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Scientific refereed journal, accredited for the purposes of scientific promotions, published by market research and consumer protection center, university of Baghdad, republic of Iraq by two numbers per year.

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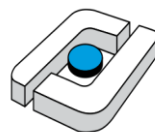


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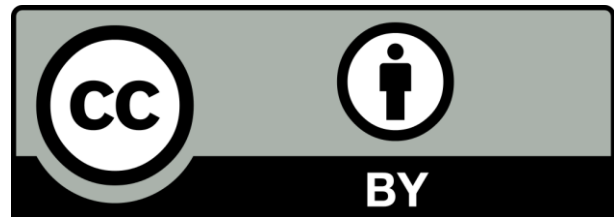


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## DEVELOPING SOFT CHEESE INDUSTRY SUPPORTED WITH MEDICINAL HERBS AS FUNCTIONAL FOOD

*Hamdia M. S. Al-Hamdani<sup>1</sup>, Sunduse H. Ahmed<sup>2</sup>, SalwaKhudadat<sup>3</sup>*

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### ABSTRACT

Herbs and spices have long been used to support various food products, including dairy products because of their flavoring, taste, texture and general appearance as well as therapeutic properties such as antioxidant activities, infections, microbes, anti-diabetes and hypertension. Therefore, this study aimed to demonstrate the effect of adding ginger, cinnamon, lycopene extract and olive oil on the physicochemical properties, the quality of the soft cheese produced and the extent of its acceptance by the Iraqi consumer, who prefers this product in abundance to other types of cheese. So, this study was prepared with ten liters of fresh cow's milk used in the manufacture of soft cheese by the dairy factory/ Abu Ghraib/ Baghdad. Standard soft cheese was processed by filtering raw milk first, heating, cooling, adding rennet, incubating, cutting, drainage the whey, salting and supplementing with different addition. Five treatments of soft cheese were made by regular method and supplemented as follows: The control treatment is to make white soft cheese without adding anything other than the basic ingredients for making soft cheese. While adding 2.5% of each of the ginger, cinnamon, lycopene and olive oil for each of the second, third, fourth and fifth treatment, to the curd of milk and supplement its manufacture from squeezing and preserving it until the necessary analyzes were done. The results of the study showed a clear and significant variance ( $P < 0.05$ ) of the percentage of fats, total solids, ash contents and calibrated acidity as the storage period of the soft cheese product increased to 21 days. The results of the statistical analysis also showed that ginger, cinnamon, lycopene and olive oil with certain concentrations had a positive effect ( $p < 0.05$ ) on the physicochemical composition of cheese and on all sensory properties. It was founded that supported cheese with cinnamon had the highest concentration in phenol contents follow: cinnamon cheese > lycopene cheese > olive oil > ginger cheese > control cheese which was 643, 564, 497, 424 and 213 mg\ kg respectively. Also, It was found that lycopene cheese appeared highest scavenging activity for free radical produced from DPPH followed by lycopene cheese, Olive oil cheese, Ginger cheese and Cinnamon cheese were 96, 94, 91 and 88% respectively. Consequently, the study concluded the importance of producing milk products fortified with medicinal plants and spices and their availability to many consumers who want to consume these fortified products to improve and preserve their health.

**Keywords:** Vital ingredient, physicochemical composition, HPLC technique.



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## الخلاصة

منذ فترة طويلة تستخدم الأعشاب والتوابل لدعم المنتجات الغذائية المختلفة، بما في ذلك منتجات الألبان بسبب النكهة والذوق والملح والمظهر العام وكذلك الخصائص العلاجية مثل أنشطة مضادات الأكسدة، والالتهابات، والميكروبات، ومرض السكري وارتفاع ضغط الدم. لذلك، تم تصميم هذه الدراسة لإظهار تأثير إضافة مستويات مختلفة من الزنجبيل والقرفة والليكوبين وزيت الزيتون إلى خثارة حليب البقر على جودة الجبن الطري الأبيض لتحسين جودته وقيمتها الغذائية والصحية، تم الحصول على عشرة لترات من حليب البقر من المزرعة المحلية في كلية الزراعة/ جامعة بغداد. تم تصنيع الجبن الطري القياسي عن طريق تصفية الحليب الخام أولاً، التسخين، التبريد، إضافة المنفحة، الحزن، التقطيع، العصر للتخلص من مصّل اللبن، إضافة الملح، والتدعيم بإضافات مختلفة. بعد ذلك، تم إجراء خمسة معاملات على النحو التالي: المعاملة الأولى هي معاملة السيطرة جبن حليب البقر الذي لا يحتوي على أي مادة مضافة، في المعاملات الثانية والثالثة والرابعة والخامسة، تمت إضافة 2.5% من الزنجبيل والقرفة والليكوبين وزيت الزيتون إلى الخثارة بعد التخمير، على التوالي، أشارت النتائج إلى أن الدهون، والمواد الصلبة الكلية، ومحتويات الرماد والحموضة لها تأثير معنوي ( $P < 0.05$ ) فترة التخزين، أظهر التحليل الإحصائي أن تدعيم التوابل بنسبة كبيرة للجبن الطري يؤثر معنوياً ( $P < 0.05$ ) بالتركيب الكيميائي للجبن، وقد وجد أن الجبن الطري الذي دعم بالدارسين محتواه عالي بالفينول > الجبن المدعم بالليكوبين > ثم الجبن المدعم بزيت الزيتون > الجبن المدعم بالزنجبيل > جبن السيطرة التي كانت 643، 564، 497، 424 و 213 ملغم /كغم على التوالي، أيضاً، تم إثبات أن الجبن المدعم باللايكوبين يمتلك أعلى نشاط في عملية التخلص من الجذور الحرة المنتجة من DPPH وأعلى تثبيط للاكسدة، يليه الجبن المدعم بالزيتون ثم الجبن المدعم بالزنجبيل ثم الجبن المدعم بالدارسين إذ كانت 96، 94، 88 و 91 ملغم /مل، وبالتالي، خلصت الدراسة إلى أهمية إنتاج هذه المنتجات المدعمة بالنباتات والأعشاب الطبية للعديد من المستهلكين الذين يرغبون في تناول هذه المنتجات الغذائية المدعمة بمكوناتها الحيوية والمحسنّة لصحتهم.

الكلمات المفتاحية: المكونات الحيوية، التركيب الفيزيائي والكيميائي، تقنية HPLC.

## INTRODUCTION

Since the pre-historical era, the milk industry has developed the cheese industry with many aspects, such as introduction of mechanization, the discovery of heat pasteurization for milk intended for the manufacture of dairy products, and the addition of pure colonies of lactic acid bacteria to milk prepared for the manufacture of soft and ripe cheeses. The food was processed with the addition of probiotic bacteria as functional foods. It was widespread popularity and acceptance for its therapeutic benefits, as it exceeded the digestive system. It also included improving immunity, its anti-cancer properties, dyspepidemia, heart disease, diarrhea and lactose intolerance (Vasiljevic & Shah, 2008). Cheese is one of the products made from raw milk and using the starter of cheese leading to the curd of the milk and then, pressing it to get rid of the excess water and then cut it and save it until it reaches the consumer and is characterized by its high content of protein, fats and calcium. Also, cheese is rich in mineral elements and vitamins that necessary for human health and for the construction of strong and healthy boon when phosphorus is available beside calcium (Chapman, 2011). The process of manufacturing cheese from cow's milk, sheep, goats, etc., and from other types of animals, is a series of processes, including the preparation of raw materials, pasteurization, coagulation, acidification, synthesis, dehydration, and moldings, compressing and salting (Effat, 2012). Since ancient times, medical and spicy plants have been used to enhanced flavor as well as therapeutic factors of most different foods, including different cheeses (Alsoufi & Aziz, 2019;

Samah&Ahmed, 2019). Many studies have shown that cheeses are fortified with herbs and spices have a significant inhibitory effect on many pathogenic microbes (Naveed *et al.*, 2013; Youssef & El-Sayed, 2018), which lead to food damage and oxidation by ingesting free radicals with their biochemical compounds (Bakheit & Foda, 2012). Because cheeses are characterized as easy to digest and absorb, they are included in the numbers of many diets, its consumption rate is great for most people and it is used in all daily meals, cheese are consumed all without any residue and the average selling price is suitable for everyone. Soft cheese is the only type available and desirable to most people in general in our country Iraq, as well as the manufacture of soft cheese is the main way to preserve excess milk in rural areas of the outskirts of Baghdad and in other provinces as well as the lack of dairy plants to use that excess milk. Previous studies have proved that herbs and spices can be added as flavoring agents, preservative, as well as their therapeutic properties as antioxidants, pressure-reduced, anti-inflammatory agents, and many other microbes. So, the wise use of medicinal herbs and nutritional spices to support soft cheese products leads to an increase in their nutritional and medicinal values and against many pathogenic microbes and as antioxidants as well as the ability to develop different dairy products of vital value added to which the consumer and eager for new products always aspires. So soft cheese was chosen in this study to supplement with ginger, cinnamon, lycopene and olive oil rich in antioxidants, phytochemicals and micronutrients, with enhanced sensory properties which qualify it to give us the qualities of therapeutic and functional foods in improving the functions of the body organs and treat a number of diseases and make the human health better.

## MATERIALS AND METHODS

### Primary raw material

Fresh cow milk, low-fat pasteurized, lactic acid bactericidal strains, microbial cheese bran that used in The State Company for Dairy Products\ Abu Ghraib\ Baghdad\ Iraq. Ginger powder, Cinnamon powder, olive oil, tomatoes, and edible salt were bought from local super market, Al-Warda in Baghdad city.

### Experimental design

Chemical analyzes of raw milk and processed cheese was performed, and cheese was also manufactured in laboratories and dairy plant\ Abu Ghraib\ Baghdad in June 2019. Five treatments of soft and processed cheese were made by regular method and support as follows: First the control treatment (Cheese Control=ChC) was to make white soft cheese without adding anything other than the basic ingredients for making soft cheese. While the other four treatments were added 2.5% of each of the Ginger (Cheese Ginger= ChG), Cinnamon (Cheese Cinnamon= ChCi), Lycopene (Cheese Lycopene= ChL) and Olive oil (Cheese Olive oil= ChO) to the curd of milk, it was lifted for 20 minutes, then after coagulation and completed manufacture then squeezing and preserving it until the necessary analyzes were done as shown in.

### Manufacturing of soft, fortified cheese with different spices

The improved method (Felix *et al.*, 2013) in making soft white cheese was followed in this study. Ten liters of fresh Cow's milk used for processing the soft cheese which were supplied from Animal farm of dairy plant\ Abu Ghraib\ Baghdad. 1.2% skimmed milk powder was added to raw milk with good stirring to homogeneity in order to obtain the desired percentage of protein\ fat to produce the soft cheese desired by the consumer. Then filtered and heated over a good pasteurization temperature of 83°C for 10 minutes, cooled to 30-40°C. Then, it was added 0.5% rennet with good stirring, and then incubated at 30°C\ 40-45 minutes until it was coagulated completely. Then the cheese curd was quietly stirred with a special knife

and the whey was removed, 1.0% NaCl was added to the curd to get the desired taste from the consumer. At this point, four treatments were made by adding 2.5% fine powdered of ginger, cinnamon, tomato lycopene extracts and olive oil were to the curd with an initial pH of 5.6. The dried spice mixes were added to the curds and kneaded for 4-5 minutes until the complete homogenization of ginger and cinnamon into the dough by kneading. Curds were squeezing for 5 hours then, each sample was divided into five almost equal cut of 250 gm each and under clean sterile conditions. Then, each piece was kept under vacuum-packaged and was storage at 7°C\ 1-21 days until it was used for analysis and sensory evaluation.

### Chemical analysis

Fat and the ash contents of milk and cheese were determined according to (AOAC, 1990). While, protein was determined by Kjeldahl method as mentioned by (AOAC, 2000). Total solids and acidity were determined according to (AOAC, 2000). All the chemical analysis was measured in duplicates at 1 and 21 days of storage for each treatment.

### Sensory evaluation

The quality of the fortified cheese samples was assessed with 2.5% of Ginger powder, Cinnamon, lycopene extract and olive oil stored for 1 and 21 days by 10 members trained for sensory evaluation of each of the following characteristics: color, flavor, texture, aroma and taste using the sensory evaluation paper according to (Larmond, 1987).

### Lycopene Purification

Lycopene purification method was installed by the research team. The silica gel was filled with a column with a distance of 1.5×10 cm. The lycopene extract was transferred to prepared column and the removal process was carried out using a solvent mixture (benzene: isopropanol 9: 1) and flow rate was 1mL\ minute (Hyeonet *al.*, 2017).

### Lycopene determination

HPLC was used to estimate the lycopene content in soft cheese by taking 2 gm of sample cheese fortified with lycopene and adding 40 mL of distilled water to the sample and mixed well, after that 40 mL of ethanol and hexane mixture were added (4:3, v\ v). The pre-stagnation solution was left\ for 30 minutes again, then the supernatant was collected and the collected residue extracted again using the same procedure described above. Likewise, the supernatants were collected again, and the supernatant solution combined with another mixture other than the previous was suspended and extracted with 10 mL of 10% NaCl solution and 15 mL of distilled water. The mixture was also left for stagnation for the fourth time for 5 minutes until the mixture was separated into two transparent layers and then the top layer was collected as mentioned above. Then all the top layers of the above extracts were collected and assembled again, vaporized under vacuum, dissolved in 1mL of hexane and filtered through 0.2 µm membrane filter, then analyzed by using HPLC (Agilent Technologies 1200 series, USA) which is equipped with UV detector and C18 column (5 µm, 4.6 mm×250 mm) (Sunfire<sup>TM</sup>C18, Water Ltd, Ireland). The homogeneous mobile phase was examined after mixing it with acetonitrile (solvent A), n-butanol (solvent B), and methyl chloride (solvent C), with quiet and homogeneous stirring and then the readings were taken using the following gradient coil: 0 minutes: 69.3% A, 29.7% B, and 1% C; 10 minutes: 67.2% A, 28.8% B, and 4% C; 20 minutes: 61.6% A, 26.4% B, and 12% C; 40 minutes: 49% A, 21% B, and 30% C; 50 minutes: 69.3% A, 29.7% B, and 1% C with flow rate 1 mL / minutes. The absorbance was measured by detection at wave length on 472 nm. The identification of lycopene was completed by comparing the peak area and retention time with a reference lycopene standard. The calibration was linear in the concentration range of 10-100 µg/mL ( $R^2=0.9991$ ) (Hyeonet *al.*, 2017).

### Determination of total phenols content

The total phenols were estimated by mixing 0.5 mL of plain soft cheese extracts and fortified cheese with different medicinal plants extract with 2.5 mL 10% Folin-Cioaltea. Add 2 mL of 7.5% sodium carbonate and keep the reaction tubes in the dark place for 40 minutes. Then measured on an appropriate wavelength of 765nm to measure the free radical (Ismail et al., 2013).

### Free radical scavenging activity (DPPH assay)

Free radical scavenging activity was determined as (Le, et al., 2007). It was determined by displacement of generated free radical with 1, 1-diphenyl-1-2- picrylhydrazyl (DPPH). It was poured 100 µL of DPPH into methanol (126 µM) in 96 mL well micro plates, as well as direct addition of 100 µL of ethanol extract at five concentrations 0, 100, 200, 300 and 400 µg/mL and then was incubated for 30 minutes at room temperature. The absorbance was measured at 517 nm using micro plate reader. As for the control sample, it was measured by using the reaction mixture with ethanol only instead of the materials that support the cheese samples. Then, DPPH assay was measured by the following equation:

$$\text{Scavenging activity (\%)} = \frac{\text{Absorbance control} - \text{Absorbance sample}}{\text{Absorbance control}} \times 100$$

Estimation the scavenging activity of any extract measured by the concentration needed to inhibit the free radicals by 50% (IC<sub>50</sub>). In other word, the concentration of extracts necessary to scavenge 50% of the produced nitric oxide (EC<sub>50</sub>) was obtained from a graph of scavenging activity (%) against a plant extract concentrations.

### Determination of cinnamon acid and methyl eugenol

The concentration of cinnamaldehyde and methyl eugenol is concentrated at a concentration of 1 - 200 µg\ mL in methanol according to (Akarca et al., 2016).

### Statistical analysis

It was used a statistical analysis program (SAS, 2012) to demonstrate the effect of the different parameters difference on the composition and sensory properties of fortified cheese by adding a specific concentration of spices used daily and healthy plants by the consumer, using the LLD test - which shows the least important difference and compares it with big differences Among the different coefficients in this study.

## RESULTS AND DISCUSSION

### Chemical composition of milk

Basic chemical composition in cheese and raw milk from which cheese was made was presented in (Table 1). It was found the percent of fat, protein, ash, total solid and titratable acidity content were 3.20, 3.50, 2.30, 9.90 and 0.13% respectively. While, it was found all that percentages increased significantly (p<0.05) to 12.50, 12.60, 2.82, 39.55 and 0.17 respectively in soft cheese due to its concentrated milk in cheese process.

**Table (1):** Chemical composition of fresh cow's milk and soft cheese.

Chemical composition (%)	Milk	Cheese	LSD value
Fat	3.20	12.50	2.95
Protein	3.50	12.60	2.88 *
Ash	2.30	2.82	0.31 *
Total Solid	9.90	39.55	5.622 *
Acidity	0.13	0.17	0.044 NS

\* (P≤0.05), NS: Non-Significant.

### Effect of storage period (days) on the chemical composition of the soft cheese

The main effect of the long storage period (days) on the chemical composition of soft, spiced fortified white cheese was presented as in (Table 2). It was found that fat contents of the cheese was decreased significantly ( $p < 0.05$ ) by increasing the time of storage. This decrease in the fat content during the storage periods of the cheese at 14 and 21 days is due to the vital activity of microorganism enzymes on the fats, which led to the breakdown of lipid molecules into fatty acids, other small fats molecules to the whey formed as a result of chemical reactions and dissolution of many different molecules according to (Kumaret al., 2013). The results of this study also showed a significant decrease ( $p < 0.05$ ) of the total solids contents of soft cheese significantly, as it was 41.25% on the first day and decreased to 38.55% for day. The results were identical to that founded by (Binet al., 2011; Landgeet al., 2011; Bhattacharyya et al., 2017). Also, a previous study attributed the reason for the decrease in the ratio of total soluble solids to the enzymatic degradation of proteins and fats available in cheese due to milk bacteria forming lactic acid and participation in making soft cheese by (Kumar et al., 2013). Ash content of the cheese samples significantly increased ( $p < 0.05$ ) from 2.82 on the first day and increased to 3.62 for day 21. The increased ash content in this study came close to that founded by (Hamid & Abdel rahmann, 2012) who attributed the reason for increasing the ash content with increasing the storage time, due to a decrease in the moisture content or increased penetration of water and serum formation due to added salt during the formation of fresh cheese curd. While, protein content was increased significantly ( $p < 0.05$ ) from 12.60 to 13.05% by increasing the time of storage from 1 to 21 days respectively. The results of this study were identical to what (Binet al., 2011) founded, which explained the reason for the decreased moisture during the storage period due to the decrease in the percentage of moisture in cheese and the increase in the amount of whey. While other previous results demonstrated a significant decrease in the percentage of protein with increasing the storage period to 21 days for soft cheese, and attributed the reason to the breakdown of the enzymatic protein into water-soluble compounds, which increased the ratio of total soluble solids (Akarca et al., 2016). As for the acidity of the cheese, there was a marked increase and a significant difference ( $P < 0.05$ ), as it was on the first day 0.17 and reached 0.34 after 21 days after storage, as is evident in Table 2. The reason for the high acidity of the cheese produced is due to the formation of lactic acid by increasing the growth of lactic acid bacteria while storing the cheese for longer periods of time, with appropriate conditions for growth in terms of temperature, pH and humidity for the growth of these bacteria and the formation of acidity (Atanu, 2017).

**Table (2):** Effect of storage period on the chemical composition of soft cheese.

Storage period(days)	Chemical composition (%)				
	Fat	Total Solid	Ash	Protein	Acidity
0	12.50 a	41.25 a	2.82 b	12.60 a	0.17 b
7	12.50 a	40.15 ab	2.86 b	12.58 a	0.20 b
14	12.25 ab	39.50 ab	2.98 b	12.60 a	0.26 ab
21	11.30 b	38.55 b	3.62 a	13.05 a	0.34 a
LSD value	1.073 *	2.138 *	0.633 *	0.582 NS	0.115 *

Means having with the different letters in same column differed significantly . \* ( $P \leq 0.05$ ), NS: Non-Significant.

### Effect of different spices addition on chemical composition of soft cheese

The effect of adding different spices on chemical composition of the soft cheese produced was presented as in (Table 3). The results of the study showed the significant increase ( $P < 0.05$ ) of the fat in cheese produced in all the different additions of ginger, cinnamon, lycopene and olive oil as shown in (Table 3). While, results of total soluble solid

indicated a significant increases ( $P < 0.05$ ) among all treatments. The ash content was increased significantly in treated cheese with ginger and cinnamon 3.22, and 2.98% respectively. The increased ash content in these two treatments is probably due to the presence of fiber in the ginger and cinnamon flour that used in this study. While the percentage of ash decreased significantly in cheese treated with lycopene extract and olive oil 2.65 and 2.66% respectively. While the results of the study showed that there was no statistically significant difference in cheese content of protein and for all additions from ginger, learners, lycopene and olive oil. The results indicated a significant decrease in the acidity content among all treatments, except for the treatment of cheese with olive oil that led to high acidity, perhaps the presence of the oil led to the microbial inhibition of the acidity microbes. Previous studies found that the protein, fat and ash content of these cheeses fortified with spices would not be significantly affected by the storage period, but the total solids and acidity were significantly affected by the addition of spices (Josipovic *et al.*, 2015).

**Table (3):** Effect of different spices addition on chemical composition of soft cheese.

Treatments	Chemical composition (%)				
	Fat	Total Solid	Ash	Protein	Acidity
Ch C	12.50 b	39.55 a	2.82 b	12.60 a	0.17 ab
ChG	12.8 b	40.50 a	3.22 a	12.88 a	0.09 b
ChCi	12.55 b	41.15 a	2.98 ab	12.65 a	0.15 ab
ChL	13.30 ab	41.35 a	2.65 b	13.05 a	0.14 ab
ChO	14.65 a	41.50 a	2.66 b	13.15 a	0.20 a
LSD value	1.769 *	2.366 NS	0.398 *	0.822 NS	0.089 *

Means having with the different letters in same column differed significantly . \* ( $P \leq 0.05$ ), NS: Non-Significant.

\*= ( $P \leq 0.05$ ), NS: Non-Significant. Means within columns bearing different superscripts are significantly different ( $p \leq 0.05$ ), S: Significantly different, NS: Not significantly different, ChC: Control soft cheese, ChG: Cheese with Ginger, ChCi: Cheese with cinnamon, ChL: Cheese with Lycopene, ChO: Cheese with Olive Oil, LS: Level of significance.

### Sensory evaluation

Herbs and spices were used to add flavor, taste and good taste to dairy products, including cheeses, to overcome the known flavor of cheese that is not desired by many consumers. Well, you know herbs and spices with therapeutic properties such as antioxidants, anti-inflammatory, anti-diabetic, antihypertensive and anti-microbial properties. Therefore, it can supplement dairy products with herbs and spices to provide it active substances, as well as increasing nutritional and medicinal values. The effect of storage period (days) on sensory characteristics of soft control and supplemented cheese product was presented as in (Table 4). The results of this study indicated that there was no significant difference ( $p < 0.05$ ) on the color characteristic of soft cheese by increasing the length of the storage period up to 21 days with Lycopene and Olive oil addition. While there was a significant difference in color by increasing the period of time especially for 21 days of storage. But the color changed a little bit with ginger, cinnamon and lycopene addition compared with control sample. While, Olive oil addition did not affect the color character compared with control sample. Usually salt is added in a very small amount to produce a good texture, taste, flavor and odor which are desired by the consumer. The results showed that there was no significant difference ( $p < 0.05$ ) for sensory characteristics such taste, texture and odor. The flavor character which decreased significantly with Ginger addition, but slight decreases with cinnamon, lycopene and olive oil addition compared with control treatment. Thus, positive effect of these spices probably due to its bioactive content that works as antioxidant and antimicrobial growth. The results of this study



were close to the results of (Naveedet *al.*, 2013;Atanu,2017).Results showed significant differences in taste by supplemented cheese with Ginger especially after 14 and 21 days of storage, while there were no significant difference in taste by supplemented with Cinnamon, Lycopene and Olive oil. These results came close to what he found (Fabiolaet *al.*, 2017), which he attributed the cause of change to each of the characteristics of taste, texture, flavor and odor, may be due to protein decomposition and lipolysis processes and peroxide flavor and sour acidic taste that occurred during storage(Josipovicet *al.*, 2015).So, it was concluded that the period from 1-14 days is the best period for storing cheese fortified with various spices in terms of most of the sensory qualities that the consumer aspires to.

**Table (4):** Effect of storage period on sensory evaluation of the supplemented cheese with different spices.

Parameters	Treatment	Storage periods (day)				LSD value
		1	7	14	21	
Color	ChC	10	9	9	7	2.18 *
	ChG	10	9	8	7	1.93 *
	ChCi	10	9	9	8	1.75 *
	ChL	9	9	9	8	1.06 NS
	ChO	9	9	9	9	0.50 NS
Taste	ChC	10	9	8	7	2.18 *
	ChG	9	9	7	7	1.79 *
	ChCi	9	9	8	8	1.15 NS
	ChL	9	8	8	8	1.08 NS
	ChO	9	9	8	8	1.15 NS
Texture	ChC	9	8	7	6	2.37 *
	ChG	9	8	8	7	1.74 *
	ChCi	9	9	9	9	0.50 NS
	ChL	9	9	8	8	1.15 NS
	ChO	9	9	9	9	0.50 NS
Flavor	ChC	9	9	8	6	2.04 *
	ChG	9	9	9	8	1.07 NS
	ChCi	9	9	8	6	2.04*
	ChL	9	9	8	8	1.15 NS
	ChO	9	9	9	8	1.06 NS
Odor	ChC	9	8	7	7	1.69 *
	ChG	9	8	8	7	1.75 *
	ChCi	9	8	8	7	1.75 *
	ChL	9	9	7	6	2.27 *
	ChO	9	9	8	8	1.15 NS

\* (P≤0.05)= Significant NS= Not significantly different, ChC= Control soft cheese, ChG= Cheese with Ginger, ChCi= Cheese with cinnamon, ChL= Cheese with Lycopene, ChO= Cheese with Olive Oil, LS: Level of significance

### Lycopene analysis

Partially purified lycopene, cinnamic acid and gingerol from lycopene, cinnamon and ginger that fortified to the soft cheese were represented as in (Figure 1, 2 and 3). These results were identical to which founded by (Hyeon-Juet *al.*, 2017).

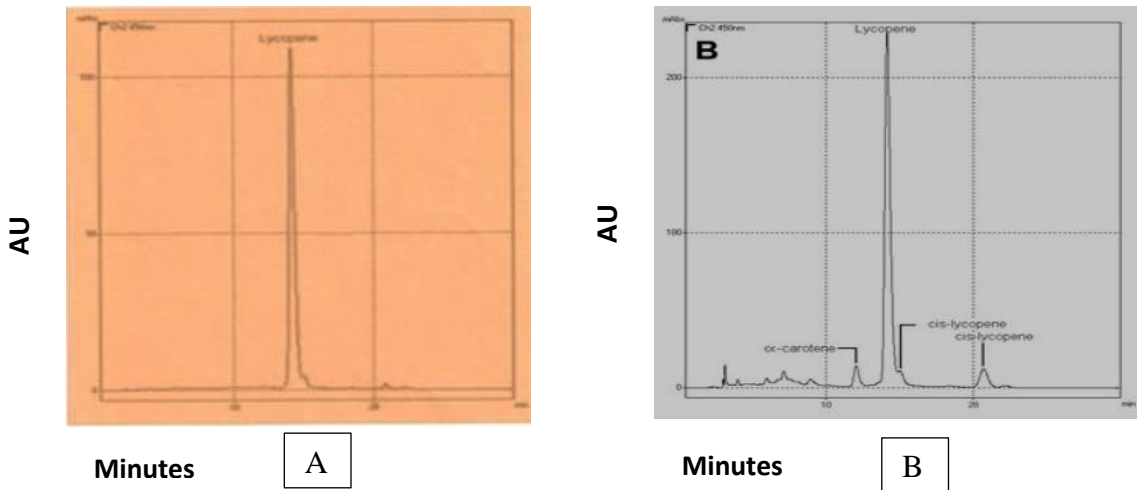


Figure (1):The diagnosis of pure lycopene A and partially purified lycopene B.

