whipping 0.5 g of the sample with 50 mL sodium phosphate buffer 0.01M at pH 7 by means of a homogenizer at a speed of 5000 rpm for 2 min, then transferring the mixture to A graduated cylinder and record the volume of foam before and after whipping, then the samples were left after conducting a foam capacity check for (1, 10, 30, 60 and 90) min, then the foam stability was estimated at all times, then the foam capacity was estimated according to the following equation:

$$\textbf{FC} = \frac{\textbf{Foam volume}}{\textbf{total volume}} \times 100$$

Statistical Analysis

The Statistical Analysis System (GenStat 12th Edition) was used to analyze the data to study the effect of different coefficients on the studied traits according to a complete random design (CRD), and the significant differences between the means were compared with the Least Significant Difference-LSD test **(Al-Rawi & Khalaf Allah, 2000)**.

RESULTS AND DISCUSSION

Functional properties

Water holding Capacity (WHC)

The results in (table 1) show ability of the protein isolate and the protein hydrolysates of the leaf powder to bind water. It was found that the highest ability to bind water was for the protein isolate (PI), reaching 3.02 mL/g, and the lowest ability to bind water was for the hydrolysate after 90 min which was1.48 mL/g. and the difference was significant with the rest of the treatments at the level ($p \leq 0.05$). The reason for this can be attributed to the high percentage of protein in the isolate, which was followed by an increase in water binding, as the ability of the protein to bind water is due to its ability to form hydrogen bonds between water molecules and polar groups of peptide chains in the protein, and this leads to an increase in the

ability of the protein to integrate with water due to its content of Hydrophilic, polar amino acids that form hydrogen bonds with water.

The results show that the water absorption capacity of the hydrolysates is low compared to the protein isolate, and this may be explained by the great ability of the protein isolate to open up and swell the peptide chain and then new sites for water binding may be due to the low concentration of polar amino acids of the hydrolysates which led to a decrease in susceptibility Carrying water. Where it was found **(Jasim & AI-Obaidi, 2022 ; Rawdkuen, 2020)** that the ability of protein to absorb water amounted to 2.31 mL/g which is among the results obtained in the study and the ability of protein to absorb water is an important characteristic in viscous foods such as soup broth and baked products, especially when its value ranges from 1.49 to 4.72 mL/g **(Nasser *et al*., 2019 ; Kareem & Shakir, 2016)**.

Table (1): The water and fat holding capacity of the leaf powder protein isolate and its hydrolysates

Samples			Water holding capacity mL/g	Oil holding capacity mL/g
T_1			3.02	2.62
T_2	30 min	A_1	2.28	1.93
	60 min	A_2	2.81	2.30
	90 min	A_3	1.48	1.41
	120 min	A_4	1.65	0.82
LSD			0.1478	0.3308

The results represent an average of three replicates T_1: represents the protein isolate. T_2: represents pepsin hydrolysis at different times. LSD value at a significant level ($p \leq 0.05$).
These results are higher than those obtained by **(Devisetti *et al*., 2016)** for Moringa seed flour and the reason may be due to the different variety as well as the different working conditions for the production of protein isolate, such as drying conditions for example as these conditions are important and have an impact on the result. As indicated by **(Stone *et al*., 2015 ; Rahman, 2018)** the water absorption capacity of the Moringa protein isolate was twice the water absorption capacity of the pea seed isolate where these differences are related to the nature and type of proteins, amino acid composition, protein formation, surface polarity in addition to the number and type of polar aggregates.

Oil holding capacity (OHC)

We note from (Table 1) that the ability of the protein isolate and the protein hydrolysates to bind the fat was good. It was noted that the highest ability to bind the fat was for the protein isolate and amounted to 2.62 mL /g, while it was the least capable of binding the fat to the enzymatic hydrolysate by the action of the enzyme pepsin for 120 min (A_4). It amounted to 0.82 mL /g, and the difference was significant with the rest of the treatments at the level ($p \leq 0.05$). As the lipid binding process is due to the non-polar side chains of the protein, which are associated with the hydrocarbon chains, and thus work to bind the lipid, as the attachment to the lipid is attributed to the presence of hydrophobic groups, and this in turn helps to form hydrophobic bonds with the lipid and increases the amount of bound lipid **(Al-Taweel *et al*., 2022 ; Jasim & Nasser, 2020 ; Jain *et al*., 2019).**

The ability of the Moringa leaves protein isolate to retain oil was 1.94 mL / g protein which is 30% higher than the Moringa seed protein isolate which was 1.9 mL /g and these

results are somewhat similar to the results obtained in this study **(Jain *et al.*, 2019 ; Chalob & Abdul-Rahman, 2018)**. As **(Kandasamy *et al.*, 2012)** indicated that the fat absorption capacity of the protein isolate of Moringa species was between 1.08- 1.34 mL /g of protein and this is less than the results obtained in this study, and the reason may be due to the variety of Moringa used in the production of the isolate. The researcher found that Moringa protein isolate has a good oil absorption capacity, as the fat absorption capacity is an important functional property that increases or improves the flavor and taste retention of different food products as the high oil absorption rate of Moringa protein isolate makes it a good ingredient in the manufacture of chilled meat especially for sausages where the protein usually by preventing the loss of fat and water to get good products.

Emulsificaation Properties

As show in (table 2) emulsifying activity and stability of the emulsion of the protein isolate and the protein hydrolysates of Moringa, where it was noted that the highest emulsifying activity was for the enzyme degraded pepsin for 60 min of hydrolysis (A_2) and it was 53.06 m^2 /g and the lowest emulsifying activity was for the pepsin hydrolyzate for 120 min (A_4) Where it was 31.50 m^2/ g and the difference was significant with the rest of the treatments at the level ($p \leq 0.05$). While the highest stability of the emulsion was for pepsin hydrolysis after 60 min of hydrolysis (A_2) and was 63.77%, while the least stability was for pepsin hydrolysis after 120 min (A_4) and it was 25.10%. Through the results, Through the results, we notice a decrease in the emulsifying activity of the protein isolate compared to the rest of the samples, and the reason for this is attributed to the high concentration of protein, which impedes the process of migration and diffusion of protein around the surface of water and oil, unlike the low concentrations of protein, which leads to the speed of its spread easily around the surface of water and oil and then leads to an increase Emulsification capacity **(Tawfeeq & Ahmaed, 2023 ; Abadi & Naser, 2019)**.

Table (2): Emulsifying activity and stability of Moringa protein isolate and its hydrolysates

Samples			Emulsion activity (m^2/g)	Emulsion stability (%)
T_1			41.45	34.52
T_2	30 min	A_1	48.08	56.73
	60 min	A_2	53.06	63.77
	90 min	A_3	34.26	33.73
	120 min	A_4	31.50	25.10
LSD			0.2329	0.3902

The results represent an average of three replicates T_1: represents the protein isolate. T_2: represents pepsin hydrolysis at different times. LSD value at a significant level ($p \leq 0.05$).

The low protein concentration works to increase the emulsification of the protein due to the increase in the area exposed to the surface, unlike the high concentration of protein that works to reduce the emulsification, which leads to improving the emulsification due to the increase in the tendency to interact with the hydrophobic lipid phase. **(Nashmi and Naser, 2022; Al-Aubadi & Al-Jobouri, 2013)**.

The results agreed with **(Adewumi *et al.*, 2022; Al–samarraie *et al.*, 2013)** when studying the functional properties and amino acid appearance of Moringa oleifera protein isolate, where it

was found that the emulsifying activity was 45.83%, while the stability of the emulsion was 47.28%. These results were higher than the results obtained by **(Bocarando-Guzman *et al*., 2022; Al-Anbari *et al*., 2019; Aziz, 2015)** when studying and comparing the physical, chemical and functional properties of flour and protein isolated from Moringa leaves, where the lowest value for emulsifying activity was found at pH 4.5- 5.5 compared to pH values While the highest value of the emulsion thiol was found at pH8 and was 31.67%, followed by 21.67% at pH 10.

Foaming Properties

As show in (table 3) the results of the foam capacity and its stability for the protein isolate and its decomposers, where the highest value of the foam capacity of the decomposer resulting from the use of the pepsin enzyme after 60 min of the reaction (A_2) was 41.17%, while the lowest value of the foam capacity was for the decomposing of the pepsin enzyme after 120 min of the reaction (A_4) and reached 13.79%, and the difference was significant with the rest of the treatments at the level of ($p \leq 0.05$). The reason for this may be due to the proximity of the pH to the point of electrical equilibrium, as the repulsion decreases and a thin and coherent film is formed as the best foam film is formed at the point of electrical equilibrium.

Table (3): Foam capacity and stability of Moringa leaves protein isolate and its hydrolysates

Samples			Foam capacity (%)	Foaming stability (%)				
				min 1	min 10	min 30	min 60	min 90
T_1			23.07	21.87	18.03	12.28	7.40	3.84
T_2	30 min	A_1	32.43	30.55	24.24	13.79	0	0
	60 min	A_2	41.17	39.02	33.33	25.37	16.66	13.79
	90 min	A_3	38.27	37.50	34.21	28.57	20.63	10.71
	120 min	A_4	13.79	12.28	7.40	0	0	0
LSD			3.404	0.121	0.120	0.129	0.070	0.076

The results represent an average of three replicates T_1: represents the protein isolate. T_2: represents pepsin hydrolysis at different times. LSD value at a significant level ($p \leq 0.05$).

As for the highest stability of the foam in the first minute of the hydrolyzed enzyme pepsin after 60 min of hydrolysis (A_2) by 39.02%, but after that it decreased to (33.33, 25.37, 16.66, and 13.79)% at each of the times (10, 30, 60, and 90) min respectively. The reason may be due to the large size of the peptide and then the formation of flexible membranes around the air bubbles and the possibility of the presence of hydrophobic amino acids, as their presence increases the stability of the foam.

The foam capacity of the enzymatic hydrolysates increased by using (alcalase, flavourzyme, protamex and neutrase) enzymes, reaching (63.9, 60.3, 61.65 and 53.1)% compared to the foam capacity of the protein before hydrolysis which amounted to 47.24%. As for the stability of the foam it was for enzyme hydrolysates alcalase has high stability during the first 10 min but the flavourzyme hydrolysates showed high stability compared to the rest of the hydrolysates after 120 min **(Khalaf & Rahman, 2015; Muhamyankaka *et al*., 2013).**

The results agreed with **(Patil *et al*., 2022; Al-Anbari et al., 2019; Khafaji & Azeez, 2008)** when studying the protein of Moringa leaves and seeds, as it showed that the stability of the protein decreased over time for those studied models. It also indicated that the low charge had an effect on the foam stability, and it was higher than the results obtained by when studying the

chemical composition and some functional properties of Moringa leaves meal, Leucina and Glyricidia, who reported a foam capacity ratio of 10% and a foam stability ratio of 2% for Moringa leaves flour **(Al-Anbari *et al*., 2019 ; Aye & Adegun, 2013)**.

CONCLUSIONS

The study showed the importance of using Moringa leaves powder protein isolate and some of its hydrolysates as an emulsifying and foaming agent in improving the quality of food products. It can be concluded from this study that the protein of Moringa leaves is a good and important source of protein and may be a viable alternative for use in food applications as a functional food due to its good functional properties.

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المجلة العراقية لبحوث السوق
وحماية المستهلك

EVALUATING THE PERFORMANCE OF DIFFERENT SOLAR IRRIGATION SYSTEMS AND THEIR EFFECT ON BEAN YIELD (*Vicia Faba… L)*

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ABSTRACT

An experiment was carried out at Al-Raed Research Station, which is located on the Baghdad-Anbar Road. It was conducted during the winter season of 2022-2023, in order to evaluate the performance of different solar irrigation systems and their effect on bean yield (*Vicia faba… L*). A randomized complete block design (RCBD) was used with three replications. The experiment consists of three factors. first factor was the solar panel type with two levels: mono-crystalline and poly-crystalline. Second factor was the irrigation system with two levels: drip and sprinkler irrigation system. The third factor was the distances between the sub-lines with three levels (40, 60, 80 cm). The results show that the monocrystalline achieved the best result by achieving the highest irrigation system efficiency (76.31%), and highest seeds yield total (6618 Kg/ha) with minimum operational costs (0.0252 $/KWh). The drip irrigation had the highest irrigation system efficiency (84.40%), and highest total seeds yield (6077 Kg/ha) with minimum operational costs (0.0340 $/ KWh). The distance (40cm) between the sublines had the highest irrigation system efficiency (76.83 %), with less operational costs (0.0398 $ / KWh).

key words: solar panels, seed yield, operational costs, drip irrigation.

[*]**The research is taken from a master's thesis by the first researcher.**

تقييم أداء أنظمة ري مختلفة تعمل بالطاقة الشمسية وتأثيرها على إنتاجية الباقلاء (*Vicia Faba…L*)

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الخلاصة

نفذت تجربة في محطة ابحاث الرائد الواقعة على طريق بغداد - الانبار خلال فصل الشتاء للموسم (2022-2023) لدراسة تقييم أداء أنظمة ري مختلفة تعمل بالطاقة الشمسية وتأثيرها على إنتاجية الباقلاء (*Vicia faba L*). تم استخدام تصميم القطاعات الكاملة المعشاة (RCBD) بثلاثة مكررات. تتكون التجربة من ثلاثة عوامل. كان العامل الأول هو نوع الألواح الشمسية بمستويين: أحادي البلورية ومتعدد البلورات. العامل الثاني كان نظام الري ذو مستويين نظام الري بالتنقيط والرش والعامل الثالث كان المسافات بين الخطوط الفرعية بثلاثة مستويات (40، 60، 80 سم) تم دراسة المؤشرات التالية: كفاءة نظام الري (٪)، مجموع محصول البذور. (كغم / هكتار)، تكاليف التشغيل (دولار/ ك.و.س). أظهرت النتائج أن أحادي البلورية حقق أفضل نتيجة من خلال تحقيق أعلى كفاءة لنظام الري (76.31٪)، وأعلى مجموع محصول للبذور (6618 كغم / هكتار) بأقل تكاليف تشغيلية (0.0252 دولار / ك.و.س). حقق الري بالتنقيط أعلى كفاءة لنظام الري (84.40٪)، وأعلى مجموع محصول للبذور (6077 كغم / هكتار) بأقل تكاليف تشغيلية (0.0340 دولار/ ك.و.س). كان للمسافات بين الخطوط الفرعية أعلى كفاءة لنظام الري (76.83٪)، مع تكاليف تشغيل أقل (0.0398 دولار/ ك.و.س).

الكلمات المفتاحية: الألواح الشمسية، حاصل البذور، تكاليف التشغيل، ري بالتنقيط.

INTRODUCTION

Human erroneous practices are considered one of the most important negatives that affect the environment and contribute to the raising rates of degrees in heat, which has contributed in the phenomenon of global warming that Iraq and the region in general suffer from **(Mahal *et al.*, 2022)**. The performance efficiency of the solar panel is one of the most important characteristics that give an indication to the quality of the solar panel, but it is affected by a number of factors, with the weather being the most influential, due to its close association with the amount of radiation reaching the panel (**Zeinaldeen, 2020)**. The use of modern irrigation systems can provide water in appropriate quantities for plants, in addition to reducing waste in irrigation water more than other irrigation systems (**Rasheed, 2021)**. Enhancing the efficiency of irrigation systems is one of the objectives that the researchers seek in order to increase the areas planted with crops and the main waist for local consumption while reducing the consumption of water resources **(Abdullah & Kadhim, 2023**). Attention to the calculations related to irrigation is one of the important things for the design and operation of irrigation projects (**Al-Kazragy, 2020)**. Increasing the productivity of agricultural crops is an objective pursued by farmers all over the world and this can be achieved by using modern irrigation methods **(Al-Lami *et al.*, 2023)**. In light of the scarcity of water, water recycling is one of the solutions that reduce pressure on water sources and is used to irrigate agricultural lands (**Rahi & Faisal, 2019)**. The use of traditional irrigation methods is one of the wrong practices by farmers, and this is due to the low operational costs compared to modern irrigation

systems (**Karim & Karim, 2020).** The sprinkler irrigation system can be considered as one of the modern irrigation methods that can be used to reduce the amount of water wasted by traditional methods and a goal to achieve security food (**Al-Mehmdy & Yacoub, 2019**). The drip irrigation system and the sprinkler irrigation system can also be considered as modern irrigation systems that have a role in rationing water waste, but the drip irrigation system is more efficient than the sprinkler system **(Khattab & El-Housini, 2019)**. The plant density significantly affected all the studied traits **(Khalaf & Hassan, , 2022).** Through a study showed that the distance between 25 cm the plants were significantly superior and gave the highest average seed yield (**Sadiq & Mohammed, 2022)**.

Research problem

Due to the challenges faced by most parts of the world, including our dear country, from the threat to food security as a result of population increase and problems related to providing the necessary water for irrigation, in addition to the obstacles to providing the energy sources necessary for the operation of various irrigation systems, and what the world in general and our country Iraq in particular is experiencing from the greenhouse effect caused by the continuous and increasing use of fossil fuel derivatives as an energy source, the subject of the research came up.

Research objective

1. Comparison the performance of two types of solar panels.
2. Comparison of modern irrigation systems (drip, spray).
3. Comparison of the effect of the variation of distances between the sub-lines on the yield of the crop.

Thus: finding the best combination between the type of solar panel and the irrigation system and the best distance between the sab-lines on the productivity of the Faba bean crop.

MATERIALS AND METHODS

An experiment was carried out at Al-Raed Research Station, which is located on the Baghdad-Anbar Road. It was conducted during the winter season of 2022-2023. in order evaluate the performance of different solar irrigation systems and their effect on bean yield (*Vicia faba… L*). A randomized complete block design (RCBD) was used with three replications. The experiment consists of three factors. first factor was the solar panel type with two levels: mono-crystalline and poly-crystalline. Second factor was the irrigation system with two levels drip and sprinkler irrigation system and third factor was the distances between the sublines with three levels (40 , 60 , 80 cm) The following traits were studied : irrigation system efficiency (%), total seeds yield (Kg / ha), and operational costs ($/ KWh). A number of agricultural operations were carried out to prepare the experimental field, the field was flooded with water, and we waited for 20 days for the field to dry completely. Because of the density of the Jungles that the Experimental field suffers from. After completing the plowing process, the rotary plow was used to smoothen the previously ploughed soil, after which the levelling and

adjustment machine was used. A group of samples were taken from the experimental field on 5/9/2022 and sent to the laboratories of the National Centre for Water Resources Management for soil analysis and to determine the physical and chemical characteristics of the experimental field as shows in Table (1). The bean crop was seeded manually at a depth of 3-5 cm, two seeds in each hole, at a distance of 25 cm between one hole and another, on 15/10/2022, weeds were removed manually and chemically.

Table (1): Physical, chemical and hydraulic preparties of the soil before planting

Soil depth	Soil articulations g.kg^{-1}			Soil texture	Field volumetric water content at 33 Kps (Cm^{3}Cm^{-3})	Volumetric water content at 1500 Kps (Cm^{3}Cm^{-3})	PH	EC dS.m^{-1}	soil bulk density Mg.m^{-3}
	sand	silt	Clay						
0 – 25	12	52	35	Silty clay loam	0.465	0.265	7.7	8	1.27
25 - 50	11	51	36	Silty clay loam	0.470	0.274	7.4	16	1.31

Characters Studied

irrigation system efficiency (%).

The efficiency of the two irrigation systems was measured according to the equations and methods used before (Al-Taif & Al-Hadithi, 1988)**.**

$Ee = Ea \times Ed \times 100$…… (1)

Where: -

Ee = Irrigation system efficiency %.

Ea = Perfusion efficiency %.

Ed = distribution efficiency %.

Perfusion efficiency was measured by the following equation:

$Ea = \left(\frac{Ws}{Wf}\right) \times 100$…… (2)

Where: -

Ea = Perfusion efficiency%

Ws = the depth of water stored in the root zone (m).

Wf = The amount of Water received from the source (m).

Distribution efficiency was measured by the equation:

$Ed = \left(\frac{1-\bar{y}}{đ}\right) \times 100$…….... (3)

Where: -

Ed = water- distribution efficiency %.

$\bar{y}$ = average numerical deviation in depth of water stored from average depth stored during the irrigation (cm).
đ = average depth of water stored during the irrigation (cm).

Total Seeds Yield (Kg / ha).

The bean crop was harvested on 3/16/2023, and the productivity was calculated on the basis of the first pound and on the basis of the average seed weight yield of three randomly selected plants from the beginning, middle and end of the experimental unit line multiplied by the plant density and then converted to ($Kg.ha^{-1}$), (**Al-Sahoki & Jiyad, 2023**).

Operational Costs ($ / KWh).

Operational costs were calculated on the basis of the energy unit (KWh), where the maximum power was measured during the operating condition and converted (Kw) and multiplied by the number of operating hours during the season, then divided the purchase price by (KWh), using the following formulas **(Maurya *et al*.,2015; Manfaluthy *et al*.,2021).**
The price per watt was calculated using the following formula:

$$O\ c = \frac{PP}{P_{mas}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where: -
O c = Operational costs ($/KWh).
PP = purchase price ($).
P_{max} = Maximum Power (KWh).
The power was calculated with the following equation:

$$Pmax = Vmp \times Imp \ldots\ldots (5).$$

Where: -
P_{max} = Maximum Power (Watt).
V_{mp} = Maximum Power Voltage (Volt).
I_{mp} = Maximum Power Current (Ampere).

RESULTS AND DISCUSSION:

Irrigation System Efficiency (%).

Table 2 show the effect of the type of solar panels on the efficiency of the irrigation system as a percentage. It is clear that there is no significant effect of the type of solar panels used on the studied characteristic, as the monocrystalline solar panels recorded the highest average values for the efficiency of the irrigation system were (76.31%), while the polycrystalline solar panels recorded the lowest average values for the efficiency of the irrigation system (74.96%).

The results also indicated in Table 2 the effect of the type of irrigation system used on the efficiency of the irrigation system in percent, as the results showed a significant effect of the type of irrigation system used on the efficiency of the irrigation system in percent, as the drip irrigation system recorded the highest. The average values of the efficiency of the irrigation system were (84.40%), while the sprinkler irrigation system (Sprinkler) recorded the lowest average efficiency by (66.87%).

Table 2 also showed the effect of the environmental distances between the sub-lines on the efficiency of the irrigation system in percentage, as the results showed that there was no significant effect of the distances between the sub-lines on the efficiency of the irrigation

system. The distance (40 cm) recorded the highest efficiency rates of (76.83%), while the distance recorded (80 cm) the lowest average efficiency was (74.73%).

The results also indicated in Table 2 the effect of the bilateral interference between the type of solar panel and the irrigation system used on the efficiency of the irrigation system %. The results indicate that there is a significant effect of the interaction between the type of solar panel and the type of irrigation system used on the efficiency of the irrigation system %. The monocrystalline solar panel (with drip irrigation system) achieved the highest average values of irrigation system efficiency (88.85%), while the monocrystalline solar panel with sprinkler irrigation system recorded the lowest irrigation system efficiency rates It reached (63.77%).

It is noted from Table 2 the effect of the bilateral interference between the type of solar panel and the distances between the sub - lines on the efficiency of the irrigation system %, The results indicate that there is no significant effect of the interaction between the type of solar panel and the distances between the sub-lines on the efficiency of the irrigation system. The monocrystalline solar panel with a distance of (40cm) recorded the highest average values for the irrigation system efficiency % which amounted to (78.28%), while the polycrystalline solar panel with a distance of (80cm) recorded the lowest average values for the irrigation system efficiency %, which was (73.24%).

Table 2 showed us the effect of the bilateral interference between the type of irrigation system used and the distances between the sub-lines, as the results indicate that there is no significant effect of the bilateral interference between the type of irrigation system and the distances between the sub-lines that the drip irrigation system achieved (Drip) at a distance of (60cm) the highest rates of irrigation system efficiency in % amounted to (85.23%), while the sprinkler irrigation system recorded with a distance of (60cm) the lowest averages of the irrigation system's efficiency in % was (65.48%).

Table 2 shows the effect of the triple interference between the type of solar panel, the type of irrigation system used, and the distances between the sub-lines on the efficiency of the irrigation system. The results showed that there was no significant difference between the triple interference between the type of solar panel, the type of irrigation system, and the distances between the sub-lines on the efficiency of the irrigation system. The results indicated that the monocrystalline solar panel with the drip irrigation system at a distance of (40cm) achieved the highest average values for the irrigation system efficiency amounted to (90.89%), while the monocrystalline solar panel with the sprinkler irrigation system recorded at the distance (60cm), the least average efficiency was (60.90%).

Table (2): The effect of the type of solar panel, irrigation system, and distances between sub-lines on the efficiency of the irrigation system (%).

Type solar panel (C)	Irrigation system (I)	Distances between sub-lines (D)			C * I	
		40	60	80		
Polycrystalline	Sprinkler	72.58	70.06	67.27	69.97	
	Drip	78.16	82.48	79.22	79.95	
Monocrystalline	Sprinkler	65.67	60.9	64.74	63.77	
	Drip	90.89	87.97	87.68	88.85	
LSD $_{C*I*D}$		6.483 $^{N.S}$			LSD $_{C*I}$	4.977
C * D						
Type solar panel (C)		40	60	80	Average type solar panel	
Polycrystalline		75.37	76.27	73.24	74.96	
Monocrystalline		78.28	74.44	76.21	76.31	
LSD C* D		4.163 $^{N.S}$			LSD $_{C}$	4.33 $^{N.S}$
I * D						
Type of irrigation system (I)		40	60	80	Average type irrigation system	
Sprinkler		69.12	65.48	66	66.87	
Drip		84.53	85.23	83.45	84.4	
LSD $_{I*D}$		5.259 $^{N.S}$			LSD $_{I}$	4.856
D						
Distances between sub-lines (D)		40	60	80		
Average distances		76.83	75.35	74.73		
LSD $_{D}$		3.057 $^{N.S}$				

Total Seeds Yield (kg / ha).

Table 3 shows us the effect of the type of solar panel used on the total seeds yield (kg/ha), as the type of panel used had a significant effect on character of the total seeds yield (kg/ha), where the monocrystalline solar panel excelled by recording the highest. The average of the total seeds yield (kg/ha) was (6618 Kg/ha), while the average total seeds yield (kg/ha) was lower for polycrystalline solar panels by (4714Kg/ha). The reason may be due to the higher efficiency of the monocrystalline panel. of the polycrystalline board, which gives a better performance to the pump responsible for the irrigation systems.

Table 3 shows us the effect of the type of irrigation system used on the character of the total seeds yield (kg/ha), where the type of irrigation system used had a significant effect on the characteristic of the total crop yield (kg/ha). It drip irrigation system excelled with, the average values of the total seeds (yield 6077Kg/ha), while the sprinkler irrigation system recorded the lowest value of the averages for the studied trait amounted to (5255Kg/ha). The cause may be

due to the fact that the drip irrigation system is rarely affected by weather factors, which has a positive impact on productivity.

Table 3 also show the effect of the distances between the sub-lines on the total seeds yield (kg/ha), as the distances between the sub-lines had a significant effect on the character seeds yield total (kg/ha), where the distance (60cm) recorded the highest value of the averages. The total seeds yield amounted to (6262 Kg/ha), while the distance (80 cm) recorded the lowest value of the relevant averages at (3950 Kg/ha).

Table 3 also shows the effect of the bilateral interference between the type of solar panels and the type of irrigation system on the total seeds yield (kg/ha). It is clear to us that the monocrystalline solar panel with the drip irrigation system had the highest average value of the total crop yield, which amounted to (7029 Kg/h), while the polycrystalline solar panel with sprinkler irrigation system achieved the lowest values of the averages of the studied characteristic was (4302 Kg/h). There was no significant interaction between the type of solar panel and the type of irrigation system used on the character of the total seeds yield (kg/ha).

Table 3 also showed us the effect of the bilateral interference between the type of solar panel and the distances between the sub-lines on the total seeds yield (kg/ha), where the bilateral interference between the type of the solar panel and the distances between the sub-lines did not have a significant effect on the characteristic of the total seeds yield (kg/ha). The monocrystalline solar panel with a distance of (60cm) had the highest average yield value of (7735Kg/ha), while the polycrystalline solar panel with a distance of (80cm) recorded the lowest average value of the total seeds yield was (3075 Kg /ha).

Table 3 shows us the effect of the bilateral interference between the type of irrigation system and the distances between the sub-lines on the total seeds yield (kg/ha) Where the table shows us that there is a significant effect of the bilateral interference between the type of irrigation system and the distances between the sub-lines on the character of the total seeds yield (kg/ha). The drip irrigation system with a distance of (40 cm) exceeded the highest average value of the total seeds yield in a record (7933 Kg/ha), While the sprinkler irrigation system at a distance of (80 cm) recorded the lowest values for the averages, the total seeds yield was (3525 kg/ha).

Table 3 shows the effect of the triple interference between the type of solar panel, the type of irrigation system used, and the distances between the sub-lines on the total seeds yield (kg/ha).The monocrystalline panels with the drip irrigation system (Drip) at a distance of (40cm) achieved the highest value for the seeds yield total rate of (9100Kg/ha), On the other hand, the polycrystalline board with the sprinkler system at a distance of (80 cm) recorded the lowest value for the total seeds yield rates, which amounted to (2717 Kg/ha). The triple interference did not have a significant effect on the studied trait.