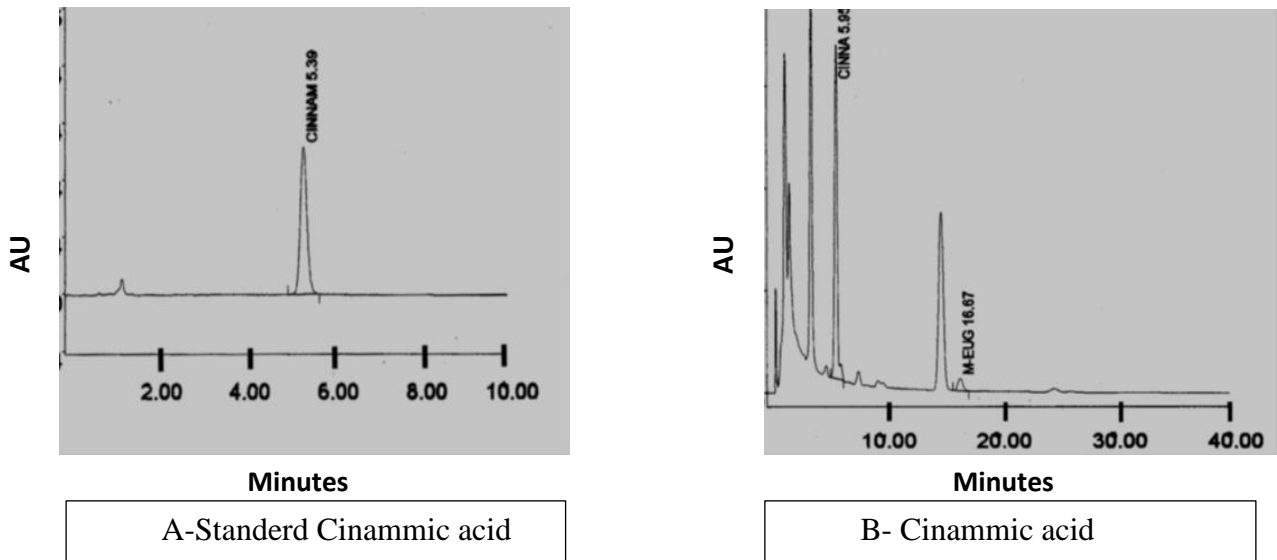


Figure (2): The diagnosis of Cinnamic acid in soft cheese supported by cinnamon.

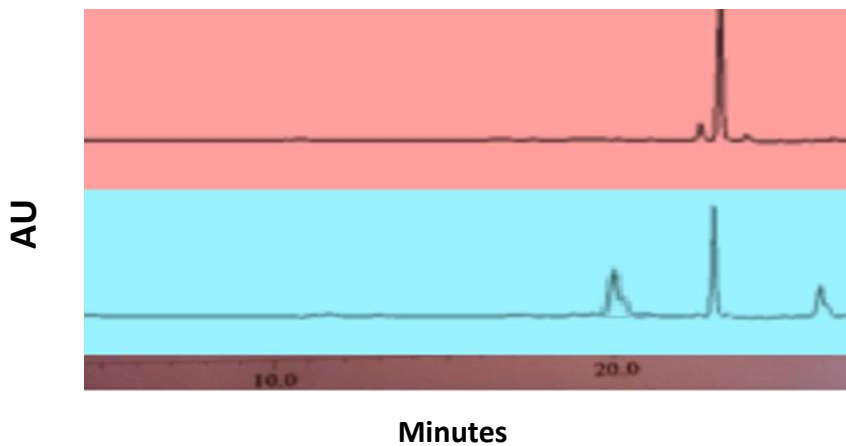


Figure (3):The diagnosis of Gingeriol in soft cheese supported by ginger, the upper shape is the standard and the lower shape is for Ginger cheese sample.

### Total phenols content

Total phenolic content and anti-oxidant activities measured with DPPH from the use of dried spices and used domestically and on a large commercial scale such as ginger and cinnamon, as well as the use of lycopene extract for tomato and olive oil presented in (Table 5). It was founded that Gingerol is the active content in ginger with 250mg\ kg concentration. While, the active content in cheese was 97mg\kg. Cinammic acid, is the active content in ginger plant extract with 125mg\kg concentration. While, the active content in cheese was 65mg\kg. Lycopene is the active content in tomato plant with 368mg/kg concentration. While, the active content in cheese was 91mg/kg. Results showed the highest total phenol content in cinnamons cheese was 643mg/kg, then decreased significantly ( $p \leq 0.05$ ) to 564mg\kg in lycopene cheese, 497mg\kg in olive oil, then 424mg\kg in ginger, and the lowest content was 213mg\kg in control treatment. From that, we conclude that it is possible to increase the biological value of cheese by strengthening food products, including the manufacture of soft cheese (Storyet al., 2010; Kumar et al., 2013) which is consumed heavily by most people and its preference over other types of cheese with types of spices and plant extracts rich in phenolic compounds, and these results were identical to what he found (Samah & Ahmed, 2019).

**Table (5):** The concentration of active ingredients in cheese supported by the active ingredients of medicinal plants.

Treatments	Bioactive content in plant	Bioactive content in plant extract (mg\kg)	Bioactive content in supplemented soft cheese (mg\kg)	Total phenols content in all treatments (mg\kg)
Ch C	-	-	-	213 d
ChG	Gengerol	250 b	97 a	424 c
ChCi	Cinnamic acid	125 c	65 b	643 a
ChL	Lycopene	368 a	91 a	564 b
ChO	Olive oil	182 c	80 a	497 a
LSD value	--	42.385 *	8.736 *	71.024 *

Means having with the different letters in same column differed significantly. \* ( $P \leq 0.05$ ).

Means within columns bearing different superscripts are significantly different ( $p \leq 0.05$ ), S: Significantly different, NS: Not significantly different, ChC: Control soft cheese, ChG: Cheese with Ginger, Chci: Cheese with cinnamon, ChL: Cheese with Lycopene, ChO: Cheese with Olive Oil, LS: Level of significance

### Effect of active group on removal of free radicals

These supplemented cheeses have an effective role in the removal of free radicals as founded in (Table 6). Results of this study showed, Lycopene was the most effective in removing free radicals, followed by olive oil cheese, ginger cheese and finally cinnamon cheese. While, the inhibition concentration on 50% in Cinnamon extract was the highest 27, followed in Ginger and olive oil 22, then followed to Lycopene extract 18 mg\mL. Olive oil is known to contain a good percentage of fatty acids rich in omega-3 fatty acids, which have a great effect on reducing fat oxidation, and it has been effectively applied to protect against fat oxidation in cheese during storage (Shan et al., 2011). Previous studies were conducted in vitro with the high potential of plant extracts as a natural preservative and antioxidants (Singhet al., 2011; Youssef & El-Sayed, 2018).

**Table (6):** The ability of the models under study to remove free radicals generated from DPPH.

Treatments	Dpph(%) needed to remove free radicals	Concentration inhibiting 50% (mg/mL)
Control cheese ©	63 e	29 a
Ginger extract	76 d	22 bc
Ginger cheese	91 ab	15 de
Cinnamon extract	69 dc	27 ab
Cinnamon cheese	88 bc	11 e
Lycopene extract	82 cd	18 cd
Lycopene cheese	96 a	9.6 e
Olive oil	74 d	22 bc
Olive oil cheese	94 ab	15 de
LSD value	7.952 *	5.478 *
Means having with the different letters in same column differed significantly. * (P<0.05).		

## CONCLUSION

From the results obtained, many studies have found that fortification of dairy products, including soft cheese, which is desirable to be consumed by most segments of human societies with medicinal plants and spices, has an important role in improving food produced with active biological components to treat many common diseases currently including malnutrition. This study demonstrated that the chemical composition of soft cheese was significantly affected ( $p < 0.5$ ) by increasing the storage time of soft cheese for more than 14 days. The study also demonstrated a decrease in the percentage of fats, total solids, ash and acidity in all types of cheese supported with different spices significantly with an increase in the storage period for more than 14 days. While, protein was not affected by different spices addition. Sensory evaluation results showed that addition of each ginger, cinnamon, lycopene and olive oil enhanced the color, flavor and odor of the white soft cheese. In addition, Ginger, Cinnamon, Lycopene and Olive oil gives a bioactive components such as gengerol, cinnamic acid, lycopene and total phenol respectively which acting an important role as highest antibacterial activity, antioxidant.

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## ELECTROCHEMICAL PREPARATION OF A MOLECULAR IMPRINTED POLYMER ELECTRODE FOR ESTIMATION OF ASPIRIN USING TWO DIFFERENT FUNCTIONAL MONOMERS.

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### ABSTRACT

For aspirin estimated, a molecularly imprinted polymer MIP-ASP electrodes were generated by electro-polymerization process, the electrodes were prepared by combining the template (aspirin) with (vinyl acetate (VA), 1-vinylimidazole (VIZ) as a functional monomer and N, N-methylene bisacrylamide (MBAA) as crosslinkers using benzoyl peroxide (BPO) as an initiator. The efficiency of the membrane electrodes was analyzed by differential pulse voltammetry (DPV). Four electrodes were synthesized using two different plasticizers, di-butyl sebacate (DBS), di-octyl phthalate (DOP) in PVC matrix. Scanning electron microscopy (SEM) was used to describe the generated MIP, studying the electrodes properties, the slope, detection limit, and life time and linearity range. The effect of PH and interferes on the efficiency of the MIP electrode was investigated. The study has shown that the molecularly imprinted electrodes have high sensitivity and responsiveness to aspirin. The DPV value was linearly dependence on the aspirin concentration and a linear curve was obtained within the range of ( $1 \times 10^{-5}$ – $5 \times 10^{-4}$ ) M of aspirin with correlation coefficients are about (0.9974, 0.9966, 0.9938 and 0.9961) with slopes value of (-21.41, -17.67, -17.47 and -18.67) and the detection limit for all electrodes ranging from ( $7.5 \times 10^{-5}$ – $1 \times 10^{-4}$ ) M. The molecularly imprinted electrode exhibited a good response with highly reproducible and no effect on interferes frequently available in pharmaceutical formulations. The approach employed is easy and fast. Also ASP membranes get a limited time of response, excellent mechanical stability, removable and are easy to construct.

**Keywords:** molecularly imprinted electrochemical sensors (MIECS), Aspirin (ASP), potentiometric method, vinyl acetate (VA) monomer, 1-vinylimidazole (VIZ) monomer.



## التحضير الكهروكيميائي لقطب بوليمر مطبوع جزيئياً لتقدير الأسبرين وباستخدام وحدتين وظيفيتين مختلفتين

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## الخلاصة

لتقدير الأسبرين، تم إنشاء أقطاب بوليمرية مطبوعة جزيئياً MIP-ASP عن طريق *Electro-polymerization*، تم تحضير الأقطاب الكهربائية عن طريق الجمع بين القالب (الأسبرين) مع (الفينيلأسيتات (VA)، 1-فينيل إيميدزول (VIZ)) كمونومروظيفي و N و N-ميثيلين بيساكريلاميد (MBAA) كموصلات متشابكة وباستخدام البنزويل بيروكساييد (BPO) كبادئ، تم تحليل كفاءة الأقطاب الكهربائية الغشائية بواسطة قياس الجهد التفاضلي (DPV)، تم تصنيع أربعة أقطاب كهربائية باستخدام ملدنين مختلفين وهما ثنائي- بوتيلسيبيكيت (DBS)، وثنائي أوكتيلفثاليت (DOP) في مصفوفة PVC، وتم استخدام الفحص المجهر الإلكتروني (SEM) لوصف MIP المتولد، ودراسة خصائص الأقطاب الكهربائية، والميل، وحد الكشف، وعمر القطب، والمدى الخطي. تم فحص تأثير درجة الحموضة وتأثير التداخلات على كفاءة القطب MIP. وقد أظهرت الدراسة أن الأقطاب التي تم طبعها جزيئياً لها حساسية عالية واستجابة للأسبرين. وكانت قيم الـ DPV هي خطية اعتماد على تركيز الأسبرين وتم الحصول على منحني خطي ضمن حدود M ( $1 \times 10^{-1}$  -  $5 \times 10^{-4}$ ) مولاري من الأسبرين مع معاملات الارتباط حوالي (0.9938 ، 0.9966 ، 0.9974) و (0.9961) بقيمة انحدار تبلغ (-21.41 ، -17.67 ، -17.47 و -18.67) وحد الكشف لجميع الأقطاب الكهربائية يتراوح من ( $7.5 \times 10^{-5}$  -  $1 \times 10^{-4}$ ) مولاري. وقد أظهرت الأقطاب المطبوعة جزيئياً استجابة جيدة مع قابلية عالية للتكرار وليس هناك تأثير للتداخلات المتوفرة بشكل متكرر في المستحضرات الصيدلانية. ان النهج المستخدم سهل وسريع. كما حصلت أغشية ASP على وقت استجابة محدود، واستقرار ميكانيكي ممتاز، وقابلة للإزالة وسهلة التركيب. الكلمات المفتاحية: أجهزة الاستشعار الكهروكيميائية المطبوعة جزيئياً (MIECS)، الأسبرين (ASP)، طريقة قياس الجهد، مونومر أسيتاتالفينيل (VA)، مونومر 1-فينيل إيميدزول (VIZ).

## INTRODUCTION

An advance in molecularly imprinted polymers (MIPs) is a useful method to a preparatory work of the polymeric materials with active sites. (Lata *et al.*, 2015). MIPs are polymers that are manufactured to create cavities with attraction to a selected "template" molecule within the polymer chains. They have been prepared by complexing the target (template) with the FM, whether by covalently or non-covalently bonding, accompanied by polymerization with large quantities crosslink's to establish a highly cross linked polymeric network. When the target-molecule is extracted from of the polymeric matrix , different recognizing locations are identified which are comparable to the target in terms of size, shape and function (Meier & Mizaikoff, 2010). MIPs have been used in various applications, including chromatography (Li *et al.*, 2014), capillary electrophoresis chromatography (Rutkowska *et al.*, 2018), quartz crystals of micro-balance (EL-Sharif *et al.*, 2015), membranes separation (Balouch *et al.*, 2019), SPE (El Nashar *et al.*, 2017; He *et al.*, 2015) and the sensors of biomimetic (Huang *et al.*, 2018; Lahcen & Amine, 2019; Malitesta *et al.*, 2017). MIPs have several benefits, including high specificity and selectivity to target molecules, greater chemical and mechanical stabilization, insoluble in DW and most organic solvents. In additional, the MIP have easily synthesis and have good mechanical properties, reliability for pressures and temperatures, and thus are cost-effectively and suitable to applicable for harmful chemicals (Al-Bayati & Aljabari, 2016; N. Zhang *et al.*, 2019)MIPs are the sensing elements of a molecular imprinting electrochemical sensor (MIECS) which are molecularly imprinted. The sensor could specifically recognise of the target molecules depending upon this cavities (active site) in the molecular structure of imprinting polymer.



Once the template have been extracted, the active site representing the spatial configuration of a target molecules will be obtained as well as the electrochemical response has been received because when MIP is associated with a particular template the electrosignal was recorded depending on the active site, as well as the targeted molecule concentrations can be estimated (Crapnell *et al.*, 2019; Jin *et al.*, 2015; Varghese *et al.*, 2019). MIECS have many characteristic includes: high selectivity, simplest techniques, lower costs, low D.L, highly stable. The combination of both the template molecules and the cavities can also be easily achieved and is carefully applicable even with harmful chemical materials. Thus, MIECS was used for optical and electrochemical applicable (Al-Bayati & Abd, 2017; Al-Bayati & Al-Safi, 2018; D'Aurelio *et al.*, 2020; Momeneh & Gholivand, 2018; Tadi & Motghare, 2016). The utilization of molecular imprinting techniques have increased significantly in recent years, thus illustrating magnificently the ability of MIP model for detection toward the target molecules (Bates *et al.*, 2017; Marc *et al.*, 2018).

As in present study, four of the MIPs have been manufactured as recognizing materials using [Vinylacetate (VA), 1-Vinylimidizol (VIZ)] as functional monomer, N, N-methylene bis-acrylamide (MBAA) is a crosslinkers also benzoyl peroxide (BPO) as an initiators using methanol as porogen solvent. The efficiency of the MIPs was tested using rebinding equilibrium assays. The highest efficiency of the MIP was selected as the identification substance in PVC membrane for the estimation of aspirin (ASP) in pharmacological samples.

Aspirin (ASP) is widely used in pharmaceutical formulations as an analgesic and antipyretic agent for relieving headaches, fever, muscle pains and inflammation severe arthritis. so the wide use of ASP had led to the therapeutic intoxication due to overdose , which can be found in individuals with chronic inflammatory diseases and routinely take ASP (Kan *et al.*, 2009; Q. Zhang *et al.*, 2020) (Figure 1) shown the aspirin structure. The aims of this research are studies the response of the MIP-sensor in presence / absences of interferences and record the analysis of the electro-chemical sensors for the estimation of ASP using MIP-sensor electrodes.

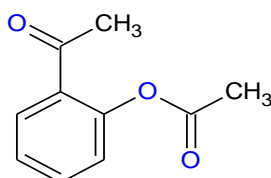


Figure (1): Aspirin structure.

## MATERIALS AND METHODS

The standard of aspirin was gained from of the government drug manufacturer (IRAQ-SDI-Samara). Aspirin tablets (100 mg) Chewable tablet, acetylsalicylic acid ((SDI)-IRAQ and Memphis/Bayer) were purchased from local pharmacies. Plasticizers: (DBS) (97.0% purity) and (DOP) (99.5% purity), were purchased from Sigma Aldrich. Other chemicals and reagents materials were obtained from Fluka, BDH and Sigma Aldrich.

## Instruments

In this work, We use potentiometric measured data by digital voltmeter (PH 211 HANA devices). An analyzer (WTW model, Germany), SCE (Gallenkamp, USA), pH meter (WTW model PH 720, Germany). UV-VIS dual-Beam system (UV-1650 PC) SHIMADZU (Japan), computer interfaced by a SHIMADZU UV investigate system (version 1.10), SHIMADZU FT-IR,-8000 (Japan), Scanning Electrons Microscopic (SEM) [JSM-6390A] (Tokyo, Japan) and sensitively balance (Electronically balance ACS120-4 Kern & Sohn GmbH, Germany). A quality of the electrodes has been monitored by measuring the potential of ASP solutions

ranging from  $5 \times 10^{-5}$  to  $1 \times 10^{-1}$  M at room temperature. The accuracy of electrode activity was measured and then the potential was registered after the internally and externally solution arrived to the equilibration.

#### Preparation of standard solutions for ISE study

100 mL of 0.1 M standard solution for aspirin was prepared by dissolving 1.802 g of aspirin in methanol. The ASP solutions ranged from ( $1 \times 10^{-5}$ - $1 \times 10^{-1}$ ) M in 100 mL, also the interferences ion ( $K^+$ ,  $Ca^{+2}$ ,  $Al^{+3}$ ) was prepared ranging from ( $1 \times 10^{-5}$ - $1 \times 10^{-1}$ ) M in 100 mL volumetric flask and 100 mL of each (methyl paraben, propyl paraben, tri sodium citrate) interferences solution was prepared from ( $1 \times 10^{-5}$ - $1 \times 10^{-1}$ ) M of a stock solution of 0.1 M interference.

#### Molecular imprinting polymer synthesis

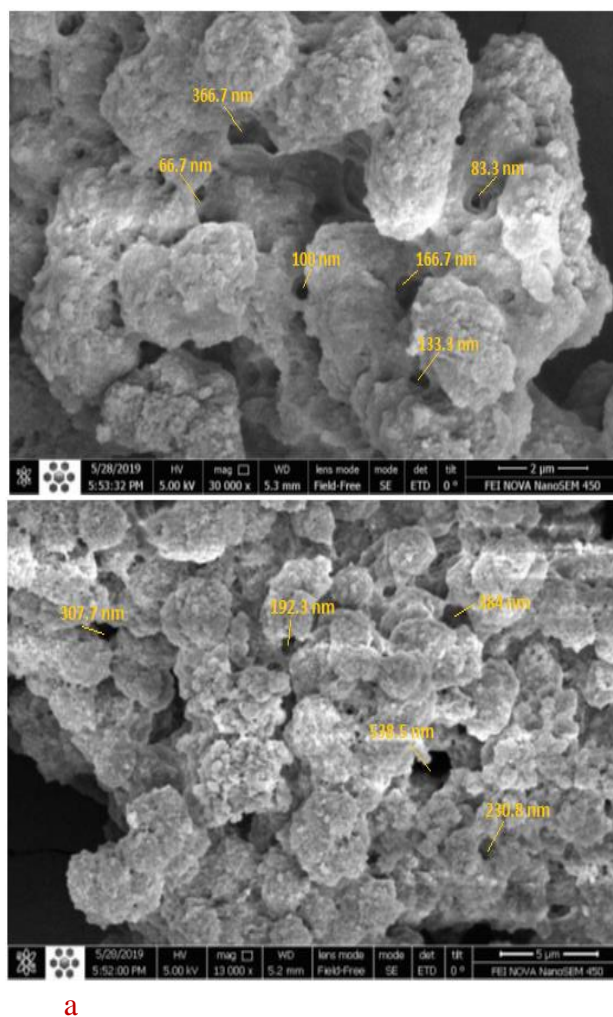
The first aspirin molecularly imprinted polymer (ASP-MIP<sub>1</sub>) prepared by mixed 0.5 mmol (0.0901 g) from aspirin then mixed with 3 mmol (0.26 g) vinyl acetate (VA) as the monomer, after that added 15 mmol (2.313 g) N,N-methylene bis-acrylamide (MBAA) to the solution as the cross-linker, followed that added (0.32 g) benzoyl peroxide as the initiator. All these materials were dissolved in  $5 \pm$  mL methanol ( $CH_3OH$ ) except the initiator have dissolved in 3 ml chloroform. While the second aspirin molecularly imprinted polymer (ASP-MIP<sub>2</sub>) were achieved by mixed 0.4 mmol (0.0721 g) from aspirin as the template, 2.4 mmol (0.226 g) 1-vinylimidizol (VIZ) as the monomer, 12 mmol (1.85 g) N,N-methylenbisacrylamide (MBAA) act as crosslinkers also (0.3 g) benzoyl peroxide as the initiator which dissolved in 5 mL of methanol ( $CH_3OH$ ). For obtaining a homogeneous solution, the mixture was stirred for 5 minutes.  $N_2$  passes for 30 minutes on the mixture to remove oxygen from the solution. After that, the solution was put at  $60^\circ C$  in a water bath. When the reaction completes the molecularly imprinted polymer became hardened, after the polymerization process the polymer was drying and crashed to obtain a polymer particle. These particles were sonicated in acetonitrile/ $CH_3COOH$  (18:2 v/v) to remove the template from MIP. The polymer was dried for (42-75) hours at room temperature, crushed and ground the polymers by mortar and pestle and sieve to get 125  $\mu m$  particle size (using 125  $\mu m$  mesh sieve); after dried completely at room temp., has been used in the membrane of the selective sensors as an active substance. To fabricate of electrode, The PVC tubing (1-2 cm long) was placed that on a glass slide and soaking it with THF. Similar to an average thickness of the PVC tubing, the membranes also was cut and pasted onto the end tube other end of that was connected to an electrode of Ag-AgCl.

#### Preparation of pharmaceutical samples.

The mortar has used to create the powder of the pharmaceutical tablets. There were two different types of ASP-tablets used it to estimate the molarity concentration of ASP-drugs. An appropriate weight of powder was then taken to prepare of ( $1 \times 10^{-3}$  and  $1 \times 10^{-4}$ ) M from the pharmaceutical sample solutions. Suitable quantity of methanol has been used to dissolve pharmaceutical samples and to complete volumes of up to 100 mL.

#### Scanning Electron Microscope (SEM)

A morphological characteristics of the MIPs before and after template removal membranes was evaluated by scanning the electron microscope using Tokyo / Japan-JSM-6390 A, in order to show the differences between both the SEM image of both the MIPs before and after the template obtained in proportion to the size and surface morphology of a polymeric particles. SEM analysis indicates that molecular imprinted polymer in surface and in cross-section, had a highly ordered and regular pore structure which serves as the sites of interaction, in (Figure 2a and b) can be seen that micro emulsion polymerization gives very small particles size around 66.8 nm to 366.7 nm for Vinyl acetate (VA) polymer and 192.3 nm to 538.5 nm for vinylimidizol (VIZ) polymer.



**Figure 2:** a. SEM of [ASP-MIP(VA)],

b. [ASP-MIP(VIZ)] obtained by bulk polymerization.

## RESULTS AND DISCUSSION

Four MIP membranes are prepared using two different monomers VA and VIZ with PVC matrix and two different plasticizers DBS and DOP, the ions selective polymer membrane is one of the most crucial components of ISEs. It isolates the internal reference solution from the external analytical sample solution. Polymeric membranes must have selectivity for different analyte ions, un-porous, insoluble in water and mechanically stable, based on the nature of the membrane material used. Other critical components of an ISE assembly are the internally and externally reference electrodes. Collection consisting of both reference electrodes and also the polymeric membrane is referred to as cell the characteristic of these membranes were studied, including: slope, detection limit, linearity, life time and the response to the Nernstian equation were investigated; the results in (Table, 1) indicated that both monomer and both plasticizers can be used for preparing effective MIP for ASP.

**Table (1):**The characteristics of ASP-MIP electrode using two different a monomer and two different plasticizer

Membranes no.	Membrane composition	Parameter			
		Slope mv/decade	Correlation coefficient (r)	Linearity range (M)	Detection limit/ M
I	ASP-MIP <sub>1</sub> (VA+MBAA+DBS)	-21.41	0.9974	1×10 <sup>-4</sup> -1×10 <sup>-1</sup>	7.5×10 <sup>-5</sup>
II	ASP-MIP <sub>1</sub> (VA+MBAA+DOP)	-17.67	0.9966	1×10 <sup>-4</sup> -1×10 <sup>-1</sup>	8.0×10 <sup>-5</sup>
III	ASP-MIP <sub>2</sub> (VIZ+MBAA+DBS)	-17.47	0.9938	5×10 <sup>-4</sup> -1×10 <sup>-1</sup>	8.5×10 <sup>-5</sup>
IV	ASP-MIP <sub>2</sub> (VIZ+MBAA+DOP)	-18.67	0.9961	5×10 <sup>-4</sup> -1×10 <sup>-2</sup>	1.0×10 <sup>-4</sup>

### Infrared spectroscopy (FTIR- Analysis)

The FTIR is a commonly utilized method of substance characterisation. The spectral generated from of the analysis provides the specific samples their identities. The peak frequency of absorption relate to signature vibration of binds between all the atoms that create up the product. The FTIR spectrum is therefore a substance characteristics and allows accurate recognition. The listed in (Table, 2& 3) showed a band that before/after removal ASP-template from MIP<sub>1</sub>& MIP<sub>2</sub> respectively.

**Table (2).** The FT-IR spectra for ASP-VA polymer before/after removal clop-template.

F.G.	ASP.	ASP-VA (MIP <sub>1</sub> ) before ASP removal	ASP-VA (MIP <sub>1</sub> ) after ASP removal
O-H str.	3200-2500	3384	3398 (enol)
C-H arom.	3016	3060	-
R-C=O.	1755	1720	-
COOH	1691	1654	-
C=C arom.	1604	1521	-
C=C aliph.	-	-	1527
COOR	-	1733	-

From this table can be seen (before ASP removal ) that the characteristically peak at ~1733 cm<sup>-1</sup> is attributable to vibrational mode of -COOR and the vibration version of amide N-C=O is allocated at ~1654 cm<sup>-1</sup> these notes indicated to fact that the interaction occurred between the template (ASP) and the monomer (VA), while after ASP removed the detected peak characteristics are missing in carboxylic spectra at ~1654 cm<sup>-1</sup>, carbonyl group at ~1720 cm<sup>-1</sup> and also missing the absorption band of C=C at ~1521 cm<sup>-1</sup>. The results indicated that the height contrast of imprinted polymer FT-IR spectra before and after template removal proves that ASP-template has been fully extracted from MIP<sub>1</sub> in the extraction stage by soxhlet.

**Table (3):** The FT-IR spectra for ASP-VIZ polymer before/after removal ASP-template

F.G.	ASP.	ASP-VIZ (MIP <sub>2</sub> ) before ASP removal	ASP-VIZ (MIP <sub>2</sub> ) after ASP removal
O-H str.	3200-2500	3300-3000	-
C-H arom.	3016	3060	3058
C-H aliph	2974-2833	2952-2866	2948
R-C=O.	1755	-	-
COOH	1691	1658	-
C=C arom.	1604	-	1525
N-C=O	-	1627	1654

The results indicate that the strongest two peaks are missing at  $\sim 1691\text{ cm}^{-1}$  and  $\sim 1755\text{ cm}^{-1}$  for carbonyl group stretching C=O and C=C aromatic stretching respectively, while the characteristically peak appears at  $\sim 1627\text{ cm}^{-1}$  due to interaction between the ASP-template and the VIZ-monomer, However when the ASP-template removed after washing noticed that the carbonyl group at  $\sim 1658\text{ cm}^{-1}$  and the hydroxyl group at  $\sim 3300\text{ cm}^{-1}$  are missing, that also indicated the template are completely extracted in the soxhlet extraction stage from MIP<sub>2</sub>.