Table (3): The effect of the type of solar panel, irrigation system, and distances between sub-lines on the total seeds yield (kg/ha).

Type solar panel (C)	Irrigation system (I)	Distances between sub-lines (D)			C * I	
		40	60	80		
Polycrystalline	Sprinkler	3690	6500	2717	4302	
	Drip	6767	5178	3433	5126	
Monocrystalline	Sprinkler	5490	8800	4333	6208	
	Drip	9100	6670	5317	7029	
LSD $_{C*I*D}$		990.4 $^{N. S}$			LSD $_{C*I}$	278.2$^{N. S}$
C * D						
Type solar panel (C)		40	60	80	Average type solar panel	
Polycrystalline		5228	5839	3075	4714	
Monocrystalline		7295	7735	4825	6618	
LSD C* D		691.5 $^{N. S}$			LSD $_{C}$	210.6
I * D						
type of irrigation system (I)		40	60	80	Average type irrigation system	
Sprinkler		4590	7650	3525	5255	
Drip		7933	5924	4375	6077	
LSD $_{I*D}$		710.1			LSD $_{I}$	277.4
D						
Distances between sub-lines (D)		40	60	80		
Average distances		6262	6787	3950		
LSD $_{D}$		594.1				

Operational Costs ($ / kwh).

Table 4 shows the effect of the type of solar panel used on operating costs $/KWh. The results indicate that there is a significant effect of the type of solar panel used on the characteristic of operating costs $/KWh. The polycrystalline solar panel achieved the highest average operating cost value (0.0874$/KWh), while the monocrystalline solar panel achieved the lowest operating cost average values (0.0252$/KWh). the reason for the low operating costs of the monocrystalline panel may be due to the lower price of the panel compared to the peak capacity.

Table 4 indicates the effect of the type of irrigation system used on operating costs ($/KWh), the results indicate that there is a significant effect of the type of irrigation system used on the characteristic of operating costs $/KWh. where the sprinkler irrigation system achieved the highest operating cost rates (0.0786$/KWh), While the drip irrigation system achieved the lowest operating cost average values (0.0340$/KWh). The reason may be due to the stability of the moisture stability, which affects the time of the irrigation periods.

It is noted from Table 4 the effect of the distances between the sub-lines on the operating costs ($/KWh)., The results indicate to us that there is a significant effect of the distances between the branch lines on the characteristic of operating costs ($/KWh). As the

distance (80cm) achieved the highest average values, operating costs amounted to (0.0725$/KWh), While the distance (40cm) achieved the least value for operating cost rates, it was (0.0398$/KWh). The reason may be due to the distance (40 cm) achieving the lowest operating cost values, due to the overlapping of the irrigation lines affecting the humidity levels.

Table 4 also shows the effect of the dual interference between the type of solar panel and the type of irrigation system used on the operating costs ($/KWh). The results indicate that there is significant effect of the dual interference between the type of solar panel and the type of irrigation system on the characteristic of operating costs ($/KWh), Where the polycrystalline solar panel with the sprinkler irrigation system achieved the highest average operating cost values (0.1247$/KWh). While the monocrystalline solar panel with the drip irrigation system achieved the lowest rates of operating costs (0.0179$/KWh).

Table 4 also indicated the effect of the bilateral interference between the type of solar panel and the distances between the sub-lines on the operating costs ($/KWh), The results indicate to us that there is a significant effect of the bilateral interference between the type of solar panel and the distances between the branch lines on the characteristic of operating costs ($/KWh). The polycrystalline solar panel at a distance of (80cm) achieved the highest values of average operating costs amounting to (0.1145$/KWh), While the monocrystalline solar panel with a distance of (40cm) achieved the lowest average operating cost values (0.0203$/KWh).

Table 4 also showed us the effect of the bilateral overlap between the type of irrigation system used and the distances between the branch lines on the operating costs ($/KWh), The results indicate to us that there is a significant effect of the bilateral overlap between the type of irrigation system used and the distances between the branch lines on the characteristic of operating costs ($/KWh). The sprinkler irrigation system at a distance of (80 cm) achieved the highest rates of operating costs amounting to (0.1053$/KWh), While the results indicate that the drip irrigation system with a distance of (40 cm) achieved the lowest rates of operating costs were (0.0288 $/KWh).

Table 4 shows us the effect of the triple overlap between the type of solar panel, the type of irrigation system used, and the distances between sub-lines on operating costs ($/KWh). The results indicate that there is a significant effect of the triple overlap between the type of solar panel, the type of irrigation system used, and the distances between the sub-lines on the studied characteristic. The polycrystalline solar panel with the sprinkler irrigation system at a distance of (80cm) achieved the highest average operating cost value of (0.1700$/KWh), While the monocrystalline solar panel with the drip irrigation system at a distance of (40cm) achieved the lowest rates of operating costs (0.0153$/KWh), The reason may be due to the achievement of the monocrystalline solar panel with the drip irrigation system at a distance of (40 cm). The technical specifications of the Monocrystalline panels and their harmony with the drip irrigation system, which is considered one of the most

important metered water irrigation systems, and the interference of moisture at the closest distance between the agricultural lines reduces the irrigation time.

Table (4): The effect of the type of solar panel, irrigation system, and distances between sub - lines on the Operating costs ($/KWh).

Type solar panel (C)	Irrigation system (I)	Distances between sub-lines (D)			C * I	
		40	60	80		
Polycrystalline	Sprinkler	0.0760	0.1280	0.1700	0.1247	
	Drip	0.0423	0.0490	0.0590	0.0501	
Monocrystalline	Sprinkler	0.0253	0.0313	0.0407	0.0324	
	Drip	0.0153	0.0180	0.0203	0.0179	
LSD $_{C*I*D}$		0.004863			LSD $_{C*I}$	0.003998
C * D						
Type solar panel (C)		40	60	80	Average type solar panel	
Polycrystalline		0.0592	0.0885	0.1145	0.0874	
Monocrystalline		0.0203	0.0247	0.0305	0.0252	
LSD C* D		0.0036			LSD $_{C}$	0.0031
I * D						
type of irrigation system (I)		40	60	80	Average type irrigation system	
Sprinkler		0.0507	0.0797	0.1053	0.0786	
Drip		0.0288	0.0335	0.0397	0.0340	
LSD $_{I*D}$		0.0032			LSD $_{I}$	0.0026
D						
Distances between sub-lines (D)		40	60	80		
Average distances		0.0398	0.0566	0.0725		
LSD $_{D}$		0.0017				

CONCLUSION

It can be concluded that the monocrystalline solar panel with the drip irrigation system at a distance of 40 cm achieved the best results in irrigation system efficiency (%), total seed yield (kg/ha), with the lowest operating costs ($/kWh).

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المجلة العراقية لبحوث السوق
وحماية المستهلك

PREPARATION AND STUDY OF NATURAL AND NANO LYCOPENE IN INHIBITING THE GROWTH OF CANCER CELLS EX VIVO IN VITRO

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ABSTRACT

The effectiveness of both natural and nano-lycopene extracted from tomato waste powder in affecting the growth of cancer cells outside the body was studied. The study included the preparation of natural lycopene extract, using the triple mixture of hexane, acetone, and ethanol in proportions 2:1:1 and drying it, then preparing the nanocomposite using the high-energy mechanical grinding technique, and its dimensions were estimated using a Scanning Electron Microscope (SEM) and which was 78nm, and the effectiveness of the two preparations was tested in Inhibition of cancer cell lines of the human mouth and skin, during three periods of time 12, 24, 72 h and at concentrations of both natural and nano-lycopene 0.0,150, 300, 600, 1200, 2400 micrograms/ml, the results of the study showed There was a significant inhibitory effect p≤0.05. for both natural and nano-lycopene in the growth of cancer cells, and nano-lycopene was significantly superior to natural lycopene for skin and mouth models, and the inhibition effect of cancer cells increased for both natural and nano-lycopene with increasing concentration and period, and the highest percentage of inhibition for natural lycopene was 71% and 79.8% While the highest percentage of inhibition for nanoscale icon was 85.2% and 93.1% at a concentration of 2400 µg/ml for 72 h and for skin and oral cancer cell lines, respectively.

Keywords: Lycopene, Lycopene nanoparticles, Anticancer, Antioxidants, Cancer cell lines.

[*] **The research is taken from a doctoral thesis for the first research.**

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تحضير ودراسة اللايكوبين الطبيعي والنانوي في تثبيط نمو الخلايا السرطانية خارج الجسم الحي In vitro

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الخلاصة

تم دراسة فعالية كل من اللايكوبين الطبيعي و النانوي المستخلص من مسحوق مخلفات الطماطة في التأثير في نمو الخلايا السرطانية خارج الجسم الحي,وتضمنت الدراسة تحضير مستخلص اللايكوبين الطبيعي بأستخدام المزيج الثلاثي من الهكسان والأسيتون والأيثانول وبالنسب (2 :1: 1) وتجفيفه ثم تحضير المركب النانوي بأستخدام تقنية الطحن الميكانيكي عالي الطاقة, وتم تقدير أبعادها بأستخدام جهاز المجهر الألكتروني الماسح وكانت (78) نانومتر وأختبرت فعالية المستحضرين في تثبيط الخطوط الخلوية السرطانية للفم والجلد البشري, وخلال ثلاث فترات زمنية (12 ,24, 72) ساعة وبتراكيز لكل من اللايكوبين الطبيعي والنانوي (0.0, 150, 300, 600, 1200, 2400 مايكروغرام / مل), أظهرت نتائج الدراسة وجود تأثير تثبيطي معنوي .($p\leq0.05$) لكل من اللايكوبين الطبيعي والنانوي في نمو الخلايا السرطانية,وتفوق اللايكوبين النانوي معنويا على اللايكوبين الطبيعي لنماذج الجلد والفم,وأزدادت فعالية التثبيط للخلايا السرطانية لكل من اللايكوبين الطبيعي والنانوي بزيادة التركيز والفترة الزمنية وبلغت أعلى نسبة مئوية للتثبيط للايكوبين الطبيعي (71% و 79.8%) فيما بلغت أعلى نسبة مئوية للتثبيط للايكوبين النانوي (85.2 %و 93.1%) وعند التركيز 2400 مايكروغرام / مل ولمدة 72 ساعة ولخطوط الخلايا السرطانية للجلد والفم وعلى التوالي.

الكلمات المفتاحية : اللايكوبين , اللايكوبين النانوي,مضادات السرطان , مضادات الاكسدة,خطوط الخلايا السرطانية.

INTRODUCTION

Lycopene is a natural carotenoid pigment produced by plants and microorganisms during the process of photosynthesis to protect them from photoactivity. It is a plant chemical primarily found in tomatoes and their products, and other plant sources including watermelon, guava, papaya, apricot, and red grapefruit. Additionally, other sources such as red carrots, rosehip, and autumn olive are among the main sources of lycopene. (**Al-Tameemi *et al.*,2023; Muna *et al*., 2023; Aziz *et al*., 2023; Fordham *et al*., 2002)**

The molecular weight of lycopene is 536.89, and the melting point of lycopene is 172-175 C. Lycopene is found in ripe tomato fruits in the form of rectangular crystals resembling needles. It is responsible for the bright red color of ripe tomato fruits. Lycopene is more soluble in organic solvents such as chloroform, benzene, hexane, ether, and ethyl acetate. It dissolves in vegetable oils but does not dissolve in water, methanol, and ethanol. **(Shi & Maguer, 2000; Asaduzzaman, 2022; Al-jubouri *et al*., 2022).**

The rates of infection and death resulting from cancer are constantly increasing, which makes cancer a major global health problem and ultraviolet radiation has increased in recent decades hit the earth's surface and depleted the ozone layer, so it is necessary to protect our skin from these rays because they cause damage to human skin such as skin cancer and hyperpigmentation and skin aging. Many active natural compounds inhibit cancer, such as lycopene, which has proven effective in protecting the skin from these rays **(Khaleel *et al*., 2019; Al-Anbari *et al*., 2019).** Lycopene is one of the biologically active compounds and one of the most important antioxidant carotenoid components in tomatoes and plays important roles in maintaining and improving human health. **(Al-Anbari *et al*., 2019; Altaee *et al*., 2020; Kanyar and Karadaş, 2023)**

Consumption of lycopene from its natural sources leads to enhanced protection of human skin from ultraviolet radiation and biological activities related to the skin and anti-aging

of the skin, and a diet rich in tomatoes has been associated with a variety of health benefits including anti-cancer properties **(Collins *et al.*, 2022; Honda, 2023;Nahla *et al.*, 2018).**

Studies indicate that lycopene accumulated in the skin can provide protection against UV rays as well as protect target molecules by suppressing free radicals, inhibiting cellular inflammatory responses, and repairing damage caused by UV rays. And that balanced nutrition is necessary to maintain healthy skin and that the loss of some nutrients leads to abnormalities in the skin **(Tarshish & Hermoni, 2023; Hamdia & Ahamed, 2023; Al-Jumaily ., 2014)**

The preparation of nanoparticles of lycopene from natural lycopene leads to an increase in its antioxidant activity and its anticancer activity in the laboratory **(Shamurad *et al.*, 2019; Khaleel *et al.*, 2019; Omran & Baek., 2021).**

The study aimed to compare the effectiveness of natural and nano-lycopene extracted from dried tomato residues in inhibiting the growth of skin and oral cancer cells. **(Al-hadedee *et al.*,2021)**

MATERIALS AND METHODS

1- **Processed tomatoes** were obtained from the farms of Karbala Governorate for the fall agricultural season 2021to2022.The dried tomato waste powder was prepared using the electric vacuum oven at 40°C until the weight stabilized. Then prepare the dried natural powder by grinding it with a mill equipped by (Monolex) company.

2 – Lycopene Extraction

Lycopene was extracted from tomato waste powder by following the method described by **(Thompson *et al.*, 2000)** by taking 1g of the dried sample and mixing it with 10 ml of a solvent mixture (acetone: hexane: ethyl alcohol) in a ratio of (1:2:1) and mixing In a vortex vibrator for 10 min, then 1.5 ml of water was added to separate the hexane layer from the acetone and ethyl alcohol layer, and mixed for another 5 min. The upper layer containing lycopene was withdrawn and kept in a dark closed vial, then it was placed in the electric oven at a temperature of 30°C until a stable weight was obtained.

3 – Preparation of Nanoparticales of Lycopene

A quantity of dried lycopene was placed in a high-energy steel ball mill was used from the German company (Retsch) and in the Ibn Al-Bitar Center, which is affiliated to the Ministry of Industry and Minerals, at a speed of 400 rpm per min for 15 min, then the crushed product was collected in sterile and opaque glass bottles and kept in refrigeration 4±2 °C, and the nanoscale dimensions were estimated using the SEM **(Ali *et al.*, 2016; Slewa & Mowsowy, 2018; Murthykumar and Malaiappan 2020)**

Preparation of cancer cell lines

The inhibitory effect of the natural and nano-lycopene extract was studied on two types of cancer cell lines, namely human squamous cell carcinoma cell line and human oral squamous cell carcinoma cell line, in passages 27 and 22 respectively.and at the Biotechnology Center Al-Nahrain University At concentrations (0.0, 150, 300, 600, 1200, 2400) µg/ml, the cells were grown in medium Rosswell Park Memorial Institute -1640 supplemented with 5% Fetal Calf Serum (FCS).

The toxic effect of culturing cells in tissue culture dishes was studied with multiple holes (Microtiter plates) 96 and the flat bottom Flat Bottom to conduct this test. The experiment included three stages:

Cells seeding

Cancer line cells were activated and proliferated for 24 h, then the growth monolayer was treated with a Trypsin-Versen solution. 25 ml of RPMI-1640 medium prepared with serum was added to each vessel and the number of cells was adjusted to 1×10^4 using a slide count. A volume of 100 µl of cell suspension was taken and distributed to the holes of the tissue culture dish. The dishes were incubated after covering them with sterile adhesive paper at a temperature of 37 °C for 24 h to allow the cells to adhere to the glass,

Preparation of experimental samples

Several concentrations of both natural and nano-lycopene extract were prepared simultaneously using a tissue culture medium devoid of fetal calf serum, and added to pits containing adherent cancer cells. Six replications were used for each treatment. The culture medium was poured into the tissue culture dishes. Column No. 1 was considered as a negative control, and 200 µl of serum-free culture medium were added to it. As for columns from (2 to 12), graduated concentrations of 200 µl / hole were added. The dishes were covered and incubated at a temperature of 37°C, for different exposure times 24, 48, 72 h.

Cytotoxicity assay

After the end of the prescribed incubation period, the contents of the dishes (the culture medium and the suspended cells) were poured out and then washed with phosphate-buffered saline three times to ensure the removal of any trace of the test material and non-adherent cells, then a volume of 10 µl of Methyl Thiazolyl Tetrazolium MTT dye solution (0.5 mg/ml) was added to each hole then left for 4 h at a temperature of 37 ° C in a carbon dioxide incubator. The cells were washed several times with a saline phosphate buffer until the excess dye was removed. After the dishes were completely dry, 100 µl of dimethyl sulfoxide DMSO were added. The results were read using an ELISA reader using a spectrophotometer on the titration dishes. Microplate spectrophotometer (ELISA) at a wavelength of 500 nm.

The inhibition rate was calculated according to the equation below:

$$\%IR = \frac{\mathrm{A-B}}{\mathrm{A}} \times 100 - 100$$

IR= Inhibitory Rate

A= Absorbancy for Negative Control

B= Absorbancy for Test

Statistical Analysis

The Statistical Analysis System **(SAS. 2018)** was used to analyze the data to study the effect of different coefficients on the studied traits according to a Complete Random Design (CRD), and the significant differences between the means were compared with the Least Significant Difference-LSD test.

RESULTS AND DISCUSSION

Figure (1) shows the image of the prepared nanostructures. It is noted that the structures fall within the nanoscale dimensions, and the average particle size is 78nm

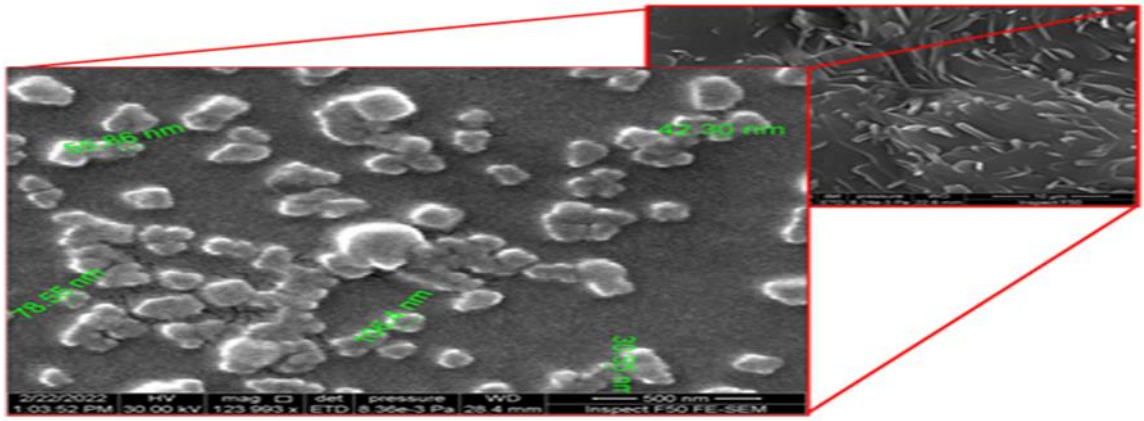

Figure (1): Lycopene nanoparticles image by SEM

As shows in the table (1) effect of adding different proportions of each natural and nano-lycopene extract in the inhibition of the human skin cancer cell line. ($p \leq 0.05$).

It is directly proportional to the increase in lycopene concentrations, and the percentage of inhibition of natural lycopene was 8.5% for 24 h and at the lowest concentration of 150 µg /ml, while it reached 71% at the highest concentration of 2400 µg /ml for 72 h. Nano-lycopene was superior to natural lycopene, as it recorded the highest level of inhibition by 85.2% at a concentration of 2400 µg/ml for 72 h. **(Haider *et al.*,2024)**

(Table, 2) also shows an increase in the percentage of inhibition of the oral cancer cell line by increasing the added concentrations, and the percentage of inhibition of natural lycopene was 7.9% at a concentration of 150 µg / ml for 24 h, while it reached 79.8% at a concentration of 2400 µg / ml for 72 h, and lycopene was superior nanoparticles to natural lycopene, as it recorded an inhibition rate of 93.1% for 72 h and at the highest concentration. **(Yaaqoob, 2022; Al-Jubouri *et al.*, 2022)**

The results indicate the effectiveness of both natural and nano-lycopene extracts in inhibiting the growth of cancer cells and the superiority of nano-lycopene over natural in both skin and oral lines **(Jasim & AI-Obaidi, 2022; Al-Anbari *et al.*, 2019; Mula and Alrubeii, 2024)**

The results agreed with what was found by **(Soares *et al.*, 2017; Doosh and Al-Mosawi, 2010)** in the inhibitory activity of lycopene extracted from tomato paste on human prostate cancer cells, at different concentrations from 500 to 5000 µg/ml and for exposure periods (24, 48, 72, 96) h.

Table (1): Comparison of the effect of natural and nanoscale lycopene on the human skin cancer cell line at different concentrations during 72 h

concentration µg/mL	inhibition%					
	Nano-Lycopene			natural lycopene		
	24 h	48h	72h	24 h	48 h	72 h
150	15.6	22.4	27.3	8.5	6.6	19.7
300	21.6	25.9	46.9	17.5	32.6	39.7
600	33.6	41.4	63.3	29.4	49.1	45.4
1200	54.6	60.7	79.1	45.5	57.1	65.3
2400	67.7	74	85.2	54.4	62.1	71
LSD	8.04 *	8.79 *	8.05 *	6.51 *	7.48 *	7.93 *
* (P≤0.05).						

Table (2): Comparison of the effect of natural and nanoscale lycopene on oral cancer cell line at different concentrations during 72 h.

concentration µg/Ml	inhibition%					
	Nano-Lycopene			natural lycopene		
	h 24	48h	72h	h 24	h 48	72 h
150	29.3	42.5	53.8	7.9	16.3	28.4
300	39.4	50.8	59.8	18.1	33.6	56.5
600	53	62.6	67.2	27	56.3	68.4
1200	66.5	77.5	86.9	50.1	63.8	75
2400	88.4	84.7	93.1	61.2	72.6	79.8
LSD	9.92 *	9.01 *	10.42 *	11.08 *	9.66 *	8.37 *
* (P≤0.05).						

(**Teodoro *et al.*, 2012**) found an inhibitory effect of lycopene on many types of cancer. The inhibitory effect depends on the type of cancer and the concentration of lycopene used.

(**Hussein *et al.*, 2023 ; Campos *et al.*, 2022**) indicated that the toxic effect of lycopene against cancer cell lines is one of the most powerful antioxidants because it contains a large number of double bonds that absorb free oxygen molecules and inhibit free radicals and is characterized by its ability High inhibition of cancer cells through its antioxidant activity, as it is toxic to cancer cells, through the mechanism of removing free radicals generated when cancer cells form (**Masoud *et al*., 2022 ; Al-Taweel *et al*., 2022**).

The inhibitory effect of lycopene is attributed to possible mechanisms represented in affecting the effectiveness of cell division through its effect on DNA replication, or one of the enzymes important in replication, or through fragmentation of the DNA strand and inducing cells to programmed death. (**Faddagh *et al.*, 2020 ; Al-jubouri *et al*., 2022 ; Usui *et al.*, 1**998). It can also work to inhibit multiple divisions of some types of human cancer cell lines and induce cells towards programmed cell death (apoptosis) and thus its role in protecting the genetic material from the effect of environmental mutagens and the ability of its components to correct genetic errors. (**Khalid *et al*., 2021 ; Shamurad *et al*., 2019; Lopus & Panda., 2006**).

The superiority of nanoparticles lycopene over natural lycopene in the toxic effect and inhibitory effectiveness against cancer cell lines could be due to its characteristics and properties in interacting with different mechanisms and methods compared to its interactions when it is in its normal dimensions due to its low size, the increased surface area of the nanoparticles and the spread of surface charges, which allows it to have a greater increase in cell activity. The number of atoms and molecules involved in the reactions. (**Sridhar *et al.*, 2021; Yaaqoob., 2022; Abdulsada *et al.*, 2023**)

CONCLUSIONS

The importance of natural lycopene extract extracted from dried tomato residues as a biologically effective compound for its inhibitory ability to inhibit the growth of cancer cells ex vivo and by increasing concentrations and exposure time. Its presence in its nano form doubles its inhibitory effectiveness against cancerous lines of the skin and mouth. Nano-lycopene achieved superiority over natural lycopene with concentrations equivalent to half the concentrations of natural lycopene in inhibiting the growth of cancer cells for both lines.

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المجلة العراقية لبحوث السوق وحماية المستهلك

EFFECT OF ADDITION OF BIOFERTILIZERS, NILE FLOWER PEAT FERTILIZER AND SPRAYING WITH ITS EXTRACT ON QUALITATIVE CHARACTERISTICS OF POTATO YIELD

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ABSTRACT

The field experiment was carried out in Horticulture Development Station in the Kut district of the Directorate of Agriculture of Wasit Governorate at spring season 2022, This study was aimed to demonstrate the effect of inoculation some Bio-fertilizers, adding Nile flower peat fertilizer and spraying with its extract on qualitative characteristics potato yield, The experiment was implemented using factorial experiment(4×3×2) within Randomized Complete Block Design with three replicates, The First factor included the inoculation with Biofertilizers and cod it (M), which is(M_0) Don't be discouraged , (M_1) inoculation the Mycorrhiza in pollen density of 25 g.tuber^{-1} ,(M_2) inoculation the Trichoderma pollen density of 4 g.tuber^{-1}and (M_3)Mixture Mycorrhiza and Trichoderma fungi together, The second factor included the addition of Nile flower peat fertilizer and cod it (N), which is (N_0) without adding, (N_1)added 15 ton.ha^{-1} and (N_2) added 30 ton.ha^{-1}, The third factor included spraying with Nile flower peat fertilizer extract and cod it (E), spraying with distilled water(E_0) and spraying with extract 2 ml.L^{-1} (E_1), The results showed that biofertilization was significantly superior in qualities characteristics of the yield represented by dry matter percentage, starch percentage ,specific density, soluble solids percentage and tuber hardness, compared with control (M0), M3 treatment produced greatest value of this characteristics which reached 16.84%, 11.01%, 1.0640 g cm^{-2}, 6.459%, 8.167 kg cm^{-2}. Organic fertilization showed significant increase in the above characteristics, N2 treatment produced greatest value which reached 17.20%, 11.33%, 1.0657g cm^{-2}, 6.633%, 8.179 , kg cm^{-2}. Spraying treatment had amoral superiority on above characteristics if produced 16.50%, 10.70%, 1.0623 g cm^{-2}, 6.407% , 7.944 kg cm^{-2}.

Keywords: Starch, Specific Density, Trichoderma, Tuber hardness.

* **Part of Ph.D. Dissertation of the first author.**

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تأثير إضافة الاسمدة الحيوية وسماد خث زهرة النيل والرش بمستخلصه في الصفات النوعية لحاصل البطاطا

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الخلاصة

نفذت تجربة حقلية في مشروع محطة تطوير البستنة في الكوت التابع الى مديرية الزراعة في محافظة واسط للموسم الربيعي لسنة 2022 ، بهدف دراسة تأثير التلقيح ببعض الاسمدة الحيوية واضافة سماد خث زهرة النيل والرش بمستخلصه في الصفات النوعية لحاصل البطاطا، نفذ البحث كتجربة عاملية (4×3×2) وفق تصميم القطاعات الكاملة المعشاة وبثلاث مكررات، شمل العامل الاول التلقيح بالاسمدة الحيوية ورمز لها (M) ، وهي (M_0)عدم التلقيح و (M_1)التلقيح بفطرالمايكورايزا بكثافة لقاحية 25 غم0درنة$^{-1}$ و (M_2) التلقيح بفطرالترايكوديرما بكثافة لقاحية 4غم0درنة$^{-1}$ و(M_3)التلقيح بخليط فطري المايكورايزاوالترايكوديرما معا، وشمل العامل الثاني إضافة سماد خث زهرة النيل ورمز لها (N) , هي(N_0)عدم الاضافة و(N_1)إضافة 15 طن هـ$^{-1}$ و(N_2) إضافة 30 طن هـ$^{-1}$, وشمل العامل الثالث الرش بمستخلص سماد خث زهرة النيل ورمز له (E)، هي الرش بالماء المقطر (E_0) والرش بالمستخلص2 مل لتر$^{-1}$ (E_1) ، أظهرت النتائج تفوق التسميد الحيوي معنويا في الصفات النوعية للحاصل المتمثلة بنسبة المادة الجافة ونسبة النشأ ونسبة المواد الصلبة الذائبة الكلية والكثافة النوعية وصلابة الدرنات مقارنة بمعاملة القياس M0 ، وتميزت المعاملة M3 بإعطائها أعلى القيم لهذه الصفات بلغت 16.84% و 11.01% و 1.0640غم سم$^{-2}$ و 6.459% و 8.167 كغم سم$^{-2}$، وأظهرت معاملات التسميد العضوي زيادة معنوية في الصفات اعلاه , واعطت المعاملة N2 اعلى القيم بلغت 17.20% و 11.33% و 1.0657غم سم$^{-2}$ و 6.633 % و 8.179 كغم سم$^{-2}$ ، وتفوقت معاملة الرش E1 معنويا في الصفات اعلاه إذ اعطت 16.50 % و 10.70% و 1.0623غم سم$^{-2}$ و 6.407% و 7.944 كغم سم$^{-2}$0

الكلمات المفتاحية: نشأ، كثافة نوعية، ترايكوديرما، صلابة الدرنات.

INTRODUCTION

The potato crop (Solanum tuberosum L.) belongs to the Solanaceae family, which includes about 90 species and 2000 species. Potato is one of the most important vegetable crops in the world, especially in the Americas, Europe and some Arab countries, due to its abundance of productivity and the diversity of environmental conditions in which it grows and its value As potatoes are grown on a large scale and in various parts of the world, it is also a strategic crop, as it lead to important role in food security and has a high nutritional value because of the elements and food compounds it contains and is involved in many food industries. Economically, the potato crop returns to farmers with a return economically in a short period of time not exceeding 120 days, and it also provides job opportunities for many other sectors and employment of the labor force, which contributes to the national income of many countries (**Andrivon, 2017; Cirocki& Golebiewska, 2019**).

The total cultivated area in Iraq amounted to 24.12 hectares, with a total production of 674.8 thousand tons, with a yield of 27.978 ton.ha-1 (**Agricultural Statistics Directorate, 2021**) ,which are low rates in relation to the cultivated area unit due to the fact that the soil conditions are not ideal and the insufficient or lack of readiness of plant nutrients And lack of interest in service and fertilization operations, and from here it was necessary to think scientifically and deliberately to increase production per unit area with the introduction of the principle of recycling and preserving the environment, as the steady increase in the use of chemical fertilizers to achieve high production per unit area leads to negative results reflected in soil pollution Groundwater as a result of washing and air pollution through volatilization, which in turn leads to economic losses as a result of the loss of the bulk of fertilizers and severe damage to human, animal and microorganism health, as well as the high cost of

manufacturing, which prompted scientists to search for methods that are safe for human health and do not cause environmental pollution. Environment through sustainable and environmentally friendly agricultural practices. Therefore, many studies have suggested trying to use beneficial microorganisms in the rhizosphere to enhance plant productivity, rehabilitate degraded lands, as well as reclaim contaminated soil and produce healthy and safe food, and its role in removing heavy elements from the soil **(Adesemoy *et al.*, 2008; Singh *et al.*, 2017; Mohammed & Al-Shamary, 2017**). Diab (2012) found that inoculation with mycorrhiza fungus as a biofertilizer to the potato plant led to an increase in the tubers content of the percentage of dry matter, total soluble solids, and amino acids compared to uninoculated plants, Saniet al. (2020) showed that the inoculation of the tomato plant with Trichoderma fungus increased the yield and improved its quality.

From the results of the research, it was found that there is a linear relationship between the components of organic soil matter and functions of soil microorganisms. Hence, the use of biofertilizers complements the action of organic fertilizers in increasing agricultural production, improving its quality, and reducing environmental pollution **(Munda *et al.*, 2016**).Therefore, the concerns in many countries of the world tended to encourage organic production and take advantage of the natural resources available for the production of organic fertilizers by recycling them for the purpose of improving the physical, chemical and biological properties of the soil and the nutrients it contains for the purpose of increasing production and improving its quality., if found **Al-Halfi & Al-Azzawi (2022)** that the addition of organic fertilizer (palm fronds waste) has improved soil properties represented by increasing the stability of soil aggregates and increasing the available water. **And Al-Dulaiml & Al-Amri (2020)** showed that the addition of Monocarps plant residues had a significant effect on the vegetative growth rate and yield and the increase in the percentage of starch in potato tubers, **Saaseea& Al-Amry (2018)** noted that the addition of organic fertilizers (humic acid) to potatoes It increased the yield and improved its quality by increasing the percentage of dry matter and the percentage of protein.

Recent research focused on the use of organic nutrients that are sprayed on the shoots according to the foliar feeding method, which is an effective method in increasing the yield and improving its quality (**Abdulrasool & Al-Malikshah, 2022; Al-Mharib *et al.*, 2022**). And **Majeed (2010)** found that when spraying potato plants with the organic nutrient Vit-org, it increased the yield and improved its quality represented by increasing the number of tubers, tuber weight, tuber content of percentage of dry matter, total soluble solids, and tuber hardness. This study was aimed to investigate the effect of adding biofertilizersand Nile flower peat fertilizer and spraying with its extract and the interaction between them on the quality of potato yield.

MATERIALS AND METHODS

The research was carried out in the project of the Horticultural Development Station in the Kut district of the Directorate of Agriculture of Wasit Governorate, with the aim of studying the effect of adding some biofertilizers and Nile flower peat fertilizer and spraying with its extract on the qualitative characteristics of the yield of potato plant, Arizona variety, for the spring season 2022.

The study was carried out using the factorial experiment with three factors within the Randomized Complete Block Design (RCBD) and with three replications.

The first factor included four treatments for adding biofertilizers and cod it (M), which are M0 (without addition), M1 (inoculation Glomus mosseaeat a pollen density of 25gtuber-1), M2 (inoculation Trichoderma harzianum fungus in at a pollen density 4g tuber-1) and M3(Mycorrhiza + Trichoderma), biological fertilizers were added at the bottom of the tubers during cultivation, The second factor included three treatments for adding organic fertilizer (Nile flower peat fertilizer, and is symbolized by and cod it N, N0 (without addition), N1 (adding 15 ton ha-1 of organic fertilizer) and N2 (adding 30 ton ha-1of organic fertilizer), organic fertilizers were added before planting by digging a 20 cm deep incision at the top of the meadow and then mixed well with the soil, The third factor included two treatments of spraying Nile flower peat extract (E), which was prepared from organic fertilizer Nile flower peat, following the method of Page et al. (1982), and its treatments are E0 (spraying with distilled water) and E1 (spraying the extract). at a concentration of 2 ml L-1), and with three sprays, the first spray is in the vegetative growth stage, the second spray is in the tuber emergence stage, and the third spray is in the tuber size increase stage.

RESULTS AND DISCUSSION

Percentage of dry matter in tubers%

The results of (table, 1) showed that biofertilization had a significant effect on the percentage of dry matter in potato tubers, the double inoculation treatment (M3) produced the highest percentage of 16.84 % compared to the control treatment, which recorded the lowest rate of 15.56%. The organic fertilization treatments also affected this characteristic significantly compared to the control treatment, which produced the lowest percentage of 15.18 %, as the treatment N2 recorded the highest percentage of 17.20 %, and the spraying treatment with Nile flower peat extract (E1) significantly affected it produced the highest percentage of 16.50%, compared to the control treatment (E0), which produced 15.89%.

Achieved the interaction coefficients between biofertilizers and organic fertilizers had a significant effect on the percentage of dry matter in tubers, as the treatment M3 N2 recorded the highest percentage of 18.06%, and the control treatment M0N0 recorded the lowest percentage of 14.65%.

The results also showed that the interaction of biofertilizers with spraying with peat extract of Nile flower had a significant effect on this trait, as the treatment M3E1 recorded the highest rate of 17.13 %, and the control treatment (M0E0) recorded the lowest rate, amounting to 15.28%.

Also, the interaction treatments of organic fertilizers and spraying with peat extract of Nile flower had a significant effect on this characteristic compared to the control treatment (N0E0), which produced the lowest percentage of 14.89 %, as the treatment N2 E1 recorded the highest percentage of 17.48%.

The results of the triple ineraction also showed a significant effect on this trait, as the treatment M3N2E1 gave the highest percentage of 18.33 %, the control treatment produced the lowest percentage of 14.38 %.

Table (1): Effect of adding biofertilizers, Nile flower peat fertilizer, spraying with its extract and the interaction between them on Percentage of dry matter in tubers % for spring seasons 2022.

M1- Myco	N	E		M
M2- Tricho	(Ton.ha^{-1})	(ml.l^{-1})		×
		E0(0)	E1(2)	N
M0 (0)	N0 (0)	14.38	14.93	14.65
	N1 (15)	15.39	15.88	15.63
	N2 (30)	16.06	16.75	16.40
M1 (25g)	N0 (0)	14.88	15.53	15.21
	N1 (15)	15.75	16.54	16.14
	N2 (30)	16.89	17.42	17.15
M2 (4 g)	N0 (0)	14.91	15.49	15.20
	N1 (15)	15.80	16.57	16.19
	N2 (30)	16.93	17.45	17.19
M3 (M1+M2)	N0 (0)	15.39	15.90	15.64
	N1 (15)	16.48	17.17	16.83
	N2 (30)	17.79	18.33	18.06
		1.037		0.733
E		15.89	16.50	
LSD$_{(0.05)}$		0.299		
				M
M	M0	15.28	15.85	15.56
×	M1	15.84	16.50	16.17
E	M2	15.88	16.50	16.19
	M3	16.55	17.13	16.84
LSD$_{(0.05)}$		0.598		0.423
				N
N	N0	14.89	15.46	15.18
×	N1	15.85	16.54	16.20
E	N2	16.92	17.48	17.20
LSD$_{(0.05)}$		0.518		0.366

Starch percentage in tubers(%)

The results of table (2) indicated that the biofertilization treatments had a significant effect on increasing the percentage of starch in potato tubers, compared to the control treatment, which produced the lowest percentage of 9.871 %, as (M3) produced the highest percentage of 11.01 %, and organic fertilization treatments significantly affected this characteristic compared to the control treatment, which produced the lowest percentage of 9.525 %, as the N2 treatment producsd the highest percentage of 11.33, and the treatment of spraying with peat extract of the Nile flower affected (E1) was significant, as it produced the highest percentage of 10.70%, compared to the control treatment (E0), which produced 10.15% .

From the data from the same table, it was found that the interaction coefficients between M and N had a significant effect on the percentage of starch in tubers, as the treatment M3N2produced highest percentage of 12.09 %, and control treatment M0N0produced lowest percentage of 9.058 %.

The interaction of biofertilizers with spraying Nile flower peat extract also achieved a significant effect on this trait, as the treatment M3E1produced the highest percentage of 11.26 %, and control treatment (M0E0) produced the lowest percentage of 9.615 %.

It was also found that the overlapping treatments of N and E had a significant effect on this characteristic compared to the control treatment (N0E0), which produced the lowest percentage of 9.272 %, as the treatment N2E1produced highest percentage of 11.58 %.

The results of the triple interaction had a significant effect on increasing the percentage of starch in the tubers, as the treatment M3N2E1 produced the highest rate of 12.33%, and control treatment produced the lowest rate of 8.813%.

Table (2): Effect of adding biofertilizers, Nile flower peat fertilizer, spraying with its extract and the interaction between them on the Starch percentage in tubers (%) for spring seasons 2022.

M1- Myco	N	E		M
M2- Tricho	(Ton.ha^{-1})	(ml.l^{-1})		×
		E0(0)	E1(2)	N
	N0 (0)	8.813	9.303	9.058
	N1 (15)	9.714	10.15	9.933
M0 (0)	N2 (30)	10.31	10.92	10.62
	N0 (0)	9.266	9.844	9.555
	N1 (15)	10.03	10.74	10.38
M1 (25 g)	N2 (30)	11.05	11.52	11.28
	N0 (0)	9.291	9.803	9.547
	N1 (15)	10.08	10.77	10.42
M2 (4 g)	N2 (30)	11.08	11.55	11.31
	N0 (0)	9.718	10.16	9.942
	N1 (15)	10.69	11.30	10.99
M3 (M1+M2)	N2 (30)	11.85	12.33	12.09
LSD$_{(0.05)}$		0.924		0.653
E		10.15	10.70	
LSD$_{(0.05)}$		0.266		
				M
M	M0	9.615	10.12	9.871
×	M1	10.11	10.70	10.41
E	M2	10.15	10.70	10.43
	M3	10.75	11.26	11.01
LSD$_{(0.05)}$		0.533		0.377
				N
N	N0	9.272	9.779	9.525
×	N1	10.13	10.74	10.43
E	N2	11.07	11.58	11.33
LSD$_{(0.05)}$		0.462		0.326

Specific density of tubers (gcm-2)

In the results of (Table, 3) it was found that the biofertilization treatments had a significant effect on the specific density of potato tubers, as the M3 treatment gave the highest value of 1.0640 gcm-2, compared to the M0 that produced the lowest value of 1.0579 gcm-2. As for the effect of the organic fertilization treatments, it was significant in this respect compared to the control treatment, which produced the lowest value of 1.0561 gcm-2, as the N2 treatment produced the highest value of 1.0657 gcm-2, and the spraying treatment with peat extract affected the flower Nile (E1) was significant, as it produced highest specific gravity of 1.0623 gcm-2, compared to the control treatment (E0), which produced 1.0595 gcm-2

With regard to the effect of the interaction between biofertilizers and organic fertilizers, it was significant in increasing the specific density of potato tubers, as the treatment M3N2produced the highest value of 1.0697 g.cm-2, and control treatment M0N0 gave the lowest value of 1.0536 gcm-2.

Also, the interaction of biofertilizers with spraying with peat extract of Nile flower had a significant effect on this trait, as the treatment M3E1 gave the highest value of 1.0653 gcm-2, and control treatment M0E0 recorded the lowest value of 1.0566 gcm-2.

The overlapping treatments of organic fertilizers and spraying with Nile flower peat extract also achieved a significant effect on this trait, as treatment N2E1produced the highest value of 1.0670 gcm-2, and control treatment (N0E0) produced the lowest value of 1.0547 gcm-2.

The results of the same table indicated that triple interaction coefficients had a significant effect on increasing the specific density in tubers compared to control treatment, which produced the lowest value of 1.0523 gcm-2, as the treatment M3N2E1 gave the highest value of 1.0710 gcm-2.

Table (3): Effect of adding biofertilizers, Nile flower peat fertilizer, spraying with its extract and the interaction between them on Specific density of tubers (g.cm-2) for spring seasons 2022.

M1- Myco	**N**	**E**		**M**
M2- Tricho	**(Ton.ha^{-1})**	**(ml.l^{-1})**		×
		E0(0)	**E1(2)**	**N**
	N0 (0)	1.0523	1.0549	1.0536
	N1 (15)	1.0571	1.0594	1.0582
M0(0)	**N2 (30)**	1.0603	1.0635	1.0619
	N0 (0)	1.0547	1.0578	1.0562
	N1 (15)	1.0588	1.0626	1.0607
M1(25 g)	**N2 (30)**	1.0642	1.0667	1.0655
	N0 (0)	1.0548	1.0576	1.0562
	N1 (15)	1.0590	1.0627	1.0609
M2 (4 g)	**N2 (30)**	1.0644	1.0668	1.0656
	N0 (0)	1.0571	1.0595	1.0583
M3	**N1 (15)**	1.0623	1.0655	1.0639
(M1+M2)	**N2 (30)**	1.0684	1.0710	1.0697
LSD$_{(0.05)}$		**0.0049**		**0.0034**
E		1.0595	1.0623	
LSD$_{(0.05)}$		**0.0014**		
				M
M	**M0**	1.0566	1.0593	1.0579
×	**M1**	1.0592	1.0623	1.0608
E	**M2**	1.0594	1.0624	1.0609
	M3	1.0626	1.0653	1.0640
LSD$_{(0.05)}$		**0.0028**		**0.0020**
				N
M	**N0**	1.0547	1.0574	1.0561
×	**N1**	1.0593	1.0626	1.0609
E	**N2**	1.0643	1.0670	1.0657
LSD$_{(0.05)}$		**0.0024**		**0.0017**

Total soluble solids T.S.S (%)

Through the results of table (4) it was found that the biofetilizers treatments had a significant effect on the percentage of T.S.S in potato tubers for the season, compared to the M0, which produced the lowest rate of 6.017%, as the M3 treatment produced the highest rate of 6.459%, the organic treatments showed a significant effect on this trait compared to the control treatment, which produced the lowest percentage of 5.866%. the N2 treatment produced the highest rate of 6.633%, and the spraying treatment with Nile flower peat extract (E1) had a significant effect as it produced the highest percentage of 6.407% compared to the control treatment (E0), which produced 6.147%.

The results of the same table showed that the M3N2 was significantly superior by produced it a rateof 6.791%, and M0N0produced the lowest of 5.528%.

With regard to the coefficients of interaction of M with E, the treatment M3E1 was significantly superior by producing it the highest rate of 6.562%, compared to the M0E0, which produced the lowest rate of 5.851%.

The treatment of interaction N and E, N2E1 showed a significant effect on this trait, as it produced the highest rate of 6.756 %, compared to the N0E0, which produced the lowest rate of 5.745 %.

The treatment M3N2E1produced the highest percentage of 6.907 %, compared to control treatment M0N0E0 which produced the lowest percentage of 5.337 %.

Table (4): Effect of biofertilizers, Nile flower peat fertilizer, spraying with its extract and the interaction between them on Total soluble solids T.S.S (%) for spring seasons 2022.

M1- Myco	N	E		M
M2- Tricho	(Ton.ha^{-1})	(ml.l^{-1})		×
		E0(0)	E1(2)	N
	N0 (0)	5.337	5.718	5.528
	N1 (15)	5.912	6.275	6.094
M0 (0)	N2 (30)	6.304	6.557	6.431
	N0 (0)	5.780	5.994	5.887
	N1 (15)	6.206	6.506	6.356
M1(25 g)	N2 (30)	6.535	6.775	6.655
	N0 (0)	5.837	6.030	5.933
	N1 (15)	6.250	6.558	6.404
M2 (4 g)	N2 (30)	6.525	6.783	6.654
	N0 (0)	6.025	6.203	6.114
M3	N1 (15)	6.372	6.575	6.473
(M1+M2)	N2 (30)	6.675	6.907	6.791
LSD$_{(0.05)}$		0.683		0.483
E		6.147	6.407	
LSD$_{(0.05)}$		0.197		
				M
M	M0	5.851	6.183	6.017
×	M1	6.174	6.425	6.299
E	M2	6.204	6.457	6.331
	M3	6.357	6.562	6.459
LSD$_{(0.05)}$		0.394		0.279
				N
N	N0	5.745	5.986	5.866
×	N1	6.185	6.478	6.332
E	N2	6.510	6.756	6.633
LSD$_{(0.05)}$		0.341		0.241

Tuber hardness (kgcm-2)

The rates presented in table (5) show that the M3 and M2 biofertilization treatments had a significant effect on increasing the hardness of potato tubers, as M3produced the highest hardness score of 8.167, kgcm-2, while the M1 treatment caused a non-significant increase in this respect, compared with control treatment that produced the lowest hardness score of 7.544 kg cm-2. As for the effect of organic fertilization treatments It was significant in this respect, as the N2 treatment produced the highest value of 8.179 kg cm-2 compared to the control treatment, which produced the lowest value of 7.458 kg cm-2, and spraying treatment with Nile flower peat extract (E1) achieved a significant effect it produced the highest hardness score of 7.944 kgcm-2 compared to the control treatment (E0), which produced 7.657 kgcm-2 .

The results of the same table showed that the interaction between M and N had a significant effect, as the treatment M3N2produced highest value of 8.640kgcm-2, compared to the M0N0, which recorded the lowest value of 7.236kgcm-2.

The interaction of M with E had a significant effect on this trait, as the treatment M3E1produced the highest value of 8.333kgcm-2 compared to the control treatment M0E0, which gave the lowest value of 7.406kgcm-2.The interaction of N and E significantly increased tuber hardness compared to the control treatment, which produced the lowest value of 7.304 kgcm-2, as treatment N2E1produced the highest value of 8.316 kgcm-2.

The results of the same table showed that the M3N2E1produced a significant increase in tuber hardness, compared to the M0N0E0, which produced the lowest value of 7.036kgcm-2, as the treatment M3N2E1produced highest value of 8.768kgcm-2.

Table (5): Effect of adding some biofertilizers, Nile flower peat fertilizer, spraying with its extract and the interaction between them on the Tuber hardness (kgcm-2) for spring seasons 2022.

M1- Myco	N	E		M
M2- Tricho	(Ton.ha^{-1})	(ml.l^{-1})		×
		E0(0)	E1(2)	N
	N0 (0)	7.036	7.437	7.236
	N1 (15)	7.429	7.648	7.539
M0(0)	N2 (30)	7.754	7.958	7.856
	N0 (0)	7.260	7.464	7.362
	N1 (15)	7.528	7.715	7.621
M1 (25 g)	N2 (30)	7.852	8.215	8.034
	N0 (0)	7.337	7.668	7.502
	N1 (15)	7.637	7.908	7.773
M2 (4 g)	N2 (30)	8.046	8.323	8.184
	N0 (0)	7.583	7.877	7.730
	N1 (15)	7.908	8.353	8.130
M3 (M1+M2)	N2 (30)	8.512	8.768	8.640
LSD$_{(0.05)}$		0.639		0.452
E		7.657	7.944	
LSD$_{(0.05)}$		0.184		
				M
M	M0	7.406	7.681	7.544
×	M1	7.547	7.798	7.672
E	M2	7.673	7.966	7.820
	M3	8.001	8.333	8.167
LSD$_{(0.05)}$		0.369		0.261

				N
N	**N0**	7.304	7.611	7.458
×	**N1**	7.626	7.906	7.766
E	**N2**	8.041	8.316	8.179
$LSD_{(0.05)}$		**0.319**		**0.226**

Interpretation Results

The increase in the percentage of indicators of the qualitative characteristics of the potato plant yield, represented by the percentage of dry matter, starch, specific density, total dissolved solids, and tuber hardness, may be attributed to the role of biofertilizers (mycorrhiza fungus) in improving the physical, chemical, and biological soil characteristics, the secretion of organic acids, enzymes, antibiotics, and the production of some plant growth regulators. Increasing the readiness and availability of nutrients, increasing the surface area of the roots, and increasing plant resistance to stress (**Al-Badawi, 2008; Al-Gamas, 2018; Al-Mamori& Abdul-Ratha, 2020**)

All this prompted the plant to produce a strong vegetative system, an increase in the efficiency and outputs of the carbon metabolism process, and a better accumulation of nutrients and complex compounds such as carbohydrates, proteins, amino acids, and organic acids in the tubers, which led to an increase in the percentage of dry matter in the tubers, and this leads to a higher dry matter index in the tubers. In tubers, an increase in the percentage of starch, total soluble solids and specific density, and these characteristics are among the quality measures of potato tubers (**Alisdair & Willmitzer, 2001; Nora *et al.*, 2017**).

The reason for the increase in the proportions of the qualitative traits of tubers of potato plants inoculated with the Trichoderma fungus may be attributed to its role in the decomposition of organic matter in the soil to its simple components beneficial to the plant, microorganisms and soil **(Bhuvaneswari *et al.*, 2014**), and works to protect and strengthen host plants as a biological resistance factor (parasitism, competition, Antagonism, decomposition) against pathogens and insects (**Saleh & Farhan, 2022**), and it works to increase the availability and readiness of the elements in the soil and increase their accumulation in plant tissues (**Fadhl & Al-Hadithi, 2016**), and it has the ability to secrete auxins and cytokines and to build a dense root complex and total Strong vegetative, characterized by plant length, wide leafy area, a high concentration of chlorophyll , which allowed for a high build-up of carbohydrates, which led to an increase in yield and an improvement in its quality (**Saeed, 2015; Yasir & Al-Salihy, 2022**).

As for the increase in the proportions of the specific characteristics of potato tubers when organic fertilizers are added, the reason may be attributed to their content of macro and micro nutrients as well as their role in increasing the readiness of the elements in the soil solution and protecting them from washing and fixing and then the ease of plant access to them, through which the plant can build A strong root system that enables it to absorb the largest amount of elements in the soil to build a dense green vegetation that results in an increase in the level of synthetic carbohydrates, proteins, amino acids, nucleic acids (RNA and DNA) and growth regulators that the plant needs for its growth and development and storing the excess in tubers, which leads to Increasing its content of carbohydrates and proteins, and then increasing its dry weight, as the tubers in their stages of development become one of the most stored parts of the plant for carbohydrates and protein, especially when nutrients are continuously available and along the growth period (**Smita *et al.*, 2017; ShaniRaj *et al.*, 2019**).

Organic fertilizers have a role in increasing the permeability of cell membranes and facilitate the transfer of nutrients, especially nitrogen, phosphorus and potassium, to other parts of the plant. The element nitrogen leads to an increase in protein as a result of its union with cationic organic acids to produce amino acids that are the building blocks of protein, and the element phosphorus is included in the composition of energy compounds ATP, NADPH, and some important organic compounds in the oxidation and reduction processes during the vital activities of the plant, such as carbon metabolism, respiration, and carbohydrate metabolism. And proteins through its activation of the enzyme nitrate reductase, as well as the elements calcium and magnesium have an active participation in the pathways of formation of sugars and protein (**Taiz & Zeiger, 2010; Sawickaetal., 2019; Ilyas *et al.*, 2021**).

The high indicators of the qualitative characteristics of potato tubers when spraying plants with Nile flower peat extract may be attributed to what it contains of nutrients (macro and micro), organic acids, amino acids, sugars and vitamins, and their effect on improving vegetative growth indicators, especially increasing levels of total chlorophyll and leafy area, which stimulated the plant to seize the largest amount From the light and then increasing the rates of the carbon metabolism process, which led to an increase in the amount of processed carbohydrates, from which the surplus is transferred to the stored part (tubers) (**Al-Sahaf, 1989**), which in turn led to a high response of the chemical characteristics of the tubers, especially the dry matter that forms the product. The final process of metabolism and metabolism, and the increase in the percentage of dry matter in the tubers results in an increase in the qualitative indicators (**Al-Zaidy & Al-Ubaidy, 2017; Al-Ubaidy *et al.*, 2019; Shayaa & Hussein, 2019; Al-Mharib *et al.*, 2021**).

CONCLUSIONS

Biofertilizers improved the quality of potato tubers, the double inoculation treatment gave the best results, also organic fertilization improved the quality of potato tubers, the treatment at a level of 30 ton. ha-1 gave best results, spraying with Nile flower peat extract improved the quality characteristics of potato tubers, the treatment of three factors interaction achieved the highest results.

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المجلة العراقيه لبحوث السوق
وحماية المستهلك

INTERNAL MARKETING AS AN ENTRANCE TO ACHIEVING SUSTAINABLE HUMAN DEVELOPMENT/ APPLIED RESEARCH IN AL-FURAT STATE COMPANY FOR CHEMICAL INDUSTRIES

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ABSTRACT

The research was conducted at Al-Furat General Chemical Industries Company (one of the formations of the Ministry of Industry and Minerals) with the aim of analyzing the relationship between internal marketing variables (employee recruitment, training, incentives, management support, clarity of marketing information) and the possibility of achieving the principles of sustainable human development (empowerment, social justice and equity)., cooperation, sustainability, safety and job stability) through an opinion poll that was distributed to a random sample of forty employees in that company. We adopted the electronic questionnaire as a tool for collecting data from that sample. The data we obtained was analyzed using percentages, trying to diagnose points. Weakness in internal marketing indicators, and this was evident from the results of the incentive incentives index and weak marketing information, as their percentages ranged from average to below that, while some of the indicators of management support and training courses for employees recorded a relatively high approval rate.

Accordingly, we recommend the need to direct the attention of the company's senior management to the indicators of incentives and clarity of marketing information, since their results were relatively lower than the rest of the indicators, knowing that these indicators are directly related or considered an important input to achieving the principles of sustainable human development such as cooperation, empowerment, safety, stability, and fairness.

Keywords: training, management support, incentives, empowerment, equity, social justice.

التسويق الداخلي كمدخل لتحقيق التنمية البشرية المستدامة/ بحث تطبيقي في شركة الفرات العامة للصناعات الكيمياوية

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الخلاصة

انجز البحث في شركة الفرات العامة للصناعات الكيمياوية (احدى تشكيلات وزارة الصناعة والمعادن) بهدف تحليل العلاقة بين متغيرات التسويق الداخلي (تعيين العاملين، التدريب، الحوافز التشجيعية، دعم الإدارة، وضوح المعلومات التسويقية) وإمكانية تحقيقها لمبادئ التنمية البشرية المستدامة (التمكين، العدالة الاجتماعية والانصاف، التعاون، الاستدامة، الأمان والاستقرار الوظيفي) بواسطة استطلاع رأي تم توزيعه على عينة عشوائية من العاملين في

تلك الشركة بلغ عددهم أربعون فردا. لقد اعتمدنا الاستبانة الالكترونية كأداة لجمع البيانات من العينة العشوائية، اذ تم تحليل بياناتها باستخدام النسب المئوية محاولين تشخيص نقاط الضعف بمؤشرات التسويق الداخلي، وهذا ما تبين من نتائج مؤشر الحوافز التشجيعية، وضعف المعلومات التسويقية، حيث كانت نسبها تتراوح بين المتوسط الى ما دون ذلك، في حين سجلت بعض من مؤشرات دعم الإدارة والدورات التدريبية للعاملين نسبة موافقة مرتفعة نسبيا. عليه، توصلنا الى مجموعة من التوصيات منها؛ ضرورة توجيه اهتمام الإدارة العليا في الشركة الى مؤشرات الحوافز التشجيعية ووضوح المعلومات التسويقية لكون نتائجها كانت اقل نسبيا من بقية المؤشرات، علما ان تلك المؤشرات ترتبط مباشرة او تعد مدخلا هاما لتحقيق مبادئ التنمية البشرية المستدامة كالتعاون والتمكين والأمان والاستقرار والانصاف.