### Effect of pH on ASP-electrodes

The pH dependency of the electrode sensor membrane was measured at a pH range of  $1 \times 10^{-4}$ ,  $10^{-3}$  and  $10^{-2}$  M aspirin concentrations (Figure 3). PH modifications were made with (HCl or NaOH) solutions. The results in (Table, 4), indicate that the potentials slightly change and remain constant from  $\sim$ pH (3.0 to 9.0). Therefore, this range can be considered as the pH of working electrode senses. The behaviors of this membrane can be explained as follow: a) the pKa of aspirin is about 3.0, i.e. at acidic pH, observed that the potential will be relatively high at this range; this might be because the membrane can responds to H<sup>+</sup> activity. b) at higher pH 9.0, it becomes increasingly dissociated for this explanation we have noticed a decline in potential.

**Table (4):** Effective pH ranges used for ASP-selective electrodes.

Membranes no.	Membrane composition	PH rang		
		$1 \times 10^{-2}$	$1 \times 10^{-3}$	$1 \times 10^{-4}$
I	ASP-MIP <sub>1</sub> (VA+MBAA+DBS)	2.5-9.0	3.0-8.5	2.5-9.5
II	ASP-MIP <sub>1</sub> (VA+MBAA+DOP)	3.0-9.0	2.5-9.5	4.0-9.5
III	ASP-MIP <sub>2</sub> (VIZ+MBAA+DBS)	3.5-9.0	3.0-9.0	3.5-10.0
IV	ASP-MIP <sub>2</sub> (VIZ+MBAA+DOP)	3.0-9.5	2.5-9.5	3.0-9.0

### Selectivity of ASP-electrodes potentiometric

The influence of interferences on the electrodes-response behavior is usually described as the selectivity coefficients. So the K<sub>pot</sub> selectivity coefficient of MIP-sensor's was analyzed using the separation solution method (SSM) and Matched Potential Method (MPM). The measured by SSM methods depend on equation of the Nickolsky-Eisenman but the SSM methods does have some disadvantages in line with interference ions (unequal charges) with non-Nernstainbehavior. Thus, a similar procedure referred to as [Matched Potential Method (MPM)] was preferred, in specific, as the primary or interference ion does not obey on the

Nernst response, or when the ions concerned is different in charge. As seen from results in (Table 5 and 6) the selectivity coefficients achieved for all prepared electrode sensors, that most of these compounds did not interfere with the response of the electrode sensors membrane.

**Table (5):** Result of selectivity coefficients for some interfering species using SSM for MIP<sub>1</sub> with different plasterer.

Interfering ions	Concentrations of Aspirin (MIP1+DBS) M: Concentrations of interference ions (M)													
	0.1		0.01		0.005		0.001		0.0005		0.0001		0.00005	
	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>
K <sup>+</sup>	-151.4	0.0078	-146.8	0.041	-143.2	0.0433	-136.6	0.131	-132.2	0.197	-127.4	0.502	-124.3	0.781
Ca <sup>2+</sup>	-157.1	0.0046	-150.4	0.006	-149.3	0.0059	-147.9	0.014	-144.1	0.016	-142.5	0.025	-138.2	0.025
Al <sup>3+</sup>	-156.3	0.0029	-153.2	0.0038	-152.6	0.0035	-150.1	0.0056	-148.7	0.007	-147.3	0.009	-145.8	0.011
M.P.	-131.7	0.0009	-130.5	0.0071	-129.4	0.0098	-127.6	0.0498	-127.3	0.116	-126.7	0.466	-126.4	0.979
P.P.	-133.8	0.0012	-131.6	0.008	-130.4	0.0109	-128.9	0.0572	-126.6	0.108	-124.2	0.356	-120.5	0.519
T.S.C.	-134.6	0.0003	-133.9	0.0005	-133.2	0.0004	-132.5	0.0008	-131.7	0.001	-129.7	0.001	-129	0.002
Interfering ions	Concentrations of Aspirin (MIP1+DOP) M: Concentrations of interference ions (M)													
	0.1		0.01		0.005		0.001		0.0005		0.0001		0.00005	
	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>
K <sup>+</sup>	-241.4	0.0105	-239.2	0.071	-238.8	0.194	-223.4	0.0996	-220.1	0.174	-219.7	0.617	-217.5	0.878
Ca <sup>2+</sup>	-238.7	0.0023	-236.4	0.005	-234.1	0.007	-231.8	0.0094	-230.3	0.015	-229.6	0.022	-226.4	0.02
Al <sup>3+</sup>	-235.5	0.001	-232.1	0.001	-230.4	0.002	-229.4	0.0022	-226.1	0.002	-225.5	0.003	-224.7	0.003
M.P.	-231.7	0.003	-229.4	0.02	-226.4	0.038	-224.1	0.1091	-221.9	0.221	-217.7	0.476	-214.2	0.571
P.P.	-230.7	0.0026	-227.4	0.015	-225.3	0.033	-221.8	0.0809	-220.7	0.189	-217.5	0.464	-215.8	0.703
T.S.C.	-233.7	0.0008	-230.1	0.001	-225.5	0.001	-224.3	0.0011	-221.2	0.001	-219.8	0.001	-219.1	0.001

**Table (6).** Result of selectivity coefficients for some interfering species using SSM for MIP<sub>2</sub> with different plasterer.

Interfering ions	Concentrations of Aspirin (MIP2+DBS) M: Concentrations of interference ions (M)													
	0.1		0.01		0.005		0.001		0.0005		0.0001		0.00005	
	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>
K <sup>+</sup>	-90	0.0007	-54.3	5×10 <sup>-5</sup>	-35.7	6×10 <sup>-6</sup>	-28.2	1.5×10 <sup>-5</sup>	-22.4	1×10 <sup>-5</sup>	-21.5	6×10 <sup>-5</sup>	-16.6	9×10 <sup>-5</sup>
Ca <sup>2+</sup>	-81.1	7×10 <sup>-5</sup>	-63.4	2×10 <sup>-5</sup>	-40	8×10 <sup>-7</sup>	-23	2.4×10 <sup>-7</sup>	-24.6	4×10 <sup>-7</sup>	-24.5	9×10 <sup>-7</sup>	-21.7	1×10 <sup>-6</sup>
Al <sup>3+</sup>	-73.6	2×10 <sup>-5</sup>	-50.6	1×10 <sup>-6</sup>	-48.1	9×10 <sup>-7</sup>	-42.4	9.6×10 <sup>-7</sup>	-37.9	6×10 <sup>-7</sup>	-31.3	5×10 <sup>-5</sup>	-29.7	7×10 <sup>-7</sup>
M.P.	-101.2	0.003	-85.3	0.003	-84.2	0.0036	-75.5	0.00752	-71.4	0.0082	-63.6	0.0155	-61.8	0.0347
P.P.	-79.8	0.0002	-70.4	0.0004	-67.5	0.0004	-60.2	0.001	-54.6	0.0009	-51.5	0.0032	-49.7	0.007
T.S.C.	-79.6	4×10 <sup>-5</sup>	-70.6	2×10 <sup>-5</sup>	-64.1	7×10 <sup>-6</sup>	-55.4	5.3×10 <sup>-6</sup>	-50.9	3×10 <sup>-6</sup>	-45.3	3×10 <sup>-6</sup>	-41.2	3×10 <sup>-6</sup>
Interfering ions	Concentrations of Aspirin (MIP2+DOP) M: Concentrations of interference ions (M)													
	0.1		0.01		0.005		0.001		0.0005		0.0001		0.00005	
	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>	E <sub>B</sub> (mV)	K <sub>A,B</sub>
K <sup>+</sup>	-112.8	8.7×10 <sup>-8</sup>	-93.9	9.6×10 <sup>-8</sup>	-91.1	8.5×10 <sup>-8</sup>	-85.7	2.8×10 <sup>-7</sup>	-84.2	6.1×10 <sup>-7</sup>	-80.7	1.5×10 <sup>-6</sup>	-77.3	2.1×10 <sup>-6</sup>
Ca <sup>2+</sup>	-116.3	4.2×10 <sup>-8</sup>	-106	4.3×10 <sup>-8</sup>	-105	3.3×10 <sup>-8</sup>	-101.2	6.0×10 <sup>-8</sup>	-97.8	7.2×10 <sup>-8</sup>	-95.5	9.4×10 <sup>-8</sup>	-92.5	9.8×10 <sup>-8</sup>
Al <sup>3+</sup>	-119.7	4.4×10 <sup>-8</sup>	-116.1	6.9×10 <sup>-8</sup>	-115.8	5.2×10 <sup>-8</sup>	-110.3	5.8×10 <sup>-8</sup>	-109.6	8.73×10 <sup>-8</sup>	-109	1.1×10 <sup>-7</sup>	-107.4	1.2×10 <sup>-7</sup>
M.P.	-163.9	4.8×10 <sup>-5</sup>	-157.8	0.00025	-154.3	0.00021	-153.2	0.00116	-151.7	0.002494	-144.6	0.004034	-139.8	0.00474
P.P.	-166.5	6.6×10 <sup>-5</sup>	-161.2	0.00039	-160.7	0.00045	-159	0.00237	-155.3	0.003888	-153.2	0.011652	-150.4	0.01751
T.S.C.	-151.3	2.2×10 <sup>-6</sup>	-148.7	3.8×10 <sup>-6</sup>	-148.2	2.8×10 <sup>-6</sup>	-142.9	3.3×10 <sup>-6</sup>	-143.9	6.0×10 <sup>-6</sup>	-141.2	5.7×10 <sup>-6</sup>	-140.5	7.0×10 <sup>-6</sup>

**Analysis of commercial tablets**

In order to illustrate the technical use of the electrochemical sensor, two tablets for (ASP) were analyzed by MIP electrodes. The solutions was obtained by dissolving the specific

weight of commercial tablets in methanol solvent and diluted so that the tablet concentration range lies within calibration plot values. The DPVs value then was recorded under precisely the same conditions. The suggested technique was used to measure the concentration of all selected drugs in two types of pharmaceutical products. In order to verify the electro-chemical detection, we have compartmented the data results for parameters RSD%, RC% and Erel% with both detection methods including: direct potentiometric, standard addition method (SAM), multi standard addition method (MSA), by using ISE, as well as titration method and optimal chromatographic conditions. The results was indicated in (Table, 7&8)

**Table (7).** Recovery results and standard deviation of ASP-drugs obtained through the use of (MIP1+DBS).

Drug	Concentration Prepared/ M	Potentiometric methods	Concentration Found/ M	%Rec.	%RE	%RSD
Aspirin material pure	1X10 <sup>-3</sup>	Direct method	1.0312×10 <sup>-3</sup>	103.1	3.12	2.31
		SAM	1.0556×10 <sup>-3</sup>	103.22	3.22	2.07
		MSM	1.0402×10 <sup>-3</sup>	104.02	4.02	0.41
	1X10 <sup>-4</sup>	Direct method	1.0300×10 <sup>-4</sup>	103.15	3.15	1.55
		SAM	1.0455×10 <sup>-4</sup>	103.41	3.41	1.1
		MSM	1.0356×10 <sup>-4</sup>	103.56	3.56	0.22
Chewable tablet, acetylsalicylic acid (SDI)-IRAQ	1X10 <sup>-3</sup>	Direct method	1.0343×10 <sup>-3</sup>	103.42	3.42	0.92
		SAM	1.0554×10 <sup>-3</sup>	104.39	4.39	0.95
		MSM	1.0332×10 <sup>-3</sup>	103.32	3.32	0.33
	1X10 <sup>-4</sup>	Direct method	1.0279×10 <sup>-4</sup>	102.79	2.79	0.77
		SAM	1.0687×10 <sup>-4</sup>	104.57	4.57	2.2
		MSM	1.0310×10 <sup>-4</sup>	103.1	3.1	0.23
acidumacetylsaicylicum, Memphis/Bayer	1X10 <sup>-3</sup>	Direct method	1.0311×10 <sup>-3</sup>	103.12	3.11	1.4
		SAM	1.0541×10 <sup>-3</sup>	104.06	4.06	1.3
		MSM	1.0394×10 <sup>-3</sup>	103.94	3.94	0.26
	1X10 <sup>-4</sup>	Direct method	1.0315×10 <sup>-4</sup>	103.15	3.15	1.55
		SAM	1.0526×10 <sup>-4</sup>	103.74	3.74	1.68
		MSM	1.0353×10 <sup>-4</sup>	103.53	3.53	0.34

**Table (8).** Recovery results and standard deviation of ASP-drugs obtained through the use of (MIP2+DBS).

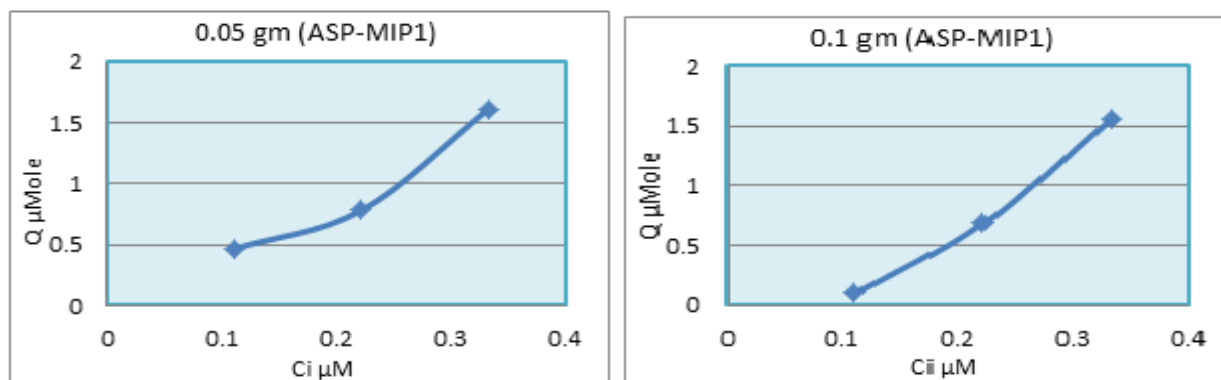
Drug	Concentration Prepared/ M	Potentiometric methods	Concentration Found/ M	%Rec.	%RE	%RSD
Aspirin material pure	1X10 <sup>-3</sup>	Direct method	1.0378×10 <sup>-3</sup>	103.78	3.78	1.71
		SAM	1.0528×10 <sup>-3</sup>	102.55	2.55	2.44
		MSM	1.0367×10 <sup>-3</sup>	103.67	3.67	0.48
	1X10 <sup>-4</sup>	Direct method	1.0370×10 <sup>-4</sup>	103.69	3.69	2.19
		SAM	1.0383×10 <sup>-4</sup>	102.42	2.42	1.79
		MSM	1.0260×10 <sup>-4</sup>	102.6	2.6	0.41
Chewable tablet, acetylsalicylic acid (SDI)-IRAQ	1X10 <sup>-3</sup>	Direct method	1.0451×10 <sup>-3</sup>	104.51	4.51	0.68
		SAM	1.0698×10 <sup>-3</sup>	104.2	4.2	2.44
		MSM	1.0323×10 <sup>-3</sup>	103.23	3.23	0.49
	1X10 <sup>-4</sup>	Direct method	1.0450×10 <sup>-4</sup>	104.53	4.53	1.51
		SAM	1.0498×10 <sup>-4</sup>	102.89	2.89	1.79
		MSM	1.0262×10 <sup>-4</sup>	102.62	2.62	0.41
acidumacetylsaicylicum, Memphis/Bayer	1X10 <sup>-3</sup>	Direct method	1.0456×10 <sup>-3</sup>	104.56	4.56	1.73
		SAM	1.0586×10 <sup>-3</sup>	103.77	3.77	2
		MSM	1.0376×10 <sup>-3</sup>	103.76	3.76	0.4
	1X10 <sup>-4</sup>	Direct method	1.0480×10 <sup>-4</sup>	104.81	4.81	1.92
		SAM	1.0626×10 <sup>-4</sup>	104.54	4.54	2.02
		MSM	1.0324×10 <sup>-4</sup>	103.24	3.24	0.47

### Adsorption Isotherm

Adsorption isotherm is useful in understanding the adsorption mechanism of the adsorption template on a polymer surface. The collected data from the adsorption isotherm equilibrium were studied to illustrate the isotherm model Langmuir or Freundlich this was accomplished by plotting the ability of binding (Q) against free drug concentration, Q is determined according to the equation below: Adsorption isotherm produced after preparation of different concentrations of standard solution at room temperature is shown in (Figure,3). Experimental results for classifying experiments have been included in the (Table, 9).

**Table (9).** Rebinding values of (ASP) using [ASP –MIP<sub>1</sub>] particles based on (VA).

Mass of MIP g	[ASP-MIP <sub>1</sub> (VA)]			
	C <sub>i</sub> mM	C <sub>free</sub> mM	Q μMole /g	Q/C <sub>free</sub> L/g
0.05	0.111	0.10866	0.4671	4.29825
	0.222	0.2181	0.7802	3.57724
	0.333	0.32494	1.6113	4.95868
0.1	0.111	0.10996	0.1043	0.94862
	0.222	0.21517	0.6834	3.17604
	0.333	0.31741	1.5592	4.91228



**Figure (3):** Binding isotherm of (ASP –MIP<sub>1</sub>) using VA as monomers.

### CONCLUSION

In this research, four electrodes were prepared based on MIP method using two monomer (VA, VIZ) and two different plasticizer (DBS, DOP), as it was observed that the interaction between template and the monomer was non-covalently, therefore the ASP-drug was extracted easily to form selective cavity for estimation commercial ASP and excellent results obtained at lowest costs and with high accuracy.

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## BANKING COMPLIANCE FUNCTION (COMPARATIVE STUDY BETWEEN COMMERCIAL & ISLAMIC BANKS)

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### ABSTRACT

Never the less, banking compliance function became one of the most important functions in banking sector according to its characteristics that considered as an interior control tools to control (executive management, departments, subsidiaries...etc) in any bank; and their compliance towards applying rules, recommendations and legislations. In addition to, estimating the risks and limited them; and controlling the anti-money laundering. Thus, these functions that covered the main concept of (Banking Compliance) would avoid the bank to be under the control of any sanctions.

**Keywords:** Banking compliance, interior control tools, anti money laundering, sanctions.



## وظيفة الامتثال المصرفي (دراسة مقارنة بين المصارف التجارية والإسلامية)

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## الخلاصة

أصبحت وظيفة الامتثال المصرفي واحدة من أهم الوظائف في القطاع المصرفي وفقاً لخصائصها التي تعتبر أدوار رقابية داخلية للرقابة (الإدارة والتنفيذية، الإدارات، الشركات التابعة... إلخ) في أي مصرف، والتزامهم بتطبيق القواعد والتوصيات التشريعات، فضلاً عن تقدير المخاطر والخدمتها، ومكافحة غسل الأموال، وبالتالي فإن هذا هو وظائف التي تغطي المفهوم الرئيسي (الامتثال المصرفي) منشأها أنتجنب البنك أن يكون تحت سيطرة أي عقوبات. الكلمات المفتاحية: الامتثال المصرفي، أدوار رقابية الداخلية، مكافحة غسل الأموال، العقوبات.

## INTRODUCTION

Banks in general considered as the most essential financial institutes in every country, according to its role and services that submitted to their clients. But never the less, with this high level of importance, there is a high level of risk if the bank does not follow the rules; more over the bank could be punished or loss its reputation towards others either the client is private, company or even country.

With the rapid changes in all economic life in general and in finance in special, the financial institutes and the controlling authority around the world always trying to find a maintenance and developing processes to limit the risk as much as possible towards shareholders, stakeholders, clients, ..etc; by taking the responsibility from the whole level of management in any bank.

## MATERIALS AND METHODS OF THE WORK

## Methodology of the article

## Problem

According to the spared amount of number of banks in Iraq during a short period of time and the new interring of foreign investment in addition to the risk of the resources of these coming money (from where); results in establishing the function of Banking Compliance; because the Iraq Banking law number 94 for 2004 and its rules number 4 for 2010, only define the function of compliance without including the risks of compliance, independence or not, and its relationships with other departments of the bank.

## Importance

Discussing one of the most important sectors of banking compliance, and its duties and its relations with other departments of the bank how compliance banking worked a supporter and tool to correct the mistakes and avoiding risks.

## Objectives

Understanding the concept of banking compliance in Iraqi banks, and its duties, roles to face risks and sections to avoid its punishments; and selecting the limitations of banking compliance as a function.

## Article Model

Using financial model to measure the results only.

## Hypothesis



Applying the roles and systems of Central Bank of Iraq CBI as well as applying the law of Anti- money laundering and Terrorism funding No., 39 for 2015; leads to limited the risks and boundaries that could banks faced.

### Methodology

Discussing & Analyzing the reports of bank's compliance of both Commercial and Islamic banks of (Cash Credit, Investments, Fixed Assets, Customer Deposits and Benefits).

### Boundaries

Two kinds of boundaries:

1. Place Boundaries: Iraq only (Commercial & Islamic banks).
2. Time Boundaries: For the years (2017-2018) according to the CBI reports.

### Location of the Study

Iraqi Bank Sector (Commercial & Islamic Banks).

### Validity and Reliability of tool and statistical methods used

Analyzing the financial reports of (Commercial and Islamic Banks).

### Relevant Pervious Studies

Reviewing the literature and how the bank compliance was used and developed through time; bank compliance is the applying of laws and systems that announced from the central bank according to its importance in doing duties with high level of ethics and responsibilities (**Pereira, 2012**). Although; it is difficult to measure the added value due to applying the bank's compliance but otherwise, it costs high amount of taxes which leads to bad situation and losing its reputation which is the worst point in business (**Al-Asraj, 2013**). According to laws and systems, it could be different due to its content, but in all cases, banks have to declare them through their bank's financial reports (**Ergys, 2016**).

One definition of compliance as Al Kasas define is (verification of the level of adopting the rules, plans and procedures) (**Al-Kasas, 2014**). Applying bank's compliance leads to effective control to avoid any limitations later (**Sloka, 2012**).

Another meaning of compliance is being sure of avoiding any kind of cheating or using these rules in a crossing path to be hidden from any law (**Hadi, 2014**).

By following the International Auditing Standard No.: (250); Management considered as the responsible side of applying the compliance.

Basel Committee on Banking Supervision BCBS defined this function as independent function to assist, select and submitting recommendations; controlling and auditing then applying the regular reports to avoid any bad situation (**Ergys, 2016**).

Under this job title, the bank compliance applying many duties (**Hilal, 2013**):

1. Studying the reports of clients.
2. Programing certain systems of bank compliance.
3. Reviewing the Anti-money laundering cases.
4. Supporting the executive management.
5. Advising the executive management with certain standards and laws to apply (**Abbas, 2012**).
6. Educate the clients and people in general of the risks of non-applying the bank compliance.
7. Documented and define every step to avoid any further compliance; if adding new banking product or service (**Enrica, 2006**).

And the responsible officer of compliance management should follow the principles of (**Systems Guide, 2008**), starting with (collecting data, preparing policies, periodic review the whole activities, updating all procedures according the two newest laws and legislations, submitting every document to the executive management as well as bank's employees,

planning for clear plan for auditing and controlling, affiliate the actual process with the planed ones, omitting and/ or adding every services or product of banks; and aligning the whole departments of banks especially the legislation's consultants either from inside/ outside the banks with the policy of bank compliance) (**Victor Livio, 2017**).

As any job function, there are many principles for applying bank compliance (**Amal, 2018**), such as systems, supervision and auditing legislations that related to all bank's activities especially the Anti- money laundering, terrorism's funding and other banking cheating processes. Rules and principles that announced by government such as company system, financial market institute, principles of foreign investment, and other rules (**Al-Asraj, 2013**). In addition to BABS principles that related to bank compliance (**Victor, 2017**), in which BABS limited them as the (10<sup>th</sup> principles) and any bank or financial institutes should follow (**Ergys, 2016**).

Al Shameri, in 2016 referred to the success of this function in which it should be completely independent from the executive management, and it should be under independent department that achieving its duties effectively and efficiently (**Al Shameri, 2016**).

## RESULTS AND DISCUSSION

The distinguished results for the years 2017- 2018 of the Commercial Banks referred to the percentage of liquidity in 2017 was 103% while in 2018 was 98% of the total bank assets, and these percentages referred that the commercial banks are completely applied the bank compliance because its under the CBI selected percentage 30%.

The bank credit of assets was in 2017 around 9% and in 2018 was around 8%, due to the policy of the bank according to the critical political situation of the country.

The percentage of the guarantees in 2017 was 62% and in 2018 was 31% in which the rest of guarantees are funded by real estate guarantees.

The insurance percentage for 2017 was 15% and for 2018 was 13%, these percentages are low with reference with the total credits.

The percentage of cash & pledge credit of the total assets for 2017, 2018 were 64 and 66%, while the CBI required percentage is 800%, so the funded percentages are not as required.

The percentage of saved liquidity for 2017, 2018 were only 1% which reflects a very low percentage that leads to risks of non-applying the bank compliance.

The percentage of capital shares was only 1% for both 2017-2018 according to the common financial crisis which caused the low prices of shares in Iraqi Stock Markets.

The distinguished results for the years 2017-2018 of the Islamic Banks referred to an obvious increasing in capital in which it was 275% for 2017 while for 2018 it was 214%, so they lead to a high level of liquidity that leads to buying the liabilities, and the bank is applying the CBI principles of bank compliance.

The percentage of cash credit of savings was 23889% in 2017 while in 2018 it was 2306%, these percentages lead to high level of risks in capital.

The percentage of insurance of guarantee was 20% in 2017, and 19% in 2018, which referred that the bank follows the CBI principles and there is no letter of credit LC for these years.

The percentage of savings was 247% in 2017 and 237% in 2018 which reflects the wrong usages of investment.

**Table (1):** Percentages calculated for the years 2017 and 2018.

Indicators	2017 (%)	2018 (%)
Liquidity	103	98
Bank credit	9	8

Guarantees	62	31
Insurance	15	13
Cash & Pledge	64	66
Saved liquidity	1	1
Capital shares	1	1
Increasing in Capital	275	214
Cash credit of saving	23889	2306
Insurance of guarantee	20	19
Saving	247	237

## CONCLUSION

With reference to Commercial bank, the liquidity credit consists of two credits of product which was 66525590 ID for 2017 and in 2018 was 88666881 ID, so the bank does not save for facing risks; and the second one was anti-product credit, which was 61555696 ID in 2017 and in 2018 it was 46589344 ID, so the bank follows the principles of CBI. The investments of shares and banking transferring was increasing in 2018 than 2017 around 352510, while the transferring was decreasing around 50% from 2017 to 2018, which referred to the positive adopting of CBI principles. In addition to the level of depreciation of fixed assets is as CBI required which was around 1846838 ID in 2018. Finally, the bank achieved around 4094163 ID in 2017 as benefits of essential activities and around 22814879 ID as benefits of nonessential activities, while in 2018 the bank achieved around 4799558 ID as benefits of essential activities and around 29659643 ID as benefits of non-essential activities, and with reference to Islamic bank, the liquidity credit was 149590833 ID for 2017 and in 2018 was 195813583 ID, both of these credits were for product accounts, in which the bank save around 1521963 ID to face the product credit for 2017 and around 3916265 ID for 2018. The amount of fixed assets in 2017 was 6971640 ID and in 2018 around 7068006 ID as fixed assets and under construction projects that referred to the level of depreciation is as CBI required.

The deposits of 2017 were 6259772 ID, while in 2018 it was 8490161 ID, so the essential deposits of 2017 were 11% and decreased to 9% in 2018, which lead to many risks in liquidity in addition to the lack of supporting the deposits. While the nonessential deposits of 2017 were 89% and in 2018 was 91% which lead to the lack of facing the client's request. Finally, the bank achieved financial inability around 3254631 ID in 2017, and around 193596ID) in 2018; this financial inability caused the lack of reputation around the foreigners.

The bank compliance becomes one of the most essential function in any banking and financial sectors to avoid the antimoney laundering and terrorism funding and other nonlegislation producers through many systems, rules and legislation that assured the ethical as well as moral financial transactions.

Most of high percentages of the capital adequacy that this study reached are according to the instability of political as well as civil society of the county; although the CBI percentage was 12% but they achieved the double; which means that these banks are lacked in investing while there is a high level of liquidity.

## RECOMMENDATIONS

To avoid these results, banks should follow the principles of CBI and going on developing the bank compliance systems to avoid any financial risks or / and crisis, moreover, the liquidity should be generated through many investing projects to reduce the pressure upon the financial budget of the country and trying to find other sources to support the financial budget through the term of participating between the government and the private banks.