الكلمات المفتاحية: التدريب، دعم الادارة، الحوافز، التمكين، الانصاف، العدالة الاجتماعية.

INTRODUCTION

Internal marketing is one of the contemporary administrative approaches. It appeared during the fifties of the twentieth century, then crystallized as a stand-alone concept at the beginning of the eighth decade of the twentieth century. This concept views workers within business organizations as internal customers whose satisfaction it is important to achieve the organization's goals. Employee satisfaction is embodied through training mechanisms, promotional incentives, management support for them, and listening to their visions in developing work, convinced of the important role they play in improving work production and productivity, on the one hand, and on the other hand, the great challenges that these organizations face, represented by technological developments and the intensification of competition, which... It requires confronting all of these issues by trying to gain the satisfaction of employees, in preparation for achieving adaptation to the challenges of the external environment in order to ensure its survival in the market. Paying attention to indicators of the organization's internal environment by applying internal marketing approaches would work to achieve the principles of sustainable human development, meaning that there is a direct link between internal marketing indicators and those principles.

Al-Furat General Company for Chemical Industries (one of the formations of the Ministry of Industry and Minerals) was chosen as a field for applying the research. In its theoretical part, we shed light on the concepts of internal marketing, its importance, sustainable human development and its principles, to clarify the importance of applying these indicators for their important role in achieving the principles of empowerment, cooperation, security, stability and fairness. Social justice is a means to achieve sustainable human development. The research was divided into four axes. Within the first axis, we presented the research methodology, then in the second axis, we highlighted the theoretical framework of internal marketing concepts, their importance, and the principles of sustainable human development. In the third axis, we addressed the applied field of research, then in the fourth axis, we presented the most important conclusions and recommendations that we reached.

First: Research Methodology

Research Problem

The research problem includes the following questions:

a. Does Al-Furat Chemical Industries Company adopt internal marketing standards?
b. Do internal marketing standards at Al-Furat Chemical Industries Company contribute to achieving the requirements of sustainable human development?

Research importance

The importance of the research is as follows:

a. The addition of knowledge that research can contribute to enriching the scientific library.
b. Providing ideas to senior management about the importance of internal marketing, and the benefits that can be achieved by companies when applying its indicators in light of the recommendations reached.
c. Opening new horizons for researchers to expand the discussion of topics related to internal marketing and its relationship to indicators of sustainable human development.

Research objectives

Emphasizing the importance of internal marketing in all its standards, in addition to alerting decision-makers in the company to the importance of developing plans and programs to pay attention to it, because of its role in developing the company and achieving the requirements of sustainable human development.

Research hypothesis

There is an application of internal marketing programs (as an independent variable) in Al-Furat General Chemical Industries Company, which works to achieve sustainable human development indicators (as a dependent variable). To test this hypothesis, sub-hypotheses were built:

a. There is a high level of mechanism for appointing employees in the company and achieving sustainable human development.
b. There is a high level of qualification and training programs in the company, achieving sustainable human development.
c. There is a high level of incentive programs in the company, achieving sustainable human development.
d. There is a high level of administrative support mechanisms for the company’s employees, and achieving sustainable human development.
e. There is a high level of clarity in the company's marketing communication mechanisms, and achieving sustainable human development.

Research analysis methodology

The descriptive analytical approach was relied upon to answer the research questions, in addition to the statistical aspect

The temporal and spatial limits of research

Spatial boundaries: Al-Furat Chemical Industries Company. Time limits: the period from January 2022 to October 2022.

Description of the sample

The research sample consisted of some employees of Al-Furat Chemical Industries Company, numbering forty individuals.

Second: Definitional concepts: internal marketing and sustainable human development

1. The concept of internal marketing

The concept of internal marketing appeared during the eighties of the twentieth century (as a relatively recent concept) and some writers have defined it as a group of activities carried out by organizations to provide their employees with the skills of a clear understanding of the mission, goals and tasks that the organization wants to achieve, through training, reward and incentive mechanisms **(Bayda & Hala, 2017**). It is also known as the mechanisms of mutual coordination between the organization and its employees to achieve external success with clients or clients. While others defined it as the efforts and activities undertaken by the organization to develop internal customers (employees) to achieve their job satisfaction, and to establish an effective communication channel between them to make each organizational unit within the organization capable of marketing its capabilities and capabilities to other units within the same organization. (**Al-Taweel. 2010; Al-Heali & Husain, 2021).**

Therefore, it can be said that internal marketing is an advanced management system whose goal is to achieve the satisfaction of employees in the organization, by developing and developing their skills by giving them knowledge of the organization's affairs, which can lead to gaining customer satisfaction in order to gain a share in the external market.

What is the importance of internal marketing?

Management scholars divide the benefits that can be obtained from applying internal marketing mechanisms in any organization into two groups: The first relates to the benefits that can accrue to the organization, while the second relates to the benefits that accrue to its employees. The following is a summary of both groups:

The first group: Benefits obtained by the organization

Management experts agree on the necessity of the organization adopting internal marketing methods, as it is one of the management methods through which it can obtain market share and a tool that enhances its competitive position. This will naturally improve its level of performance, guarantee appropriate profits, and also achieve its set goals.

Therefore, it can be said that applying internal marketing mechanisms for the organization will have a positive impact through three axes:

• **Managing changes**: The organization may suddenly resort to making fundamental changes in the nature of its work, without these changes being planned, for example introducing new production lines or using advanced technology with the aim of raising the level of its profits or keeping pace with sudden developments in the market. Here, internal marketing has an essential role. By accepting and making these changes successful, if the organization develops and develops a culture of dialogue, which leads to communication with employees and giving them sufficient confidence to present ideas and initiatives that contribute to the development of work **(Hussein &Lafta. 2019)**

• **Improving and building the organization's image in the market**: Internal marketing has an essential role in improving and building the organization's image in the market, which contributes to strengthening its strategic position, entering the market effectively, facilitating access to various resources at lower costs, and directing individuals' behavior to increase productivity, as internal marketing has an important role through Internal communication and introducing the organization's points of distinction to its employees, so it is said that all employees are potential ambassadors for the organization (**Lafta, 2016).**

The organization's strategy: Internal marketing mechanisms work to reduce the intensity of functional conflict within the organization, through the prevailing spirit of cooperation and coordination, which contributes to implementing its strategy in the best way, as well as implementing the promises that the organization makes, whether those promises concern its employees, or what is related to the environment External.

2. The importance of internal marketing for the organization's employees

The organization's employees obtain many benefits and advantages as a result of implementing internal marketing programs, the most important of which are:

• **Achieving employee satisfaction**: One of the most important advantages that result from applying internal marketing programs is achieving satisfaction and a sense of stability among employees. By researching the needs and requirements of employees and working to meet them by the organization, this will contribute to gaining employee satisfaction (**Lafta *et al*., 2021).**

• **Developing and improving the level of employee performance**: Employees are the first to benefit from internal marketing programs because they work to provide all appropriate conditions, in addition to training and development programs and other activities that fall within internal marketing programs that contribute to improving their performance and thus increasing their pride in the work they perform in a way that works to achieve Developing human capital to activate the organization's activity through an effective marketing vision. (**Al-Heali & Husain. 2021; Shahwani *et al*., 2020)**

• **The employee's feeling of the meaning of the job**: Internal marketing programs generate a feeling in the employee of the importance of the tasks and duties that he performs within the organization to achieve its goals. Thus, his view of the meaning of work changes, which makes him seek self-realization and excellence at work. Accordingly, the importance of internal marketing can be divided into two levels. The first is strategic and aims to create an organizational environment in which workers' awareness of the importance of customer service prevails. As for the second, it is tactical and its importance lies in paying attention to the internal work environment **(Halili & Abdel-Razzaq. 2018).**

Therefore, internal marketing is of great importance because it aims to satisfy the needs and desires of working individuals, to achieve job satisfaction and achieve the organization's goals.

Internal marketing mechanisms

The most important internal marketing mechanisms can be summarized as follows:

• **Employee recruitment policy**: The employee recruitment policy is one of the most important internal marketing mechanisms, as it is the key to the organization's success and a means that enables it to achieve competitive advantage by selecting the competent and appropriate human element for the job. The proper selection and distribution of human resources within the organization, each according to his ability, and placing the right person in the right place reflects positively on the performance of the employees in the organization. Hence, the selection process receives great care and attention from the organization due to the negative consequences of the wrong choice.

• **Qualification and training programs**: Qualification and training programs for workers are one of the means for them to acquire skills and develop their abilities, and in-service training is a necessity imposed by developments in work in the organization, and the changes that occur in the market force the organization to implement advanced production and service systems, provided that all of this is governed in light of the needs. the Actual.
• **Incentives and rewards**: If the wage or salary is the compensation that an individual receives as a value for the job he occupies, then the incentive is the return that he receives as a result of excellence in performance, and therefore we find that the organization's applications for the practice of internal marketing is to care for its employees and not only ask them, but also reward them. If a service organization wants its work team to be oriented towards customer service, it must make every effort to develop an effective reward system, acknowledge the efforts of its workers, and treat them as part of the organization's family (**Lafta & Hussein, 2017**).
• **Marketing information**: The existence of an interactive, mutual communication of information between management and workers requires establishing a mechanism in which marketing information is disseminated among them. Communication tools with employees are the tangible and most important aspect of internal marketing, as we note that most organizations, especially service ones, give great attention to internal communication in order to Providing employees with the necessary information about work to develop programs and services to deliver high-quality service, as well as accessing and expanding internal communications, is an important matter in building and providing service. It has been shown experimentally to meet customer expectations, and thus employees are well aware of their role. Employee activities are considered essential to provide quality service, and based on It is assumed that the marketing information system contributes to disseminating information to employees, so that they are able to provide services to customers. This requires management to provide marketing information that is quantitative and qualitative, and characterized by accuracy, so that it includes the services that the organization intends to promote so that they are aware of them so that they contribute to Providing benefits to customers.
• **Internal communication**: It is the work network through which information is collected and transmitted easily and conveniently within the organization, to contribute to effective decision-making.
• **Administrative support**: Senior management's full support for its employees with the aim of motivating them to complete work with enthusiasm and confidence by providing an appropriate environment for work, in which a culture of trust and cooperation prevails, exerting more effort and working in a single team spirit, and achieving continuous communication and attention in addition to personal relationships with employees **(Mohamed & Hamid, 2022).**

Sustainable human development

The concept of sustainable human development is based on two basic principles: The first is that humans are the focus of the development process. True development cannot be achieved without the development of the human element **(Hussein, 2017)**. The second is that development must guarantee the rights of the current generation and future generations, which means that the goal of sustainable human development is to expand the options and capabilities of individuals, through distribution mechanisms. The returns to growth are fair and just among members of society **(Al-Bustani, 2009**; **Shaban, & Hussien, 2020**)

Sustainable human development is concerned with achieving the following: (**United Nations Development Programme, 1997; Al-Douri & Saleh, 2019; Al-Heali, *et al.*, 2022)**

a. Cooperation: It focuses on and is concerned with the work mechanisms of individuals within the framework of their cooperation and interaction to achieve the goals of the organization or institution in which they work, and enhances their sense of belonging to it.
b. Social justice and equity: Achieving social justice through individuals' access to health, education, and training services and an income consistent with the work they do.
C. Safety and stability: It focuses on individuals obtaining an element of safety from everything that threatens their lives from sudden fluctuations, which works to achieve their stability, which will naturally contribute to increasing their productivity.
d. Sustainability: concerned with ensuring that the current generation receives the benefits of development, without compromising the rights of future generations.
e. Empowerment: Providing the opportunity for individuals to build their educational and cognitive capabilities with the aim of expanding their options in a way that develops work methods towards making changes to improve the quality of the product provided to the customer, which means the participation of employees in making decisions that achieve the organization's goals.

Third: Results

1- Description of the research sample

The research was applied in Al-Furat Chemical Industries Company, which is one of the formations of the Ministry of Industry and Minerals, to a random sample of workers whose description is shown in (Table, 1).

Table (1): Personal information of the research sample.

Gender									
Male				**Female**					
Frequency		%		Frequency		%			
23		57.5		17		42.5			
Age									
36-45 year		**46-55 year**				**56-60 year**			
Freq.	%	Freq.	%			Freq.	%		
16	40	21	52.5			3	7.5		
Academic achievement									
PhD		**master**		**BSc.**		**Diploma**		**Secondary school**	
Freq.	%	Freq.	%	Freq.	%	Freq.	%	Freq.	%
1	2.5	4	10	30	75	3	7.5	2	5
الخبرة									
less than 5 years		**11-15 years**				**16 years or more**			
Freq.	%	Freq.	%			Freq.	%		
1	2.5	3	7.5			36	90		

(Table, 1) includes the demographic distribution of the research sample, where we note:

a. Males constituted about 57.5% of the sample, while females constituted about 42.5%.

b. The age group of 46-55 years constituted the largest percentage of the research sample, reaching about 52.5%, and the age group of 56-60 years was the least, amounting to about 7.5%.

c. The percentage of those holding a bachelor's degree was about 75%, which means that three-quarters of the research sample held a preliminary certificate, while the percentage of those holding a doctorate degree was about 2.5%, which is the lowest percentage, while the rest of the certificates were distributed to holders of a master's degree, 10% and 7.5% for diploma certificates. And the secondary school certificate is 5%.

d. Regarding years of experience, it was highest for workers with years of experience of about 16 years or more, at 90%, followed by 7.5% for those with years of experience from 11-15 years, and the percentage of those with less than five years was the lowest, as they constituted about 2.5%.

Table (2): Distribution of the research sample according to participation in training courses

Participation in training courses	(%)
yes	90
No	10

We notice from the data in Table 2 that the largest percentage of the company's employees have participated in training courses, as they constituted about 90% of the sample. This is one of the positive things that works to provide them with the required skills, whether in their field of work or to develop their personalities in a way that helps the company achieve its goals.

Table (3): The relationship between internal marketing standards and sustainable human development indicators

		Agree	Neutral	not Agree
Sustainable human development indicators	**Internal marketing standards**			
	First: The mechanism for appointing employees	(%)	(%)	(%)
Equality	1. The competence and skills that I possess were the reason why I was chosen by the company's management for the current job.	67.5	25	7.5
Fairness	2. My educational qualifications match the position I hold.	82.5	5	12.5
Fairness	3- The experience I have is appropriate for the job I hold.	87.5	10	2.5
Enable	4. I have full knowledge of my current job duties.	87.5	12.5	-
Fairness	5. The company's principle to achieve its goals is to put the right person in .the right place	42.5	45	12.5
Enable	6. There is a job description for every employee in the company.	65	27.5	7.5
Fairness and empowerment	The average	72	21	7
	Second: Training and qualification			
Enable	1. There is an annual plan to train the company's employees.	82.5	10	7.5
Enable	**2**. The company organizes training courses for new employees before they join work.	52.5	20	27.5

Enable	**3.** There is a connection between the nature of my work and the training courses I take.	70	22.5	7.5
Enable	4. I gained new skills as a result of joining training courses.	75	17.5	7.5
Empowerment and sustainability	5. The company's senior management is keen to develop my skills and abilities in customer service.	62.5	27.5	10
halves	6. The company offers material or moral incentives when I excel in the training courses in which I participate.	40	32.5	27.5
Empowerment, sustainability and equity	The average	64	22	14
	Third: Encouragement incentives			
halves	1. The company grants material and moral incentives to employees who provide outstanding results (based on objective criteria).	42.5	35	22.5
halves	2. Nepotism plays a role in the company's employees receiving incentives (cash amounts or letters of thanks and appreciation).	25	42.5	32.5
Safety and stability	3. The wages I receive are commensurate with the nature of the work I perform in the company.	70	22.5	7.5
Safety and stability	4. There is a health insurance system for the company's employees.	57.5	20	22.5
cooperation	5- The company's management shares personal occasions with me (whether happy or sad).	72.5	20	7.5
halves	6- There is annual leave in place for employees according to the company's regulations and laws.	47.5	40	12.5
Cooperation, fairness, safety and stability	The average	52.5	30	17.5
	Fourth: Administrative support			
cooperation	1- My direct supervisor helps me find solutions to the difficulties I face during work.	90	-	10
Empowerment and equity	2. The company provides the necessary facilities for better work performance.	75	15	10
Safety and stability	3. I am introduced to all the benefits and programs available within the company.	60	30	10
cooperation	4. There are good relations between the direct manager and subordinates in the company.	85	12.5	2.5
Enable	5. 5- I can express my point of view at work to my direct supervisor without hesitation.	92.5	2.5	5
cooperation	6- Working as a team spirit is prevalent within the company.	77.5	17.5	5
Cooperation, empowerment, fairness, safety and stability	The average	80	13	7
	Fifth: Clarity of marketing information			
Enable	1. The company's employees are fully aware of customers' needs.	62.5	30	7.5
Enable	2. The company's senior management informs its members of the products it intends to promote.	77.5	15	7.5
Enable	3. The company's senior management provides complete information about new services by all means.	62.5	30	7.5
cooperation	4. The company allows workers to communicate with each other to exchange information	75	25	-
Enable	5. The company's senior management listens to employees' suggestions and studies them in depth.	62.5	25	12.5
Enable	6- The company's management holds periodic meetings with employees to listen to their proposals.	62.5	15	22.5
Empowerment and collaboration	The average	67	23	10

(Table, 3) shows the results of analyzing the questionnaire data that was distributed to a random sample of the company's employees. Through these results, we attempt to shed light on

the dimensions of internal marketing applied in the company and the possibility of it being a gateway to achieving sustainable human development indicators, as follows:

a. Mechanisms for appointing employees in the company

The highest approval rate was 87.5% for the two indicators: "I have full knowledge of my job tasks" and "My experience is compatible with the job I hold," as both dimensions are linked to the principles of social justice, equity, and empowerment (sustainable human development indicators), which means that the company was able to achieve sustainable human development indicators. Through the mechanisms you use to appoint workers or distribute them to various jobs, this is of course linked to expanding the capabilities of workers and ensuring that they perform the tasks entrusted to them to the fullest extent. It also achieves the principle of social justice and equity.

The criterion "My degree and experience are compatible with the job I hold" received an approval rate of 82.5%, which is a very good percentage, due to its direct connection to achieving the principles of sustainable human development, which is social justice and equity.

The sample members were divided regarding the application of the criterion "The company's principle is embodied in placing the right person in the right place," as the percentage of those who agreed constituted about 42.5%, and the percentage of those who were neutral and disagreed was 45% and 12.5%, respectively. This result is due to the connection of this criterion with one of the principles of sustainable human development, which is social justice and fairness, which means that this dimension, as one of the dimensions of internal marketing, did not contribute to achieving the principle of fairness. Therefore, the company's senior management is required to pay attention to addressing this by changing its work mechanisms by appointing workers in various positions.

The criteria "The competence and skills that I possess were the reason for my selection by the company's management for the current job" and "There is a job description for every employee in the company" also received a moderate degree of approval, as the percentage of those who agreed was about 67.5% and 65%, respectively, and both are related to the principles of social justice. Fairness and empowerment, and this also indicates the existence of a defect on the part of the company's management in this aspect, and requires treatment before it becomes more severe.

The arithmetic rate of the sample's responses with the word "agree" to the six paragraphs was about 72%, and these paragraphs had a correlation with the indicators of equity and empowerment (sustainable human development indicators), which indicates the need to pay more attention to this topic, especially to those paragraphs whose percentage was relatively small, so it cannot be accepted. The hypothesis that states that there is a high level of appointment mechanisms and achieving sustainable development indicators, is that the level was not high, but good.

b. Training and qualification

Most of the internal marketing standards within training and qualification are linked to the principle of empowerment as one of the principles of sustainable human development. We note that the highest percentage, 82.5% of the sample, agreed to "the existence of an annual plan to train employees in the company." This, of course, is a positive indicator, meaning that there is Interest in the field of training workers and providing them with the skills required to perform the work assigned to them.

Regarding the new skills that workers acquire from their participation in training courses, we note that 75% of the sample responded with approval, then the approval rate was about 70% due to the connection between the nature of the workers' work and the course in which they participate, and these two indicators are related to the principle of empowerment.

The approval rate for one of the internal marketing indicators was about 62.5% for the statement, "The company's senior management is keen to develop my skills and abilities in customer service." This is related to the principle of empowerment. This indicates the need for the company to be interested in holding training courses for employees aimed at developing the capabilities of employees in the field of customer service. This is very necessary because the customer is the one who works to sustain the company's work.

Regarding the indicator "The company provides material or moral incentives for the excellence of participants among the company's employees within the training course programs," it obtained an approval rate of 40% from the sample. This is a negative indicator that senior management must pay attention to its relationship to fairness in order to achieve justice. Therefore, it is not Attention must be paid to this aspect because it will contribute effectively to achieving sustainable human development indicators.

We also note the company's lack of interest in putting new workers in training courses before they join their new jobs, as the percentage of those who agreed was about 52.2%. This is of course closely related to the principle of empowerment, so it is necessary to pay attention to this aspect, as it works to expand the capabilities of workers and contributes to increasing productivity and achieving the quality element.

Therefore, it can be said not to accept the research sub-hypothesis, which states that there is a high level of training and qualification mechanisms and the achievement of sustainable human development indicators. The rate obtained was 64% with an average degree, which indicates the need for the company to give importance to this topic.

c. Incentives

The results of Table 3 regarding the dimension of incentives and their relationship to indicators of sustainable human development indicate that the highest percentage of agreement for the research sample was 72.5% on "The company's management shares with me personal occasions, whether they are happy or sad." This is a good indicator, especially in the area of its connection to cooperation. As one of the principles of sustainable human development.

As for the indicator "The level of wages I receive is consistent with the nature of the work I perform" and "A health insurance system is available for workers," the approval rate for them was 70% and 57.5%, respectively, and both are related to security and stability.

We conclude from this that the company's management must review the wage standard as one of the internal marketing standards, and try to address it through rewarding rewards for workers who complete their work or innovate work mechanisms that contribute to increasing productivity, as well as the importance of moving to provide a health insurance system, through national insurance companies, and both. Treatments would achieve the satisfaction of the company's employees.

We note within the third criterion, "Favouritism and its relationship to providing material and moral incentives," that 25% of the sample agreed, compared to 42.5% and 32.5% whose answers were neutral and disagreed, respectively. This indicates that three-quarters of the sample were between rejecting and neutral, so there is a clear division in the application. This indicator, which is linked to one of the principles of sustainable human development,

which is fairness, which means that this topic must be given importance by senior management, to achieve employee satisfaction, and then achieve sustainable human development. Likewise, with regard to the indicator of annual leave given to workers, we note that the sample is divided between agree, neutral, and reject, with percentages of 47.5%, 40%, and 12.5%, respectively. This, of course, is linked to fairness, as one of the principles of sustainable human development. This requires more attention to this aspect.

Therefore, the sub-hypothesis that states that there is a high level of the standard of incentives that the company grants and indicators of sustainable human development cannot be accepted, because this dimension received an acceptable rate, as the acceptance rate for the company's applications was 52.5%.

d. Management support. The percentage of those who agreed was about 92.5% of the sample with the statement "I can express my point of view at work in front of my direct supervisor without hesitation." This is an important indicator because it is linked to management, which gives freedom of expression to workers, and the relationship of this to the empowerment indicator, which contributes to expanding workers' capabilities and options. To get to the best of them.

The percentage of those who agreed with the criterion "My direct supervisor helps me find solutions to the difficulties I face during work" was about 90%, and this criterion is directly linked to the cooperation index as one of the indicators of sustainable human development. Likewise, with regard to the same cooperation indicator, we note that about 85% of the sample agreed with the statement, "There are good relations between the direct manager and subordinates in the company." This is, of course, an important indicator that works in favor of strengthening the principle of internal marketing for employees and achieving the principles of sustainable human development.

The percentage of those who agree that the company provides all facilities to achieve better performance for employees was about 75%, and this is related to the principles of fairness and empowerment. The percentage of those who agree that work is carried out in the spirit of one team was about 77.5%, and this is related to cooperation as one of the principles of sustainable human development.

The percentage of those who agreed with the phrase "I am introduced to all the benefits and programs available within the company" was about 60%, and this is related to security and stability. This requires the company's senior management to give this issue importance, whether through meetings with employees to introduce them to the details of work, as well as paying attention to simplifying work procedures and organizing Introductory brochures and brochures about jobs and their descriptions, in addition to the continuous rotation of workers to learn about the details of the company's work.

The sub-hypothesis of the research can be accepted, which states that there is a high level between the application of internal marketing programs in terms of supporting management and achieving sustainable development indicators, due to the arithmetic rate of those who agreed to its paragraphs in this axis, as their percentage constituted about 80%. Despite this, the company must define the employees. With all the privileges, rights and programs available in the company, since the percentage that received approval was average.

H. Clarity of marketing information. It is clear from the data in Table 3 that more than three-quarters of the research sample agreed with the statement, "The company's senior management informs its members of the products it intends to promote," as their percentage

reached 77.5%. This is a positive paragraph, especially since it is linked to empowering employees and informing them of the mechanisms Working in the company with the aim of increasing their capabilities. Then came the item "The company allows employees to communicate with each other to exchange information" at about 75%, and this is related to cooperation as one of the indicators of sustainable human development.

The percentage of approval reached about 62.5% for each of the internal marketing indicators: "The company's employees are fully aware of customers' needs," "Senior management listens to employees' suggestions," and "The company's management holds periodic meetings with employees to listen to their suggestions," each of which is linked to empowerment as one of the principles of development. Sustainable humanity, therefore it is necessary for senior management to pay attention to applying these two indicators in the company.

Thus, it can be said that the sub-hypothesis was not met, as it stated that there is a high level of application of internal marketing standards with regard to the clarity of marketing information and achieving sustainable human development indicators, as the arithmetic average for the percentage of those who agreed reached about 67%, which is an average rate. Accordingly, it will not be possible to To achieve the associated human development indicators in the company.

Fourth: Conclusions and recommendations

a. Conclusions

1. Internal marketing is one of the advanced administrative systems that aims to achieve employee satisfaction in business organizations.
2. The administrative system for internal marketing includes a set of indicators, including training, qualification, incentive rewards, appointment mechanisms, and administrative support, and everything that can contribute to achieving employee satisfaction within business organizations.
3. Some of the administrative support index mechanisms used at Al-Furat Chemical Industries Company achieved the highest approval rate, as the approval rate for the mechanism "I can express my point of view on issues related to work to my direct supervisor without hesitation" reached a percentage of 92.5, followed by a percentage of 90 for the indicator that my direct supervisor helps me in Finding solutions to the difficulties I face during work, which is of course a positive indicator because it is linked to the principles of achieving sustainable human development, which are the principles of cooperation and empowerment.
4. The results achieved for the incentive incentives and rewards index were not at the required level in Al-Furat Chemical Industries Company, as its mechanisms received varying degrees of approval, ranging from good to average, and some of them reached a percentage of about forty percent.

b. Recommendations

1. The need for the company to find appropriate mechanisms regarding the incentives allocated to employees because they contribute to achieving their satisfaction, and then link them to indicators of sustainable human development in terms of fairness, safety and stability.
2. The company needs to focus attention on the issue of clarity of marketing information.

3. Paying attention to organizing training courses for new employees in the company, as it is important in providing them with the required skills that will enable them to perform the work that will be allocated to them.
4. Listening to the opinions of employees and holding periodic meetings are among the indicators of achieving internal marketing. The company's management must set timetables for this, because it will inform the company of all the problems that may occur in order to find solutions to them, before they escalate and reach the crisis stage.
5. The company must study the weak points and try to find appropriate solutions for them, and it can seek the help of academic experts to contribute to this.

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Ibrahim & Khalaf

المجلة العراقيه لبحوث السوق
وحماية المستهلك

SYNTHESIS AND CHARACTERIZATION OF SOME HETEROCYCLIC COMPOUNDS DERIVED FROM METOCLOPRAMIDE DRUG AND MEASUREMENT of BIOLOGICAL ACTIVITY

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ABSTRACT

This study included the synthesis of some new Schiff bases and heterocyclic compounds [1-6] from the reaction of metoclopramide with some aromatic aldehydes by the classical Schiff method, then the Schiff bases were treated with chloroacetyl chloride and thioglycolic acid to obtain cyclic derivatives of beta-lactams [5,6] and 4-thiazolidinone [3,4], respectively. These derivatives were characterized by their melting points by FT IR, 1H NMR. Some compounds prepared were evaluated for their antioxidant activity by oxidation with 2,2-diphenyl-1-picrylhydryl (DPPH). Comparison of the antioxidant activity of the bioactive molecules of the compounds [3–6] with that of the conventional drug metoclopramide showed encouraging results.

Keywords: Thioglycolic acid, Chloroacetyl chloride, Antioxidant, Schiff base.

تحضير وتشخيص بعض المركبات الحلقية غير المتجانسة المشتقة من عقار الميتوكلوبراميد وقياس فعاليتها البايلوجية

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الخلاصة

تضمنت هذه الدراسة تخليق بعض قواعد شيف الجديدة و المركبات الحلقية غير المتجانسة من تفاعل ميتوكلوبراميد مع بعض الألدهيدات العطرية بطريقة شيف الكلاسيكية، ثم تمت معالجة قواعد شيف بكلورو اسيتايل كلورايد وحامض الثايوگلايگولك للحصول على مشتق حلقي من بيتا -لاكتام [5،6] ومشتق 4-ثيازوليدينون [3،4] ،على التوالي. شخصت هذه المشتقات بنقطة انصهار, FT IR، ^{1}H NMR. تم تقييم بعض المركبات المحضرة في المختبر من حيث نشاطها المضاد للأكسدة في المختبر باستخدام (DPPH-2،2 diphenyl-1-picrylhydryl). أظهرت مقارنة النشاط المضاد للأكسدة للجزيئات النشطة بيولوجيًا للمركب [3-6] مع نشاط عقار ميتوكلوبراميد التقليدي نتائج مشجعة.

الكلمات المفتاحية: حامض الثايوكلايكولك، كلورواسيتايل كلورايد، مضادات اكسدة، قواعد شف.

***The research is taken from a master's thesis, the first research.**

INTRODUCTION

The continued existence of humans requires the presence of heterocyclic compounds. The use of synthetic heterocyclic compounds has a substantial influence on a wide variety of goods, including medications, chemotherapy drugs, dyestuffs, photographic chemicals, co-polymers, and a few more **(Al-Adhami & Al-Majidi, 2021)**. In the field of chemistry, a lactam ring that has four members is referred to as a beta-lactam (-lactam) ring.**(Joule et *al.*, 2020**). The term "lactam" refers to a cyclic amide, and beta-lactams get their name from the fact that the nitrogen atom is connected to the beta-carbon atom rather than the carbonyl atom. The 2-azetidinone is the simplest β-lactam that may be created. β-lactams are major structural components of pharmaceuticals**(Fisher *et al.*, 2005)**and have number of pharmacological activities**(Kaur *et al.*, 2020)**, anti-bacterial**(Mishra *et al.*, 2020)**, anti-fungal**(Jarrahpour et *al.*, 2017)**, and anti-inflammatory activities**(Arefi *et al.*, 2020)**. The general structure of 2-Azetidinone, and simplest β-lactam is shown in (Figure, 1).

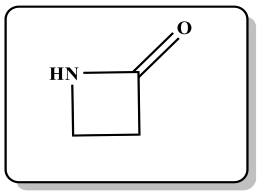


Figure (1): structure of 2-Azetidinone.

Thiazolidinones are derivatives of thiazoles which belong to one of the most intensively investigated classes of five-member heterocyclic compounds having a carbonyl group in addition to nitrogen and sulfur **(Cheddie *et al.*, 2020)**. In the field of medicinal chemistry, these substances are regarded as privileged scaffolds. and pharmacological because of their various biological activities**(Haroon *et al.*, 2021)**. Various isomers of thiazolidinone includes: thiazolidin-2-one, thiazolidin-4-one, thiazolidin-5-one, 2-thioxo-thiazolidin-4- one and thiazolidine-2,4-dione are associated with numerous pharmacological properties**(Nirwan *et al.*, 2019)**,

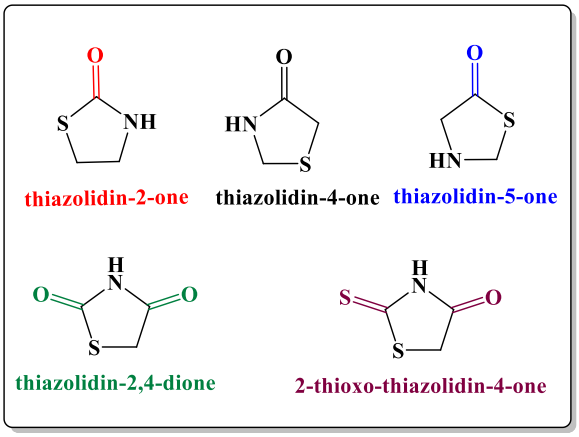


Figure (2): Some different isomers of thiazolidinones.

In particular, thiazolidinone derivatives have attracted interest due to the vast variety of biological features that they possess, in addition to the fact that they are included in the

structural makeup of a great deal of naturally occurring items. They are also the fundamental building blocks of the structures of a wide variety of pharmaceutically active drugs.**(Nirwan *et al.*, 2019)**

EXPERIMENTAL

MATERIALS AND METHODS

The BDH, Fluka, Merck, and Sigma Aldrich firms provide the chemicals that are utilized in this investigation, and those chemicals are used without any additional purification. In addition to this, the melting points that were recorded according to the point of electrothermal melting equipment remained correct. SHIMAZU FTIR-8400 Fourier transform infrared spectrophotometer was used to record FTIR spectra of the produced compounds in the spectral region of (4000-600) cm-1 using KBr discs. The 1H-NMR spectra were obtained by utilizing TMS as an internal reference and DMSO-d6 as a solvent throughout the recording process using a BRUKER 400MHz instrument located in Iraq.

Synthesis of 5-chloro-N-(2-(diethylamino)ethyl)-4-((4-(dimethylamino) benzylidene) amino)-2-methoxybenzamide and (E)-5-chloro-N-(2-(diethylamino)ethyl)-2-methoxy-4-((4-nitrobenzylidene)amino)benzamide (Hamid & Obaid, 2020)

In a (50 mL) round bottom flask, the compound Metoclopramide (0.0044 mol), with equimolar amount of different substituted aromatic aldehydes (0.0044 mol) were added, in (10 mL) absolute ethanol and (2-3) drops of a catalyst glacial acetic acid. The mixture was refluxed in water bath for (6-7) h. The excess solvent was evaporated under reduced pressure. The generate product was dried and further purification was done using recrystallization from dioxan. Some of the physical properties of compounds [1,2] and yield are listed in Table (1.1).

Synthesisof 5-chloro-N-(2-(diethylamino)ethyl)-4-(2-(4-(dimethylamino) phenyl)-4-oxothiazolidin-3-yl)-2-methoxybenzamide and 5-chloro-N-(2-(diethylamino)ethyl)-2-methoxy-4-(2-(4-nitrophenyl)-4-oxothiazolidin-3-yl)benzamide [3,4](Ahamad & Hussain, 2015)

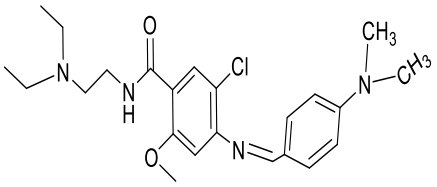
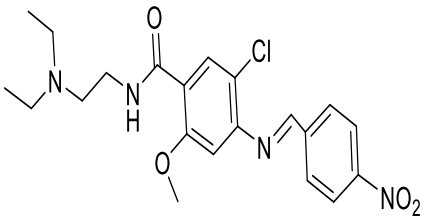
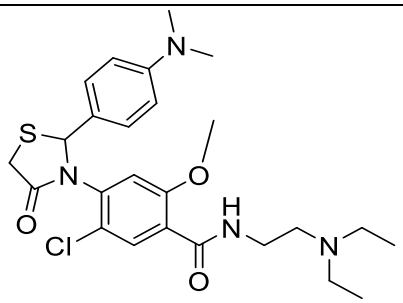
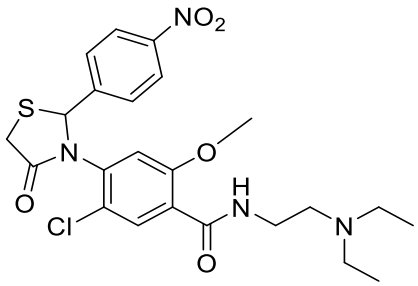
Ethanol with a combination of Schiff bases [1,2] (0.001mol) and an excess of thioglycolic acid (0.002mol). The response was allowed to reflux for a while (18-20h.). After evaporating the solvent, the residue was neutralized with a solution of sodium bicarbonate at a concentration of 5% in order to eliminate any excess thioglycolic acid. After being filtered, the precipitate that had formed was then washed with water many times before being recrystallized from chloroform**(Ali & Hassan, 2022).** Table contains a listing of the compounds' (3, 4) physicochemical features (1.1).

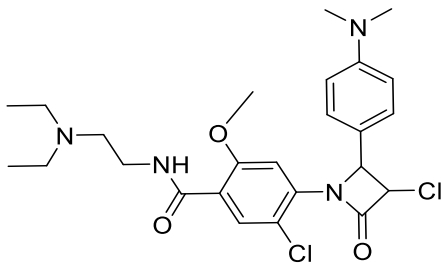
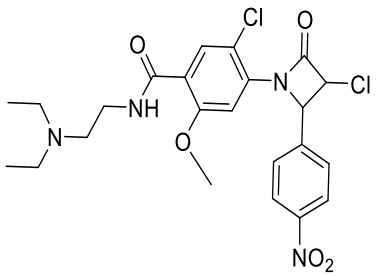
Synthesis of 5-chloro-4-(3-chloro-2-(4-(dimethylamino)phenyl)-4-oxoazetidin-1-yl)-N-(2-(diethylamino)ethyl)-2-methoxybenzamide and 5-chloro-4-(3-chloro-2-(4-(dimethylamino)phenyl)-4-oxoazetidin-1-yl)-N-(2-(diethylamino)ethyl)-2-methoxybenzamide[5,6](Sankar *et al.*, 2019)

After adding a combination of Schiff bases derivatives [1,2] with equimolar volumes of chloroacetyl chloride (0.2 mL, 0.0016 mol) and triethylamine (Et3N) (0.0016 mol) in 10 mL of THF as a solvent, the final product had 0.0016 mol of chloroacetyl chloride and 0.0016 mol of triethylamine. After that, the mixture was heated for (14-16) hours while in a reflux state. The contents were allowed to come down to room temperature, after which the precipitate that was

generated was filtered, washed with cold water, and then subjected to further purification using the recrystallization process with the use of ethanol**(Mousa & Jassim, 2021)**. The table below contains a few examples of the physical characteristics of compounds [5,6]. are listed in Table1.

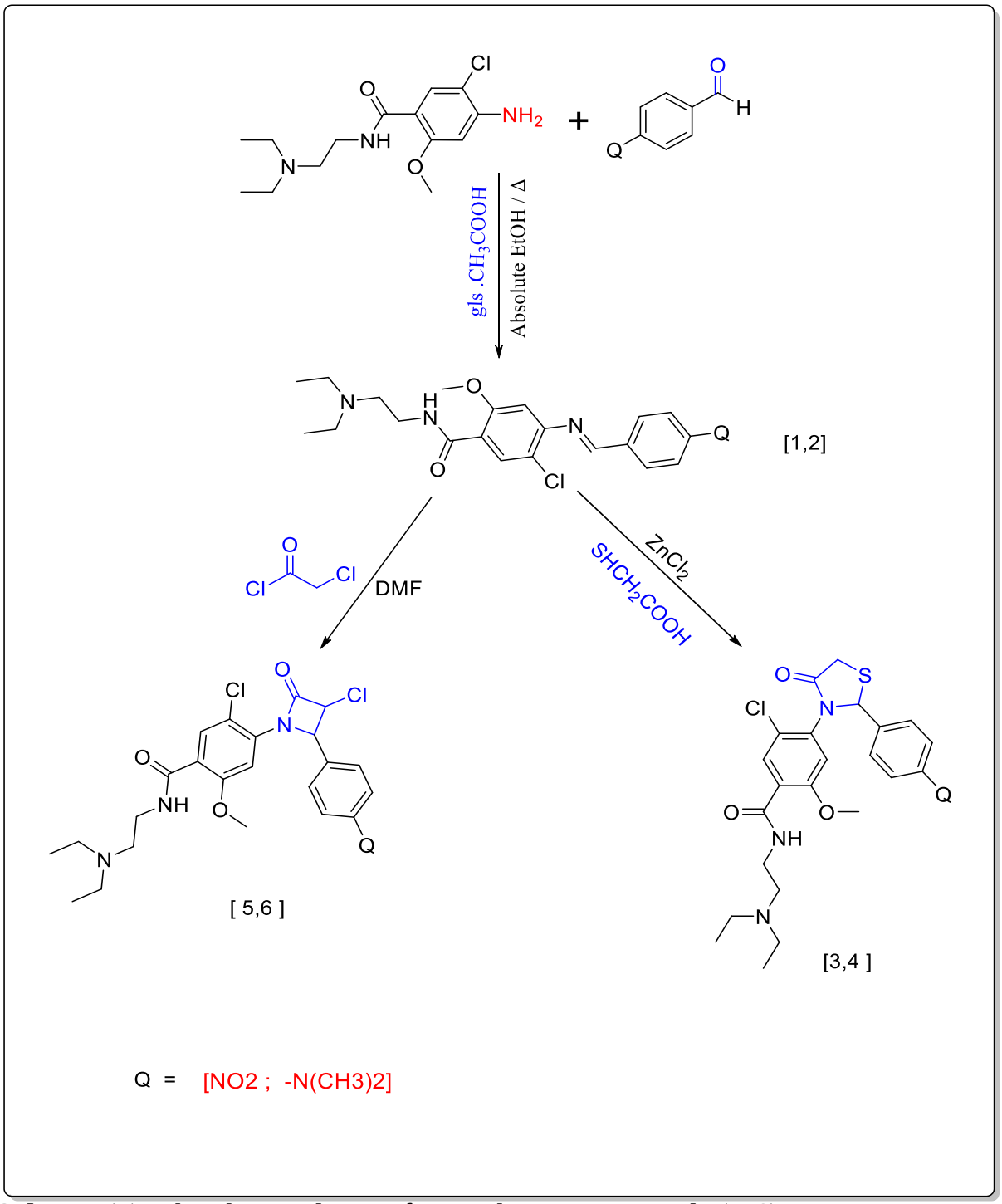
Table (1): presents a few of the produced compounds' [1-6] observable physical characteristics.

N0.	Structure and Name	Formula and M.wt(g\mol)	M.P °C	Color	Yield (%)	Solvent of Rec
1		$C_{23}H_{31}ClN_4O_2$ 430	107-112	red	95	Ethanol
2		$C_{21}H_{25}ClN_4O_4$ 433	93-103	yellow	91	Ethanol
3		$C_{25}H_{33}ClN_4O_3S$ 505	118-122	yellow	86	Dioxane
4		$C_{23}H_{27}ClN_4O_5S$ 507	180-183	Grey	77	Dioxane

5		$C_{25}H_{32}Cl_2N_4O_3$ 507	135-142	Light Brown	76	THF
6		$C_{23}H_{26}Cl_2N_4O_5$ 509		Light Brown	72	THF

.

3.RESULTS AND DISCUSSION

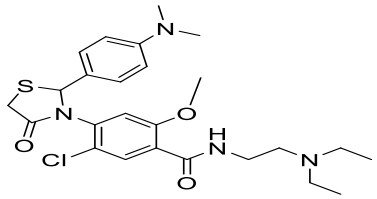
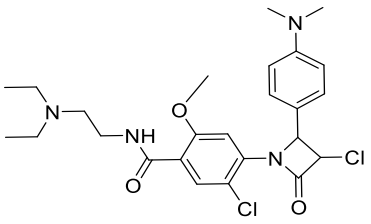


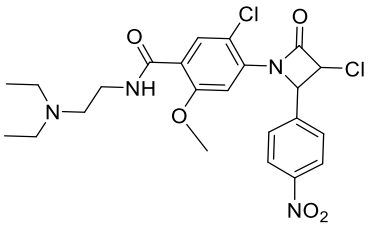
Scheme (1). The chemical steps for synthesis compounds (1-6).

Table (2): FT-IR spectral data (cm^{-1}) for the compounds [1-6]

Comp. No.	νC-H Aromatic	νC-H Aliphatic	νC-O	νC-Cl	C-N	ν C=C Aromatic	νN-H Amide	νC=O Amide	Other bands (ν)cm^{-1}
1	3004	2977 2881	1234	850	1303	1595	3290	1708	1639 (νC=N)Imine
2	3105	2970 2985	1246	856	1344	1604	3221	1693	1647 (νC=N)Imine 1523 Asym. ν (NO2) 1413 Sym.ν (NO_2)
3	3055	2979 2941	1174	821	1305	1579	3220	1716	-
4	3005	2900 2804	1176	813	1251	1604	3221	1678	1523 Asym. (NO_2) 1365 Sym. (NO_2)
5	3109	2931 2868	1245	856	1309	1604	3259	1693	1525 Asym. (NO2) 1344 Sym. (NO_2) 1415 N-N
6	3105	2970 2889	1338	854	1270	1639	3290	1689	-

Table (3): ^{1}H NMR spectral data (δ ppm).

Compound Number	Structure	^{1}H NMR spectral data (δ ppm)
1		9.0 (s,1H, NH), 7.0-8.0 (m, 6H, Ar-H), 4.0 (s, 3H, OCH_3), 3.5 (m, 4H, N-$2CH_2$), 2.7 (s, 2H, O=C-CH_2), 2.6 (m, 4H, $2CH_2$), 4.16 (s, 1H, N-CH thiazolidine ring), 1.12-2.3 (m, 12H, 4-CH_3)
2		8.5(s,1H ,NH), 7-8 (m,6H,Ar-H), 5.3(s,1H,N-CH-C=O), 5.44(s,1H,CH-Cl) 4(s,3H,O-CH_3), 3.5 (m , 2H ,NH-CH_2), 2.6(m,4H,N-CH2), 2.2(m, 4H , $2CH_2$), 1.1-2.1(m , 12H , $4CH_3$)

3		8.6(s,1H ,NH), 7-8 (m,6H,Ar-H) 5.16(s,1H,N-CH-C=O), 5.45(s,1H,CH-Cl) 3.90(s,3H,O-CH_3),3.7(m ,2H,N-CH_2), 2.7 (m, 4H , 2CH_2), 1.2(m , 6H , 2-CH_3)

The second part of our work includes the synthesis of Heterocyclic compounds. The FT-IR spectra of these compounds showed absorption bands in the range of 3457 cm^{-1} attributed to the N-H stretching vibrations. The absorptions in the 1467 cm^{-1} range are caused by aromatic C=C stretching vibrations. The absorption in the range between 1309 and 1270 cm^{-1} is due to C-N stretching vibrations, and the range between 1716 and 1689 cm^{-1} is for C=O amid vibrations. The absorptions from 856 cm^{-1} refer to C-Cl vibrations. The ^{1}H NMR spectrum of compound **1** shows signals at 1.12 and 2.3 ppm for the four CH_3 groups. Signals at 2.6, 2.4, and 3.5 ppm are attributed to the CH_2 groups .The chemical shift of the methoxy group was at 4.0 ppm. Mutilate signals from 7.0-8.0 are belong to the aromatic protons. The singlet signal at 9.0 ppm is for the N-H proton . The ^{1}H NMR spectrum of compound **2** shows signals at 1.1 and 2.52 ppm for the four CH_3 groups. Signals at 2.6, 2.8, and 3.5 ppm are attributed to the CH_2 groups. The chemical shift of the methoxy group was at 4.0 ppm. Mutilate signals from 7.0-8.0 are belong to the aromatic protons. The singlet signal at 8.5 ppm is for the N-H proton. The ^{1}H NMR spectrum of compound **3** shows signals at 1.2 ppm for the two CH_3 groups. Signals at 2.7, 2.8, and 3.7 ppm are attributed to the CH_2 groups. The chemical shift of the methoxy group was at 3.90 ppm. Mutilate signals from 7.0-8.0 are belong to the aromatic protons. The singlet signal at 8.6 ppm is for the N-H.

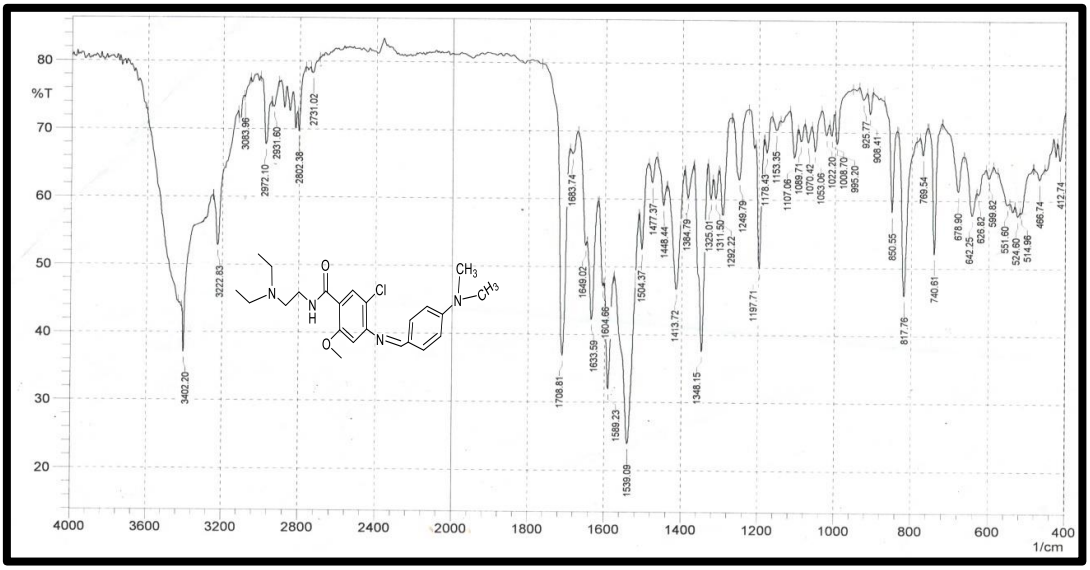


Figure (3). FT-IR spectrum for compound 1.

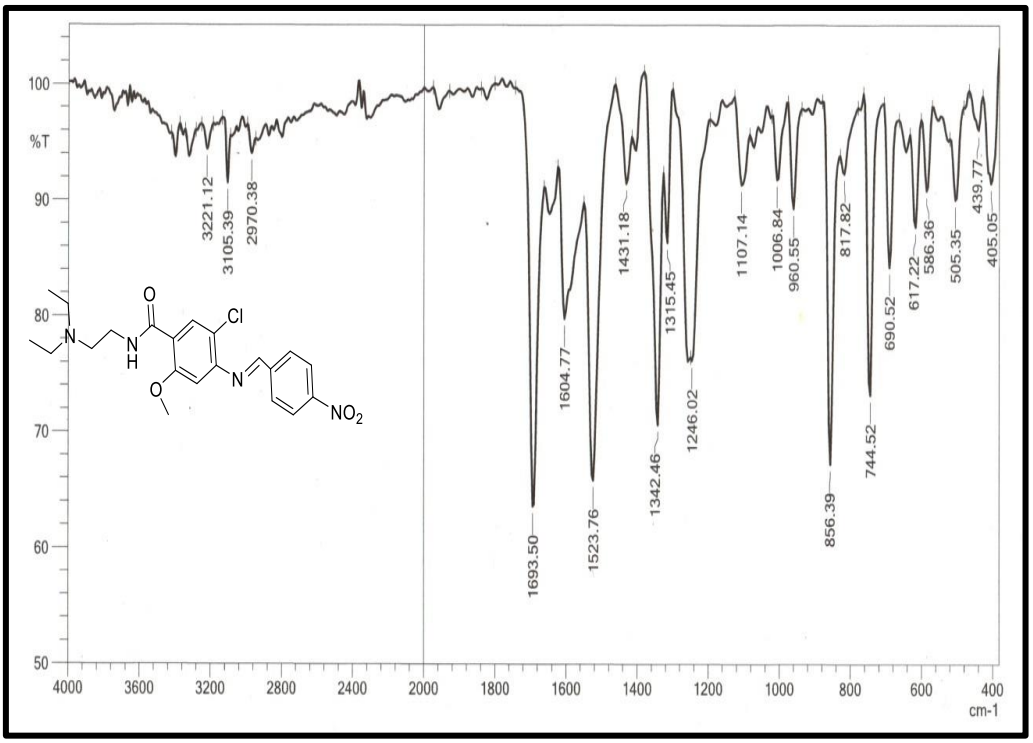


Figure (4): FT-IR spectrum for compound 2.

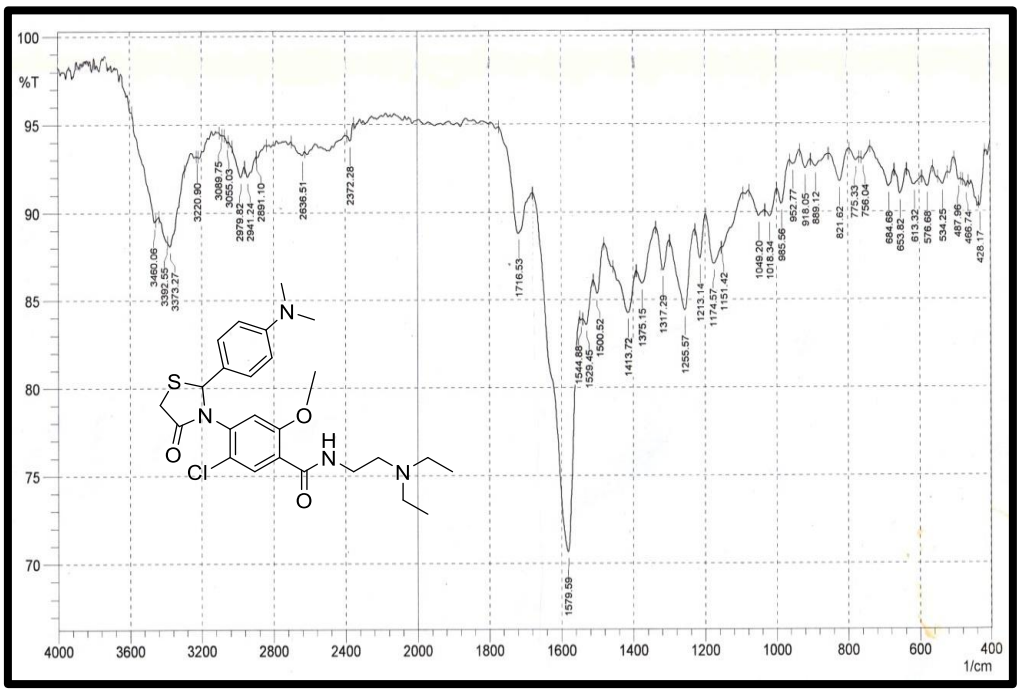


Figure (5). FT-IR spectrum for compound 3.

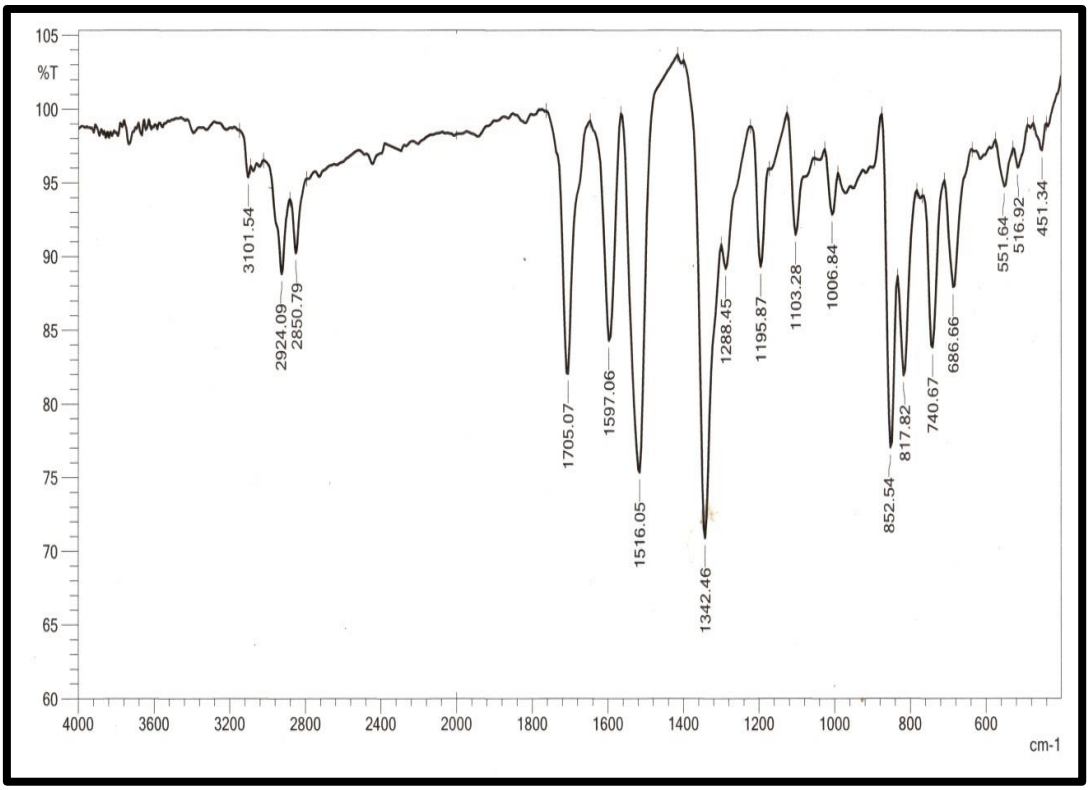


Figure (6): FT-IR spectrum for compound 4.