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## THE ROLE OF SATELLITE CHANNELS IN FORMING TRAFFIC AWARENESS AND PREVENTING ACCIDENTS - A FIELD STUDY

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### ABSTRACT

The importance of the research comes from dealing with the problem of lack of traffic awareness, which causes accidents and the occurrence of human and material losses, and the research aims to study the role of satellite channels in forming traffic awareness among the public, and a sample was chosen from Baghdad consisting of (280) individuals, male and female, and used the questionnaire tool. To obtain the data, which included several questions, the results were analyzed statistically and several results were reached, the most important of which is that there is an interest among the public in following traffic programs at a rate of one to two hours to receive information through traffic programs and to identify and apply general rules, and it was found that there is a statistically significant correlation between the extent of follow-up. The sample is for traffic programs and the extent to which satellite channels spread traffic awareness among individuals, and there is a significant correlation between the sample's intensity of watching traffic programs and the level of obtaining information about traffic awareness from television, and there are differences between the sample averages towards the role of satellite channels in spreading traffic awareness.

**Keywords:** Television, audience, traffic, awareness, field.



## دور القنوات الفضائية في تشكيل الوعي المروري والوقاية من الحوادث- دراسة ميدانية

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## الخلاصة

تأتي أهمية البحث من تناوله اشكالية غياب الوعي المروري الامر الذي يتسبب في وقوع الحوادث وحدوث الخسائر البشرية والمادية، ويهدف البحث لدراسة دور الفضائيات في تشكيل الوعي المروري لدى الجمهور، وتم اختيار عينة من مدينة بغداد مكونة من 280 فردا من الذكور والاناث واستخدمت اداة الاستبانة للحصول على البيانات والتي تضمنت اسئلة عدة تم تحليل النتائج احصائيا وتم التوصل الى نتائج عدة اهمها ان هناك اهتمام لدى الجمهور في متابعة البرامج المرورية بمعدل ساعة الى ساعتين لتلقي المعلومات عبر البرامج المرورية والتعرف على القواعد العامة وتطبيقها، وتبين وجود علاقة ارتباط دالة احصائيا بين مدى متابعة العينة لبرامج المرور ومدى قيام الفضائيات في نشر الوعي المروري لدى الافراد، كما ان هنالك علاقة ارتباط دالة بين كثافة مشاهدة العينة لبرامج المرور ومستوى الحصول على المعلومات عن الوعي المروري من التلفزيون، ويوجد فروق بين متوسطات العينة نحو دور القنوات الفضائية في نشر الوعي المروري.

الكلمات المفتاحية: التلفزيون، الجمهور، المرور، التوعية، ميدانية.

## INTRODUCTION

Awareness is one of the most important jobs carried out by the media in various fields, and it is concerned with providing news and topics to the receiving audience. Therefore, the media undertakes a social responsibility to spread traffic awareness among the public, which is the subject of research by presenting a field study of a sample of the community in order to identify the level of traffic awareness and the role of satellite channels in its development.

FIRST: METHODOLOGY OF RESEARCH  
RESEARCH PROBLEM

The research problem can be identified by asking the question: What is the role of satellite TV in forming traffic awareness and preventing accidents? And the following sub-questions:

What are the levels of traffic awareness of the audience receiving the traffic satellite programs?

What is the role played by satellite channels in spreading traffic knowledge?

How well does the sample follow up on TV traffic programs?

What are the causes of traffic accidents?

What are the methods that contribute to spreading traffic awareness?

## RESEARCH IMPORTANCE

The importance of the research lies in dealing with one of the important problems and issues after the increasing number of traffic accidents and the resulting human and material losses that negatively affect the development and development of society, so the importance is to spread traffic awareness, which is one of the important concepts that require further research and study Also, this research is an attempt to spread awareness, education and guidance by shedding light on the role of the media, especially television, and the research deals with this topic with a scientific treatment that contributes to awareness and reduces traffic accidents that occur as a result of lack of awareness and perception of traffic instructions and laws, and linking the role of television programs to traffic awareness With the audience.

## SEARCH OBJECTIVES

1. Knowing the extent of traffic awareness among the public.

2. Defining the role of satellite channels in spreading traffic awareness.
3. Clarify the extent of the sample's follow-up to TV traffic programs.
4. Knowing the extent to which the public depends on satellite channels to obtain traffic information.
5. Determine the causes of traffic accidents.
6. Determining the means of spreading traffic awareness.

### **SEARCH HYPOTHESES**

1. There are differences in the demographic characteristics of the sample towards watching TV traffic programs.
2. There is a significant correlation between the sample's intensity of watching traffic programs and the level of obtaining information about traffic awareness from television.
3. There are differences between the sample averages in the role of satellite channels in spreading traffic awareness.

### **RESEARCH METHODOLOGY**

The descriptive survey method was used, which is the most appropriate method for research to obtain information to study the phenomenon, its characteristics and characteristics.

### **RESEARCH COMMUNITY AND SAMPLE**

The research community is represented in the center and outskirts of Baghdad. An intentional sample was taken from vehicle drivers, and the sample consisted of (280) individuals, males and females with different characteristics.

### **SEARCH TOOL**

The observation tool was used with the aim of obtaining information on the characteristics of the research sample, the factors affecting it, and following up on television programs that pertain to traffic. The questionnaire form tool that was presented to the experts, was modified and distributed by (280) forms ready for analysis.

### **SEARCH LIMITS**

Spatial domain: The spatial domain is determined by the most urgent in the city of Baghdad, the periphery and the center.

The temporal domain: The temporal domain of slaughter is determined in the period 1/1/2020 - 31/12/2020.

### **VALIDITY AND RELIABILITY TEST**

The apparent validity of the questionnaire was tested by presenting it to specialized professors who gave their scientific observations that were evaluated from the questionnaire questions and were finalized.

Stability was tested using (Alpha Cronbach), and the stability factor was (80.5).

### **STATISTICAL METHODS**

Use the statistical program spss in analyzing the results of the questionnaire. It used the test factor chi, the Pearson factor test, the ANOVA test and the Alpha Cronbach test for the stability of the test.

Sample traffic television programs:

The Safety Way Program: A weekly program that will be shown on Afaq satellite channel, which will be shown on Wednesday at 7 pm

Traffic pause program: a weekly program that is shown on the Iraqi satellite channel.

### **DEFINITION OF TERMS**

Role: A role is defined as being related to specific attitudes, specific behaviors, and the position of the individual through the job he performs in society (Shabani, 2006).

Satellite TV: They are channels broadcast from the satellite network whose paths are defined at a certain angle that transmit signals in specific directions as they rotate around the earth (**Abdul Nabi, 2010**).

Consciousness: Consciousness is defined as the state in which a person's mental state is aware of his natural surroundings through the senses possessed by the perceptions and the senses possessed by the person (**Khadour, 2007**).

Traffic accidents: are the damages resulting from a collision between one vehicle and another, or vehicles and public property, which leads to slight or large material and moral losses that may lead to human life as a result of deviation from the road or failure to abide by traffic rules (**Al-Asiri, 2009**).

### PREVIOUS STUDIES

- The study **Fatiha (2012)** entitled (The Role of the Media in Awareness and Prevention of Traffic Accidents in Algeria) The study aimed to know the means used by the media in traffic awareness. A comparative exploratory study was conducted between rural and urban areas. The most prominent of which is the presence of media influences on people, some of which are social, including economic and cultural, and there are influential factors and variables, each of which has its role in influencing the personality of the individual in addition to the effects of the family, institutions and others, and the results of the causes of accidents vary between urban and rural.
- The study **Architecture (2018)** entitled (The Role of Jordanian Television in Providing University Youth with Traffic Safety Information) The study aimed to find out the intensity of university youth's viewing of programs and brochures on traffic safety on Jordanian television and the sources of information for Jordanian university youth and the motives for watching traffic safety programs. The study consisted of 400 respondents and came out with results, the most important of which is that the intensity of Al-Hinah's follow-up to traffic programs on Jordanian television has a role in the level of information that Jordanian youth get.
- The study of **Illah (2019)** entitled (The role of the media in shaping traffic awareness and reducing accidents from the youth point of view), which is a field study aimed at knowing the role of the media in increasing traffic awareness and avoiding accidents, and identifying differences in demographic data in the level of awareness. The sample is from 200 respondents using a simple random sampling method. The study yielded a set of results, the most prominent of which is the positive role of the media in increasing traffic awareness, and that there are differences in the sample's attitudes towards the role of the media in traffic awareness.

### THEORETICAL FRAMEWORK

Satellite television channels are characterized as an effective means of influencing the audience due to the advantages they receive in terms of image and sound, keeping up with events in the world at the fastest possible speed, in addition to the persuasive and influential ability.

The media plays an important role in awareness, as it is one of the basic functions that the media performs due to the distinct characteristics that it possesses, and through the days of its role in achieving social responsibility towards society and developing individuals' sense of responsibility and integrating them in turn in order to evaluate incorrect behaviors and spread positive behaviors through communication and media programs. Educational, to avoid traffic accidents (**Obakat, 2007**).



The media also has the ability to influence behavior, opinions and ideas by achieving specific goals (**Ahmad, 2017**).

The media works to provide the public with facts and information, which leads to the formation of knowledge and perception to form a public opinion on various problems and issues (**Fatiha, 2012**).

The various media outlets provide and communicate information to individuals and educate them, and contribute effectively to the development of various projects (**Shaban, 2017**).

It is known that it affects the attitudes of the masses through the programs they provide, which is reflected in both positive and negative human behavior (**Shaban, 2019**).

**Traffic awareness:** Traffic awareness targets a person who has comprehensive knowledge of traffic culture from traffic rules, regulations, roads and vehicles, knowledge, awareness and learning come from the accumulation of information and experiences in the individual and from the daily practice of the general life of the individual (**Khadour, 2007**).

Traffic awareness includes the ability of a person to understand and know aspects related to the traffic aspect, its rules, and the routes of roads and vehicles.

Traffic awareness spreads knowledge among all citizens by practicing positive behaviors to adhere to traffic rules and regulations, and correcting their misconceptions by persuading people to provide sound information to achieve safety and public safety (**Bayan, 2010**).

The importance of traffic awareness is evidenced by preventing traffic accidents, adhering to the etiquette and ethics of driving vehicles and traffic instructions, and obeying general laws.

Traffic awareness is linked to respect for the provisions of the law and its legislation and to refer to it, and it is above all a kind of prevention of traffic accidents, knowledge of signs and regulations, which makes the individual be positive in his behavior and adherence to the traffic system (**Hamrani, 2010**).

Traffic awareness aims to form cognitive and intellectual trends by providing information and developing positive behaviors that replace negative ones in order to achieve the principle of traffic safety (**Tabani, 2012**).

It is necessary for awareness to have basics in the social and educational upbringing of the individual, and it must be placed within the media education in educational institutions and schools due to its importance in the formation of the individual's personality and his social responsibility towards the traffic field (**Al-Saied, 2008**).

## PRACTICAL SIDE

### 1. Demographic data for the sample:

**Table (1):** the sample description.

Details	Categories	Number	Percentage (%)
Type	Males	183	65.4
	Female	97	34.6
Age categories	20-30	83	29.6
	31-40	89	31.8
	41-50	52	18.6
	51 or more	56	20
Educational level	primary	5	1.8
	Medium	19	6.8
	Junior high	80	28.6
	diploma	15	5.4
	BA	131	46.7

	Postgraduate	30	10.7
Social status	Unmarried	80	28.6
	Married	150	53.6
	absolute	46	16.4
	Widower	4	1.4

It is evident from the results of (Table, 1) describing the sample that the percentage of males is (65.4%) by the number (183) while the percentage of females was (34.6%) with (97) from the sample. The highest percentage of the sample is for the age group (31-40) with a percentage of (31.8%), followed by the age group (20-30) at a rate of (29.6%) with (83), and the group (51 and more) got (56) with (20%), and the category (41-50) came with (52). (18.6%). As for the educational level of the sample, it came first in the bachelor's category with (131), at a rate of (46.7%), followed by the preparatory category with (80) and at (28.6%), and the number of those with higher degrees was (30) at a rate of (10.7%) of the sample. The marital status of the sample members is that a married group got (150) at a rate of (53.6%), and the category was unmarried with (80) at a rate of (28.6%), then followed in the ranking by an absolute group with (46) at a rate of (16.4%).

#### 2. Watching satellite channels:

**Table (2):** the extent of watching satellite TV.

Watch satellite TV	duplicates	Percentage (%)
Always	143	51
Sometimes	127	45.4
Scarcely	10	3.6
Total	280	100

(Table,2) the extent of the sample's viewing of satellite television, and the results indicate that the majority of the sample, with a total of (143) frequencies, (51%), answered they always watched satellite channels, while the total number of (127) frequencies (45.4%) answered that they sometimes watched satellite channels.

#### 3. The extent to which traffic programs are monitored on satellite television:

**Table (3):** the extent of follow-up of traffic programs on satellite television

The extent to which traffic programs are monitored	duplicates	Percentage (%)
Always	118	42.1
Sometimes	141	50.4
Scarcely	21	7.5
Total	280	100

(Table,3) the extent of the sample's follow-up to TV traffic programs, so the highest percentages of those who answered sometimes followed traffic programs with a total of (141) at a rate of (50.4%), and a total of (118), with a percentage of (42.1%), answered that they always follow the traffic programs in satellite channels.

#### 4. Hours of watching traffic programs:

**Table (4):** the sample viewing intensity for traffic TV programs.

Watch traffic hours	duplicates	Percentage (%)
Less than an hour	134	47.8
One to two hours	146	52.2
More than two hours	-	-
Total	280	100

(Table, 4) the intensity of the sample's viewing of TV traffic programs that the highest percentage of the sample watched from one to two hours, with a total of (146), at a rate of (52.2%), and that a total of (134), or (47.8%), watched traffic programs from one to two hours.

#### 5. Traffic programs provide what meets the public's need for knowledge:

**Table (5):** the extent to which traffic programs meet the public's need for knowledge.

Traffic programs provide what meets the needs of the public	duplicates	Percentage (%)
Always	133	47.5
Sometimes	136	48.6
Scarcely	11	3.9
Total	280	100

(Table,5) the extent to which traffic programs meet the public's need for knowledge through the topics they provide, as the highest response rate is sometimes a total of (136) with a rate of (48.6%), while a total of (133) responded with a rate of (47.5%) that traffic programs always Provide what meets the needs of the public of knowledge.

6. The media has a major role in spreading traffic awareness:

**Table (6):** the level of obtaining traffic awareness information from television.

The level of obtaining information on traffic awareness from television	duplicates	Percentage (%)
Always	148	52.9
Sometimes	122	43.6
Scarcely	10	3.6
Total	280	100

(Table,6) the level of obtaining information about traffic awareness from television from the viewpoint of the sample, as the highest response rate is always a total of (148) with a rate of (52.9%), while those who answered sometimes total (122) at a rate of (43.6%).

7. Causes of exposure to traffic accidents:

**Table (7):** the causes of traffic accidents.

Causes of traffic accidents	duplicates	Percentage (%)
Lack of awareness of traffic laws and regulations	97	34.6
Indifference and lack of responsibility	67	23.9
The constant traffic jam on the streets	6	2.1
Weak traffic control	3	1.1
Poor enforcement of strict laws on violators	42	15
Taking drugs while driving a vehicle	44	15.7
Talking on a mobile phone while driving a vehicle	21	7.5
Total	280	100

(Table,7) the reasons for the occurrence of traffic accidents that the highest percentage was obtained by the reason (lack of awareness of traffic laws and regulations) with a total of (97), at (34.6%), followed by the reason (indifference and lack of sense of responsibility) with a total of (67) at a rate of (23.9%) The reason (drug use while driving the vehicle) came to a total of (44), at a rate of (15.7%), and the reason (weak enforcement of strict laws on violators) got a total of (42) by (15%), and a total of (21) indicated (7.5%). The reason is (talking on a mobile phone while driving a vehicle) in the occurrence of traffic accidents, and I got the lowest answers (constant traffic congestion on the streets) and (poor traffic control).

8. Methods of spreading awareness and traffic culture:

**Table (8):** methods of spreading awareness and traffic culture.

Methods of spreading awareness and traffic culture	duplicates	Percentage (%)
Follow TV programs related to traffic	35	12.5
Developing a sense of social responsibility among community members	35	12.5



Adhere to traffic rules and regulations	71	25.4
Increase traffic awareness among the public through posters and pictures	48	17.1
Familiarity with traffic lights on the roads	54	19.3
Practicing road ethics and manners and driving vehicles	37	13.2
Total	280	100

(Table,8) methods for spreading awareness and traffic culture show that the highest percentage is (adherence to traffic rules and regulations) with a total of (71) at a rate of (25.4%), and the answer (familiarity with traffic signs on the roads) got a total of (54) at a rate of (19.3%) While a total of (48) responded with (17.1%) to (increase traffic awareness among the public through posters and pictures), and a total of (37) indicated (13.2%) on (practicing ethics and road manners and driving vehicles), and the two partners got (follow-up programs Television specializing in traffic) and (developing a sense of social responsibility among community members) on a total of (35), with a percentage of (12.5%).

#### 9. The role of satellite channels in spreading traffic awareness:

**Table (9):** the calculation of the arithmetic mean and the standard deviation to test the scale of the role of satellite channels in spreading traffic awareness.

The role of satellite TV in spreading traffic awareness	mean	standard deviation
Satellite channels contribute to spreading traffic awareness	2.892	0.309
Satellite TV provides the largest amount of traffic knowledge	2.778	0.494
Poor traffic programs are the reason for the lack of traffic awareness	2.807	0.395
Most of the information I knew about traffic laws was from traffic programs	2.782	0.413
Taking the advice provided by traffic programs and civilized awareness	2.796	0.429
Adherence to traffic instructions reduces accidents	2.839	0.367
Traffic accidents cause social and material losses to people and society	2.871	0.335
Provide strict security measures that regulate traffic issues	2.650	0.477
TV programs reduce traffic accidents	2.635	0.768

It is clear from (Table,9) the arithmetic mean and standard deviation of the role of satellite channels in spreading traffic awareness, as it was found that the phrase (satellite channels contribute to spreading traffic awareness) is the sum of those who agree with it (250) with a ratio of (89.3) and got the arithmetic mean (2.892), which is greater than the value The hypothesis mean (2) and the value of the standard deviation (0.309), and the sum of (228) with a percentage of (81.4) agrees with the phrase (satellite channels provide the largest amount of knowledge about traffic) with the value of the arithmetic mean (2.778) which is greater than the value of the hypothesis (2) and the value of The standard deviation is (0.494), and as for the phrase (poor traffic programs cause a lack of traffic awareness), the total of (226) who answered that they agree with it with a percentage is (80.7) with the value of the arithmetic



mean (2.807) which is greater than the value of the hypothesis (2) and the value of the standard deviation (0.395), and the phrase (most of the information I know about traffic laws was from traffic programs) got a total of (219) neutral with it with a ratio of (78.2) with the value of the arithmetic mean (2.782) which is greater than the value of the hypothesis (2) and the value of the standard deviation (0.413), and the total of (226) corresponds to (80.7) with the phrase (taking the advice provided by traffic programs A civilized awareness) of the value of the arithmetic mean (2.796), which is greater than the value of the hypothetical mean (2) and the value of the standard deviation (0.429), and the sum of (235) of (83.9%) indicated that they agree with the phrase (compliance with traffic instructions reduces accidents) with the value of the arithmetic mean (2.839) which is greater than the value of the hypothetical mean (2) and the value of the standard deviation (0.367), and the total of (244) at a rate of (87.1%) agrees with the phrase (traffic accidents cause social and material losses to people and society) with the value of the arithmetic mean (2.871), which is greater From the value of the hypothesis mean (2) and the value of the standard deviation (0.335), and the total of (182) at (65%) agrees with the phrase (providing firm security measures that regulate traffic issues) with the value of the arithmetic mean (2.650) which is greater than the value of the hypothesis (2) The value of the standard deviation is (0.477), and the sum of (228) indicates (81.4%) who do not agree with the phrase (TV programs limit traffic accidents) with the value of the arithmetic mean (2.635) which is greater than the value of the hypothesis (2) and the value of the standard deviation (0.768) While the total of (162), by (57.9%), agrees with the statement (Do not rely on obtaining controls and traffic information from the media) in the mean value The arithmetic (2.307) is greater than the hypothetical mean value (2) and the standard deviation value (0.870).

### Hypothesis test

The first hypothesis: There are differences in the demographic characteristics of the sample towards watching TV traffic programs

**Table (10):** differences in the demographic characteristics of the sample and the extent of follow-up programs to pass the CHI test.

The value of the chi	Significance level p-value	Level of morale	Degree of freedom	Relationship type
54.212	0.000	0.05	6	Function

(Table, 10) that the value of the chi parameter test (54,212) with significance level (0.000) is smaller than the level of significance (0.05), which indicates proof of the hypothesis and that the demographic characteristics of the sample formed significant differences towards following TV traffic programs. The existence of a statistically significant correlation relationship between the extent of the sample's follow-up to traffic programs and the extent to which satellite channels spread traffic awareness among individuals.

The second hypothesis: There is a significant correlation between the extent of the sample's follow-up to traffic programs and the level of obtaining traffic awareness information from television

**Table (11):** the relationship of the correlation between the intensity of the sample's viewing of traffic programs and the level of obtaining information about traffic awareness from television.

Pearson correlation coefficient value	Significance level p-value	Level of morale	Relationship type
0.700	0.000	0.05	Function

(Table,11) that the value of the Pearson correlation coefficient (0.700) with the level of significance (0.000) is smaller than the significant value (0.05), which indicates the validity of the hypothesis. Television.

The third hypothesis: There are differences between the sample averages in the role of satellite channels in spreading traffic awareness

**Table (12):** differences between the sample averages in the role of satellite channels in spreading traffic awareness.

Test value (F)	Significance level p-value	Level of morale	Degree of freedom	Relationship type
295.268	0.000	0.05	1	Function

It is evident from (Table,12) that the value of (F) test is equal to (295.268) and the degree of freedom (1) at the level of significance (0,000), which is smaller than the level of significance (0.05), which indicates the existence of differences between the sample averages towards the role of satellite channels in Spread traffic awareness.

## RESULTS AND CONCLUSIONS

1. Traffic awareness is of great importance as perception and understanding of traffic laws and traffic instructions reduce the occurrence of traffic accidents and abide by and respect the rules and regulations.
2. The results show that there is a large percentage of viewership of satellite channels, as 51% of respondents said they always watch satellite channels, and this indicates the importance of television as a public means that has its place and influence on the public in disseminating and consolidating information.
3. The percentage of (50.4%) of the sample follow traffic programs, which indicates the existence of good follow-up of traffic programs.
4. There is a large percentage of the public following traffic programs at a rate of one to two hours at a rate of (52.2%), and this indicates the presence of interest from the public in receiving information through traffic programs and getting acquainted with the general rules and their application.
5. The results indicate that traffic programs sometimes meet the needs of the public for information by 48.6%. This indicates that the programs need to develop the type of information they provide, and they must follow the needs of the public in the programs and endeavor to present them.
6. The absence of traffic awareness among the public is evident by identifying the causes of traffic accidents, most notably the lack of awareness of laws and traffic regulations by (34.6%), and indifference and lack of sense of responsibility by (23.9%).
7. The public needs specific methods to spread traffic awareness and reduce the occurrence of traffic accidents by adhering to traffic rules and regulations by (25.4%), and familiarity with traffic signs on the roads by (19.3%), as well as increasing traffic awareness among the public through posters and pictures by a percentage (17.1%)
8. It is evident that the public has a strong agreement regarding the role of satellite channels in spreading traffic awareness.
9. Proving the hypothesis that there is a statistically significant correlation between the extent of the sample's follow-up to traffic programs and the extent to which satellite channels spread traffic awareness among individuals.
10. The results of the validity of the hypothesis that there is a significant correlation between the sample's viewing intensity of traffic programs and the level of obtaining traffic awareness information from television.

11. Proving the hypothesis that there are differences between the sample averages regarding the role of satellite channels in spreading traffic awareness.

### RECOMMENDATIONS

1. Intensifying traffic awareness programs through audiovisual media.
2. The interest of educational institutions to place traffic awareness materials within the school curricula to teach them to school students.
3. Carrying out future studies interested in researching traffic awareness from various new aspects.

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## SYNTHESIS, CHARACTERIZATION, AND STUDY THE BIOLOGICAL ACTIVITY OF SOME SCHIFF'S BASES, AND 1,3 - OXAZEPINE COMPOUNDS DERIVED FROM SULFAMETHOXAZOLE DRUG

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### ABSTRACT

This study including synthesis of some new Schiff bases compounds [1-6] from the reaction of Sulfamethoxazole drug with some aromatic aldehydes in classical Schiff base method then treatment Schiff bases with succinic anhydride to get oxazepines rings [7-11] These derivatives were characterized by melting point, FT-IR, <sup>1</sup>H NMR and mass spectra. Some of synthesized compounds were evaluated in vitro for their antibacterial activities against three kinds of pathogenic strains *Staphylococcus aureus*, *Escherichia coli* and *Pseudomonas aeruginosa* by agar diffusion disk method, and against the fungal species (*Candida*). The results showed that some of these derivatives have good antibacterial activities compared to biological activity of parent drug.

**Keywords:** Sulfamethoxazole, Schiff base, 1,3-Oxazepine, antibacterial activity.



## تحضير تشخيص ودراسة الفعالية البيولوجية لبعض مركبات قواعد شف و3,1-اوكسازيبين المشتقة من دواء السلفاميثاكسازول

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**البحث مستل من أطروحة دكتوراه للباحث الأول**

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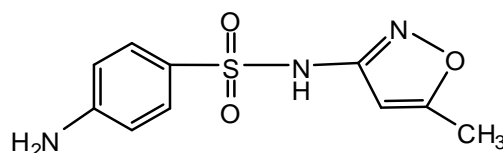
### الخلاصة

تضمنت الدراسة تحضير بعض المركبات الجديدة لقواعد شف (1-6) من تفاعل دواء السلفاميثاكسازول مع بعض الالدهايدات الاروماتية بالطريقة التقليدية لقواعد شف، وبعد ذلك تم مفاعلة قواعد شف المحضرة مع انهدريدالاسكسنيك لتعطي مركبات 3,1-اوكسازيبين (7-11)، وشخصت هذه المركبات بقياس درجات الانصهار واطياف الاشعة تحت الحمراء والرنين النووي المغناطيسي للبروتون والكتلة، وتم تقدير الفعالية الحيوية المضادة للبكتريا لبعض المركبات المحضرة ضد ثلاث أنواع سلالات مرضية لبكتريا *Staphylococcus aureus* و *Escherichia coli* و *Pseudomonas aeruginosa* وضد نوع من الفطريات (*Candida*) باستخدام طريقة أنتشار القرص في الاكار، وكشفت النتائج بعض هذه المركبات أظهرت فعالية جيدة ضد هذه البكتريا وللفطريات مقارنة بالدواء المشتقة منه. الكلمات المفتاحية: السلفاميثاكسازول، قواعد شف، 3,1-اوكسازيبين، الفعالية الحيوية المضادة للبكتريا.

### INTRODUCTION

4-Amino-N-5-methyl-3-isoxazolybenzenesulfonamide is the common name of Sulfamethoxazole, but N1-5-Methyl-3-isoxazolyl sulfanilamide, is the IUPAC name (Figure 1). This drug was considered by previous working groups (Lyon, 1980; Lyon, 1987). Sulfamethoxazole is an antibiotic that has been used since the 1990s to treat various general injuries in humans and different species. There was more use in the treatment of acute infections of the urinary tract. Also Sulfamethoxazole is used against gonorrhoea, meningitis, respiratory infections and prevention of poor meningococcal meningitis. Given the relatively unfavorable, pattern of tissue delivery, antibacterial medication that is widely used to treat various systemic infections worldwide with trimethoprim or pyrimethamine. With methoprim, the mixture is used mainly to treat the device's inflammation. Sulfamethoxazole of chloroquineresistant plasmodium falciparum malaria (Lyon, 1980; Gennaro, 1995; Elggellal & Alshadly, 2014).

Researches on complexes of sulfamethoxazole have a lot of physiological and pharmacological due to complexes of sulfa drugs have been discovered to be more bacteriostatic than the medication themselves (Alias et al., 2015; Al-Khodir, 2015).



**Figure (1):** The structure of Sulfamethoxazole.

A heterocyclic compound consisting of an oxygen atom at site 1 and a nitrogen atom at position 3 in addition to five carbon atoms is 1,3- Oxazepine compound. In 1965, the oxazepine derivative was introduced use to relieve psychoneurosis symptoms marked by anxiety and stress (Kuluod & Hamid, 2013). Oxazepine derivatives has been shown to display a broad range of biological activities, including antibacterial, antifungal, hypnotic relaxant, muscle inflammatory and antiepileptic activities (Taha, 2017).

## MATERIALS AND METHODE

### Materials and measurements of physical

Both reactants and solvents used in this study were reagent grade and are available from companies such as Sigma-Aldrich, BDH and Fluka. Sulfamethoxazole was obtained from Samara, Iraq.

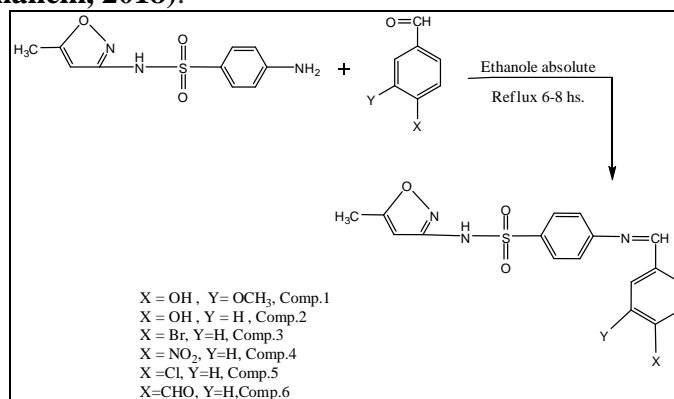
Melting points have been registered and are uncorrected using a hot stage Gallen Kamp melting point apparatus. SHIMADZU model FT-IR-8400S was used to receive the FT-IR spectrum. On the BRUKER model Ultra shield 300MHz spectrophotometer. <sup>1</sup>H-NMR spectra were obtained in the DMSO-d<sub>6</sub> solution with the TMS as the internal standard. Mass spectra were recorded using Mass Spectrometer, Agilent Technology (HP) at Tehran University, Central Lab, Iran.

### Common technique of preparing of Schiff bases (1-6)

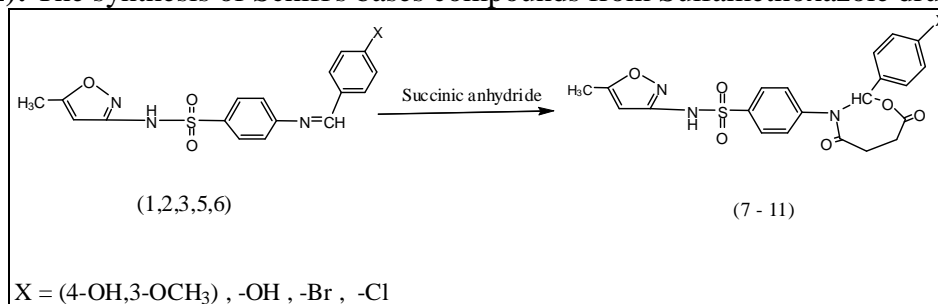
For 6-8 hrs, a mixture of Sulfamethoxazole (0.0039 mole, 1 g), and various aromatic aldehydes (0.0039 mol) in absolute ethanol (15 mL) and 3 drops of glacial acetic acid were refluxed (Jassim & Ali, 2018; Abdullah *et al.*, 2013). The mixture was cooled and the solid was purified after the end of the reaction, checked with TLC ethanol: benzene (1:1), then recrystallized from ethanol and collected by filtration, as shown in (Scheme 1) (Table 1) describes the physical properties of these compounds.

### Preparation 1,3-oxazepin-4,7-dione derivatives (7-11)

In 15 mL of dry benzene, Schiff base (1,2,3,5-6) (0.0005 mol) was combined with phthalic anhydride (0.0005 mol) and then refluxed for 18-20 hrs, evaporating the excess solvent. The solid product was washed with distilled water and then purified and recrystallized, as illustrated in (Scheme 2) (Table 2) lists the physical properties (Tawfiq *et al.*, 2012; Muhsen *et al.*, 2017; Sallal & Ghanem, 2018).



**Scheme (1):** The synthesis of Schiff's bases compounds from Sulfamethoxazole drug.



**Scheme (2):** The synthesis of 1,3-oxazepin-4,7-dione derivatives (7-11) by reaction Schiff's bases compounds with succinic anhydride



**Table (1):** Physical properties of compounds of the Schiff base(1-6).

Comp. No.	Structure of Compounds	Molecular Formula	Molecular Weight (g/mole)	Yield (%)	M.P. (°C)	Colour	Rf
1		C <sub>18</sub> H <sub>17</sub> N <sub>3</sub> O <sub>5</sub> S	387.40	76	172-174	Dark yellow	0.74
2		C <sub>17</sub> H <sub>15</sub> N <sub>3</sub> O <sub>4</sub> S	420.28	81	192-194	Pale yellow	0.76
3		C <sub>17</sub> H <sub>14</sub> N <sub>3</sub> O <sub>3</sub> SBr	420.28	81	196-198	Pale yellow	0.76
4		C <sub>18</sub> H <sub>14</sub> N <sub>4</sub> O <sub>5</sub> S	386.28	83	88-90	Yellow	0.77
5		C <sub>17</sub> H <sub>14</sub> N <sub>3</sub> O <sub>3</sub> SCl	375.78	86	137-139	Pale Yellow	0.81
6		C <sub>28</sub> H <sub>24</sub> N <sub>6</sub> O <sub>6</sub> S <sub>2</sub>	604.56	85	> 250(dec)	Orange	0.58

**Table(2):** The physical properties of 1,3-oxazepin derivatives (7-11).

comp. no.	Structure of Compounds	Molecular Formula	Molecular Weight (g/mole)	Yield (%)	M.P. (°C)	Colour	Rf
7		C <sub>22</sub> H <sub>21</sub> N <sub>3</sub> O <sub>8</sub> S	487.98	74	165-167	yellow	0.76
8		C <sub>21</sub> H <sub>19</sub> N <sub>3</sub> O <sub>7</sub> S	457.40	75	135-137	Dark yellow	0.90
9		C <sub>21</sub> H <sub>18</sub> N <sub>3</sub> O <sub>6</sub> SBr	520.28	73	154 - 156	Orange	0.82
10		C <sub>21</sub> H <sub>18</sub> N <sub>3</sub> O <sub>6</sub> SCl	475.78	74	140-142	Yellow	0.94
11		C <sub>36</sub> H <sub>32</sub> N <sub>6</sub> O <sub>12</sub> S <sub>2</sub>	704.56	73	210-212	Orange	0.77

## RESULTS AND DISCUSSION

The Schiff's bases compounds of sulfamethoxazole (1-6) were synthesized in good percentage from the reaction of Sulfamethoxazole with different aromatic aldehydes in absolute ethanol as a solvent. These compounds have been synthesized according to the steps described in (Scheme 1).

The physical properties described in (Table1) and spectral methods, such as FT-IR and some of them by <sup>1</sup>H-NMR, verified the structures of (1-6) compounds. Spectra of compounds (1-6) displayed characteristic absorption bands at 1614-1627cm<sup>-1</sup>, 2976-2993cm<sup>-1</sup>, 2839-2891cm<sup>-1</sup>, 3066-3086cm<sup>-1</sup> and 1465-1593cm<sup>-1</sup> due to stretching vibrations for (C=N)-asymmetrical, (C-H) aliphatic, symmetrical (C-H) aliphatic, (C-H) aromatic and (C=C) aromatic. These and other bands as shown in (Figures 2,3 and 4).

At 1735-1774cm<sup>-1</sup>, 2854-2997cm<sup>-1</sup> and 1265-1267cm<sup>-1</sup>, 1693-1706cm<sup>-1</sup> and 1157-1161cm<sup>-1</sup> to stretch vibrations (C=O) lactone (C=O), lactame, (C-H) aliphatic, (C-N) and (C-O-C) band, the FT-IR spectra of oxazepin compounds (7-11) displayed characteristic absorption bands. These and other bands are shown in (Table 4), as shown in (Figure 5).



The <sup>1</sup>H-NMR spectra of compounds (1, 3 and 6) showed the signal at 2.24, 2.25 and 2.31 ppm due to proton of (CH<sub>3</sub>) group and the singlet signal at 6.11, 6.05 and 6.08 ppm due to proton of isoxazol ring, the multiplet signals at δ[(7.30-7.83), (7.33-7.85), (7.44-7.47)] ppm, the singlet signal appeared at δ (8.40, 8.58, 8.73) ppm which could be assigned to proton of (N=CH) group that could be assigned to protons of benzene rings, and the singlet signal at δ (10.92, 11.42, 10.91) ppm. suggested the attribution of the proton of (NH) group of sulfonamides shown in (Figures 6, 7 and 8).

The <sup>1</sup>H-NMR spectra of compounds (7 and 10) showed the signal at 2.26 and 2.29 ppm due to proton of (CH<sub>3</sub>) group, and signal at 2.06-2.79 ppm due to the protons of oxazepine ring (CH<sub>2</sub>), while the other signals are listed in as shown in (Figures 9 and 10).

The compound 1 mass spectrum (Figure 11), shows the molecular ion at m/z = 387.4, and the compound 3 mass spectrum, (Figure 12), shows the molecular ion at m/z = 420.28.

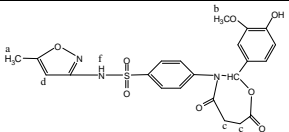
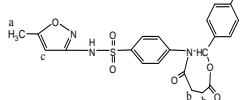
**Table (3):** The FT-IR distinguishing bands of Derivatives (1-6).

Deriv. No.	Molecular Formula	v (C-H) Ar.	v (C-H) Aliph.	v (C=N)	v (C=C) Ar.	another bands
1	C <sub>18</sub> H <sub>17</sub> N <sub>3</sub> O <sub>5</sub> S	3084	2993, 2891	1618	1467-1593	<b>v (C-O-C)</b> (1265, 1029) <i>P-OH</i> (3588)
2	C <sub>17</sub> H <sub>15</sub> N <sub>3</sub> O <sub>4</sub> S	3066	2981, 2854	1618	1465-1593	<i>P-OH</i> (3589)
3	C <sub>17</sub> H <sub>14</sub> N <sub>3</sub> O <sub>3</sub> SBr	3086	2987, 2848	1627	1473-1579	<i>P-Br</i> 684
4	C <sub>18</sub> H <sub>14</sub> N <sub>4</sub> O <sub>5</sub> S	3086	2976, 2875	1616	1465-1593	<i>P-NO<sub>2</sub></i> (1533, 1342)
5	C <sub>17</sub> H <sub>14</sub> N <sub>3</sub> O <sub>3</sub> SCl	3088	2991, 2839	1616	1467-1593	<i>P-Cl</i> 786
6	C <sub>28</sub> H <sub>24</sub> N <sub>6</sub> O <sub>6</sub> S <sub>2</sub>	3086	2991, 2879	1614	1469-1581	

**Table (4):** The FT-IR distinguishing bands of derivatives (7-11).

Comp.No.	Molecular Formula	v (C-H) aromatic	v (C-H) aliphatic	v (C=O) lactone	v (C=O) lactame	v (C-N)	v (C-O-C)	Other Bands
7	C <sub>22</sub> H <sub>21</sub> N <sub>3</sub> O <sub>8</sub> S	3078	2987, 2893	1745	1701	1161	1267	<i>p-OH</i> 3550
8	C <sub>21</sub> H <sub>19</sub> N <sub>3</sub> O <sub>7</sub> S	3092	2931, 2854	Overlap with C=O lactame	1705	1157	1265	<i>p-OH</i> 3464
9	C <sub>21</sub> H <sub>18</sub> N <sub>3</sub> O <sub>6</sub> SBr	3093	2997, 2897	Overlap with C=O lactame	1705	1161	1265	<i>p-Br</i> 686
10	C <sub>21</sub> H <sub>18</sub> N <sub>3</sub> O <sub>6</sub> SCl	3093	2926, 2854	1735	1693	1157	1265	<i>p-Cl</i> 792
11	C <sub>36</sub> H <sub>32</sub> N <sub>6</sub> O <sub>12</sub> S <sub>2</sub>	3095	2922	1774	1706	1161	1265	-----

**Table 5 :** <sup>1</sup>H-NMR Spectral information for selected derivatives.

Deriv.No.	Derivative Structure	<sup>1</sup> H-NMR parameters (ppm) δ-H
7		a(2.26)(s,3H,CH <sub>3</sub> );b(3.81)(s,3H,CH <sub>3</sub> );c(2.06 – 2.39)(t,4H,2CH <sub>2</sub> );d(6.07)(s,1H, isoxazol ring); (6.54 - 8.58)(m,8H, for both two benzene ring);f(11.32)(s,1H,NH)
10		a(2.29)(s,3H,CH <sub>3</sub> );b(2.54-2.79)(t,4H,2CH <sub>2</sub> );c(6.08)(s,1H, isoxazol ring);7.40(s,1H, CH of oxazepin ring);7.44-7.96(m,8H, for both two benzene ring);10.95(s,1H,NH)

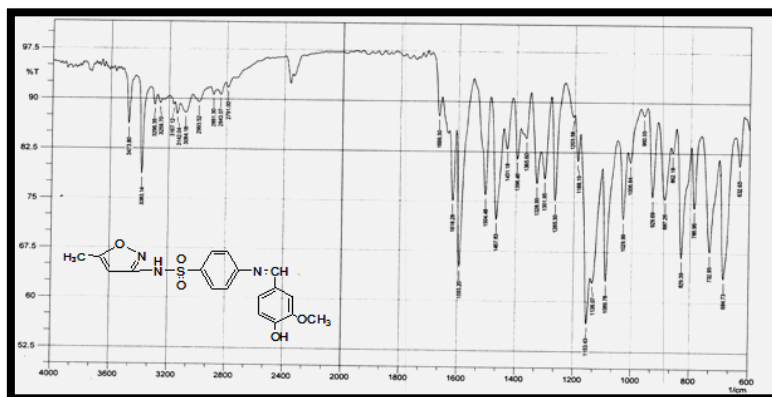


Figure (2): FT-IR Spectrum of compound 1 .

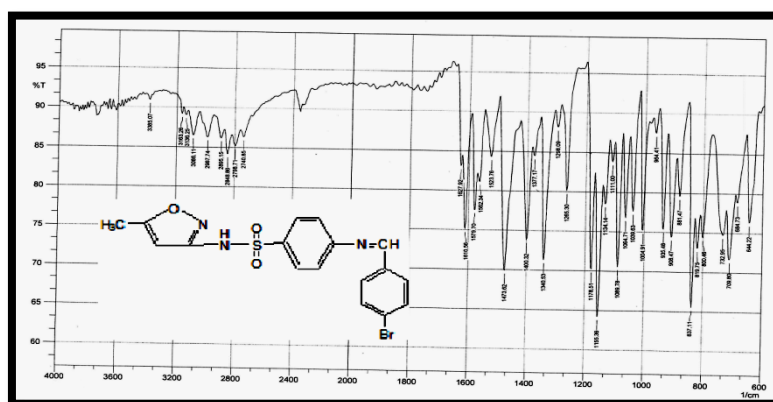


Figure (3): FT-IR Spectrum of derivative 3 .

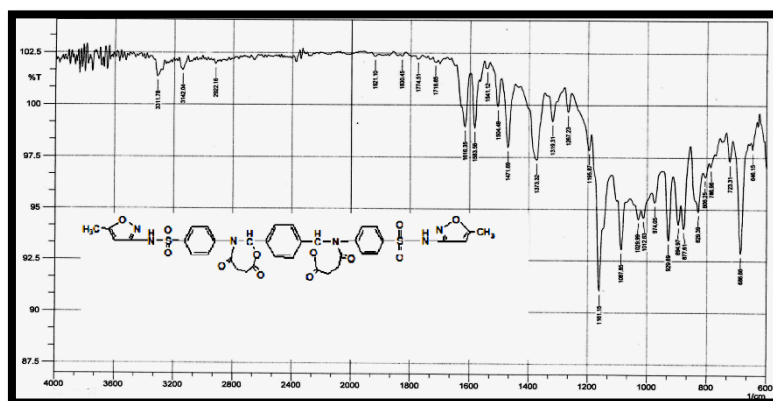


Figure (4): FT-IR Spectrum of derivative 6 .



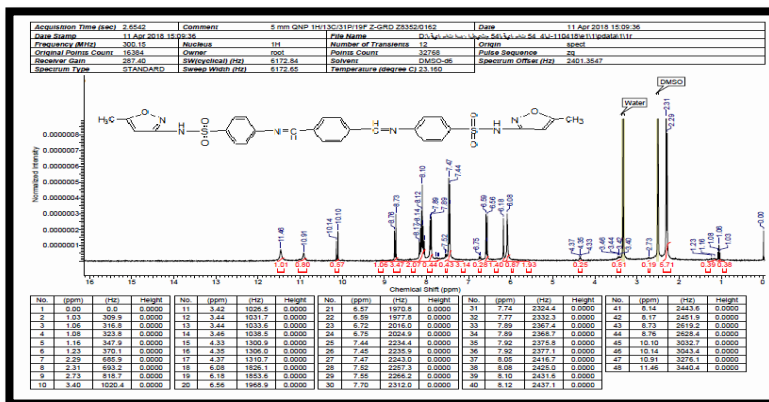


Figure (8): <sup>1</sup>H-NMR chart for derivative 6.

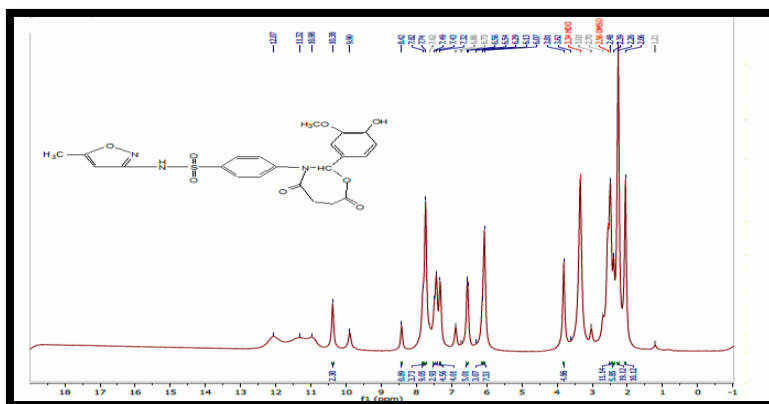


Figure (9): <sup>1</sup>H-NMR chart of derivative 7.

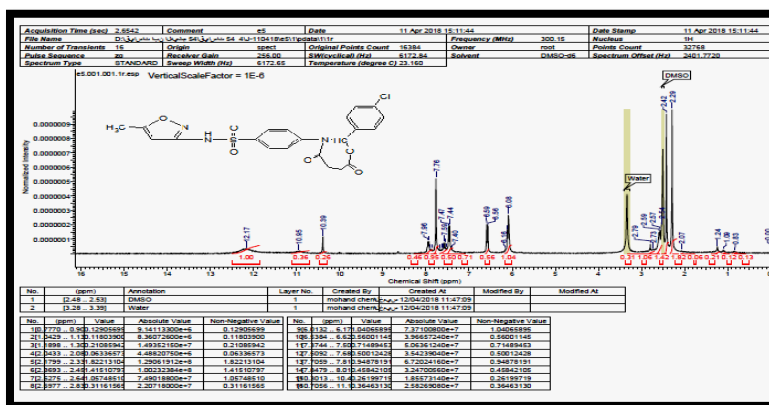


Figure (10): <sup>1</sup>H-NMR chart of derivative 10.

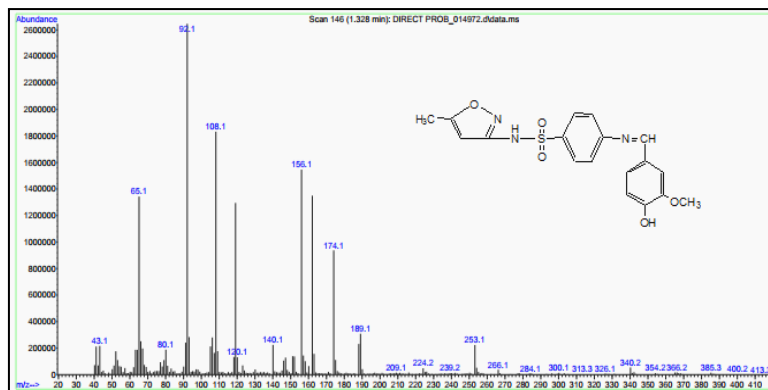


Figure (11): mass chart of derivative 1.

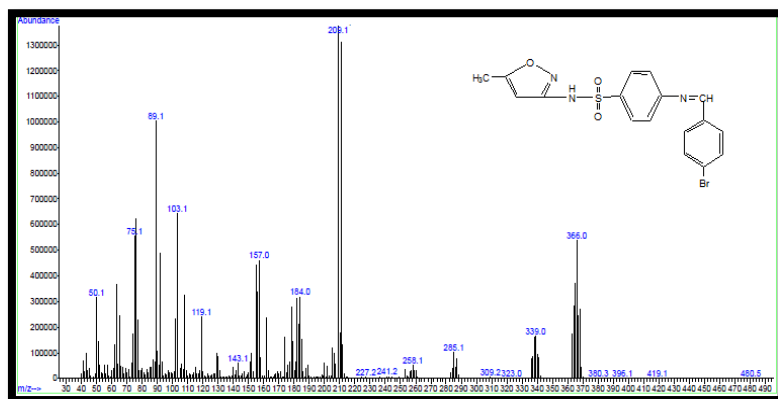


Figure (12): mass chart of derivative 3.

## BIOLOGICAL ACTIVITY

### Anti-bacterial activity

Three forms of pathogenic strains (*S. aureus*, *E. coli* and *P. aeruginosa*) were used to test the antimicrobial activity of the synthesized compounds (2, 4, 7, 10 and 11) using the agar diffusion process. Appropriate spaced separate holes were created by Mueller Hinton agar (6mm in diameter) appropriate spaced separate holes were filled with 0.1 mL concentration of prepared compounds that dissolve in DMSO before spreading the bacteria on agar. These plates were incubated for 24 hr at 37°C, the bacteria growth inhibition zone around the hole observed and measured in millimeter of diameter (Entesar & Enaam, 2017; Saleh & Ali, 2020). Results and clarification are given in (Table 6).

### Anti-fungal activity

The antifungal activity was tested against the fungal species, *C. albicans* at 10mg/mL concentration of some prepared compounds using DMSO as solvent. The results were reported as zone of inhibition compared to standard Nystatin as antifungal drugs. The results obtained showed that some of these derivatives showed commensurable activity like exhibited by (Table, 7).

**Table (6):** Antimicrobial Activities of several prepared derivatives.

Deriv. No.	Sample No. (In image)	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>	<i>Pseudomonas aeruginosa</i>
2	9	+++	++	++
4	8	+++	++	++
7	3	+++	++	++
10	10	+++	++	---
11	15	+++	++	---

(++) = 18- 24mm, (+++) = 28-33mm.

**Table (7):** Anti-fungicide activity for the prepared derivatives(6,7,and8) .

Deriv.No	<i>Candida albicans</i>
6	S
7	R
8	S
Nystatine	S

S: Sensitive,R: Resistance.

## CONCLUSION

The results indicate that the synthesized compounds (2, 4,7,10 and 11) have a microbial activity against the tested organisms up to 3.2mg/ disk.Thesederivatives showed higheffect against *S. aureus*and moderately activity against*E.coli*and *P.aeruginosa*.the fungal species, *Candida albicans* showed higher sensitivity toward the compounds 6 and 8 more than compound 7 .

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## EXTRACTION AND CHARACTERATION OF MUCILAGE EXTRACTED FROM MUSTARD SEEDS

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### ABSTRACT

The mucilage was isolated from mustard seeds and identification by some different methods like, thermo gravimetric, FTIR, X-ray powdered, proton NMR, FTIR spectra of the three gums contain different functional group in the gums, major peaks bands noticed were belong to OH (3410.15 – 3010.88) group from hydroxyl group, CH aliphatic (2925-2343.51), C-O (1072.42-1060.85) group and C=O 1743.65, Thermochemical parameters of mucilage was evaluated and compared with the standard gums, Results indicated the mucilage was decomposed in 392°C and mass loss 55%, The X ray process found the mucilage had single not sharp peak at 19.9265° in highest 53.35 cts, Also the standard gums indicted not sharp peaks at 20.94°, 19.04° in highest 47.78, 52.84 cts separately, mucilage examination using nuclear magnetic resonance revealed the presence of glucose, rhamnose, galacturonic acid compared with two standard gums contain similar polysaccharide. It was concluded The results that had that mucilage had good advantages, which might be used in various food industries showed that mustard gum is beneficial in food manufacturing.

**Keywords:** Mustard seeds gum, characterization, thermogravimetric analysis, X-ray diffraction, nuclear magnetic resonance.





## استخلاص وتشخيص الهلام المستخلص من بذور الخردل

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البحث مستل من أطروحة دكتوراه للباحث الأول

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## الخلاصة

فصل الهلام من الخردل وتم تشخيصه باستعمال بضعة تقنيات مختلفة مثل التحليل الحراري المسعري والتحليل الحراري الكتلي، الأشعة تحت الحمراء وحيود الأشعة السينية والرنين النووي المغناطيسي، طيف الأشعة تحت الحمراء للصبوغ الثلاثة يتضمن مجاميع وظيفية مختلفة في هذه الصمغ، الحزم الرئيسية تشير الى مجموعة الهيدروكسيل (3410.15-3010.88)، المجموعة الأليفاتية (2925-2343.51) ومجموعة الكربونيل (1072.42-1060.85) ومجموعة الكربوكسيل 1743.65، قدرت الخصائص الحرارية للهلام وقورنت مع الصمغ القياسية، اشارت النتائج ان الهلام يتفكك عند درجة حرارة 392م مع فقدان بالوزن مقداره 55%، بينت نتائج حيود الأشعة السينية ان هلام الخردل يمتلك قمة ضعيفة عند الزوايا 19.9265 ارتفاعها 53.35 درجة/ثانية، بينما تمتلك الصمغ القياسية قمتين ضعيفتين عند الزوايا 19.04 و 20.94 ارتفاعها 47.78 و 52.84 درجة/ثانية على التوالي، اظهر تحليل الرنين النووي المغناطيسي ان الهلام يحتوي على كلوكوز، ارامينوز وحامض كالاكتورونك بينما يحتوي الصمغ العربي والاكاسيا على نفس السكريات، بينت النتائج ان الهلام يمتلك مميزات تجعله مفيد في صناعة الاغذية.

الكلمات المفتاحية: صمغ بذور الخردل، تشخيص، تحاليل حرارية، حيود الأشعة السينية، الرنين النووي المغناطيسي.

## INTRODUCTION

Gum are considered to be pathological products by formed injury to the plant or due unfavorable condition such as dehydration or distraction of cell wall, While mucilage's are normal products of metabolism (physiological materials) form without the cell (Reddy & Manjunath, 2013; Bhosaleet al., 2014). Gums have many uses in the food manufacturing as stabilizers, emulsifier, gelling material by improving the consistency of these products, such as delaying the recrystallization of starch during storage , increasing water retention, so they are used in low calorie foods (Mahmood et al., 2015; Hemedat & Mohamed, 2010; Amehet al., 2015). Many applications of gums are used in manufacture of ice cream, meat products, sweets, drinks, canned products, backed products, dairy products and sauces (Edwards, 2007; Decade et al., 2012; Noolrailaet al., 2015). Mustard *Sinapis alba* L. is a spring crop growing in cold and moderate areas, belong to the Crusadeas family which includes the cauliflower and broccoli, one of the early crops known as human cable (Lorenzo, 2008). Gum contain a mixture of polysaccharides that are mainly composed of glucose and polysaccharides acid such as galacturonic acid, glucuronic acid, galactose and rhamnose linked to glucose units by  $\beta$ , 1-4 glycosidic bonds in addition to the association of ether groups. The resulting gum is a viscous solution with a weak gel, and it was used in many meat products, as flavor agent, emulsifier and in cosmetics (Izydorczyk et al., 2005; Repin, 2016; Kay, 2016). The search aimed to isolated mucilage from mustard seeds and evaluated by different methods and its effect on its characterization.

## MATERIALS AND METHODS

Mustard seeds were brought from the local market in Basrah city. It was crushed using an electrical mill. Two standard gum were got from the chemical BDH company, England. The chemical and reagents used were laboratory grade.

### Extraction of Mucilage

Mustard mucilage was extracted according to the method by Nazni&vigneswar (2014). A 100gm of the mustard powder was added to distilled water in a ratio (1:10 w/v), The mixture was mixed using shaking for 4h at 40°C. The mucilage was filtered through muslin to eliminate the insoluble solids. The extract was precipitated by using ethanol (99%). Mucilage was dried in oven at temperature 40-45°C and stored in airtight bottle.

### Characterization

#### FTIR Analysis

The FT-IR Spectrum of The gums was Recorded in chemistry department, college of education for pure science, university of Basrah. FTIR spectrometer (Shimadzu. Japan) using potassium bromide disc prepared from powdered gums mixed with dry KBr.