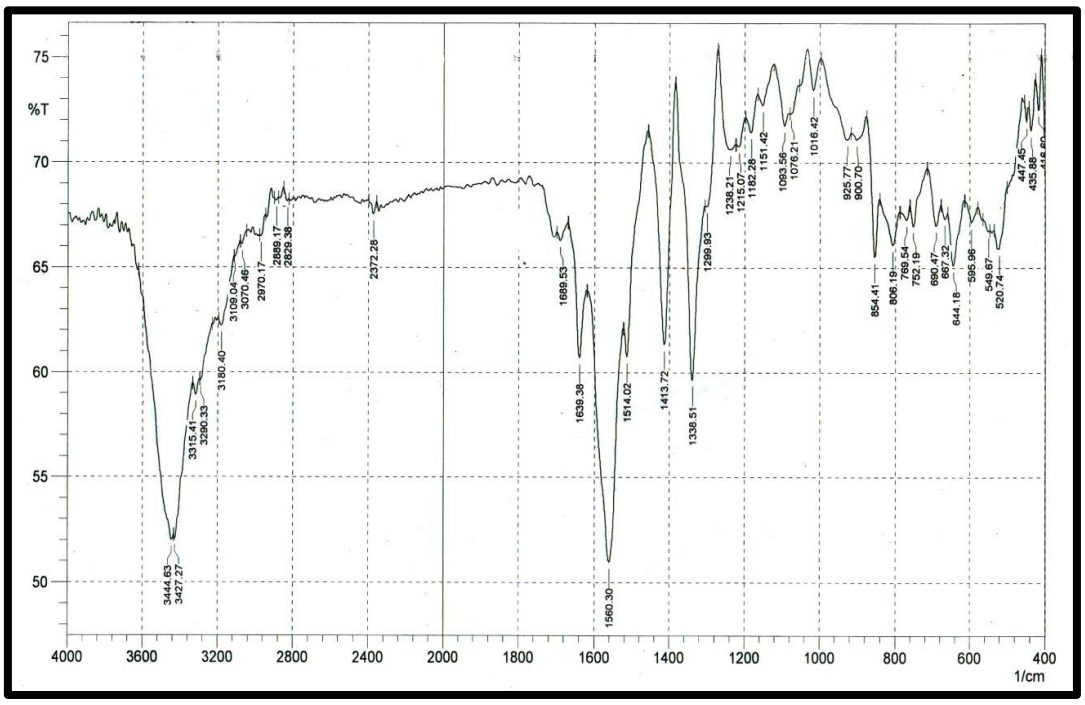


Figure (7): FT-IR spectrum for compound 5.

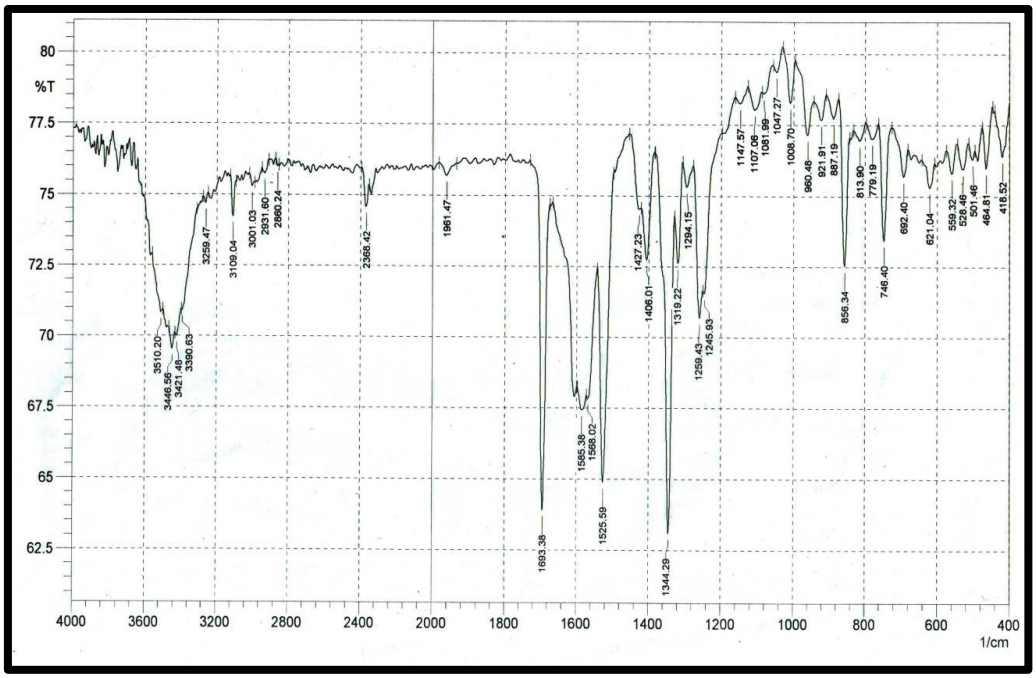


Figure (8): FT-IR spectrum for compound 6.

Antioxidant activity[3,4,5,6] DPPH Radical Detoxificatio Activity(Olszowy & Dawidowicz, 2018):

1- DPPH (1،1-Diphenyl-2-picrylhydrazyl): Re-treated in 100 mL ethanol, keeping solution protected from light..
2- Various concentrations (100, 50, 25)mg/ml from some of the produced chemicals ppm were made. It was made by mixing 1milligram of the compound with10milliliters of ethanol tomake100parts per million, which was then diluted to 50, 25 parts per million, etc.
3- Ascorbic acid (vitamin C): Similar concentrations was prepared.

Table (4): Inhibition percentage (I%) of compounds 3-6.

Compound number	25 (mg/mL)	50 (mg/mL)	100 (mg/mL)
3	71.03	84.29	88.2
4	79.21	77.81	85.17
5	43.18	59.12	78.57
6	54.07	65.82	83.53
Ascorbic acid	80.95	89.25	93.54

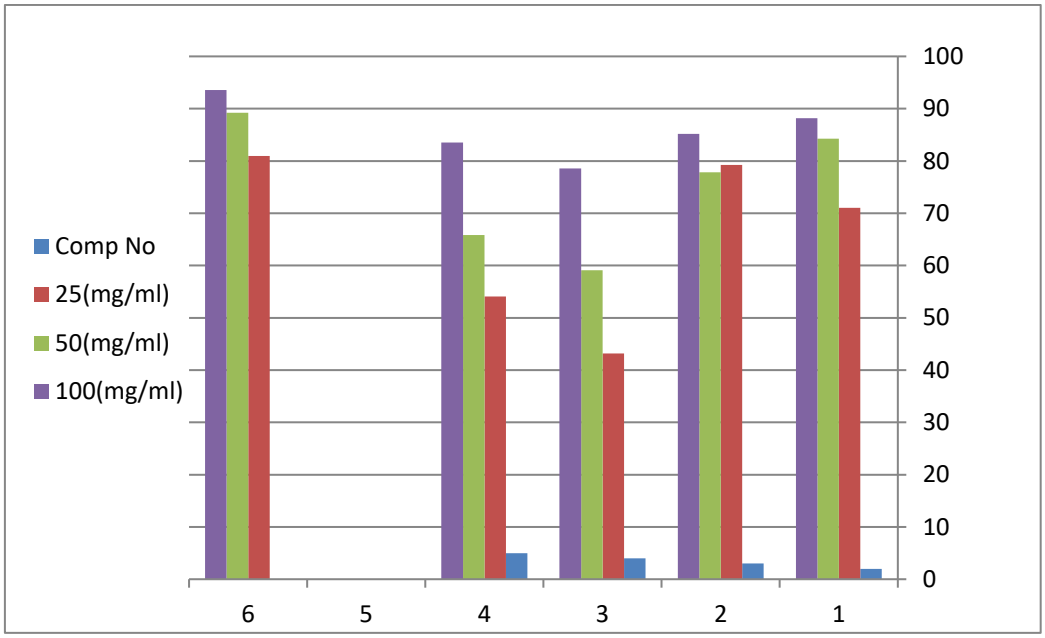


Figure (9): Inhibition percentage (I%) of compounds 3-6.

(DPPH) and a few other methods, all of the compounds were tested to see whether they had the ability to function as antioxidants when the experiment was conducted in vitro. The favorable findings were found when the antioxidant activity of the compound bioactive molecules [3-6] was compared to that of the traditional drug metoclopramide.

CONCLUSION

In this work, some heterocyclic compounds were prepared by the interaction of the drug metoclopramide with some derivatives of aldehydes and ketones, where the antioxidants of these prepared compounds were measured.

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Abdul-Razaq &et al.,

المجلة العراقيه لبحوث السوق
وحماية المستهلك

STUDYING THE EFFECT OF USING OLIVE OIL AGAINST MICROBES THAT CAUSE SKIN INFECTIONS

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ABSTRACT

Modern nutritional sciences look at the fruits of olives and Olive oil as essential products for human life due to the richness of its fruits in basic compounds such as oil, proteins, carbohydrates, mineral salts and some other vitamins, which Iraqi tables are almost devoid of as a consumable material, either in the form of green or black olives or their oil, hence the idea This study aimed at evaluating some types of commercial olive oil samples extracted from olive fruits as a biomaterial against some types of microorganisms represented by Gram-negative *Pseudomonas aeruginosa* (*Pseu aeruginosa*) and *Staphylococcus aureus* (*Staph aureus*) as Gram-positive. Characterized at the level (P≤0.01) for the concentrations of the oil extract (25, 50 and 100)% used in the study on both types of target bacteria in the study, and thus the possibility of using this extract, which has proven its inhibitory effectiveness as an antidote against some types of gram-positive and gram-negative bacteria.

Keywords: Olive oil, Baghdad's local markets, skin infection.

دراسة تاثير استعمال زيت الزيتون ضد الميكروبات المسببة للالتهابات الجلدية

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الخلاصة

تنظر علوم التغذية الحديثة الى ثمار الزيتون وزيت الزيتون على انها منتجات ضرورية لحياة الانسان نظرا لغنى ثمارها بمركبات اساسية كالزيت والبروتينات والكاربوهيدرات والاملاح المعدنية وبعض الفيتامينات الاخرى والتي لاتكاد تخلو موائد العراق منها كمادة استهلاكية اما على شكل زيتون اخضر او اسود او زيته, ومن هنا جاءت فكرة هذه الدراسة التي هدفت الى تقييم بعض انواع عينات زيت الزيتون التجارية المستخلصة من ثمار الزيتون كمادة حيوية مضادة لبعض انواع الاحياء المجهرية المتمثلة في بكتريا *Pseudomonas aeruginosa (Pseu aeruginosa)* سالبة لصبغة كرام وبكتريا *Staphylococcus aureus* (*Staph aureus*) كموجبة لصبغة كرام, اظهرت نتائج الدراسة وجود فروق معنوية على مستوى (P≤0.01) لتراكيز مستخلص الزيت (25, 50 و100)% المستعملة بالدراسة على كلتا نوعي البكتريا المستهدفة في الدراسة, وبالتالي امكانية استعمال هذا المستخلص الذي اثبت فعاليته التثبيطية كمادة مضادة تجاه بعض انواع البكتريا الموجبة والسالبة لصبغة كرام.

الكلمات المفتاحية: زيت زيتون ، أسواق بغداد المحلية ، التهابات جلدية.

INTRODUCTION

The Olive plant *Oleaeuropaea* belongs to the family *Oleaceae*, and is a perennial olive plant, evergreen is characterized by a huge stem and longitudinal lanceolate leaves, 5.7 cm long, with opposite edges sharply tapering, silvery-green in light, the flowers are greasy-white and the fruits are stoned ,its length is about 1 cm, green in color at the beginning of summer, but it turns black at the beginning of summer maturity (**Rotondi *et al.*, 2004; Ray *et al.*, 2019**). Due to its nutritional and medicinal benefits, the olive tree and the oil it yields hold a prominent standing among fruit trees. It can be found in places with a variety of climates, including humid & arid regions (**Gorzynik-Debicka *et al.*, 2018**). Olive oil is the fruit juices oil, which itself is separated from the other constituents of the producing olive fruit using extraction method (**Abdelhafez *et al.*, 2017**). Olives are distinguished by the fact that they contain oils, which in turn consist of multiple components such as compounds volatile aromatics, phenolic compounds, and triglycerides of all kinds, mono, di, and tri It also contains fatty acids, vitamins such as E and D, phospholipids, saponins and others (**Cicerale *et al.*, 2012**) and (**Di Bene *et al.*, 2012**). The natural olive oil extract inhibits some types of bacteria that cause skin inflammation, in addition to inhibiting the growth of some bacteria on culture medium such as *Pseu aeruginosa*, olive fruit oil works To reduce high blood pressure, olive oil have effectiveness Protective against blood clots, as it is important in the analysis of fats and reduces platelet aggregation It also has an anti-cancer and anti-inflammatory effect (**Hohmann *et al.*, 2015; Gabriel *et al.*, 2019**). Numerous research studies have investigated the chemical and physical qualities, antioxidant substances of olive oil. Olive oil contains the following chemicals, together with their ratios: palmic, palmitoleic, margaric, margarolic, stearic, linoleic, linolenic, and henric. (**Rubio *et al.*, 2014; Alvarez-Laderas *et al.*, 2020)**. The objective of this research was to assess the efficacy of Olive oil as a treatment ingredient in preventing bacterial infections of the skin.

MATERIAL AND METHIODS

Sample collection

A total of Ten Olive oil samples from different brands were randomly collected from Baghdad's local markets, samples are given in (**Table, 1**).

Table 1: Olive oil samples used in this study

Code	Trade mark	Country	Expiration date
O1	Rafael salgadors	Spain	2022/9/22-2024/8/22
O2	KDD	Kuwait	2022/4/1-2023/10/1
O3	Mazola	Lebanon	2022/1/26-2024/1/25
O4	Al-motawasset	Syria	2022/6/8-2024/6/7
O5	Fourati	Tunis	2022/7/2-2024/7/1
O6	Afia	Turkey	2022/2/12-2024/2/11
O7	Zer	Turkey	2022/9/14-2024/9/13
O8	Aljabal teeba	Lebanon	2021/2/15-2023/2/14
O9	Fil	IRAQ	2022/2/24-2024/1/23
O10	Hemani	Pakistan	2021/1/-2023/12

Microorganisms and Culture Conditions

Two bacterial isolates were obtained, one positive for Gram-positive staining, including *Staph aureus* and the other negative for Gram-positive including *Pseu aeruginosa* They were obtained from the laboratories of the College of Science / University of Baghdad as

isolates that cause skin infections. They were used in order to qualitatively detect the inhibitory effectiveness of Olive oil extract. These two isolates were active on the medium. Luria Broth (LB) for 18 h at 37°C **(Rubio *et al.*, 2014).**

Determination of the Antibacterial Susceptibility

The Well diffusion method was used to detect the inhibitory activity of Olive oil extracts, as 0.1 ml of pathogenic bacterial isolates activated with an approximate number of inoculum 1.5 x 10^8 cells / mL were spread on sterile N.A solid agar media cast in sterilized Petri dishes using L- shape, holes were made on the surfaces of the cultured media with a cork borer, and specific amounts of 50 microliters of different concentrations of extracts were placed, including (25, 50 and 100)%, in duplicate for each concentration, in addition to the positive control treatment free of the extract. The plates were incubated at a temperature of 37°C for 24 h for pathogenic bacterial isolates and at a temperature of 28°C for 48 h for yeast. The diameter of the inhibition zone was measured around each hole using a graduated ruler (**Fratianni *et al*., 2016**; **Alvarez-Laderas *et al.*, 2020).**

STATISTICAL ANALYSIS

The Statistical Analysis System- SAS (2018) program was used to detect the effect of difference factors in study parameters. Least significant difference –LSD test (Analysis of Variation-ANOVA) was used to significant compare between means in this study.

RESULT AND DISCUSION

The antibacterial ability of Olive oil was evaluated at concentrations (25, 50,100) % against some types of gram-positive and gram-negative bacteria, proved its inhibitory effectiveness against the bacteria as shown in (Tables 2).

Table (2): Diameters of inhibition zones (mm) for the growth of bacteria treated with different concentrations of olive oil (25, 50,100) %.

Code	Organism						LSD value
	Staph aureus			***Pseu aeruginosa***			
	25%	50%	100%	25%	50%	100%	
O1	14	18	25	-	11	13	5.61 *
O2	10	12	14	-	9	10	4.95 *
O3	11	10	12	8	11	10	3.79 *
O4	10	16	22	7	9	13	5.66 *
O5	11	13	19	-	14	16	5.02 *
O6	13	19	21	11	14	16	4.78 *
O7	15	18	23	-	9	11	6.13 *
O8	13	19	28	10	16	22	5.75 *
O9	15	17	26	12	11	15	6.37 *
O10	11	14	17	-	7	14	4.82 *
LSD value	4.39 *	5.22 *	5.84 *	3.92 *	4.28 *	4.5 1 *	----
** (P≤0.01).							

As shown in Table 1, the olive oil extracts of O1, O4, O6, O7, O8, O9 significant differences at the level (P≤0.01) in its higher inhibitory activity against *Staph aureus*, while the

brands O2, O3, O5 and O10 showed the least inhibition against the same bacteria, while the O8 model showed a significant difference at the level of (P≤0.01). the highest in inhibition against *Pseu aeruginosa*, while the least inhibition was observed for the O2 brand. O3 against the same bacteria. This result was agreement with **Manuel *et al.*, (2019**), he found The different concentration for inhibit the growth of the pathogenic bacteria strains was active for all the olive oil extracts against *S. aureus* This confirms that polyphenols present in the olive oil have a general capacity to inhibit the growth of pathogenic or unwanted microorganisms. A lot in vitro research studies have demonstrated that certain olive oil polyphenols may inhibit the growth of many kinds of bacteria, including those that cause some skin infections and digestive problems as well as cancer-causing bacteria like Helicobacter pylori and other types of peptic ulcers (**Lazzez *et al.*, 2008;Rubio *et al.*, 2014; Manuel Silvana *et al.*, 2019**).This result was agreement with **Fratianni *et al.*,(2019**), who found the *Pseu aeruginosa*, a well-known pathogen equivalent to *E. coli*, was also able to be inhibited by several extra virgin olive oil extracts. Since it could form biofilms, which raises its resistance to conventional medicine. 4.9 g of the olive oil extract were generally extremely successful at preventing the growth of all bacterial strains, with an inhibition zone not less than 10.67 *Staph aureus*. Overall, inhibitory zones generated by 4.9 g of the polyphenol extract was superior to 17 mm, when evaluated against *Staphylococcus aureus*. Olive oil extracts in the amount of 4.9 g created zones which were not more than 12.67 mm, which was less effective, that a microorganism's vulnerability or susceptibility to a certain drug or natural extract might not only be linked to its species or genus but, in certain circumstances, to its strain as well, this result was disagreement with (**Ombra *et al*., 2016 ;Cerulli *et al*., 2017; Karygianni *et al.*, 2019).**

CONCLUSION

The results of the study showed that the commercial olive oil extracts with different concentrations were different in their inhibitory activity against some types of Gram-positive and Gram-negative bacteria that cause skin infections, which indicates the possibility of using extracts of this oil against infection with other types of bacteria that cause skin infections.

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المجلة العراقيه لبحوث السوق
وحماية المستهلك

EVALUATION OF THE EFFICIENCY OF CHITOSAN PRODUCED FROM THE STALKS OF *AGARICUS BISPORUS* BROWN AS AN ANTIFUNGAL AGAINST *ASPERGILLUS FLAVUS* AND REDUCING AFLATOXIN B1

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ABSTRACT

The aim of this study was to benefit from the remnants of edible mushrooms production farms in the production of bioactive compounds such as chitosan and characterization it, and then evaluating the efficiency of chitosan in inhibiting growth of *Aspergillus flavus* (*A. flavus*) and preventing it from producing aflatoxin B1 toxins. The prepared chitosan diagnosed via Fourier Transform Infrared (FTIR). Chitosan have been tested in inhibiting *A. flavus* in the concentrations 0.5, 1, 1.5 and 2%. The chitosan inhibition rate of the fungus *A. flavus* has reached 16, 36, 53 and 100% respectively. The effectiveness of chitosan tested in preventing *A. flavus* from producing Aflatoxin (AFB1) in the concentrations 0.5, 1 and 1.5 %. The rate of Aflatoxin B1 production inhibition by chitosan has reached 72.7 ،86.7 and 100% respectively after 21 days of incubation at 25°C after estimating Aflatoxin B1 via High-Performance Liquid Chromatography (HPLC).

Keywords: *Agaricus bisporus*, Chitosan, Aflatoxin B1.

تقييم كفاءة الكايتوسان المنتج من سيقان الفطر *Agaricus bisporus* Brown البني كمضاد فطري تجاه *Aspergillus flavus* واختزال سم الأفلاتوكسين B1

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الخلاصة

هدفت الدراسة إلى الاستفادة من مخلفات مزارع إنتاج الفطر *Agaricus bisporus* Brown (*A.bisporus*) الصالح للأكل في إنتاج المركبات النشطة حيوياً مثل الكايتوسان ومن ثم تقييم كفاءته في تثبيط نمو فطر *Aspergillus flavus* واختزال سم الأفلاتوكسين B1. وجرى تشخيص الكايتوسان بتقانة طيف الاشعة تحت الحمراء (FTIR) Fourier Transform InfraRed ٪. وتم اختبار الكايتوسان في تثبيط فطر *Aspergillus flavus* بتركيز 0.5 و 1 و 1.5 و 2٪. بنسبة تثبيط الكايتوسان للفطر *A. flavus* 16 و 36 و 53 و % على الترتيب.كما اختبرت فاعلية الكايتوسان في منع هذا الفطر من إنتاج سم الافلاتوكسين B1 بتركيز 0.5 و 1 و 1.5٪. اذ بلغت نسبة التثبيط 72.7 و 86.7 و 100 % على التوالي بعد 21 يوم من الحضن في درجة حرارة 25 مْ بعد تقدير سم الافلاتوكسين B1 بأستعمال تقنية الكروموتكرافي السائل عالي الأداء High-Performance Liquid Chromatography (HPLC).

الكلمات المفتاحية : *Agaricus bisporus*، شيتوسان، الأفلاتوكسين B1.

[*]**The research is extracted from the doctoral thesis of the first researcher.**

INTRODUCTION

The chitosan produced from Mushrooms is characterized by many features such as it is hygienically safe as well as the availability of the Mushrooms resources all over the year with the possibility of harvesting the biomass at a low cost via simple fermentation, and producing chitosan from Mushrooms resources does not require demineralization with the possibility of producing high-quality chitosan via treating with acids and bases which makes the process of producing chitosan from Mushrooms resources economically friendly **(Dhillon *et al.*, 2013) .** The demand for chitin and chitosan produced from Mushrooms resources has increased recently according to its distinctive physiochemical characteristics preferred to those produced by traditional crustacean resources and because of the seasonal and limited availability of the crustacean resources as well as the heterogeneous physiochemical characteristics and inconsistent levels to the extent of removing acetyl groups as well as high molecular weight as the traditional method of producing chitin and chitosan reduced the possibility of using it industrially, so fungal chitosan has become a topic of discussion for many modern studies **(Gapsari *et al.*, 2020)**. Many studies which were conducted in Iraq succeeded in extracting chitosan from Mushrooms resources especially the edible Mushrooms such as *Agaricus bisporus* and using chitosan healthily or nutritionally **(Aldulimy *et al.*, 2021; Al Fatima *et al.*, 2021; Fadhil and Mous, 2020)**. Chitosan and its products have got big attention academically and industrially as chitosan has an antifungal activity against plant pathogens and it is used in food industry **(Al-Aubadi, 2021; Al- Aubadi *et al.*, 2020; Salman & Al-aubadi, 2010)**. and chitosan is used medically and pharmaceutically, and in preparing anti-microbial casings as well as manufacturing plasters and removing heavy minerals from water **(Ismail *et al* ., 2015; Taha *et al.*, 2019; Mohsen & Ali, 2022; Yonis *et al.*, 2019)**. The anti-Aflatoxin biocides are used in different parts of the world, and some countries are about to register biocides domestically as an approach towards developing economically friendly antifungal compounds to control the food pollution by fungi. Many studies referred to the role of chitosan in food manufacturing, fungi inhibition and preventing the production of mycotoxins which have cumulative and carcinogenic effects when having them with food item. So, the study aimed to benefitting from the residues of edible mushrooms production farms in producing bioactive compounds such as chitosan and studying its physicochemical properties by FTIR. then evaluating the efficiency of chitosan in inhibiting the growth of *A. flavus* and preventing it from producing Aflatoxin B1.

MATERIALS AND METHODS

Chitosan resource

The residues of *A. bisporus* brown from farms in Baghdad where chitosan was extracted and some of its characteristics were studied according to the study been conducted by **Shahadha *et al.*, (2023)**.

Characterizing chitosan

Chitosan prepared from the stalks of *A. bisporus* brown are characterized by using Fourier transform infrared spectroscopy (FTIR) by mixing dry chitosan with dry potassium bromide in a rate 1: 5 with a ceramic mortar and pestle for 2 min and pressing the mixture by using a hydraulic compressor belongs to FTIR at a pressure of 8 bars for 60 sec. The disc is put in FTIR for being analyzed by using a frequency range between 400 – 4000 cm^{-1} **(Sivakami *et al.*, 2013)**.

Activation of fungus *A. flavus* isolation

The isolation of *A. flavus* is obtained that is characterized on the genetic level and isolated from the local wheatgrass which go back to the marketing of 2020 from a number of silos in Baghdad, and producing Aflatoxin B1 is confirmed by a study conducted by **Mohamed & Al–Shamary, (2022)** in Department of Food Sciences, College of Agricultural Engineering Sciences, University of Baghdad. Fungi isolation of *A. flavus* is activated by using Potato Dextrose Agar (PDA) media, then the plates are incubated at 25°C for 5 d.

Adding chitosan to PDA media

The media is prepared by adding chitosan in concentrations 0.5, 1, 1.5 and 2% for chitosan individually to the PDA media that is sterilized by autoclave at 15 lb (121ºC)for 15 min after cooling it to 50ºC Note that this quantity is calculated from the total size of the media in one flask, as the final size of one media reached 100 mL. then the medium PDA is poured in petri dishes of 9 cm, and then left to solidify **(Saharan *et al.*, 2013)**.

Evaluation of chitosan efficiency in *Aspergillus flavus* growth inhibition on PDA media

A potency test for the extracted chitosan and chitosan nanoparticles that is prepared from the stalks of *A. bisporus* brown is made according to the method mentioned by **El-Mohamedya *et al.*, (2019)** and the inhibition rate is calculated according to the equation described by **(Dewi & Nur , 2017)**.

$$\% \textbf{ inhibition } = \frac{B-A}{B} \times 100$$

where

A: diameter of the growth colony in the test plates

B: diameter of the growth colony in the control plates

spore suspension preparation

Spore suspension was prepared as described by **Cortés-Higareda *et al.*, (2019)**. The spores were counted using hemocytometer and the amount of spores for was adjusted to 10^7 spore/mL.

The inhibition of producing of aflatoxin B1 from *A. flavus* by using chitosan

The test is made according to the method mentioned by **Meng *et al* ., (2020)**. the media is prepared by adding chitosan in the concentrations 0.5, 1 and 1.5 % to the Potato dextrose broth (PDB) media that is sterilized by autoclave 15 lb (121ºC) for 15 min in flasks of capacity 250 mL, as the concentrations are added to both types of chitosan after cooling the PDB media to 50ºC, in each flask by three replicates for each concentration and three replicates are left without adding chitosan as a control treatment. The flasks containing the modified PDB media are inoculated by adding chitosan with 1 ml of the spore suspension of *A. flavus* as each 1 mL contains (1×10^7 spores / mL) and the flasks are incubated in 25ºC for 7 d for producing Aflatoxin.

Extraction of Aflatoxin B1

Extraction of Aflatoxin B1 was carried out according to method of **Kollu *et al.*, (2009)**. the flasks content is filtered by filter papers Whatman No.1 to get rid of fungal biomass. 25 mL of filtrate was transferred to a 250 mL separation funnel, then 100 mL of chloroform was added, the mixture was shacked, expelling gases accumulated in separating funnel as needed, and leaving the separation funnel for 15 min. The lower layer chloroform is collected and passed through filtering papers that having 10 g of anhydrous sodium sulfate Na_2SO_4 which spread by a sterilized spreader to form a homogenous layer above filtering papers. 10 mL of chloroform are added to the upper layer in the separation funnel and the funnel is shacked to

expel the collected gases then the funnel is left on the holder until the two layers separate, the lower layer is collected and passed through filtering papers that contain anhydrous sodium sulfate then the obtained layer of chloroform is collected and evaporated in the rotary evaporator until it gets dry, then it is kept in small pipes and covered with aluminum foil to prevent its exposing to light, and it is kept in freezer in -18^{o}C.

Identification of Aflatoxin B1 using HPLC

Aflatoxin B1 is characterized according to the method mentioned by **Cota-Arriola *et al.*, (2011)**. using a high-performance liquid chromatography HPLC, mobile phase uses acetonitrile : distilled water (60 : 40) by injecting 50 µL from the sample and the flow rate of mobile phase is 1.2mL/min , and a florescence detector is used to detect fungal toxin according to the wavelengths (Ex=365 nm , Em = 455 nm).

Diagnosis was made based on a match between Retention time (RT) between extracted AflB1 and AflB1 standard. Concentration of AflB1 was calculated according to the following equation:

$$\textbf{concentration}\ (\mu\textbf{g/mL}) = \frac{\textbf{standard concentration x sample's curve area}}{\textbf{standard poison's curve area}} \times \textbf{Dilution factor}$$

RESULTS AND DISCUSSION

characterization of chitosan

The result showed that the numbers of the functional groups of the chitosan extracted from *Agaricus bisporus* brown, (Figure 1) comparing with the spectra of FTIR for a commercial chitosan sample as a standard sample, (Figure 2). the samples of chitosan under consideration give a similar shape of commercial chitosan.