#### Thermal Analysis

Thermo stability of three gums were carried on and recorded in AmirKabiruniversity, Mahshahr. The samples was put in a platinum pan, the sample were scanned from 30to 700°C and sealed at heating rate 10°C 1 min, nitrogen was used with the flow ratio of nitrogen at a rate 80 ml/ min.

#### XRD measurement.

XRD measurement of the samplerecorded in department of physical, college of education for pure science, university of Basrah, by using XRD System, X per T-pro pw 3050H-NMR analysis, H-NMR spectra of the gums was analyzed in Iran by using two devices, namely a Bruker AVANCE 400 MHZ spectrometer and a Bruker AVANCE 500 MHZ spectrometer.

## RESULTS AND DISCUSSION

### FTIR Analysis

FTIR spectrum of the different gums are shown in (Table 1) and (Figures 1, 2 and 3) characteristic band at 3010.88 and 3410.15 cm<sup>-1</sup> referred to the OH bending of water, The boardpeak round 2343.51 and 2925 cm<sup>-1</sup> was assigned to alkane CH group, The band at in the region between 1616.35 and 1735.93, 1429.25-1668.43, 1743.65 cm<sup>-1</sup> belong to C=O in the FTIR spectra of mustard mucilage, Arabic gum and acacia.

**Table (1):** FTIR characteristics of three gums.

Wave number cm <sup>-1</sup>			Functional groups
Mustard mucilage	Arab gum	Acacia gum	
3410.15 – 3010.88	3412.08	3406.26	OH
1072.42-1060.85	1033.85-1014.56	1064.71-1029.99	C-O
1743.65	1616.35 –1735.93	1668.43-1429.25	COO
2925 - 2343.51		2910-58	CH
677.01- 155.36	970.19 - 493.27	808.17-709.80	C-H out of plane

The characteristic band at 1072.42-1060.85 cm<sup>-1</sup> attributed to the carbonyl, the finger print region of mustard spectra contain of many board bands in the region 677.01-493.27cm<sup>-1</sup> which the finger print region of standard gums spectra consist of 2-5 peaks at range 970.19-493.27cm<sup>-1</sup> for Arabic gum, 808.17-709.80cm<sup>-1</sup> for acacia gum, the FTIR spectra of samples contain similar functional groups, the mustard mucilage was closes to the Arabic gum. This results were agreement by Daoubet *al.*(2016) who illustrated functional groups of acacia gum, the band between 3290-3305 was assigned to hydroxyl group, band at 2926 belong to arabinose and galactose, peak at 1411-1563 was assigned to COO indicate the presence of

glucuronic acid and also were agreed with **Bilal et al.(2015)** who indicate the refined Arabic gum contain functional groups like  $3634.01\text{cm}^{-1}$  was assigned to the OH while the peaks at  $2851.35, 2914.16$  and  $2671.98\text{cm}^{-1}$  attributed to the alkane CH group, the band in  $1663.66\text{cm}^{-1}$  was belong to C=O indicated to glucuronic acid.

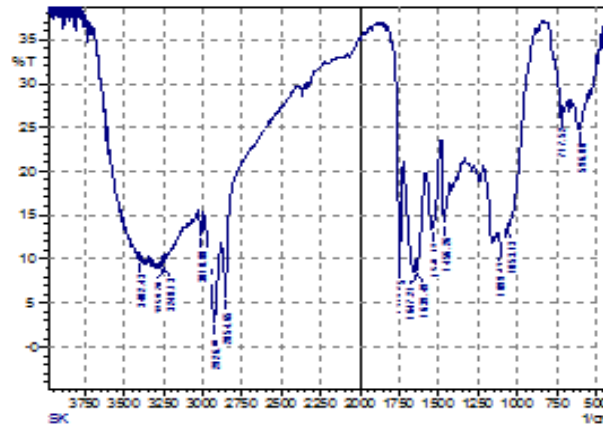


Figure (1):FTIR spectra of mustard seed gum.

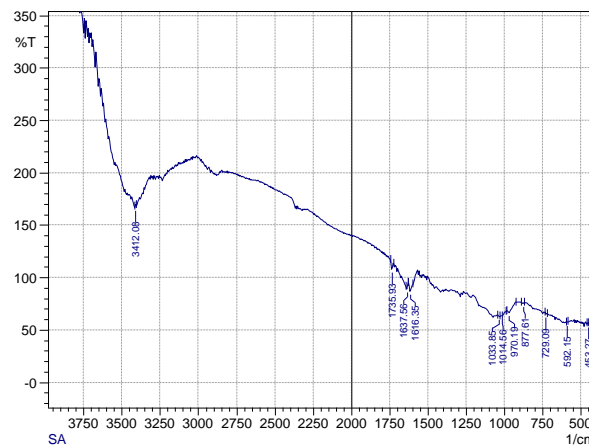


Figure (2): FTIR spectra of Arabic gum.

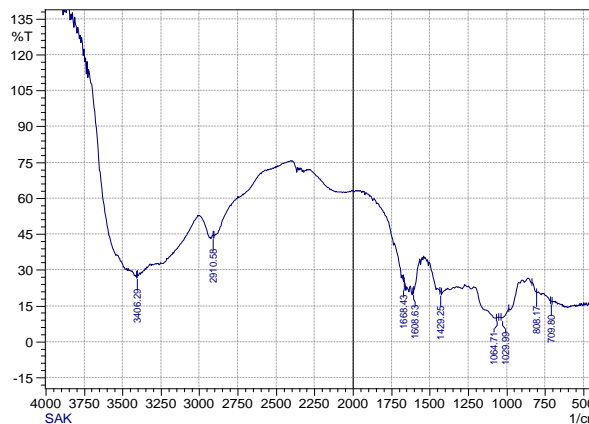
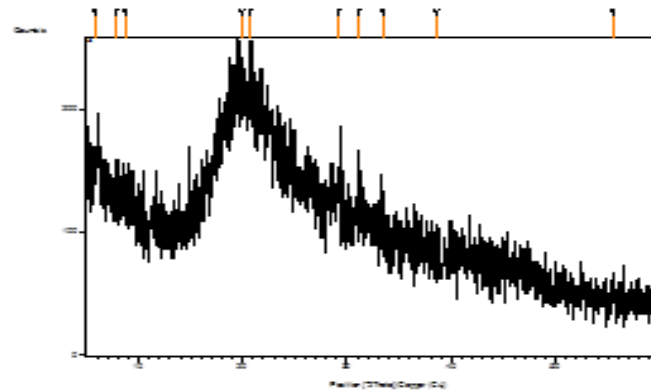


Figure (3):FTIR spectra of acacia gum.

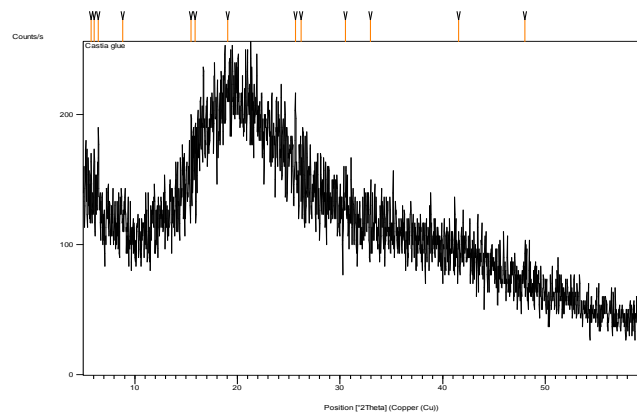
**XRD mearment**

XRD analysis of the three gums were presented in(Figures 4, 5 and 6) the XRD analysis of mustard mucilage showed a sharp peak was noticed at  $19.9265^\circ$  in highest 53.35 cts .This indicates that mustard mucilage was a partially crystalline.

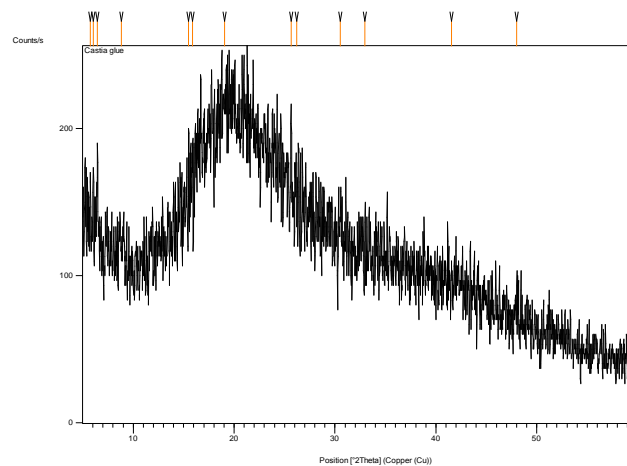


**Figure (4):** X-ray spectra of mustard mucilage.

Compared the XRD spectra of standard gums contain two peaks were not sharp in the highest 47.78, 52.84 cts at 20.94° and 19.04°, and this indicates to the amorphous nature.



**Figure (5):**X-ray of Arabic gum.



**Figure (6):** X-ray of Acacia gum

The result were in agreement with the finding by **Zaharuddin et al.(2014)**that okra gum had one weak peak, also results were agree with **Nqwuluka et al. (2014)** who showed the usual gums are amorphous or semicrystalline. X-ray were used to examine the degree of crystalline of materials. The absence of sharp peaks in the spectrum of ray indicated that gums are amorphous. So most of the natural gums were amorphous and had partial crystalline.

### DSC analysis

DSC Thermo gram of mustard mucilage were presented in (Figure 7), the mucilage had a sharp peak was presented at 610°C, compared with Arabic gum and acacia gum which had was similar were the peak at 300°C, that were presented in (Figures 8 and 9), loss of water led to appear peak.

### TGA analysis

Thermogram of three gums were presented in (Figures 7, 8 and 9) of gums, the first stage of decomposition of mustard mucilage, taking place between 30 to 700°C with a maximum decomposition occurs at 392°C with weight loss 53%.

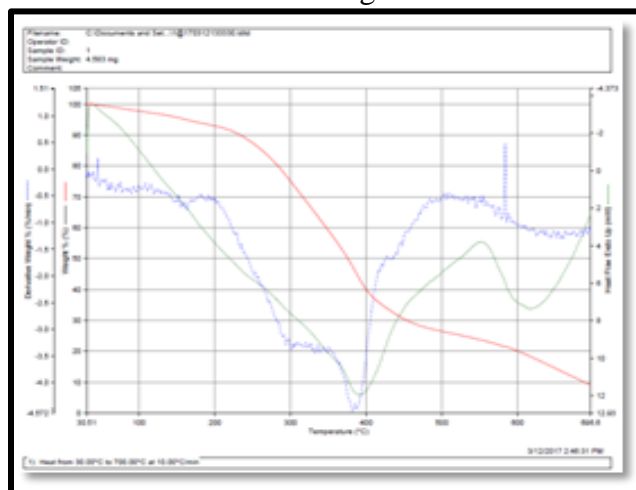


Figure (7): The rmogram of mustard mucilage.

The degradation process of Arabic gum and acacia gum occurs in the same temperature at 300°C with mass loss percentage of 53 and 45%.

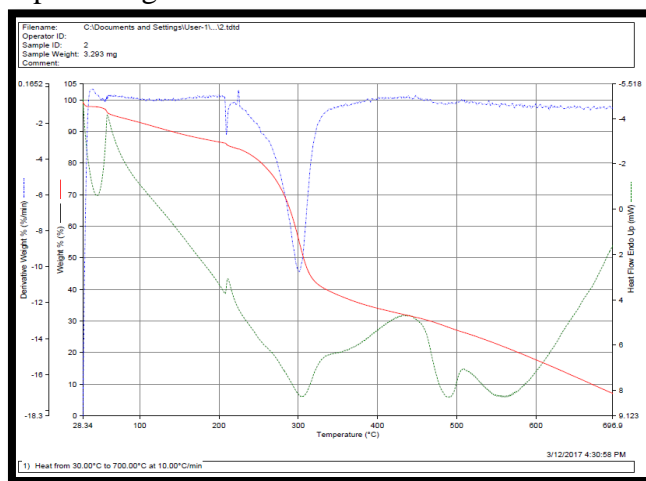
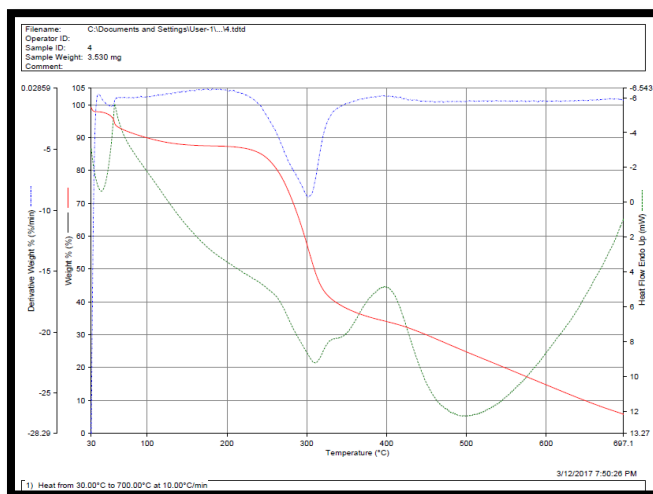


Figure (8): The rmogram of Arabic gum.



**Figure (9):**The rmogram of acacia gum.

The result showed that mustard mucilage had high thermal stability, which helps in its uses in the food industry.

**H-NMR analysis**

The result of H NMR spectrum of mustard mucilage was shown in (Table 2 and Figure10), the major indicators at chemical shift between 1.971-1.76 ppm assigned to CH<sub>3</sub> of rhamnose, indicators around at 3.98-3.087ppm in mustard mucilage assigned to CH<sub>2</sub> of galactose, The indicators arise at 4.187 ppm that refers to COO group.

**Table (2):** Individualities of H-NMR of three gums.

	Chemical shift( ppm)		Functional group	
	Mustard gum	Arabic gum		Acacia gum
	5.309 – 5.10	5.4	5.4	OH
	1.971 - 1.76	1..084-1.07.6	1.19	CH <sub>3</sub>
	2.761- 2.493			CH <sub>2</sub> O
	4.187	4.03		C=O
	3.98 – 3.087			CH <sub>2</sub>
				CH

The results were agreed with **Daoub et al. (2016)** who indicated the H-NMR spectrum of acacia gum contain CH<sub>3</sub>of rhamnose and C=O group of galactronic acid.

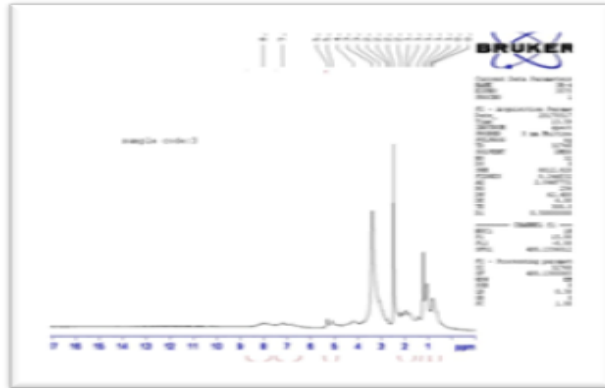


Figure (10): Nuclear magnetic resonance of mustard mucilage.

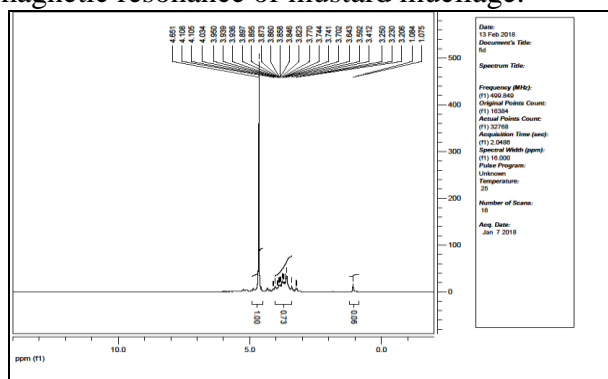


Figure (11): Nuclear magnetic resonance of Arabic gum.

The H NMR spectrum of standard gum in (Table 2 and Figures 11 and 12) indicated the presence of signals at chemical shift 1.19 ppm belong to CH<sub>3</sub> of rhamnose, indicators round at 3.73-3.85 ppm belong to CH<sub>2</sub>OH of galactose.

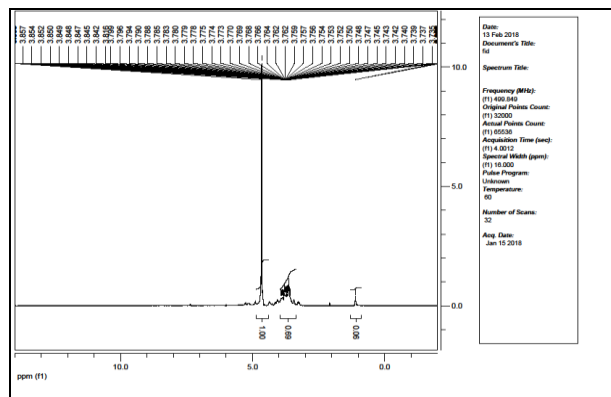


Figure (12): Nuclear magnetic resonance of acacia gum.

The H-NMR spectrum of the studied gums showed the presence of main sugars spectrum of acacia gum contain CH<sub>3</sub> of rhamnose and C=O group.

**CONCLUSION**

FTIR analysis has been used for the characterization of gums contain different group such O-H, C-H, C=O, COO that are presence in the structure of gums, XRD result indicate that mustard mucilage was showed has weak peak which show a semicrystalline compared Arabic gum and acacia gum were low crystallinity. Thermochemical parameters of mustard

mucilage was examined by TGA, DSC. Results indicated that mustard mucilage had good thermal stability. H-NMR analysis of studied gums contain monosaccharide. Major monosaccharide of mustard mucilage were glucose, rhamnose, galactose, glucuronic acid and galactronic acid, while the standard gums had the similar monosaccharide like galactose, glucuronic acid and arabinose sugar, So it was concluded that mustard gum could be used in many different food industries.

## ACKNOWLEDGEMENTS

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## SPECTROPHOTOMETRIC DETERMINATION OF PURE SULFAMETHOXAZOLE IN PHARMACEUTICAL PREPARATIONS BY OXIDATIVE COUPLING REACTION

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### ABSTRACT

A new, simple, rapid and sensitive spectrophotometric method for the determination of sulfamethoxazole in both pure form and pharmaceutical preparations has been reported. The adapted technique based on utilization 4-aminobenzene sulfonic acid as a new modern chromogenic through an oxidative coupling reaction with sulfamethoxazole and potassium iodate in basic media to form orange soluble dye product with absorption maxima at 490 nm. Subject to Beer's law in the range 2–32 μg mL<sup>-1</sup>. The values of molar absorption coefficient (ε) and correlation coefficient were found to be 9.118 × 10<sup>3</sup> and 0.9999 respectively whereas the Sandels index was 0.02778 μg.cm<sup>-2</sup>.

**Keywords:** Spectrophotometric, pharmaceutical, sulfamethoxazole, oxidative coupling.



## تقدير الطيفي للسلفاميثوكسازول النقي في المستحضرات الصيدلانية عن طريق تفاعل الاقتران التأكسدي

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## الخلاصة

يتضمن هذا الجزء تطوير طريقة طيفية سريعة وحساسة لتقدير كميات ضئيلة من السلفاميثوكسازول (SMZ) في محلول مائي قاعدي، تعتمد هذه الطريقة على الاقتران التأكسدي لل SMZ مع الكاشف 4-امينو بنزين حامض سلفونيك بوجود العامل المؤكسد يودات البوتاسيوم لتكوين ناتج برتقالي اللون ذائب بالماء مستقر، يعطي اعلى امتصاص عند الطول الموجي 490 نانومتر ويخضع لقانون بير بحدود (2-32) مايكروغرام/ مل، وبلغت الامتصاصية المولارية  $9.118 \times 10^3$  لتر/ مول . سم ، ودلالة ساندل 0.02778 مايكروغرام/ سم<sup>2</sup> وحد الكشف 0.063 مايكروغرام/ مل، والحد الكمي 0.2173 مايكروغرام/ مل، الانحراف القياسي النسبي بين (0.5923-1.4662)% ومعدل الاسترجاعية 100.24%، معامل التقدير 0.9999 وطبقت هذه الطريقة المقترحة بنجاح لتقدير (SMZ) في مستحضراته الدوائية. الكلمات المفتاحية: المطيافية، الأدوية، السلفاميثوكسازول، اقتران التأكسدي.

## INTRODUCTION

Sulfamethoxazole (SMZ) is an isoxazole (1,2-oxazole) compound having a methyl substituent at the 5-position and a 4-aminobenzenesulfonamido group at the 3-position. It has a role as an antibacterial agent. IUPAC Name is [4-amino-N-(5-methyl-1,2-oxazol-3-yl)benzenesulfonamide] an antibiotic usually used in the cases of urinary tract, bronchitis and prostatitis (Ma et al.,2007; Kazanjian et al., 2008; Amaliet al.,2019). It has been recorded to be effective against both gram negative and gram positive bacteria, such as *Listeria momocytogene* and *E. Coli*. (Kazanjian et al., 2008). After being introduced in the United States in 1960 (Amaliet al.,2019),it is now mostly used in combination with trimethoprim, abbreviated as (SMZ-TMP) (Brunton et al., 2011). This combination (formulation) is included registered in WHO model list of essential medicines as a prime choice treatment for urinary tract infection. is also known as: sulfamethalazole, sulfisomezole (Roth et al., 2018) and Sulfamethazole. (Figure 1) shows the structural formula of sulfmethaxazole.

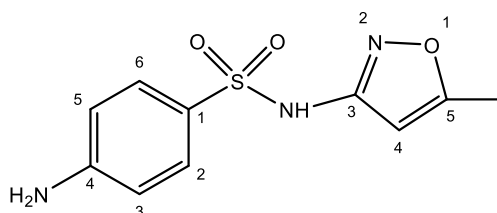


Figure (1):The structural formula ofSulfamethazole.

Various analytical methods for determining SMZ have been recorded, including differential calorimetry scanning (Agafonova et al.,2013), spectrofluorimetry (Chen et al.,1999), potentiometric titration (Fritz et al., 1952), sequential injection chemiluminescence (Soto et al., 2005; Aintisaret al., 2019), capillary zone electrophoresis (Lietal., 2008), reverse phase high performance liquid chromatography (Soto et al., 2005), micellarelectrokinetic chromatography (Berzas Nevado et al.,2005), spectrophotometry

(Hamzahet *et al.*, 2014) and nuclear magnetic resonance (Salem *et al.*, 2012). The system used by the Bratton and Marshall was considered to be the most popular colorimetric method used for sulfa drugs (Zhou *et al.*, 2009).

## MATERIALS AND METHODS

### Instruments

Absorbance measurements were performed using T92 + Spectrophotometer wavelength at the range (190-900) nm. (Beijing, China). Measurement was performed by a spectrophotometer in an analytical chemistry laboratory. (Table 1) shows the chemicals used.

**Table (1):** Chemicals.

Chemicals	M.wt (g/mol)	Chemical Formula	Assay (%)
Sulfamethoxazole (SMZ)	253.28	C <sub>10</sub> H <sub>11</sub> N <sub>3</sub> O <sub>3</sub> S	99
Iodate Potassium	214	KIO <sub>3</sub>	99
4-aminobenzenesulfonic acid	173	C <sub>6</sub> H <sub>7</sub> NO <sub>3</sub> S	98
Sodium Hydroxide	40	NaOH	98

### Reagents

- All chemicals used in this study were of analytical grades.
- The stock solution for sulfamethoxazole (100 µg/mL) was prepared by dissolving 0.10g in 5 mL of ethyl alcohol sulfamethoxazole and completing the volume in 100 mL of volumetric flask with distilled water.
- Sodium hydroxide solution (0.2 M) was prepared by dissolving 0.8 g of NaOH in a specified amount of distilled water in a volumetric flask of 100 mL and then the volume was completed to the mark.
- Aminobenzenesulfonic acid solution ( $2 \times 10^{-2}$  M) was prepared by dissolving 0.346 g of 4-aminobenzenesulfonic acid in distilled water and then progressing to the point of mark with the same solvent in a 100 mL volumetric flask.
- Oxidizing agent (KIO<sub>3</sub>) ( $2 \times 10^{-2}$  M) solution was prepared by dissolving 0.428 g of potassium iodate powder in distilled water filled in a 100 mL volumetric flask to the point of the mark.

### Pharmaceutical preparations of sulfamethoxazole

Pharmaceutical preparations from industrial sources were collected as follows:

- Trimoks Pills of (SDI, Samarra-Iraq): 400 mg sulfamethoxazole and 80 mg trimethoprim for each Pills.
- Sulprim syrup (Jerusalem Pharmacy products Co.Ltd-palestine): 200 mg sulfamethoxazole and 40 mg trimethoprim for each 1 mL of syrup (100 mL).

It was prepared by taking 1 mL of the Sulprim and diluting it with a small amount of distilled water and filtering and the complement filtrate with distilled water to the mark to obtain a concentration of 100 µg / mL.

The principle of the method is based on the coupling of the reagent (4-aminobenzene sulfonic acid) with (Sulfamethoxazole) in the presence of potassium iodate as oxidizing agent in basic medium.

### Preliminary study

We put (1 mL,  $2 \times 10^{-2}$  M) of reagent 4-aminobenzenesulfonic acid and add an oxidizing agent potassium iodate (1 mL,  $2 \times 10^{-2}$  M) to 2 mL Sulfamethoxazole (100 µg/ mL) an orange product was formed. The intensity of the color increased in the presence of 1 mL (0.2 M) of NaOH solution. Absorbance measurements were performed after diluting the solution with

distilled water in a 25 mL volumetric flask. The maximum absorbance value was measured against an blank solution at a wavelength of 490 nm.

## RESULTS AND DISCUSSION

### Study of the optimal reaction conditions

Experiments were performed using 2 mL of Sulfamethoxazole 100 µg/ mL in a final volume of 25 mL and the absorbance values of the solutions was measured at 490 nm wavelength versus a blank solution.

### Effect of the best coupling reagent

Series of experiments were held (performed) by mixing 1mL of each of the reagent solutions shown below in (Table 2) with 1 mL of ( $2 \times 10^{-2}$ M) of potassium iodate as oxidizing agent solution with 2 mL of (100µg/ mL) sulfamethoxazole solution in the presence of 1 mL NaOH solution 0.2 M and the absorbance values were recorded at 490 nm wavelength.

**Table (2):** Effect of the best coupling reagent.

Reagent $2 \times 10^{-2}$ M	Variable	Absorbance
<i>P</i> -nitro aniline	SB	0.15
	BW	0.07
<i>O</i> -aminophenol	SB	0.17
	BW	0.08
<i>P</i> -aminophenol	SB	0.18
	BW	0.097
Resorcinol	SB	0.163
	BW	0.09
4-aminobenzen sulfonic acid	SB	0.520
	BW	0.091

S= Sample , B= Blank , W= Water

### Oxidizing agent optimization:

1 mL,  $2 \times 10^{-2}$  M of series of oxidizing agents were added to 1 mL, 4-amino benzene sulfonic reagent solution, 2 mL of (100µg/ mL) sulfamethoxazole solution and 1 mL, 0.2 M NaOH solution in 25 mL volume flask followed by absorption measurements. For each sample, the blank solution was fixed at wavelength (800-400) nm. The best oxidizing agent was noted to be  $KIO_3$  for giving the maximum absorption value of the colored product at the wavelength 490 nm. The results are shown in (Table 3).

**Table (3):** Test from best oxidizing agent.

Oxidizing agent $2 \times 10^{-2}$ M	Absorbance		$\lambda$ max(nm)
	Blank	Sample	
Potassium Iodate	0.091	0.524	490
Potassium per Sulphate	0.040	0.290	427
Ammonium per Sulphate	0.042	0.192	466
Ammonium ferric Sulphate	0.039	0.018	518

### Effect of the coupling reagent quantity

Various aliquots of the conjugating reagent (4-aminobenzenesulfonic acid) solutions (0.3-2.1) mL with concentration equal to ( $2 \times 10^{-2}$  M) were mixed with (1 mL,  $2 \times 10^{-2}$ M)  $KIO_3$ , (1mL, 100µ g/ mL) sulfamethoxazole solution and (1 mL, 0.2 M) NaOH solution. (Table 4) shows that the maximum absorption value was obtained using 1 mL of the reagent 4-amino benzenesulfonic acid solution.

**Table (4):** Effect of the coupling reagent quantity used.

mL of Reagent $2 \times 10^{-2}$ M	Absorbance	
	BW	SB
0.3	0.075	0.352
0.6	0.080	0.447
0.8	0.092	0.514
1.0	0.088	0.522
1.3	0.108	0.510
1.5	0.113	0.483
1.8	0.081	0.425
2.1	0.097	0.315

#### Effect the base used for coupling

One mL of different types of bases (strong and weak) has been used; optimum results were recorded in the case of NaOH as shown in the (Table 5).

**Table (5):** Effect of the base used in the coupling.

Base Solution Used 0.2 M	NaOH	KOH	Ca(OH)	NH <sub>4</sub> OH
Absorbance	0.519	0.467	0.376	0.189

#### Effect of the amount of base used

Various quantities of the used base (NaOH) have been added that have been selected to find the optimal amount that gives the highest absorption of the formed product. Maximum absorption was recorded in the case of (1 mL) with pH=11.86. This volume was adopted in the subsequent experiments and all the results are shown in the (Table 6).

**Table (6):** Effect of quantity of the base used.

mL of NaOH 0.2 M	Absorbance		pH
	BW	SB	
0.2	0.108	0.351	11.72
0.4	0.109	0.405	11.77
0.6	0.098	0.471	11.83
0.8	0.103	0.516	11.84
1	0.104	0.525	11.86
1.2	0.102	0.502	11.87
1.4	0.095	0.484	11.89
1.6	0.082	0.425	11.91
1.8	0.071	0.368	11.93
2	0.103	0.216	11.94

#### Sequence of addition

Sulfamethoxazole (D), reagent solution 4-aminobenzen sulfonic (R), potassium iodate solution (O), and sodium hydroxide (B) alkaline solution were added to each other in different sequence keeping the same volume and concentration thereof. Maximum absorption was recorded in the sequence mode of addition (IV) as shown in (Table 7).

**Table (7):** Effect of order sequence of addition.

Order Number	Order of addition	Absorbance	
		BW	SB
I	D+O+B+R	0.041	0.438
II	B+D+R+O	0.020	0.227
III	O+R+B+D	0.052	0.348
IV	R+O+D+B	0.097	0.523

### Effect of temperature

The temperature of mixture and the colored product formed was varied at the range 10-70°C. Maximum and stable absorption of the formed colored product solution was observed at the temperature range 20-30°C. 25°C was chosen to be the optimal temperature for the reaction mixture. The details of the results are shown in the (Table 8).

**Table (8):** Effect of the temperature.

Temperature (°C)	15	20	25	30	35	40	50	60	70
Absorbance	0.42	0.49	0.523	0.51	0.44	0.35	0.28	0.15	0.08

### Stability of the reaction product:

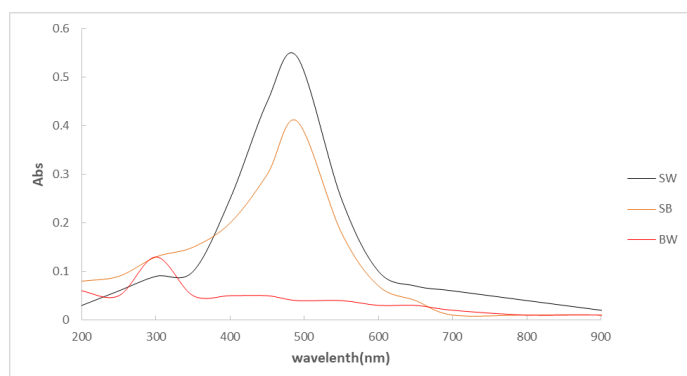
The stability of the reaction product was studied throughout observing the values of absorption of the formed colored solution at duration of time 5-60min using 2 mL, 100µg/mL of the sulfamethoxazole solution and 2 mL of the drug. Results exhibited in (Table 9) shows that 60 min was very sufficient time period to hold the measurements.

**Table No (9):** Effect of time on absorption of the formed product.

µg /mL of SMZ	Absorbance/ min. Standing time									
	5	10	15	20	25	30	45	60	75	
2	0.520	0.521	0.521	0.522	0.521	0.521	0.522	0.519	0.320	

### Ultimate absorption spectrum

The ultimate absorption spectrum shown in (Figure 2) was achieved on adopting the optimum conditions: 25 mL of aqueous solution comprising of (1mL,  $2 \times 10^{-2}$  M) 4-amino benzenesulfonic acid, (2mL, 100 µg/ mL) sulfamethoxazole, (1mL,  $2 \times 10^{-3}$ M) KIO<sub>3</sub>, (1mL, 0.2M) NaOH. Maximum absorption was recorded against blank solution for orange product solution at the wavelength 490 nm. The emergence of a peak of 490 nm indicates electronic transitions  $n \rightarrow \pi^*$ ,  $\pi \rightarrow \pi^*$

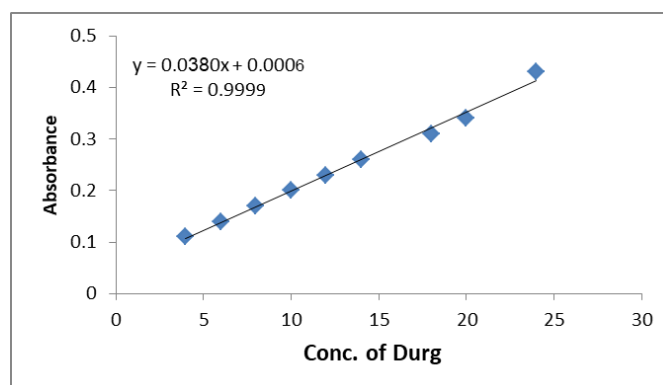


**Figure (2):** Ultimate normal spectrum.

### Approved working method and calibration curve

Subsequent to the determination of the optimum conditions, standard calibration curve was obtained by application of the following procedure of mixing:

Series of aliquots 0.5-8 mL of 100 µg/mL Sulfamethoxazole were added to a number of 25 mL volumetric bottles each containing (1 mL,  $2 \times 10^{-2}$  M) 4-aminobenzenesulfonic reagent, (1 mL,  $2 \times 10^{-2}$  M) of the oxidizing agent potassium iodate, and (1 mL, 0.2 M) of a (NaOH) base solution. The volume of the formed mixture was completed by distilled water to the mark point. Absorption measurements were performed for each solution against the official solution at the wavelength of 490 nm.



**Figure (3):** Calibration curve.

The standard curve obtained which follows Lambert-Beer's law in the limits of concentrations (2-32) µg /mL. The linear regression was utilized to calculate the equation constants that are: Molar absorption coefficient  $\epsilon$ ,  $9.118 \times 10^3$ , correlation coefficient 0.9999, The Sandelis  $0.02778 \mu\text{g}\cdot\text{cm}^{-2}$

### Accuracy and precision

The accuracy of the method represented by relative error (E%) and recovery(Rec%) was calculated to estimate sulfamethoxazole and the compatibility of the method represented by relative standard deviation (RSD%) (measuring three different concentrations) of Sulfamethoxazole 100 µg/ mL, as it appears from the results obtained. (Table 10) it demonstrates that the technique has good accuracy and precision.

**Table (10):** The accuracy and precision of the method \*

Conc. Of SMZ Present (µg/mL)	Conc. of SMZ Measured(µg/mL)	RE (%)	Recovery(%)	Average of Recovery (%)	RSD(%)
9	8.88	-1.33	99.18	100.24	0.5923
16	15.97	-0.187	100.5		0.6615
32	32.54	1.687	101.02		1.4662

\*Each value averages five readings

### Detection Limit

The detection limit was calculated for at the wavelength of 490 nm, by measuring the absorption of the lowest concentration (2 µg/ mL) taken from the calibration curve depicted from (for) seven readings under the same conditions. Results exhibited in (Table 11).

$$D. L = 3 \times S \times \text{Conc.} / \bar{x}$$

$$Q. L = 10 \times S \times \text{Conc.} / \bar{x}$$



**Table (11):** Detection Limit

Conc.(µg/mL)	$\bar{X}$	S	D.L.(µg/mL)	Q.L.(µg/mL)
2	0.122	0.001326	0.063	0.2173

Then

**Conc.:** The lowest concentration of L in the calibration curve.

**$\bar{X}$ :** The absorption rate for a series of measurements of no less than seven values.

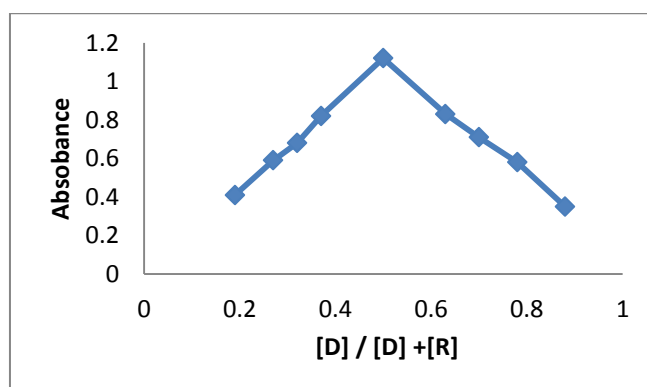
**S:** Standard deviation.

**D.L.:** Detection limit.

**Q.L.:** Quantitative limit.

### The nature of the formed product

To identify the nature of the formed product and the ratio of the drug's attachment to the detector, both continuous changes method (the Jop method) and molar ratio method (Niknia, 2018) have been applied. Different volumes of drug solution ranging from 1-9 mL were placed into series of 25 mL volumetric flasks containing decreasing volumes of the reagent 9-1 mL. The rest of the additives were in the optimal sizes according to the adopted method of work in this study, followed by filling the bottles to the mark with distilled water. According to absorption measurements of the solutions at 490 nm against their blank solutions the following (Figure 4) was depicted for JOP method. 1:1 ratio found to be the optimal mixing ratio.



**Figure (4):** The JOP method.

According to this method mixture comprising of Sulfamethoxazole with the 4-amino benzenesulfonic reagent in the presence of the oxidizing agent potassium iodate, and 1 mL of a NaOH-base solution 0.2M was estimated. To ensure that the interaction ratio between Sulfamethoxazole and the reagent 4-aminobenzen sulfonic is 1:1, the molar ratio method was used and as follows: 2 mL of the drug solution were placed in a series of 25 mL volumetric bottles containing different volumes of reagent solution 0.2-2 mL with the remaining additives at the optimum sizes, and diluted with distilled water to the point of the mark. Absorption measurements of these solutions at the wavelength of 490 nm against the formed solution for each of them were held. Molar ratio was found to be consistent with the method of continuous changes. (Figure 5) shows that the ratio is 1:1.

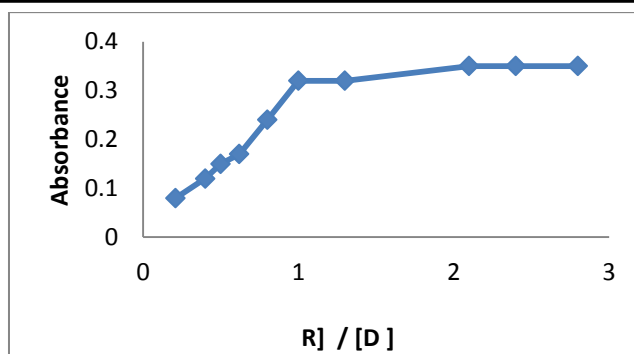


Figure (5): Shows that the molar ratio of Sulfamethoxazole.

The reaction equation is as (Figure 6).

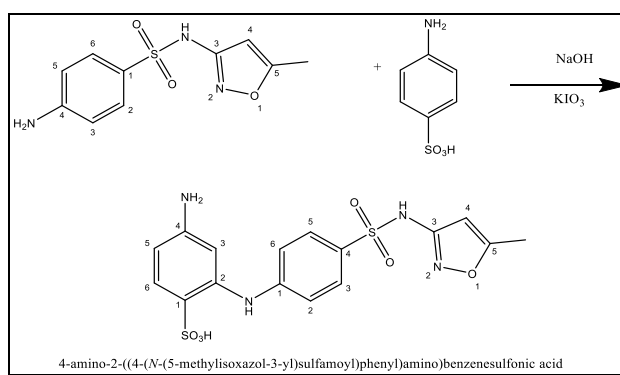


Figure (6): Reaction scheme.

### Applications

This method can be applied to the following pharmaceutical preparations containing: Sulprim syrups (Jerusalem Pharmaceuticals Co.Ltd-palestine):200 mg sulfamethoxazole and 40 mg trimethoprim for each 5 mL of suspension 100 mL.

### The direct method

Three different concentrations were taken from the solution of each product (Suspensions ) whose preparation is indicated in its preparation in the paragraph, namely 10,20, 30µg/mL. The solutions were treated following the same steps in preparing the titration curve and measuring the absorbance for them at the wavelength of 490 nm against the blank solution. An average of five readings was calculated for each, as well as the calculation of retrospective and RSD according to the results shown in (Table 12).

Table (12):The direct method.

Con.of (SMZ) (Syrup)	Con.of (SMZ) Measured	RE(%)	Recovery(%)	Average of Recovery (%)	RSD(%)
10	10.14	1.4	101.4	100.405	0.1163
20	20.01	0.05	100.05		0.3519
30	29.93	-0.233	99.766		0.5685

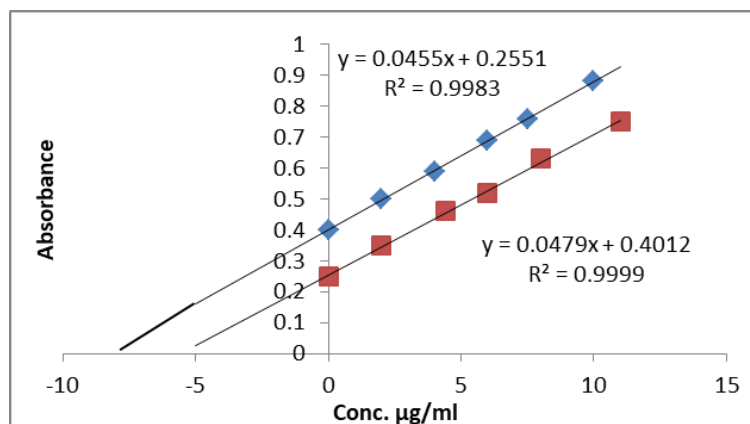
The results exhibited in the above table show that the proposed method was successful in estimating the pharmaceutical preparations that contain them. The value of the recovery rate was 100.405% for syrups.

### Standard additions method

The standard addition method was used to demonstrate the efficiency of the proposed method, as the method includes the addition of constant quantities (1.3, 2.0 mL) of prepared pharmaceutical solutions at a concentration of 100µg/mL in two series of volumetric bottles of 25mL in capacity and adding increasing volume 1, 1.5, 2.5 and 3.5 mL of a standard solution of 100 µg/mL concentration and leaving one of the bottles without addition. The absorbance of the solutions was measured against the blank solution at the 490 wavelength. The results are shown in (Table 13) and (Figure 7).

**Table (13):** Standard additions method.

Pharmaceutical preparation	Amount taken (µg/mL)	Amount measured	Recovery (%)
Sulprim suspensions	5	4.9	98.33
	9.8	9.7	98.86
Pharmaceutical preparation	Amount taken (µg/mL)	Amount measured	Recovery (%)
Sulprim suspensions	5.2	5.6	107.69
	8	8.37	103.75



**Figure (7):** Direct method.

From the results shown in the (Table 13), it is shown that the standard addition method is well in agreement with the direct method, within the range of acceptance of the error, which indicates that it is the satisfactory method.

### Statistical evaluation of the results of the proposed method

A comparison was made between the proposed analytical method and the standard method to find out the accuracy and validity of the analytical application of the proposed method by applying the following two tests. The obtained result was less than the tabular value for F, t. F=4.36, t=2.84 (which is required). While the tabular value for F, t was F=7.25, t=4.776 at confidence limit 95% and for four degrees of freedom. Consequently, these values demonstrate the success of the proposed method as shown in (Table 13).

**Table (13):** Statistical evaluation.

Preparation	Normal value	Recovery (%) $\pm$ RSD	
		Proposed method	Literature method
Sulprim suspensions	200/40mg	100.08 $\pm$ 0.56	103.56 $\pm$ 1.57
		t= 2.84	t= 4.776
		F= 4.36	F=7.25

## CONCLUSIONS

Many reagents were used as an oxidative coupling for this drug. Many methods were used to determine this drug and its properties. This paper proposes an oxidative coupling reaction of sulfamethoxazole with 4-aminobenzenesulfonic acid with potassium iodate in basic media to form an orange soluble dye product has been used. According to the findings, the current method is suitable for the routine analysis of this drug in its pharmaceutical preparations and its pure form. This method has also been characterized by linearity, accuracy, and high compatibility, and the procedure does not require special conditions, like temperature or pH limit. The technique was very simple, fast, cheap and fairly selective than some of the colorimetric methods published.

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## A COMPARATIVE STUDY OF THE CONSUMERS ATTITUDE ON THE MEAT HYGIENE SLAUGHTERED IN AND OUTSIDETHE SLAUGHTER HOUSEES

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### ABSTRACT

The study was conducted to assess the attitude and awareness of a sample of people regarding the indiscriminate slaughter and its effects on health and the environment compared with slaughtering in a slaughterhouse. The sample consisted of 120 persons from six equal professional groups contacted with the butchery labour (livestock keeper, truck driver, butcher, veterinarian, shopkeeper and consumer). The age ranged 22-76 years old, mean  $52 \pm 10$  years, lived  $\geq 5$  years in the Baghdad city. The results showed that there is a preference for slaughtering inside the slaughterhouse due to the presence of veterinary examination, slaughtering and preparing meat in a healthy, easy-to-clean places, unlike the indiscriminate slaughter that took place on the sidewalks of streets or in front of butchers' shops or at the entrances of their homes in front of people and passers-by. The results also showed that there is a great spread of the indiscriminate slaughter phenomenon throughout Baghdad governorate, coinciding with the lack of health awareness, lack of attention by citizens, weak monitoring authorities, and a great waste of secondary waste resulting from indiscriminate slaughter, such as leather, wool and blood.

**Keywords:** Slaughterhouses, indiscriminate slaughter, awareness.



## دراسة مقارنة لرأي المستهلك في صحة اللحوم المذبوحة في المجازر وخارجها

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## الخلاصة:

أجريت الدراسة لتقييم موقف ووعي عينة من الناس بظاهرة الذبح العشوائي وتأثيراته على الصحة والبيئة مقارنة مع الذبح داخل المجزرة، وتكونت العينة من 120 شخصاً من ست مجموعات مهنية متساوية ذات صلة بمهنة القصابة (مربي مواشي، سائق سيارة حمل، جزار، طبيب بيطري، صاحب متجر، مستهلك)، وتراوحت أعمار المشاركين 22-76 سنة بمتوسط  $10 \pm 52$  سنوات يعيشون بمدينة بغداد منذ  $5 \leq$  سنوات، وأظهرت النتائج أن هناك تفضيل للذبح داخل المجزرة وذلك لوجود الفحص البيطري وذبح وتجهيز اللحوم في أماكن صحية سهلة التنظيف على عكس الذبح العشوائي الذي كان يحدث على أرصفة الشوارع أو أمام دكاكين الجزارين أو عند مداخيل بيوتهم أمام الناس والمارة، كما أظهرت النتائج أن هناك انتشاراً كبيراً لظاهرة الذبح العشوائي في أنحاء محافظة بغداد، تزامن مع قلة الوعي الصحوي للامبالاة من قبل المواطنين وضعف الجهات الرقابية وإهدار كبير للنفايات الثانوية الناتجة عن الذبح العشوائي مثل الجلود والصفوف والدم.

الكلمات المفتاحية: مسالخ، ذبح عشوائي، توعية.

## INTRODUCTION

In the early nineteenth century, the slaughterhouse was known as the largest institution of transition from the agricultural system to the industrial system, accompanied by increased urbanization, technological developments, and attention to hygiene (Brantz, 2008). Meat elements play an important role in the human diet by providing a good source of high-quality protein as well as beneficial fatty acids and a variety of micronutrients for optimal health (Wyness, 2016).

A cholera outbreak in the 1840s eventually brought public health concerns about slaughtering animals in the city which led to the close of the live animal part of the Smithfield Market, it is necessary to build slaughterhouses outside city centers and monitor the impacts on air and water quality in the surrounding area (Kalof, 2007).

In a typical slaughterhouse, carcasses are slaughtered, prepared, stored and classified under safe conditions and in a sanitary environment (Kayikci et al., 2019). On the contrary of that, the indiscriminate slaughter is an unacceptable health violation and a source of danger to public health, the meat is unknown source and the slaughtered animal isn't examined by the veterinarian, as well as displaying meat and hanging it on irregular and unhealthy places, the animal may have had a communicable disease and contact with harmful insects. These places certainly not healthy or designated for slaughtering as what occur in the official slaughterhouses. In Iraq the indiscriminate slaughterer is considered a person who is not officially authorized and does not have a health certificate from the Ministry of Health confirm free from diseases (Federal Office of Supreme Audit, 2017)

In the slaughterhouse, large quantities of by-products, such as skin, bones, internal organs, fat tissue, horns and hooves, are disposed of which can be converted into useful products for use in other sectors such as in the manufacture of animal feed, fertilizers, leather and wool industries etc. (Al-Saffar & Abdul Hussein, 2020)

Indiscriminate slaughter produces many materials containing nitrogen and phosphorus that can be extremely hazardous to the environment and human health when they enter water systems in large quantities. For this reason global environmental laws have defined the presence of animals in rural areas and stayed away from the urban communities (Matchawet

*al.,2019*). This study aimed to estimate the consumers' attitudes towards meat slaughtered inside and outside the slaughterhouse, and to evaluate the role of the authorities, and the consumer interaction with this uncivilized behaviour.

## METHODOLOGY

**Design of the study:** Descriptive analytical study.

**Sample of the study:** 120 of six equal occupational groups (livestock breeder, truck driver, butcher, veterinarian, shop owner and consumer citizen).

**The setting of the study:** The data were collected from June to September 2020. The mean age of the sample was 52 years, range 22-76±10 SD, lived ≥ 5 years in Baghdad. Consent was obtained from all the participants before conducting the questionnaire.

**Place of interviewing:** Participants were interviewed in several areas in Baghdad, such as near the slaughterhouses, street, Animal selling places, the butcher shop, workers in this sector. Performing a questionnaire which included two parts:

Part 1: Demographic questionnaire: Information collected from each participant included: gender, age, educational level, occupational status, residence as shown in (Table, 1)

Part 2: Questionnaire: Information was utilized about the reality of the situation of indiscriminate slaughter in Baghdad city and how the authorities dealt with it and the extent of the citizen's interaction and awareness with this situation involve rejection or acceptance as shown in (Table 2).

**Statistics:** The data were analyzed in SPSS software version 22, for comparison between variables used the chi-square test at a level of significantly lower than 0.05 (*Wuet al.,2016*).

**Table (1):** Characteristics of the study sample.

Characteristics of the Study Sample	No of Cases	(%)
<b>Gender</b>		
Male	89	74
Female	31	26
<b>Age (years)</b>		
	Mean: 52, (22-76) yrs., ±10 SD	
20-29	3	
30-39	18	15
40 -49	33	27.5
50-59	34	28.5
60-69	21	17.5
≤ 70	11	9
Total	120	100
<b>Education</b>		
Illiterate	10	8
Learner	14	12
Primary	26	22
Secondary	38	31
Collectors	32	27
Total	120	100
<b>Residencies</b>		
Al-Karkh	81	67
Rusafa	39	33
Total	120	100





## RESULTS AND DISCUSSION

Demographic characteristic of those included in the questionnaire were 74% males and 26% female their ages ranged between 20-70 years, and from different educational levels, the highest was secondary graduates 38%, followed by a university graduate 32% and the lowest rate of illiteracy 8%. As for residence, the majority were from the Karkh area 67%, then from Rusafa 33% as shown in (Table 1). Meat is a meal that the human being cannot dispense with since the beginning of creation and up to the present day, as the demand has become more and more, spatially are guaranteed and developments in the food industries. Beef and sheep meats were and still the favorite of everyone's human in most religions due to their delicious and favorite taste (Wyness, 2016). This was in agreement with the results of this study when the majority of the sample preferred eating the red meat 70%, and the rest preferred eating white meat 30%. About the red meat the majority of people prefer sheep meat 65% the other prefers the beef 35%. According to participants' opinion, most of the animals slaughtered in the butcher shops are sheep 46% as for poultry 41 and 13% for cattle. The butchers is slaughtering, hanging and processing the carcass on public roads, sidewalks, squares, and gardens. Most of the sample lives far from the slaughterhouse site, on the other hand, most of them live near the butcher's shops, and most of these shops contrary to environmental laws and the conditions of complete safety, although the butchers wash their stores constantly and use the sterilizers, the insecticides spray and rat poisons as shown in (Figure 1). This is contrary to what previous studies indicated about the environmental laws which undertake, the butcher's shops are designed to sell meat only and prevent the slaughter in it or the near because this has great health risk. What encouraged the increase in the situation was the failure to apply the fines imposed by law, as indicated by 72% of the sample (Broadway, 2000)

The results of the questionnaire showed the percentage of community rejection of random slaughter is 61 for those who do not believe that there is an effect of random slaughter on the health of the consumer as shown in Table 2. Furthermore, the community rejection of the indiscriminate slaughter was compared in this study with the occupational and the educational level groups as shown in (Table 3) there was no significant value for rejection this situation and this indicates the lack of societal awareness of this condition, which is culturally unacceptable. So, it's very important to raise the community awareness and the knowledge about the dangers of indiscriminate slaughter on the physical and psychological health (Cadmus, 2010).

About the slaughterhouses, approximately three quarters of the sample agreed that there are no solid waste incinerators, no recycling of this waste in any way and no treatment of heavy water resulting from the slaughtering, these matters pose major health and economic problems. In countries with an economic outlook this large quantity of by-products, such as skin, bones, internal organs, fat tissue, horns and hooves, are disposed of which can be converted into useful products in other sectors such as the manufacture of animal feed, fertilizers, leather and wool industries etc. (Sabri, 2013).

From the beginning at the farm to the ending on the consumer's table, the meat passes through many stages that are in contact with environmental effects, which are either microscopic and macroscopic organisms or non-organisms substances such as toxins and other chemicals (Matchawet *al.*, 2019). By meats, many zoonotic diseases pose a threat to human health, such as emerging bacterial diseases (Colibacillosis, Listeriosis and Salmonellosis) or by viruses such as Hepatitis type E, Rift Valley fever or parasitic disease such as worms include a large group of flatworms and roundworms like hookworm and codfish, also, there is a belief that cancerous bovine leukosis may be transmitted to humans through the meat and dairy products. So the risks of contamination are high in the peri-urban environment, especially where high densities of humans and livestock (Alabbody, 2018).

In Iraq among the legal violations, as shown by previous studies, 63% of the red meat slaughterhouses are within the basic design of the municipal borders. The Iraqi laws on massacres stipulated that the location of the slaughterhouses must be outside the basic design of cities and far from the municipal borders of cities with a distance not less than one kilometer from the population centers where the number of houses exceeds 20. But because the expansion of Baghdad city and the extension of the horizontal building, the slaughterhouse became inter the municipal borders of the city and this is a big problem resulting from poor planning officials (Aldoskyet *al.*, 2015). Some problems and obstacles prevented the development of government slaughterhouses, such as failure to provide the sustainability and maintenance of the slaughterhouses, the use of slaughterhouses by terrorists in hot spots, building a slaughterhouses in the others ownership, lack of training courses for slaughterhouse workers and a lack of monetary portion.(Al-Anbariet *al.*, 2014)

The study appeared there was a veterinary examination of the animal in the slaughterhouses to make sure it's healthy before and after slaughtering, the veterinarian puts a health stamp on the meat (Figure 1).While the indiscriminate slaughter had been lack of control and they are illegal which cause many problems, as the meat may not be suitable for human consumption, or some livestock may be sick or dead, in addition to the residues and unpleasant odors that cause many health problems (Federal Office of Supreme Audit, 2017).

Every profession has rights and duties, the duties must be obeyed , as indicated by the International Labour Organization (ILO). This labor should not cause any physiological and psychological problems (Alabbody, 2021).In this study, 68% of the sample confirmed that indiscriminate slaughtering causes psychological harm to children and women. Many studies have indicated to the excessive psychological damage on children and some adults in minute of appearance the blood when these animals are slaughtered, it causes fear or refuses to eat the meat, and it may push the person when scenes of violence are repeated, the behavior may become hostile to others, eventually becoming an aggressive character who is not afraid to see blood (Muller, 2018).

**Table 2:** The questionnaire to the sample regarding slaughter and meat hygiene.

The questions	No of Cases	(%)
Which the most types of meat you prefer?		
Red	70	84
White	30	36
Total	100	120
Which types of red meat you prefer?		
65	78	Sheep
35	42	Beef
100	120	Total
Do you prefer slaughtering in the slaughterhouse or outside it?		
75	90	Slaughterhouse
25	30	Outside the slaughterhouse
100	120	Total
Are butcher's shops available in your district?		
92	110	Yes
8	10	No
100	120	Total
Is indiscriminate slaughter residues close to your home?		
31	37	Yes
69	83	No
100	120	Total
What are the most types of animals slaughtered in your area?		