The active group which represents stretching band Hydroxyl appeared for extracted chitosan and commercial chitosan at the wave numbers 3353.95 and 3350.52 cm^{-1} respectively. While the stretching frequency of the group N-H at frequencies 3291.79 and 3289.20 cm^{-1} for extracted and commercial chitosan respectively. This result agrees with what **Poverenov *et al.*, (2018)** mentioned when testing the chitosan extracted from the stalks and fruiting body of champignon. The amide band of the produced chitosan appeared from the stalks of *A. bisporus* brown, commercial chitosan at frequencies 1643.93 and 1638.77 cm^{-1} respectively which represent carbonyl group (C=O) the (AmideI) whose absorbance value on the wave number 1640 – 1700 cm^{1}.

The bands at the wave number 1586.90 and 1587.75 for the extracted chitosan and commercial chitosan respectively refer to the group (N-H) in the second amide bond (Amide II). While the bands whose absorbance value appeared at the wave number 1373.39 and 1372.06 for the extracted chitosan and commercial chitosan respectively represent the bending vibration for the group C-N (Amide III). The band whose absorbance value appeared at the wave number 829.43 and 893.14 cm^{-1} represent glycosidic bond β- (1,4) in the extracted chitosan and commercial chitosan respectively **(Wu *et al.*, 2019)**.

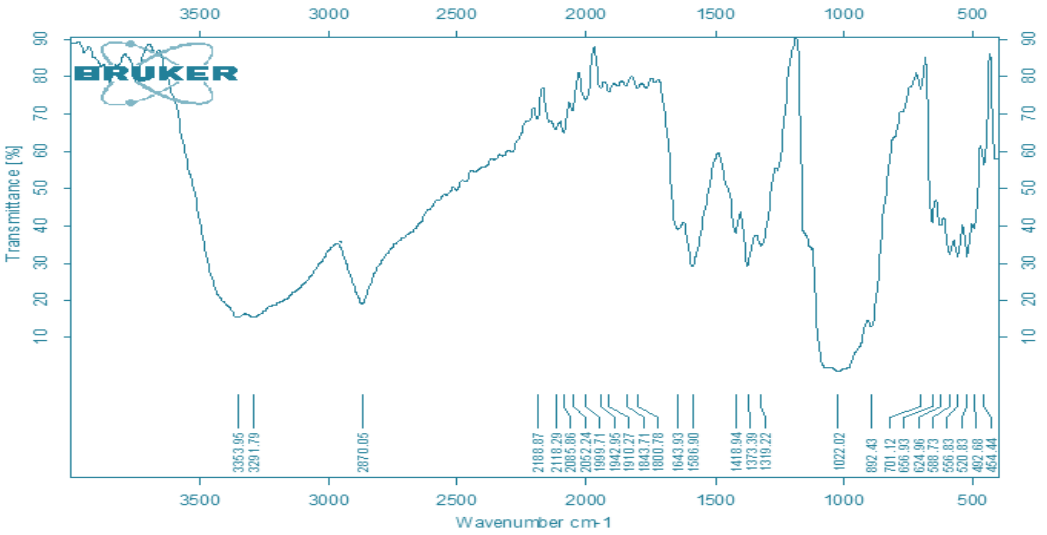

Figure (1): Infrared spectra of the chitosan extracted from the stalks of *A.bisporus* brown.

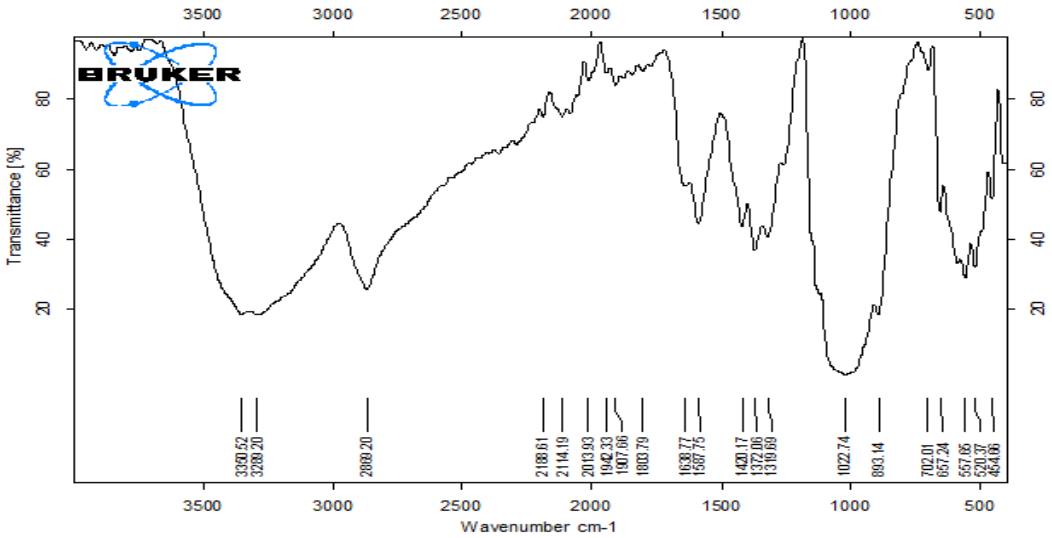

Figure (2): Infrared spectra of commercial chitosan.

Evaluation of chitosan efficiency in *Aspergillus flavus* growth inhibition on PDA media

It is clear from (Figure 3) that the inhibiting potency of chitosan increases directly with increasing concentration. The results show that adding chitosan to culture medium in concentrations 0.5, 1, 1.5 and 2% leads to inhibiting the growth of *A. flavus* 16, 36, 53 and 100 % respectively. these results correspond with what **Dewi & Nur, (2017**) found while he was studying the most potent concentration of chitosan towards some types of *Aspergillus*.

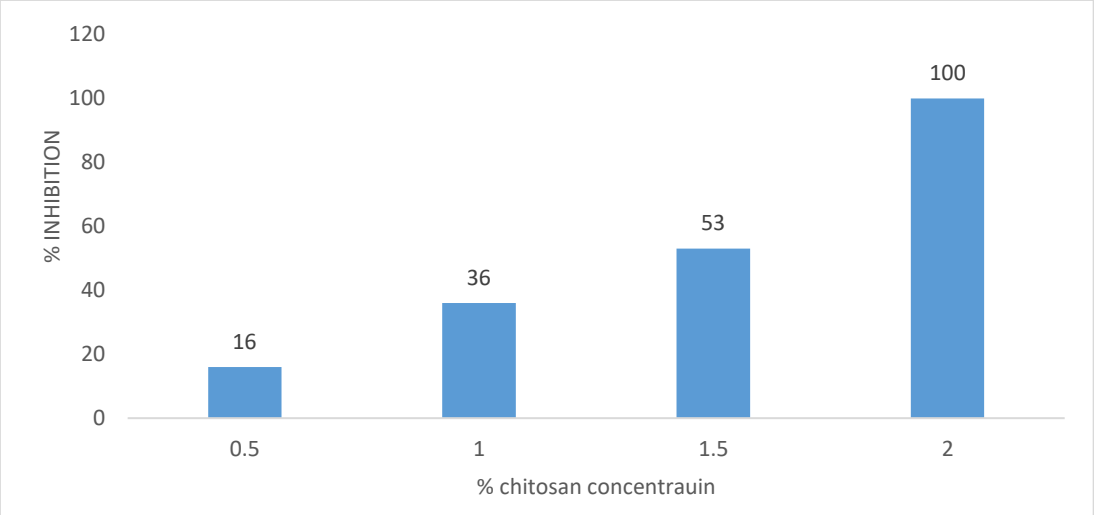

Figure (3): The effect of adding chitosan on the growth of *Aspergillus flavus* by using PDA media

The results contradict the study carried out by **Bukola *et al.*, (2023)**, which included a study of the inhibitory effectiveness of chitosan extracted from shrimp peels against *A.flavus*, which found that the highest inhibition rate was at a concentration of chitosan 0.5 mg / mL, which amounted to 61.7%, in While the higher concentrations 0.25, 0.75 and 1 mg / mL gave a lower inhibition rate, which amounted to 6.80, 20.4 and 12.2 %, respectively, and the reason for this result was explained by the fact that it is possible that chitosan has a stimulating effect on the defensive enzyme as another mechanism for the high activity of the fungus towards chitosan **(Xing *et al.*, 2015)**. This variation in the inhibitory activity of chitosan may be due to several reasons, including those related to the target isolate of the fungus. The varying tolerance of *A. flavus* isolates to different concentrations of chitosan may also be due to the difference in the unsaturated fatty acid composition, which is an essential part of the phospholipids in the layer bilayer lipids in the cell membrane which represent an important factor affecting membrane stability and fluidity, in general, the antifungal activity of chitosan is attributed to the ability of chitosan to easily penetrate fungal cell membranes and then bind to specific enzymes responsible for fungal growth and thus reduce its activities. In addition, increasing the concentration of chitosan increases the density of the cation charge $NH3^{+}$ in the chitosan solution, which can It easily attaches to the fungal membrane and changes its permeability, which in turn leads to the death of microorganisms **(Yien *et al.*, 2012)**.

The inhibition of producing of aflatoxin B1 from *A. flavus* by using chitosan

The rate of inhibiting the production of aflatoxin B1 from *A. flavus* in concentrations 0.5, 1 and 1.5 % for chitosan reaches 72.7, 86.7 and 100 % respectively (Figure 4). These results indicate that in addition to the ability of chitosan to inhibit fungal growth and aflatoxin production, chitosan has the ability to reduce Aflatoxin This result agrees with what was mentioned by **Solís-Cruz *et al.*, (2017)** which indicated that chitosan has the ability to reduce aflatoxin B1 by up to 34%.

The potency of chitosan in reducing Aflatoxin B1 may be due to the capability of these substances to adsorb Aflatoxin by forming bonds between the potent groups of chitosan with

other groups on Aflatoxin that are different in charge, which leads to the conversion of Aflatoxin into less-toxin compounds or toxin reduction, as chitosan contains a group of positive amines while Aflatoxin B1 contains negative oxygen. Aflatoxin, according to its combination, provides 6 positions of oxygen atoms that are available to react with the amino group in chitosan. Mostly, the reaction occurs between chitosan and Aflatoxin B1 at the oxygen atom number 6 according to energy and the negative charge which is higher than the oxygen in that position. Reaction occurs in the other positions depending on energy, so it is possible to say that the possibility of adsorption happens depending on energy, so chitosan can be an electrostatic attracting factor that is responsible for adsorption **(Juarez-Morales *et al.*, 2017)**.

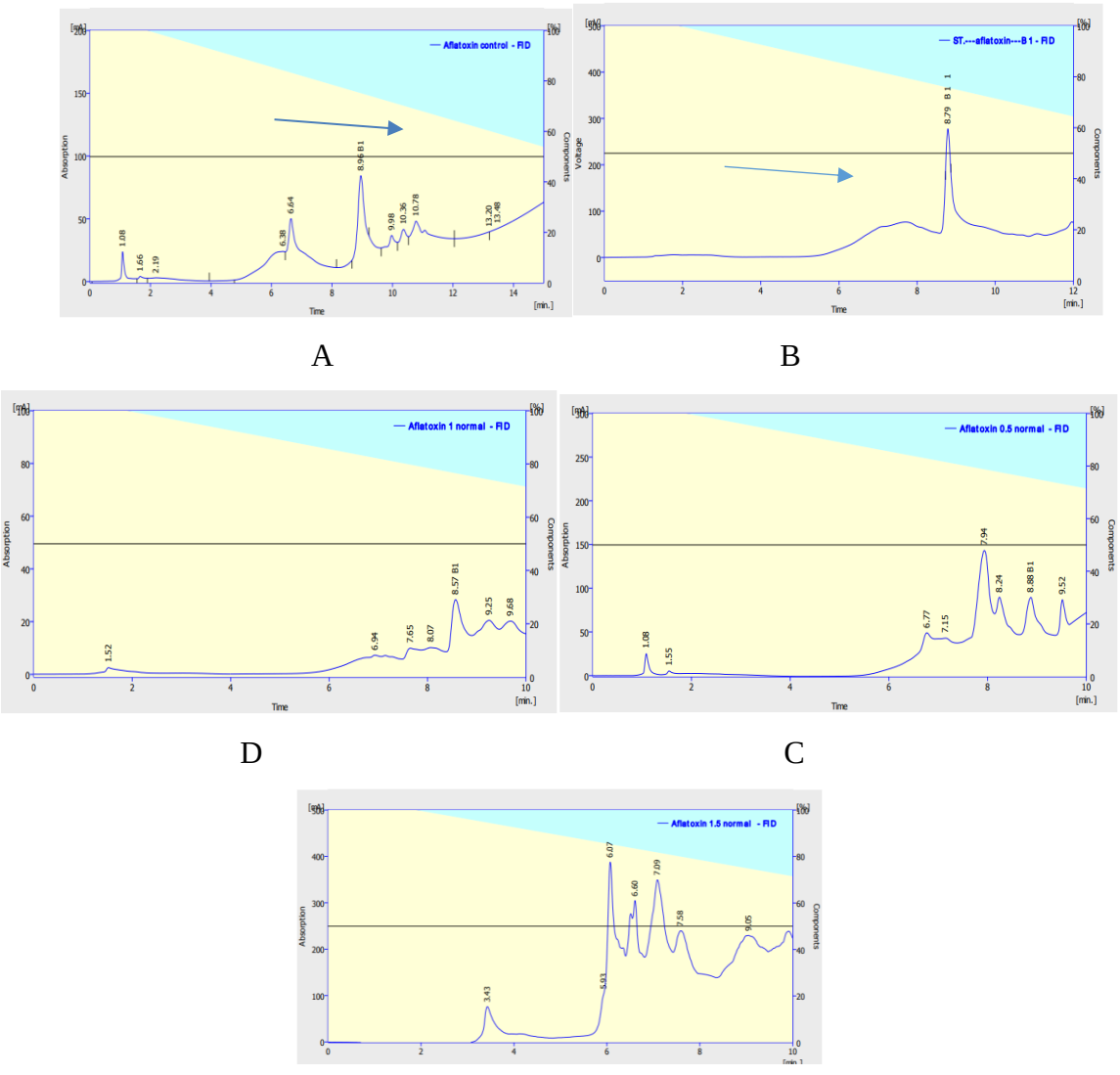

Figure (4): The effect of chitosan in inhibiting the production of Aflatoxin B1 from *A. flavus* by HPLC, (A): Standard Aflatoxin B1 poison, (B) Control coefficient, (C) Chitosan in concentration 0.5%, (D) Chitosan in concentration 1% (E) Chitosan in concentration 1.5%.

CONCLUSIONS

The results of this research showed that chitosan possesses a high inhibitory activity against *Aspergillus* fungus, and it also has effectiveness towards inhibiting the production of aflatoxin B1, as there was a direct relationship between the concentrations of chitosan and inhibiting the growth of the fungus and preventing it from producing the toxin AFB1.

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Ali & Shaker

المجلة العراقيه لبحوث السوق
وحماية المستهلك

(REVIEW ARTICLE)

GLUCOSE- 6-PHOSPHATE DEHYDROGENASE DEFICIENCY AND FAVISM

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ABSTRACT

Favism disease, also known as hemolytic syndrome (the breakdown of red blood cells) affects some people (especially male children) when they consume the fava beans (fava bean, broad bean) because they contain high concentrations of pyrimidine glycosides (vicine and convicine) or they take some medications or an imbalance in metabolism or infections. causing the generation of harmful oxygen forms, as these people suffer from a deficiency in the glucose-6-phosphate dehydrogenase (G6PD) enzyme, which is responsible for the availability of, NADPH, which is important in providing reduced glutathione forms, (GSH), as the latter contributes to the conversion of H_2O_2 into O_2 and H_2O, and thus prevents the harmful effects of oxidation in red blood cells, represented by their destruction. Symptoms include nausea, pale jaundice, and dark urine. the severity of the disease varies between patients, and the severity of episodes can vary in the same patient, therefore, diagnosing G6PD deficiency and educating the patient regarding safe and unsafe medications and foods is critical to prevent recurring episodes.

Key words: Vicine, Fava bean, Favism.

الفافزم ونقص انزيم glucose-6-phosphate dehydrogenase

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الخلاصة

مرض الفافزم او ما يطلق عليه بمتلازمه انحلال الدم (تكسر كريات الدم الحمراء) يحدث عند بعض الاشخاص (خصوصا الاطفال الذكور) عند تناولهم الباقلاء بسبب احتواءها على تراكيز عالية من الكلايكوسيدات البيرميدينيه (الفايسين والكونفايسين) او تناولهم بعض الأدوية او حدوث خلل في التمثيل الغذائي او الالتهابات المسببة لتوليد صور الاوكسجين المضر. اذ يعاني هؤلاء الاشخاص من نقص في انزيم glucose-6-phosphate dehydrogenase (G6PD) والمسؤول عن توافر NADPH المهم في توفير صور الكلوتاثيون المختزل (GSH) حيث يساهم الاخير في تحويل مركب H_2O_2 الى H_2O و O_2، وبذلك يمنع مضار الأكسدة في كريات الدم الحمراء والمتمثلة بتدميرها، تشمل الاعراض الغثيان ،الشحوب ،اليرقان والبول الداكن، تختلف شده المرض من مصاب الى اخر كما تختلف نوبات المرض في نفس المريض لذلك يعد تشخيص نقص الانزيم وتثقيف المريض فيما يتعلق بالأدوية والأطعمة الأمنه وغير الأمنه امر بالغ الأهمية لمنع تكرار نوبات المرض.

الكلمات المفتاحية: فافزم، باقلاء، فايسين.

INTRODUCTION

Pythagoras of Samos may have been the first person firmly assert that fava beans may be dangerous and even fatal to humans in the fifth century B.C. (**Chu *et al.*, 2017; Di Meo & Venditti, 2020) ,** he was unaware that the hazard was dependent on the genotype of the individual consuming the beans. This became clear only after G6PD deficiency was discovered in 1956. (**Karafin & Francis, 2019)**. Glucose-6- phosphate Dehydrogenase (G6PD) is the enzyme that catalyzes the first step of the hexose monophosphate shunt., which results in the formation of NADPH. This mechanism eventually protects RBCs from oxidative stress by reducing reactive oxidant species **(Salles *et al*., 2020; Sköld *et al*., 2017).**

G6PD insufficiency is widespread, however it is more frequent in, Southern Europe, Africa, the Middle East, Oceania and Southeast Asia (**Zuccotti *et al*., 2014**). G6PD insufficiency manifest itself clinically in a variety of ways with varying degrees of severity. G6PD insufficiency is categorized as class I-IV by the World Health Organization based on the severity of the G6PD deficiency. People in class II have a significant lack of enzymes, with G6PD activity less than 10% of what it should be. People in Class II have random hemolytic episodes, which usually happen after they are exposed to things that stress oxidants, like fava beans (as in this case) or oxidant drugs. G6PD deficiency can also be categorized by the G6PD gene variants found in certain ethnic groups, such as the class II Mediterranean-type G6PD deficiency. **(Di Meo & Venditti, 2020; Chu *et al*., 2017).**

Although G6PD deficiency is an X- linked recessive condition, most female carriers do not experience the primary clinical signs **(Puspitasari,2017; Prabhu & Rajeswari, 2018**). The two most important parts of favism are the red cell and the bean. Favism defies the conventional division between intraerythrocytic and extraerythrocytic causes of acute hemolytic anemia because it only appears when a person with G6PD-deficient red cells is exposed to specific substances present in fava beans, specifically Vicine and convicine, two - glucosides present in fava beans in high concentrations (up to 2% in dry weight) **(Johns & Hertzler, 2021**).

When fava beans are eaten, the glucosidases in both the fava bean and the digestive tract break down vicine and convicine into divicine (2,6, diamino 4,5-dihydroxypyrimidine) and isouramil (6-amino ,2,4,5 trihydroxypyrimidine), which are then released (Favism induced factors). The antifungal and pesticide properties of these highly reactive redox compounds probably help keep fava beans from going bad, but the compounds can also cause a favism attack (**Karafin & Francis, 2019).**

This article aims to explain the favism disease, the causes of its symptoms, method of diagnosis and the type of food that should be avoided.

Convicine and Vicine

The vicine [2,6-diamino - 4,5 -dihydroxypyrimidine5 - (-D- glucopyranoside)] and convicine [2,4,5-trihydroxy-6-aminopyrimidine 5-(-D-glucopyranoside] are made up of one molecule of glucose coupled to one pyrimidine nucleoside (aglycones) (**Cardador-Martinez *et al*., 2012**). These substances are virtually exclusively found in the legume species Vicia faba, which is a member of the vetch family. Several species of the Vicia genus, such Vicia narbonsensis, have also been discovered to contain trace levels of convicine and vicine (0.1 mg/g) (**Pavlik *et al*., 2002**). The unusual class of antinutritional substances known as convicine

and vicine is almost entirely confined to the genus Vicia. These are the primary causes of the favism medical condition **(Ray & Georges, 2010**).

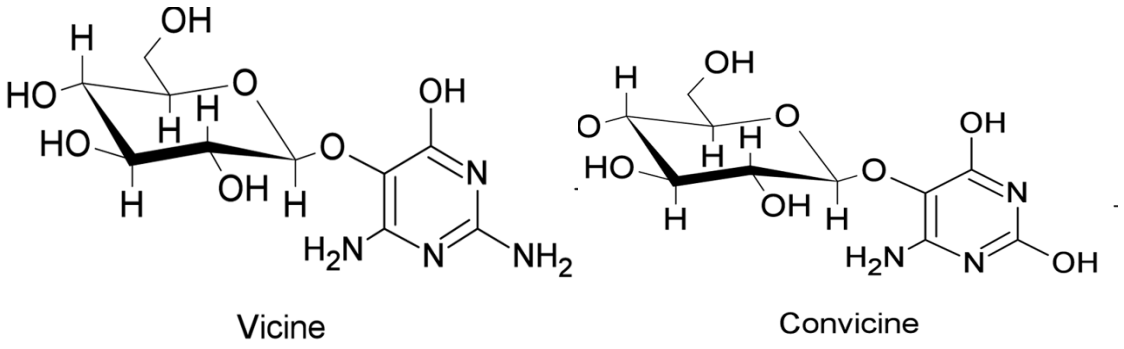

Figure (1): structural formulas of vicine and convicine (**Ray & Georges, 2010**)

How Vicine and Convicine Play a Role in Favism

Vicine and convicine are broken down into divicine and isouramil by -glycosidase, the enzyme made by anaerobic micro flora in the intestinal tract. These compounds are aglycone derivatives, cause favism, a genetic disease that leads to acute hemolytic anemia (**Mckay, 1992**). In natural red blood cells, the oxidative effect of aglycones is quickly taken care of by the effect of compound NADPH, which is the result of the pentose phosphate pathway. But in red blood cells that aren't natural and are sensitive to favism, the effect of compound NADPH can’t get rid of the oxidative effect of aglycones because there isn't enough G6PD and NADPH. So, the aglycones cause change GSH into GSSG (**Multari *et al*., 2015**).

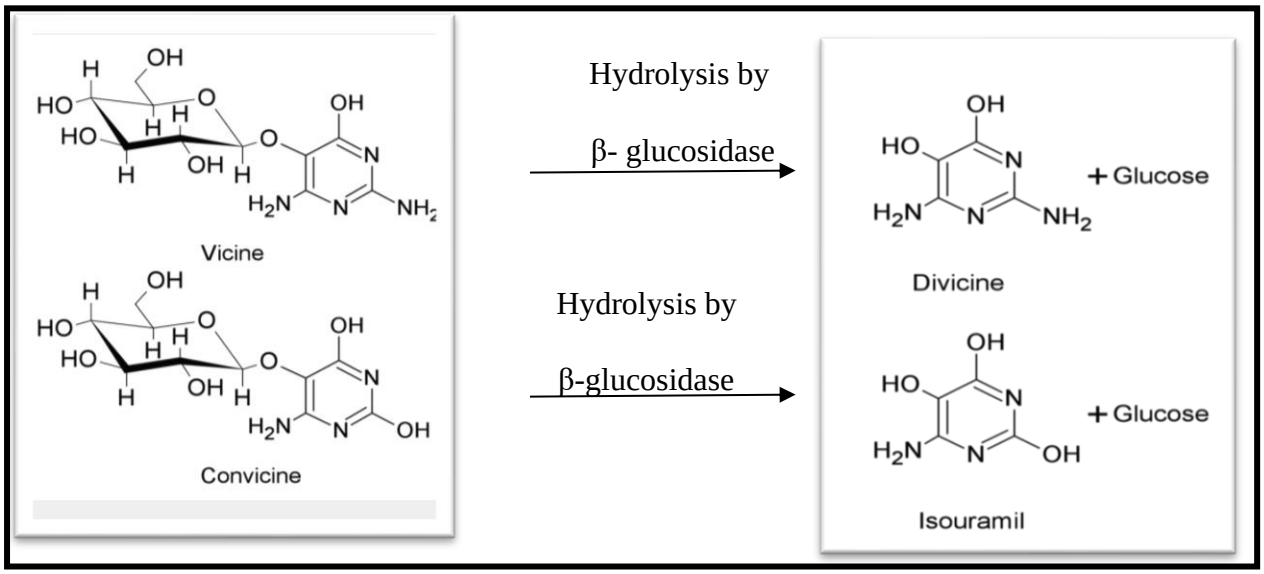

Figure (2): Enzymatic hydrolysis of vicine and convicine to divicine and isouramil (Mckay, 1992).

Glucose-6-phosphate Dehydrogenase Deficiency and Favism

The G6PD enzyme is involved in catalyzing the first step in the pentose phosphate pathway (PPP), which leads to formation of antioxidants that protect cells from oxidative damage. This process produces NADPH, which keeps the reduced glutathione (GSH) inside the cell. Reduced glutathione functions as an antioxidant, protecting cells from oxidative damage **(Luzzatto & Arese, 2018**) (Fig 2).

As a result, a patient with G6PD deficiency is unable to shield their red blood cells (RBC) from oxidative stress caused by several medications, metabolic disorders, infections, or fava bean ingestion (**Cappellini & Fiorelli, 2008**). In the majority of cells, other metabolic pathways support the production of the required intracellular NADPH. In contrast, RBC have no alternative means of producing NADPH.

Thus, G6PD deficiency in RBC results in death. The conversion of oxidized glutathione (GSSG) into the tripeptide known as GSH requires the assistance of NADPH, this tripeptide functions as a reducing agent along with the enzyme glutathione peroxidase for the detoxification of hydrogen peroxide. This mechanism results in the conversion of GSH into GSSG, which lowers the levels of GSH. In the presence of NADPH, glutathione reductase catalyzes the conversion of GSSG to GSH, which results in the regeneration of GSH **(Njalsson& Norgren, 2005**).

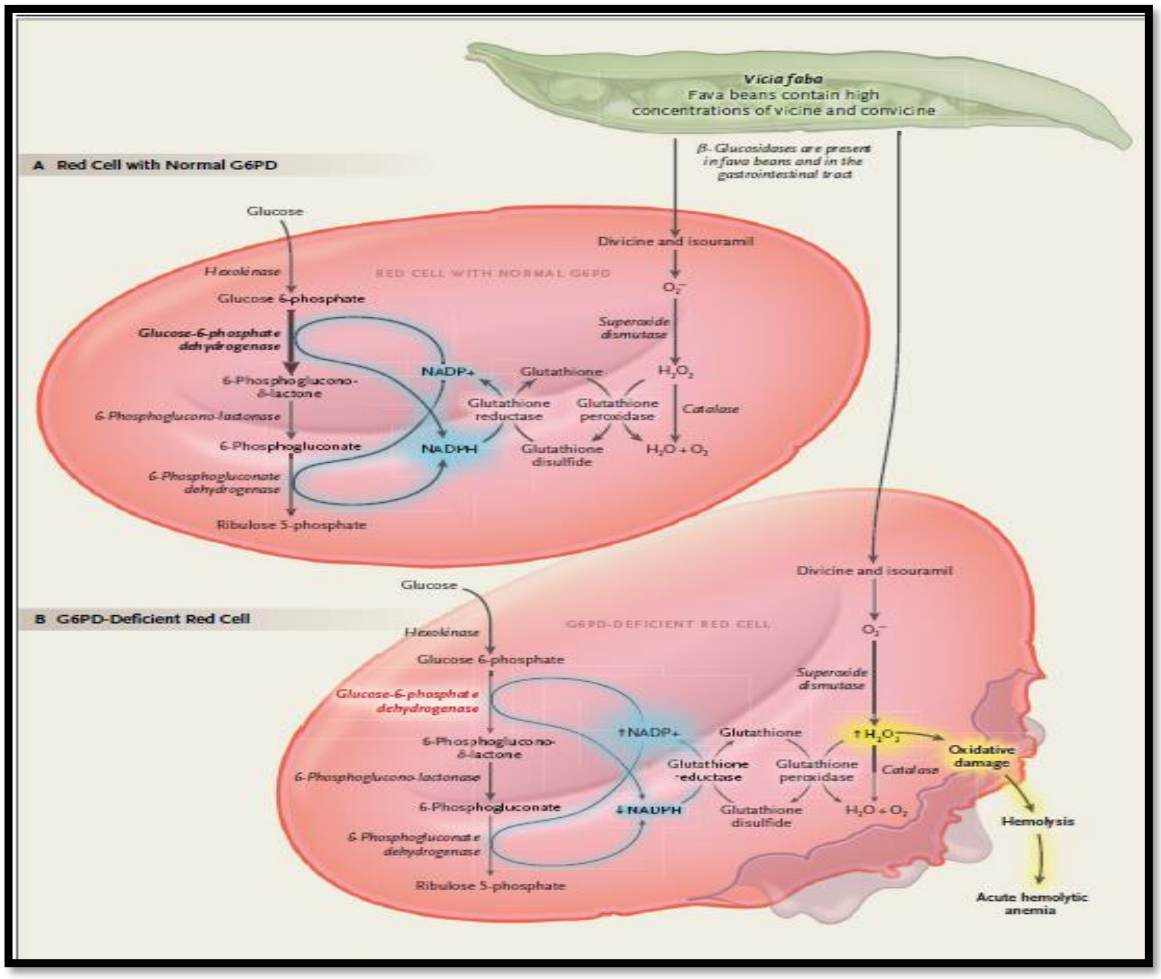

Figure (3): Biochemical role of G6PD enzyme in the cells (**Luzzatto & Arese, 2018**)

Processing is used to eliminate convicine and vicine

Convicine and vicine are stable in heat and water, which makes them hard to get rid of. several studies, most of which focused on soaking (**Jamalian & Ghorbani, 2005; Al-Salmani & Musa**, **2015),** roasting and cooking (**Juma'a, 2010; Cardaror-Martinez *et al.*, 2012**), and fractionation, have looked at how soaking, roasting, and cooking, as well as fractionation, can reduce the amount of vicine and convicine in a plant **(Coda *et al*., 2015).** A number of processing methods, such as enzyme treatments **(Pulkkinen *et al.*, 2016)**, fermentation **(Goyoaga *et al*., 2008),** and germination (**Coda *et al*., 2015; Rizzello *et al*., 2016)**, can cause the hydrolysis of the - glycosidic link. Vicine and convicine are broken down by hydrolysis, but at the same time, the aglycones are released. Most studies have been about getting rid of convicine and vicine, so they haven't looked into how the toxic aglycones are released.

G6PD Deficiency Diagnose

1-peripheral smear

Patients who show signs of acute hemolysis are given this diagnosis. A peripheral smear may show RBCs that have a blister-like appearance (blister cells) and RBCs with Heinz bodies, which are denatured hemoglobin particles that can only be seen with special stains. In individuals with an intact spleen, these cells are removed, thus although they may be evident early in the hemolytic episode, they do not persist (**Ghergurovich *et al*., 2020**).

2-G6PD activity

G6PD activity can be checked but during and right after a hemolytic episode, tests may give false-negative results because of the destruction of older, less healthy RBCs and the production of reticulocytes, which are high in G6PD. So, testing may need to be done again a few weeks after the acute event (**Liu *et al*., 2015).**

Symptoms

Symptoms may appear in 5 to 24 h. Jaundice, dark, crimson urine, pallor, headaches, weariness, fast pulse, dyspnea, stomach discomfort, headache, vomiting, nausea, and a high fever are among the symptoms (**Vottonen, 2018**).

When red blood cells break down, they leave behind a yellow substance called bilirubin. When a person with G6PD has a hemolytic crisis, however, bilirubin levels rise above what is normal. This makes the skin and eyes turn yellow (**Crépon *et al*., 2010).** In severe cases, the symptoms can lead to hemolytic anemia, which is followed by hemoglobinuria.

Types

According to the body's activity of the G6PD enzyme, there are five different types of G6PD deficiency:

Class 1: Chronic hemolytic anemia, with a G6PD enzyme activity under 10%, This indicates that red blood cell degradation is greater than red blood cell regeneration.

Class 2: 10% or less G6PD enzyme activity; disintegration of red blood cells only after being exposed to trigger foods, medications or illnesses.

Class 3: 10–60% G6PD enzyme activity, and the only time symptoms show up is when there are infections.

Class 4: More than 60% activity of the G6PD enzyme, but only mild symptoms.

Class 5: higher G6PD enzyme activity than healthy people, and people often don't know they have this condition because they don't have any symptoms.

Classes one through three are clinically important. This means that when exposed to triggers, there is a high chance of hemolytic anemia, which often needs medical help and treatment (**Crépon *et al*., 2010; Gulewicz *et al*., 2014).**

What to eat and what to avoid

Patients with G6PD deficiency may prioritize eating antioxidant-rich foods to minimize oxidative damage, protect red blood cells, and improve overall health **(Stone *et al*., 2020).** Glutathione replenishment is insufficient in patients with G6PD deficiency, making it essential to maintain vitamin D levels in the body. Current research suggests a relationship between G6PD deficiency and vitamin D levels (**Richardson *et al*., 2021; Subramani *et al*., 2020).**

People with a G6PD deficiency who eat fava beans are more likely to get hemolytic anemia (**La Vieille *et al*., 2019**). Also five-year study of one thousand G6PD-deficient people

showed that the following foods caused hemolytic anemia in some people: broad beans, peanuts, lentils, green peas, black peas, and chickpeas (**Luzzatto & Arese, 2018**).

Substances to avoid using

The following compounds might exacerbate G6PD signs: Naphthalene is a chemical found in home items including mothballs, Toluidine blue is a dye used in several scientific experiments and henna, a plant-based dye commonly used for body art (**Malik *et al.*,2020)**.

drug to avoid

Drug is the least common reason why people with G6PD get hemolytic anemia. But here are the kinds that people with G6PD deficiency should avoid: Diclofenac sodium, Co-trimoxazole, Nitrofurantoin, Dapsone, Acetylsalicylic acid, Rasburicase, Acalypha indica, Ibuprofen, Primaquine, Methylene blue, and Phenazopyridine (**Georgakouli *et al*., 2019).**

Vitamin C (ascorbic acid)

Vitamin C works in the body as an antioxidant that dissolves in water. It can easily get rid of hypochlorite and reactive oxygen and nitrogen species. The products of oxidation with one and two electrons are easy to make again with glutathione and NADPH **(Johns & Hertzler, 2021).**

People who don't have enough G6PD have a less efficient pentose phosphate pathway that turns NADP into NADPH. This uses up the antioxidant glutathione and raises the levels of free radicals and oxidative stress, which break down red blood cells and cause hemolysis. Even though vitamin C is part of the antioxidant defense system, putting erythrocytes in a solution with 0.2mM vitamin C caused oxidative stress and used up glutathione. The pentose phosphate pathway, on the other hand, became more active **(Liu *et al*., 2015).**

In vitro tests with erythrocytes from healthy people and people with G6PD deficiency showed that a solution of 5mM ascorbate alone or in combination with divicine from fava beans increased the production of hydroxyl radical markers, which was then made stronger by the addition of chelated iron EDTA (**Harcke *et al*., 2019).**

CONCLUSIONS

Favism results from a sensitivity to the ingestion of fava beans when red blood cells lack glucose-6-phosphate dehydrogenase, in addition, other substances outside fava beans, such as a broad variety of medications and industrial pollutants, may cause favism in G6PD-deficient people. Children between the ages of two and six are more likely to experience it, and boys are more affected than girls. A reduction in the amount of vicine and convicine is caused by several manufacturing operation, including heating, boiling, and soaking the beans, which lowers the risk of infection. The most common therapeutic intervention is a blood transfusion, and a recovery time of two to three days is anticipated.

RECOMMENDATIONS

The families can be given instruction about the condition and the offending agents of hemolysis., Detailed printed leaflets about the condition and the offending agents should be available in order to be given to the families., Kits for assessing the level of G6PD should be available all through the year, and especially during the fava season, Blood units should be available in a good supply in all pediatric hospitals, in order that the families will not suffer a lot in finding compatible blood for their ill children, laboratory evidence of G6PD deficiency requires particular attention of the public., Health education sessions and further

epidemiological studies are required because early detection and prevention is the key strategy for successful management and control of this genetic disease.

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المجلة العراقيه لبحوث السوق
وحماية المستهلك

(ARTICLE REVIEW)

AN ANALYSIS OF CONSUMER BEHAVIOR IN MODERN MARKETS

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ABSTRACT

Quantitative research methods are crucial in understanding consumer behavior in modern markets. These methods involve collecting and analyzing numerical data to uncover patterns, trends, and correlations related to consumer preferences and choices. Researchers can gain insights into consumer identity, values, taste, consumption experiences, and even topics such as invisible brands, material culture, and fetishes in contemporary consumption by utilizing statistical models. Additionally, quantitative research methods allow for the examination of consumer sentiment, brand engagement, and market maven behavior, providing valuable information for marketers and businesses.

The application of quantitative research methods in consumer behavior studies not only enhances our understanding of consumer preferences but also enables the integration of past findings with new observations, driving advancements in the field of consumer research. This integration is essential for effectively capturing the dynamic nature of consumer behavior, especially in response to global events and societal changes.

Keywords: Consumption, Qualitative research methods, Marketers and Businesses

دراسة تحليلية لسلوك المستهلك في الأسواق الحديثة

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الخلاصة

تعتبر طرق البحث الكمي حاسمة في فهم سلوك المستهلك في الأسواق الحديثة. تتضمن هذه الطرق جمع وتحليل البيانات الرقمية للكشف عن الأنماط والاتجاهات والارتباطات المتعلقة بتفضيلات المستهلك واختياراته. يمكن للباحثين اكتساب رؤى حول هوية المستهلك وقيمه وذوقه وتجاربه الاستهلاكية وحتى موضوعات مثل العلامات التجارية غير المرئية والثقافة المادية والأهواء في الاستهلاك المعاصر من خلال استخدام النماذج الإحصائية. بالإضافة إلى ذلك، تسمح طرق البحث الكمي بفحص مشاعر المستهلك والتفاعل مع العلامة التجارية وسلوك خبراء السوق، مما يوفر معلومات قيمة للمسوقين والشركات.

إن تطبيق طرق البحث الكمي في دراسات سلوك المستهلك لا يعزز فهمنا لتفضيلات المستهلك فحسب، بل يتيح أيضًا دمج النتائج السابقة مع الملاحظات الجديدة، مما يؤدي إلى تقدم في مجال أبحاث المستهلك. يعد هذا التكامل ضروريًا لالتقاط الطبيعة الديناميكية لسلوك المستهلك بشكل فعال، وخاصة في الاستجابة للأحداث العالمية والتغيرات المجتمعية.

الكلمات المفتاحية: استهلاك، طرق البحث الكمي، المسوقين والشركات

INTRODUCTION

Consumer behavior analysis is a multifaceted field that integrates insights from various disciplines such as economics, psychology, sociology, and anthropology. Understanding consumer behavior is crucial for businesses as it influences marketing decisions and strategies. The process of decision-making in purchasing, as described by the model of Engel and Blackwell, involves stages such as problem recognition, information analysis, evaluation of solutions, choice, and post-purchase evaluation (**Irina & Georgiana, 2007**). Moreover, the interconnected nature of the modern world underscores the impact of global events on consumer behavior, emphasizing the need to integrate past research with new observations to advance our understanding (**Malter *et al.*, 2020**).

These foundational concepts and theories provide a framework for comprehensively analyzing consumer behavior in modern markets, setting the stage for the subsequent discussions in this essay.

Consumer behavior analysis is a crucial aspect of contemporary business, with its roots tracing back to the 1960s when it was initially considered an applicative domain of psychology. The study of consumer behavior encompasses a multidisciplinary approach, integrating economics, psychology, sociology, and anthropology. As highlighted by Rabontu and Boncea (**Irina & Georgiana 2007**), understanding consumer behavior is vital for businesses as it influences marketing decisions, brand positioning, market segmentation, product development, and advertising strategies. Moreover, the interconnected nature of the modern world, as noted by Malter et al. (**Malter *et al.*, 2020**), means that global events can significantly impact consumer behavior, emphasizing the need to integrate past findings with new observations and research to advance our understanding of consumer behavior. This historical context and contemporary significance lay the foundation for comprehending the complexities of consumer behavior in modern markets.

Theoretical Framework

The theoretical framework of consumer behavior analysis encompasses various perspectives and models that contribute to understanding consumer behavior in modern markets. This includes integrating past findings with new observations and fresh research to advance our understanding of consumer behavior (**Malter *et al.*, 2020**). The study of consumer behavior is essential for enterprises as it influences marketing decisions such as brand positioning, market segmentation, product development, and advertising strategies (**Irina & Georgiana, 2007**). The multidisciplinary nature of consumer behavior analysis draws from economics, psychology, sociology, and anthropology, with a focus on the decision-making process, factors influencing purchasing behavior, and the analysis of consumer behavior. Understanding these theoretical underpinnings is crucial for comprehending the complexities of consumer behavior in modern markets.

Consumer Behavior Theories

Consumer behavior theories encompass a wide range of perspectives that contribute to the understanding of consumer actions and decision-making processes. The study of consumer behavior is interdisciplinary, drawing from economics, psychology, sociology, and anthropology, and plays a vital role in shaping marketing decisions for enterprises. The decision-making model by Engel and Blackwell outlines the stages of consumer behavior,

encompassing the recognition of the problem, information analysis, solution evaluation, choice, and results (**Irina & Georgiana, 2007**). Additionally, external factors such as society, culture, and group interactions, as well as internal factors like values and personality, significantly impact consumer behavior (**Azevedo *et al*., 2008**).

The analysis of consumer behavior is crucial for enterprises in understanding marketing decisions, including brand positioning, market segmentation, product development, and advertising strategies. The multifaceted nature of consumer behavior theories underscores the importance of considering both internal and external influences on consumer decision-making processes.

RESEARCH METHODOLOGY

The research methodology for studying consumer behavior involves a multi-faceted approach to understanding the decision-making process and cognitive components that influence purchasing and consumption decisions. The selection of research methods is grounded in the need to comprehend the complex nature of consumer behavior, which is influenced by stimuli and responses. According to (**Irina & Georgiana, 2007**), the study of consumer behavior encompasses the cognitive processes leading to the act of purchase, emphasizing the significance of understanding sensation, attention, and perception in consumer decision-making. Additionally, (**Azevedo *et al*., 2008**) highlight the use of non-random and snowball sampling techniques in their research, demonstrating the importance of strategic sampling in capturing diverse perspectives on consumer behavior. Furthermore, the employment of closed-ended questionnaires in their study signifies a structured approach to gathering data on consumer buying behavior in fashion retailing.

Factors Influencing Consumer Behavior

Consumer behavior is a complex process influenced by various factors. According to Rabontu and Boncea, consumer behavior is shaped by endogenous variables such as personal characteristics and cognitive processes, as well as exogenous variables including environmental and socio-demographic factors, incomes, prices, social group influences, and media. Additionally, the analysis of information pursued by each consumer involves the role of three psychological concepts: sensation, attention, and perception. Sensation corresponds to the awakening of the senses determined by stimuli, while perception represents the process of acknowledging variables of the environment, and attention is the process that defines the information to be approached. Furthermore, the model of Engel and Blackwell describes consumer behavior as a multi-stage process, including problem recognition, information analysis, evaluation of possible solutions, choice, and results. Understanding these factors is crucial for enterprises in making informed marketing decisions, as consumer behavior reflects people's conduct when it comes to purchasing and consuming goods and services (**Irina & Georgiana, 2007**). This multidisciplinary field encompasses economics, psychology, sociology, and anthropology, with research focusing on the process of decision-making in purchasing, the factors influencing purchase behavior, and understanding the consumer.

Psychological Factors

Psychological factors play a crucial role in shaping consumer behavior in modern markets. Research has shown that consumer behavior is influenced by cognitive and emotional

determinants, which impact their choices and preferences in the marketplace (**Marian *et al.*, 2022**). The affective perspective emphasizes the confluence between mental processes, personality, and consumer behavior, highlighting the significance of motivational factors in online consumer behavior and purchase decisions. Additionally, the study demonstrates a shift in consumer satisfaction from being driven by physical needs to psychological needs, particularly in the online environment, leading to adaptive modifications in purchasing patterns and behavior. Furthermore, the psychological profiles of online buyers have been validated through statistical tests, revealing a positive attitude towards online purchases, with different age groups showing varying inclinations to shop online.

Consumer behavior is also influenced by endogenous and exogenous variables, encompassing personal characteristics, cognitive processes, and environmental factors such as socio-demographics, incomes, and prices of merchandise (**Irina & Georgiana, 2007**). The process of consumer behavior involves the perception of stimuli, information processing, attitude, motivation, and manifest behavior, which includes the act of purchasing or using a product. Moreover, psychological concepts such as sensation, attention, and perception significantly impact the information consumers seek, with individuals selectively interpreting stimuli that attract their attention and correspond to a state of internal disequilibrium. Understanding these psychological factors is essential for businesses to comprehend and respond effectively to consumer behavior in modern markets.

Consumer Decision-Making Process

The consumer decision-making process involves several stages that consumers go through when making purchasing choices. These stages include problem recognition, information search, evaluation of alternatives, purchase decision, and post-purchase evaluation (**Goodhope, 2013**). During the problem recognition stage, consumers identify a need or want that can be satisfied through a purchase. This is followed by an information search where consumers seek out information about the available options. The next stage involves the evaluation of alternatives, where consumers assess the different options based on various factors such as price, quality, and personal preferences. Subsequently, the purchase decision is made, and finally, consumers engage in post-purchase evaluation to assess their satisfaction with the chosen product or service.

Furthermore, the consumer decision-making process is influenced by the processing capacity of consumer information (**Sandoval & Ferdaous, 2015**). The analysis of consumer behavior and information processing reveals that consumers do not always have unlimited information processing capabilities. Instead, they may rely on heuristic rules and make choices based on imperfect information. This challenges the traditional assumption of rationality and perfect information processing. Understanding the cognitive processes involved in consumer decision-making is crucial for marketers to tailor their strategies to effectively reach and influence their target audience.

Stages of Decision-Making

The consumer decision-making process encompasses several distinct stages that individuals navigate when making purchasing decisions. These stages typically include problem recognition, information search, evaluation of alternatives, purchase decision, and post-purchase evaluation (**Sandoval & Ferdaous, 2015**). Each stage involves specific

cognitive and behavioral activities, such as recognizing a need or desire for a product, seeking information about available options, assessing the features and benefits of different alternatives, making the actual purchase, and reflecting on the satisfaction or dissatisfaction with the chosen product after its use. It's important to note that the decision-making process is influenced by various factors, including the individual's interpretive capacity, information-processing capabilities, and external communication infrastructures (**Gambo *et al.*, 2013**). Therefore, understanding these stages and the underlying cognitive processes is crucial for businesses aiming to effectively market their products and services to consumers.

Impact of Technology on Consumer Behavior

Technological advancements have significantly impacted consumer behavior in modern markets. The availability of Big Data about online and offline behavior has led to a substantial increase in the use of secondary data for consumer research (**Malter *et al.*, 2020**). Furthermore, methods in computer science have advanced the ability to efficiently analyze large corpuses of unstructured data, enabling a better understanding of how the "new" consumer interacts with other consumers and companies in the current marketplace. Additionally, Lapina (**Lapina, 2017**) highlights that information and communication technologies have caused profound changes in traditional models of consumer behavior, forming new patterns of consumption. The globalization of advertising practices has also increased its influence in various aspects of life, transforming traditional models of consumer behavior and forming consumerism, which is a specific source of social conflicts and tensions.

These insights underline the critical role of technology in shaping consumer behavior and the need for a better understanding of how it will continue to evolve in the future. Understanding these changes can provide valuable insights for businesses and researchers alike, especially in the context of long-term sustainability, social equality, and ethical business practices.

E-commerce and Online Shopping

(**Krypton, *et al.*, 2018**) emphasizes the impact of e-marketing variables on consumer behavior, highlighting the influence of personal, psychological, and cultural factors. The study underscores the convenience and interactivity offered by social media platforms, which not only facilitate transactions but also enhance relationships and enable information exchange between buyers and sellers. Additionally, Praneeth et al. (**Praneeth *et al.*, 2019**) stress the increasing role of online shopping environments in shaping consumer motivations and behaviors. Their review emphasizes the complexity of understanding online consumer behavior and the diverse reasons driving consumers to shop online, indicating the need for a nuanced approach to analyzing the impact of e-commerce on consumer behavior. These findings underscore the multifaceted influence of e-commerce and online shopping on consumer behavior, emphasizing the interplay of technological, social, and psychological factors in shaping modern purchasing patterns.

Cross-Cultural Consumer Behavior

Cross-cultural consumer behavior plays a crucial role in understanding consumer preferences and decision-making processes in diverse market environments. Cultural factors have a significant influence on consumer behavior, shaping attitudes, information processing,

and purchasing decisions. According to Rabontu and Boncea (**Irina & Georgiana, 2007**), consumer behavior encompasses the totality of consumer acts and focuses on cognitive components, emphasizing the interdependency of various factors. The systemic approach to understanding consumer behavior likens the consumer to a 'black box,' where behavior is a response to external stimuli and internal processes.

Moreover, Hallab's (**Hallab, 2009**) study on acculturation to global consumer culture (AGCC) and ethnic identity (EID) highlights the impact of local and global cultural influences on consumer behavior. The findings underscore that despite globalization, consumer behaviors vary across different cultures and can be influenced by local, global, or both cultural factors. This emphasizes the importance of studying cross-cultural consumer behavior to comprehend the complex interplay of local and global cultural forces on consumer preferences and decision-making processes. Understanding these dynamics is essential for businesses operating in diverse markets to tailor their marketing strategies effectively.

Cultural Influences on Buying Behavior

Cultural influences play a significant role in shaping consumer behavior, impacting individuals' choices in the marketplace. According to (**Irina & Georgiana, 2007**), consumer behavior is influenced by cultural norms, values, and traditions, which shape the psychological components of sensation, attention, and perception. This influence is further emphasized by (**Hallab, 2009**). The study categorizes products into culture-bound and culture-free categories, demonstrating how consumption patterns are influenced by cultural factors such as food, clothing, personal care products, and household appliances. The findings suggest that local products are more closely associated with ethnic identity, while certain products like consumer electronics may transcend cultural influences.

These insights underscore the intricate relationship between culture and consumer behavior, emphasizing the need for a comprehensive understanding of cultural influences in modern markets.

Ethical Considerations in Consumer Behavior Research

Ethical considerations are paramount in consumer behavior research, as they shape the methods and outcomes of studies that seek to understand and influence consumer actions. Researchers and businesses must adhere to moral and professional standards when conducting consumer behavior research. This involves obtaining informed consent from participants, ensuring their privacy and confidentiality, and avoiding any form of coercion or deception in data collection and analysis (**Malter *et al.*, 2020**). Moreover, ethical considerations extend to the dissemination of findings, requiring transparency and accuracy in reporting research results to avoid misleading consumers or stakeholders.

The multidisciplinary nature of consumer behavior research, drawing from fields such as economics, psychology, sociology, and anthropology, underscores the need for researchers to navigate ethical challenges with sensitivity and expertise (**Irina & Georgiana, 2007**). The evolution of consumer behavior research has been driven by global events and technological advancements, emphasizing the importance of continually integrating past findings with new observations to advance the understanding of consumer behavior in an ethical and responsible manner.

Ethical Issues in Marketing

Ethical issues in marketing encompass a wide range of considerations, from the promotion of controversial products to the use of consumer data for targeted advertising. (**Cohen, 2007**) highlights that marketers often face ethical dilemmas when influencing consumer behavior, as the line between ethical and unethical practices is not always clear. This is particularly evident in areas such as pricing, branding, marketing to children, puffery, and stereotyping. Furthermore, (**Laczniak & Murphy, 2006**) discuss the ethical implications of technology-aided marketing practices, emphasizing the concerns surrounding consumer data collection and profiling. They note that many consumers feel a loss of control over their personal information and express discomfort with certain marketing techniques that they perceive as intrusive and exploitative.

These references underscore the complex ethical landscape that marketers navigate, prompting discussions on the responsible promotion of products and the ethical use of consumer data for targeted marketing campaigns. The evolving nature of marketing technology further complicates these ethical considerations, raising new questions and challenges for marketers and regulatory frameworks alike.

Brand Loyalty and Consumer Behavior

Brand loyalty is a crucial aspect of consumer behavior that significantly impacts market dynamics and company performance. Factors influencing brand loyalty include customer satisfaction, perceived quality, product involvement, and brand trust (**Saidu, 2014**). Research has shown that brand loyalty plays a vital role in a company's advertising strategies, with loyal customer bases contributing to increased market share and higher returns on investment. Additionally, brand loyalists can enhance a company's bargaining power with suppliers, partners, and channels, positively affecting shareholder value and reducing associated risks.

Moreover, the concept of brand loyalty is multifaceted and has been measured in various ways, such as analyzing repeat purchases and assessing brand commitment and positive attitudes toward a brand (**Oates, 2018**). Companies need to consider a balanced approach of qualitative and interpretive methods to understand how consumers interact with their preferred brands, as this can lead to fostering true brand loyalty and ultimately impacting profitability.

Factors Influencing Brand Loyalty

Brand loyalty is a critical aspect of consumer behavior, influencing purchasing decisions and long-term consumer attachment to specific brands. Factors influencing brand loyalty encompass a range of elements that contribute to consumer preferences and repeat purchases. These factors include customer satisfaction, perceived quality, brand trust, and product involvement (**Saidu, 2014**). In addition, Dai and Chen (**Dai & Chen, 2017**) emphasize the significance of previous usage experience in shaping brand loyalty, highlighting the role of cognitive, effective, conation, and action loyalty in consumer behavior. Moreover, the evolving nature of the drivers of brand loyalty, such as perceived risk, inertia, habit, and satisfaction, underscores the dynamic and complex nature of consumer attachment to brands.

Understanding these factors is vital for marketers and managers, as it enables them to predict and influence consumer purchasing behavior. By recognizing the drivers of brand loyalty, companies can tailor their strategies to enhance consumer attachment to their brands,

ultimately leading to increased market share and higher returns on investments. Therefore, a comprehensive understanding of the multifaceted factors influencing brand loyalty is essential for businesses seeking to thrive in modern markets.

Consumer Behavior in Sustainable Markets

Consumer behavior in sustainable markets is significantly influenced by environmental concerns and sustainability principles. The societal marketing concept, which emphasizes serving and satisfying human needs while considering society's long-term interests, has gained traction in the modern market landscape (**Hunt & Reynolds, 2009**). For instance, companies like the Body Shop have successfully addressed cosmetic needs while meeting environmental concerns, positioning themselves as ethical brands and appealing to consumers seeking products with good environmental credentials. Additionally, the commercial success of Toyota's Prius, a hybrid electric car, highlights the growing consumer demand for sustainable products. This shift in consumer preferences necessitates that product designers and marketers prioritize sustainable design and "green" issues, integrating them into product development and marketing strategies.

Moreover, marketing plays a crucial role in driving green purchase decisions, as evidenced by the need for well-planned communication to increase awareness about sustainability and the benefits of green products (**Trivedi, 2015**). Salesmen also have a significant impact on consumer behavior by guiding customers towards green products, educating them about the advantages of sustainability, and encouraging altruism, ultimately driving green purchases. These insights underscore the importance of marketing strategies tailored to meet the evolving consumer preferences for sustainable products in modern markets.

Green Consumerism

Green consumerism, a key aspect of sustainable markets, is driven by the motivations and behaviors of consumers who prioritize environmentally friendly products and businesses. Research has shown that there is a segment of consumers who are willing to pay more for environmentally friendly products, reflecting a growing environmental attitude and ecological behavior among certain consumer groups (**Akif H. *et al.*, 2012**). This presents opportunities for green marketing, particularly among young consumers who are increasingly conscious of environmental issues and seek out pro-environmental products (**Ergen *et al.*, 2014**).

Moreover, studies have indicated that consumer attitudes and behaviors towards green practices are influenced by factors such as environmental knowledge, concern, and activism, highlighting the complex interplay of individual and societal influences on green purchasing decisions. As businesses aim to capitalize on the growing demand for environmentally friendly products, understanding the dynamics of green consumerism becomes crucial for designing and marketing greener products that align with consumer values and preferences.

CONCLUSION

In conclusion, the analysis of consumer behavior in modern markets has highlighted several key findings and implications for businesses and future research. The use of technology has significantly impacted research methodology, leading to an increased reliance on secondary data due to the availability of Big Data about online and offline behavior (**Malter *et al.*, 2020**). Moreover, advancements in computer science have enhanced the ability to analyze large corpuses of unstructured data in an efficient and rigorous manner. The emphasis on managerial relevance and the understanding of how the "new" consumer interacts with other consumers and companies over time are identified as crucial areas for future research. Additionally, the study of consumer behavior is essential for explaining the mechanism of purchase and consumption decisions, with a focus on the cognitive components of human behavior (**Irina & Georgiana, 2007**).

Overall, the current trends in real-world consumption, coupled with the increasing number of digitally native consumers, underscore the importance of understanding and predicting the evolution of consumer behavior in the future. As such, the implications drawn from this analysis have significant relevance for businesses and point to the need for continued research in consumer behavior to address the challenges and opportunities presented by modern markets.

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