41	50	Poultry
46	55	Sheep
13	15	cattle
100	120	Total
Do you think there is community interest to reduce indiscriminate slaughter?		
39	47	Interested
61	73	Careless
100	120	Total
Do you think there are a healthy incinerators of the solid waste of the massacre?		
35	42	Yes
65	78	No
100	120	Total
Do you think there are liquid waste treatment units for the massacre?		
20	24	Yes
80	96	No
100	120	Total
Do you think there is a usage of carot accessories like leather and viscera?		
29	35	Yes
71	85	No
100	120	Total
Do you think there are fines for the offending butchers?		
28	34	Yes
72	86	No
100	120	Total
Is indiscriminate slaughter cause psychological harm to children and women ?		
68	82	Yes
32	38	No
100	120	Total



**Figure (1):**Some unhealthy cases and practices 1,2,3 from indiscriminate slaughter and others from slaughtering in slaughterhouse which are somewhat healthy and correct with some indicators 3,4,5.

- 1: Slaughter at the entrance of the house.
- 2: Slaughter in the butcher shop in the market, as seen by the sellers and shoppers.
- 3: Slaughter on the street.
- 4: Slaughter sheep in the slaughterhouse.
- 5: The carcass is hanging in the slaughterhouse for the processing.
- 6: The suspended carcasses are examined by a veterinarian.

**Table (3):** Indicates the level of acceptance or rejection of the sample for slaughter outside the slaughterhouse, depending on the level of education and occupation by the Chi-square test.

The society rejection of indiscriminate slaughter	Education	Rejection No. & (%)	$X^2$ test: P value (df) Asymp. Sig. (2-sided)	Occupation	Rejection No.& (%)	$X^2$ test P value (df) Asymp. Sig. (2-sided)
		Illiterate	5(50)	<b>10.080a (12) 0.60</b>	Livestock owners	8(40)
	Reads & writes	4(29)	Truck drivers		5(25)	
	Primary	10(39)	Butchers		8(40)	
	Secondary	14(60)	Veterinarians		8(40)	
	Collectors	14(47)	Shop owners		10(50)	
			Citizens		8(40)	

The results showed that the educational level and the type of the occupation did not have a significant impact on the extent to which people accept or reject the case of random slaughter. This coincided with the fact that consumers were not concerned with the sources of processing and marketing of meat, while the percentage of the sample was 61% indifferent to this issue.

## CONCLUSION

In Iraq, the behaviour of the indiscriminate slaughter is scattering until it became the rule and the other is exceptions because of the weakness of the state's policy in planning by the relevant authorities in activating and developing the work of slaughterhouse model and the lack of awareness of the community and the impact on health and the environment. Non-use of carrot waste such as tanning and leather processing and the use of waste as fertilizer. The recommendation is to establish several modern slaughterhouse in Baghdad outside the basic design of the city at a distance of not less than one kilometer from the nearest gathering and to activate the work of the massacres by facilitating the process of carcasses reaching the slaughterhouse and taking advantage of the attachments of slaughtering and pursuing the violators who carried out the indiscriminate slaughter and imposing a fine on them.

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## STUDYING THE EFFECT OF FLOUR AND LUPINE PROTEIN CONCENTRATE INCORPORATION ON PHYSICAL, CHEMICAL AND SENSORY PROPERTIES OF BISCUIT

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### ABSTRACT

The purpose of this study was to determine the effect of lupine flour (L.f) and lupine protein concentrate (L.P.C) incorporation on chemical, nutritional and sensory qualities characteristics of biscuit (L.P.C) was prepared by isoelectric precipitation method. A standard recipe for biscuit preparation by wheat patent flour used as the control. Wheat flour in the control treatment was replaced with (L.f) and (L.P.C) at levels 10, 20 & 30%. Chemical composition of (L.f), (L.P.C) and biscuit treatments were studied. Results showed that protein contents were 35.35 & 75.80% for (L.F) and (L.P.C), respectively. While they amounted to 14.70, 16.16 & 18.61% for (L.f) incorporated biscuits and 15.20, 18.09 & 21.08% for (L.P.C) incorporated biscuits at the substitution levels studied, respectively compared 12.43% control. Results also indicated contents of total dietary fibers and tannins in (L.f), (L.P.C) and biscuits prepared. Sensory evaluation of biscuit treatments revealed that there was significant decrease at substitution level up to 30% of (L.f) except color score, while all scores of sensory properties were improved significantly at all substitution levels of (L.P.C). Spread ratio was affected adversely by incorporation of (L.f) and slightly when (L.P.C) used. Results showed a reduction in biscuit tenderness during storage. Reverse to the above statement with 30% incorporation of (L.f) while increased at (L.P.C) treatments. The study demonstrated that (L.f) and (L.P.C) can be incorporated into biscuits formulation by replacing up to 20, & 30% of wheat flour control 12.43%, respectively to increase dietary fiber and protein contents.

**Keywords:** Lupine flour, protein concentrate, chemical nutritional, sensory properties.



## دراسة تأثير الطحين والمركز البروتيني لبذور الترمس على الخصائص الفيزيائية والكيميائية والحسية للبسكويت

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## الخلاصة

حدد تأثير دمج دقيق الترمس (Lf) وتركيز بروتين الترمس (L.P.C) على الصفات الكيميائية والغذائية والحسية للبسكويت المحضر بطريقة الترسيب الكهروضوئي من خلال وصفاً قياسية لتحضير البسكويت بدقيق القمح المستخدم كعنصر اساسي، إذ تم استبدال دقيق القمح بـ (Lf) و (L.P.C) عند المستويات 10 و 20 و 30٪، وتمت دراسة التركيب الكيميائي لكل من (Lf) و (C.L.P) ومعاملات البسكويت، وأظهرت النتائج أن محتوى البروتين كان 35.35 و 75.80٪ لكل من (Lf) و (L.P.C) على التوالي، بينما بلغت 14.70 و 16.16 و 18.61٪ للبسكويت المضاف له (Lf) و 15.20 و 18.09 و 21.08٪ للبسكويت المضاف له (L.P.C)، كما أشارت النتائج إلى محتويات إجمالي الألياف الغذائية والعفص في (Lf) و (L.P.C) والبسكويت المحضر، وأظهر التقييم الحسي معاملات البسكويت وجود انخفاض معنوي عند مستوى ابدال حتى 30٪ من (Lf) باستثناء درجة اللون، بينما تحسنت جميع درجات الخصائص الحسية معنوياً في جميع مستويات ابدال لـ (L.P.C)، وتأثرت نسبة الانتشار سلباً بدمج (Lf) و قليلاً عند استخدام (L.P.C)، وأظهرت النتائج انخفاضاً في طراوة البسكويت أثناء التخزين، وأوضحت الدراسة أن (Lf) و (L.P.C) يمكن إدخالهما في تركيبة البسكويت عن طريق استبدال ما يصل إلى 20 و 30٪ من دقيق القمح بنسبة 12.43٪ على التوالي لزيادة محتوى الألياف الغذائية والبروتين.

الكلمات المفتاحية: طحين الترمس، مركز البروتين، الخصائص التغذوية الكيميائية، الخصائص الحسية.

## INTRODUCTION

Legumes are considered of a great importance for human nutrition because of their high protein content .And due to their technological characteristics, their use has been carried out not only by consumption by their whole grains but they are used for production flour, protein concentrate and protein isolate which improve nutritional properties that are added to them. (Polit *et al.*, 2019). Among the famous nutritional pulses in the world, lupine is considered as an important source of vegetarian protein. Lupine is planted for improvement of soil structure, animal feed as well as human food (Maghaydah *et al.*, 2013). Many studies, reported the use of lupine flour (L.f) and lupine protein concentrate (C.L.P) in production of pastries, like noodles, bakeries like bread, cake, biscuit and sausage. In addition to its content of about 40% protein and essential amino acids, it is also considered a good source of lipids richened with unsaturated fatty acids, as well as fibers, minerals, vitamins and antioxidants which assist in protection of human from cardiac diseases, hypertention, diabetes, osteoporosis and cancer (Kohajdova *et al.*, 2011; Mattila *et al.*, 2018). Studies proved that both protein and fibers of lupine had physiological advantages of body, but lupine use became limited because of presence of antinutritional factors like phytic acid, alkaloids, saponins and enzyme inhibitors which affected protein digestibility. So production of protein concentrates or isolates is considered a good method to decrease these matters. Incorporation of lupine flour or protein to wheat based foods such as biscuits, has a potential to increase dietary fiber or protein contents, respectively. The amount of high protein and high dietary lupine flour that can substitute wheat flour represent a compromise between nutritional improvement and achievement of satisfactory and physical properties of dough & its many prodded (Jayasena & Nasar, 2011).

Commercially, the world market of biscuit was projected to reach \$43 billion by the year 2015, and may be multiplied at 2025, and this is primarily driven by the changing consumer trends toward healthy food options & supplemented products with many differ



natural heady plants (Anonymous, 2010). Lupine seeds contain a percentage of tannins and antioxidants that benefit human health in eliminating oxidative stress in the human body. This research aimed to prepare (L.f) and (L.P.C) incorporate them in biscuit making and study some its chemical, nutritional and organoleptic characteristics.

## MATERIALS AND METHODS

### Preparing the raw materials for biscuit production

Sweet lupine seeds were purchased from AL-Shorja Market, Baghdad, imported from Egypt. Patent wheat flour (Kuwait flour mills and bakeries Co.) sugar, milk, shortening (Turkish origin) baking powder (Arab food industries Co. Amman, Jordan). All chemical and reagents are chemical grade.

### Preparation of lupine flour

To prepare (L.f) 2.5 kg of seeds were ground by metal mortar then by coffee mill and sieved to pass 60 mesh sieve. Flour sample were stored by using plastic containers in freezer until utilization in different tests.

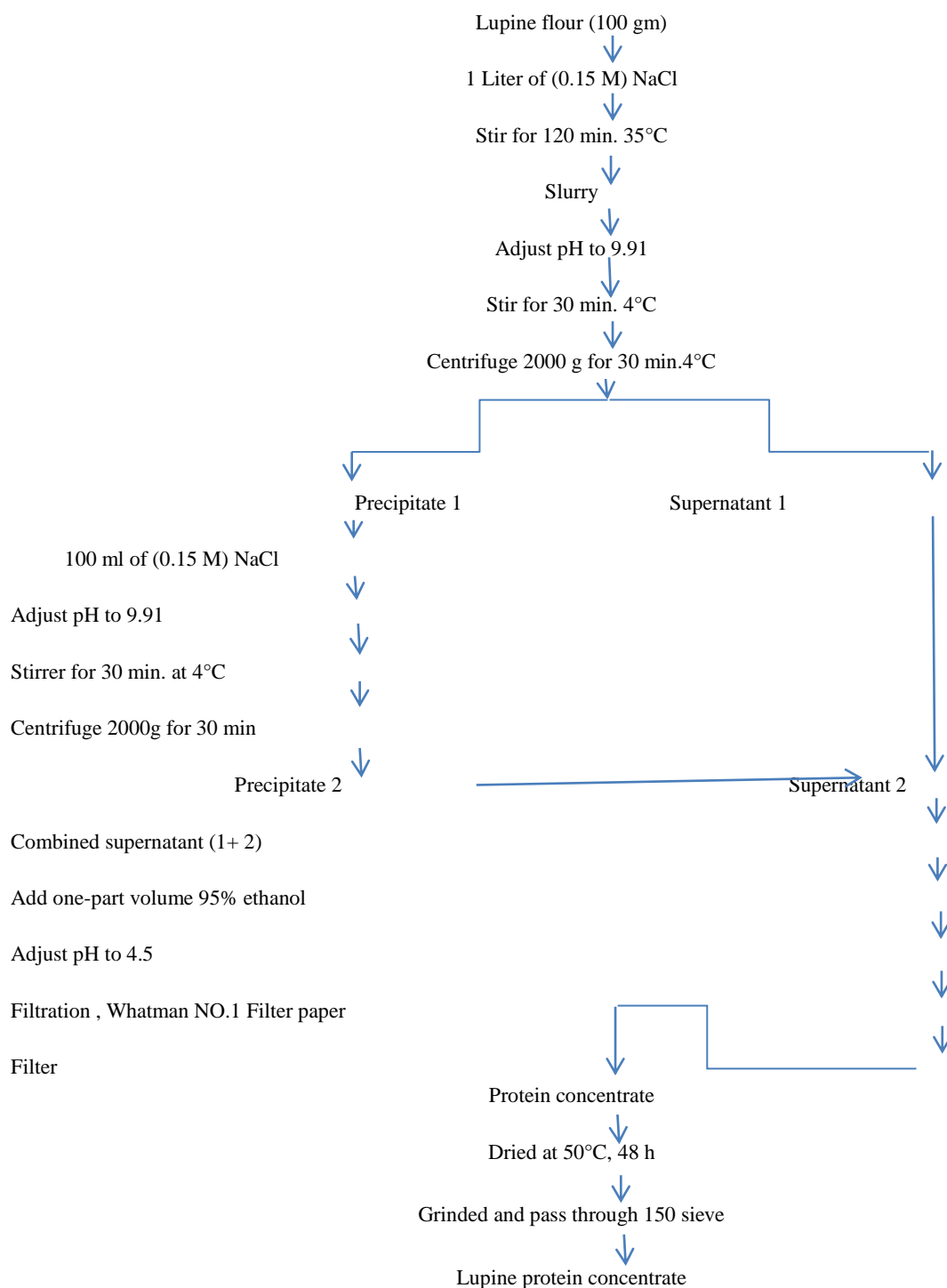
### Preparation of lupine protein concentrate:

Lupine protein concentrate was prepared by isoelectric precipitation as reported by Muneet *al.* (2013) with some modification of sample weight and reagent volumes a sample of 100 gm (L.f) was mixed with 1 liter of 0.15 M sodium chloride solution and stirred for 120 min at 35°C. pH was adjusted to 9.91 and mixture was further stirred for 30 min at 4°C. The slurry which was resulted was centrifuged at 2000 g for 30 min. The precipitate obtained after recovering supernatant, was dissolved in the initial sodium chloride solution at the above liquid to solid ratio under stirring. The pH was a digested to the initial value and slurry stirred for 30 min at 4°C and then centrifuged as explained previously. Supernatant of the two alkaline extraction were collected and one part volume of ethanol (95% v/v) added. The pH was adjusted to 4.5 during stirring. Precipitated proteins were recovered under vacuum using filter paper (Whatman No:1). The protein concentrate was dried at 50°C for 48 h in an air oven and then ground. The resultant powder was kept in plastic containers until using, (Figure 1) shows the production scheme for lupine seed protein concentrate.

### Biscuit making

Biscuit was made according to this recipe as flour basis: wheat patent flour: 100%, sugar: 47.5%, shortening 28.4%, salt 0.9%, baking powder 2%, milk 10%, water 25% which its amount was changed as replacement of (L.f) and (C.L.P) levels were changed, replacement levels of (L.f) and (C.L.P) were 10, 20, and 30% of wheat flour. Water absorption of wheat patent flour was 58% as flour basis. Absorption of (L.f) and (C.L.P) were 4.1, 3.05 gm/gm, respectively. Biscuit formula components were mixed by electrical mixer to get a creamy appearance. The resultant dough was spread, thickness of 7 mm using a hand roller and cut to pieces of 30 mm diameter. The pieces were placed on trays greased with shortening, baked at electrical oven on 200°C for 10 min. Biscuits were cooled at room temperature, kept in polyethylene bags and stored for analysis. Treatment were denoted as follows: control, T1, T2, T3 substitution levels, (L.f), T4, T5, T6: substitution levels of (C.L.P), respectively (Lopezmetal., 2019).





**Figure (1):** Schematic of lupine concentrate normal.

### Spread ratio calculation

Spread ratio was measured according to (AACC 10-50.2000) by estimation diameter and thickness as average of seven pieces of biscuit by vernier. Spread ratio was determined by the equation:

$$\text{spread ratio} = \frac{\text{diameter (width)}}{\text{thickness}}$$

### Tenderness of biscuit

Tenderness of biscuit was measured by using a local instrument contained a metal cone which can go down over the piece of biscuit, the depth of the hole formed was measured as millimeters by a ruler fixed inside the instrument.

### Sensory evaluation

Fifteen panelists were asked to record their assessment of biscuits color, texture, flavor and appearance scored as: 20, 30, 30 and 20, respectively, Oneway analysis of variance (ANOVA) was performed to test differences between treatment followed by Duncan's analysis (Kefale&Yetenayet, 2020).

### Chemical analysis

Moisture was determined by air-oven method at 105°C/ min. Lipids content was estimated by using petroleum ether extraction in soxhlet instrument. Total nitrogen was determined by Kjeldahl method, protein content was calculated as N×5.7 for wheat flour and N×6.25 for lupine flour and its products, respectively. Ash was determined by incineration sample in a muffle. All these determination were methods conducted to (AOAC). Carbohydrates were estimated by difference. Total dietary fiber was quantified by chemical method described by AOAC. Tannins was determined by the method described by Van-Burden & Robinson (1981).

## RESULT AND DISCUSSION

### Chemical composition of lupine flour and lupine protein concentrate

The results are shown in the (Table 1). chemical composition of control. The moisture content of whole meal flour was 10.93%, which is a high percentage compared to legume flour, which has a lower moisture content. The proportion of ash and fat in whole wheat flour was less than that of lupine seed flour 1.64 and 1.60%, respectively, which is close to its percentage in the protein concentrate of lupine seeds. While the protein content of whole meal flour is 12.50%, and carbohydrates its proportion of 73.33%, which is higher than that of lupine seed flour (Lin *et al.*, 2019). (L.f) and (L.P.C). The decrease of moisture, fat and ash in (L.P.C) compared to (L.f) was due to the partial removal of them during protein extraction. Protein content of (L.f) 35.35% was at the range shown by Monteiro *et al.* (2014). When comparing (L.f) in our study with other legumes, it had higher protein content than lentils, peas and chickpeas but lower than soybean (Maphosa & Jideani, 2017).

It was for (C.L.P) revealed that its content of moisture, fat, ash and protein were in supplier 4.30, 1.33, 1.18 and 75.80%, respectively. The protein content of (L.P.C) was higher compared with previous studies (Evangelista *et al.*, 2004; Sujaket *et al.*, 2006). The method of protein extraction is important to obtain a good protein concentrate. Many papers indicated that to prepare concentrate of higher protein contents from lupine it is necessary further to process flours to remove some of low molecular weight components. Fat contents of (L.f) and (L.P.C) were 8.57 and 1.33%, respectively. Content of fat in current study was at the range reported. In general, lupine oil is characterized by a balance fatty acid composition with total saturated fatty acids of 10% and total unsaturated fatty acids of 90% of which 32 to 50% is oleic acid, 17-47% is linoleic acid and 3-11% is linolenic acid (Kohasdova *et al.*, 2011). Various researchers measured carbohydrates with fibers, while others reported their content alone. Author links open overlay pane Maphosa & Jideani (2017) found carbohydrate content equivalent to 71% of the seed weight, and its content of raw fiber was 1.7%. Lupine seed contained little quantities of starch 5-12% and higher levels of soluble non-starch polysaccharides 30-40%.

**Mierlitaet al.(2018)** production of (L.P.C) normally decreased amount of available carbohydrates, it reached 17.39%.

**Table (1):** Chemical composition of lupine flour and its protein concentrate

Component (%)	Wheat flour	Lupine flour	Lupine protein concentrate
Moisture	10.93	5.51	4.30
Ash	1.64	2.92	1.18
Protein	12.50	35.35	75.80
Fat	1.60	8.57	1.33
Carbohydrates	73.33	47.65	17.39

Values are average of duplicate

### Chemical composition of biscuits

The chemical analysis of biscuit & different incorporation (or supplemented) with L.f and L.P.C was presented in (Table 2). Results indicate low increase in moisture and ash of biscuits prepared with the results indicate that the percentage of humidity in baskets was close in its proportions and that the highest averages were T3, T6 and reached 2.99 and 2.98% respectively, as for the means significantly, they were in the standard treatment 2.64% (**Polit et al., 2019**).

The percentage of ash in the samples of biscuit varied, the mean was the most high transaction T3, 3.75% as for the lowest means, it was for the transaction C 2.10%. The percentage of protein in biscuits had different averages, as the highest of those averages were significant differences T6 21.08%, as for the lowest averages, they were C 12.43%. The increase of protein quantity due to lupine incorporation emphasized the potential to use it as a source of protein in biscuit and other pastries. Lupine represented a good balance of essential amino acids (**Drakoset al., 2007; Naumann et al., 2017**). It was considered to be a good source of lysine. But **Monteiro et al. (2014)** reported that cultivars they studied had lower protein quality in relation to the presence of essential amino acids, but with good digestibility when compared to other legumes. The fat percentage of biscuits had high averages and the highest averages were at T3 25.01, while the lowest averages were significantly lower C 21.65%. Biscuits prepared with (L.f) and (C.L.P) represented a good source of energy due to its high content of oil ranged 18.5-19.10% which contained high content of C18:1 and C18:2 fatty acids (**AL-Hamdani, 2017**). No problem in storage of biscuits may be expected due to high content of oil, because of low moisture content of the product as well as vacuumed packing and low acidity of oil extracted, indicating no danger of rancidity in biscuits (**Rutkowskiet al., 2016**). The averages of carbohydrates were different, as the results indicated that the highest averages increased in the mean C 61.18%, while the lowest averages were treated T3 49.64%.

**Table (2):** Chemical analysis of biscuit treatment.

Treatment	Moisture (%)	Ash (%)	Protein (%)	Fat (%)	Carbohydrates (%)
C	2.64 d	2.10 f	12.43 f	21.65 f	61.18 a
T1	2.77 c	2.25 e	14.17 e	22.63 c	58.18 b
T2	2.89 b	2.76 b	16.16 c	23.75 b	54.44 e
T3	2.99 a	3.75 a	18.61 b	25.01 a	49.64 f
T4	2.79 c	2.33 d	15.20 d	21.75 e	57.93 c
T5	2.86 b	2.45 c	18.09 b	21.90 d	54.70 d
T6	2.98 a	2.55 c	21.08 a	21.99 d	51.40 e

Values are the average of duplicates Similar letters indicate that there are no significant differences between the parameters ( $p < 0.05$ ) according to Duncan s test.

### Fibers and tannins in lupine flour, lupine protein concentrate and biscuit treatments

The results in (Table, 3) show the percentage of fibers and tines in lupine seed flour, the protein concentrate of lupine and the biscuit made from them. as it was noticed that the fiber content of lupine seed flour was the highest of the average protein concentration of lupine and reached 10.62 mg/g, while the average percentage of protein concentrate in lupine seeds was 0.22 mg/g, this percentage of fiber in the protein center is very small compared with flour of lupine seeds, because of high content and good quality of lupine fiber, lupine flour is usually added to many foods such as bakeries. good quality of lupine fiber came from its white color and its high water absorption as well as the nutritional value (Monteiroet al., 2014).

In the (Table 3), the percentage of fiber in the produced biscuit, The results showed that there were significant differences in their averages, as the most high were in T1, T2, and T3. T3 and it ranged between 4.23-6.84 mg/g, while the least studied averages for biscuits were low, it was C 3.01mg/g, whereas the mean protein concentrations ranged between 3.25-3.79 mg/g. The high fiber content is due to the bits of the husks of the lupine seeds, also, a portion of the fiber is found in the endosperm of the seed. and he indicated Pisarkova&Zraly (2010) that the extraction rate for flour may be inefficient, which increases the percentage of fiber in the flour, and that the fiber is part dissolved in water and the other part is insoluble, which makes its ratio different between mixtures.

The results showed in the ratio of tannins to lupine seed flour and the protein concentrate of lupine seeds and biscuits produced from different mixtures of them significant differences in their averages, as it was observed that the averages of the tannins for the protein concentrate were higher and reached 0.95 mg/g, while the mean of lupine seed flour was lower than that and reached 0.83 mg/g, as for the results of tannins in the produced biscuits, the highest averages were higher in treatment T6, while the lowest averages were in C, while in lupine seed flour biscuits, they were between that, as they ranged between 0.212-0.076 mg/g. Tannin was considered one of phytochemicals which have antioxidant capacity due to their classification as polyphenols. So lupine products may be source of tannins as antioxidants as well as flavonoids (Kohajdovaet al., 2011; Mattilaet al., 2018). According to results in (Table 4), there was no adverse effects for higher content of tannins on sensory evaluation, and this may consider a superior property for lupine addition to biscuits formulations.

**Table (3):** Total dietary fiber and tannins of lupine flour , its protein concentrate and biscuits treatments.

Treatment	Total dietary fiber (mg/g)	Tannins (mg/g)
L.f	10.62	0.83
C.L.P	0.22	0.95
Biscuit C	3.01 g	0.076 g
T1	4.23 c	0.149 f
T2	5.32 b	0.199 d
T3	6.84 a	0.212 c
T4	3.25 f	0.166 e
T5	3.55 e	0.266 b
T6	3.79 d	0.332 a

Values are the average of duplicates Similar letters indicate that there are no significant differences between the parameters ( $p < 0.05$ ) according to Duncan s test.

### Sensory evaluation of biscuit

The results of the sensory evaluation in (Table 4), which was conducted by ten evaluators from the college of agriculture, Tikrituniversity, showed that the averages had significant differences in the studied sensory characteristics of the produced biscuits, so the color trait had the highest mean increases in treatment C and T6 and reached 19, or

less. Averages of significant differences in T3 and 14, this may be due to yellowness caused by natural yellow pigments present in (L.f). This finding was agreed with **Jayasena & Nasar (2011)** who reported that there was an improvement in color with (L.f) substitution, it was found that the color of the biscuit was linked to the high level of proteins. The color of the biscuit becomes darker as the levels of proteins in the composition increase due to the interaction of amino acids of proteins with reducing sugars during baking in the Maillard reaction **Mahgoub et al. (2015)**.

The texture characteristic of biscuits had the highest averages for treatment C and T6 and reached 28, while the lowest averages for produced biscuits were for treatment T3 and reached 19. This may be referred to whiteness of the lupine protein concentrate. These results were in agreed with observations found by **Eman & Ahmad (2012)** who used lupine flour and its protein isolate in cake making. Generally speaking, the incorporation of protein concentrate, and isolates of legumes were better than their flours in respect of protein fortification of certain foods. Taste is the primary factor that determines the acceptability of any product which has the highest impact as far as market success of product is concerned (**Yilma & Admassu, 2019**).

As for the flavor characteristic of the produced biscuits, the highest averages were in the C and T6 treatment, reaching 28, while the lowest averages were in the decrease in the T3 treatment, reaching 20. The decrease of spread ratio probably caused by the formation of an elastic network, that causes shrinkage after baking due to the higher content of fibers in (L. f) and due to the higher content of protein in (L.P.C) These factors reduced diameter of biscuit, there for reduced spread ratio. the flavor of food products with added legumes can be improved by using traditional processing techniques such as soaking, fermentation, roasting, boiling and other processes (**Joshi & Awasthi 2020**).

The outward appearance of the produced biscuit had its averages showed significant differences, as the results showed that the highest averages were in treatment C and reached 19, while the lowest averages were significantly lower in treatment T3 and reached 11. The general appearance is important for the consumer to accept the product, and the shape of the biscuit produced is affected by the addition of flour in different proportions due to the presence of fibers in the flour, and the Glutennetwork is important in showing the desired shape of the product (**Adbelgadiret et al., 2019**).

The prevalence rate in the produced biscuit was the highest mean significantly in treatment C, reaching 4, while the lowest mean significant decrease was for treatment T3, reaching 2.90. The approximation of the diffusion ratio in the processed biscuits due to the consistency of the dough and the strength of the gluten network in the dough, and the shape of the biscuit cutting is consistent as a result of the correct cutting of the pieces, and the fermentation period is short, which helps not to damage the shape of the pieces (**Ahmad et al., 2019**).

**Table (4):** Sensory evaluation and spread ratio of biscuit.

Treatment	Color (20)	Texture (30)	Flavor (30)	Appearance (20)	Spread ratio
C	19 a	28 a	28 a	19 a	4.00 a
T1	17 b	26 c	26 b	16 d	3.57 c
T2	18 b	26 c	25 c	17 c	3.32 d
T3	14 c	19 e	20 d	11 e	2.90 g
T4	18 b	25 d	24 c	17 c	3.60 b
T5	18 b	27 b	27 b	18 b	3.12 e
T6	19 a	28 a	28 a	18 b	3.05 f

Values are the average of duplicates Similar letters indicate that there are no significant differences between the parameters ( $p < 0.05$ ) according to Duncan s test.

### Tenderness of biscuits

Results in (Table 5) revealed tenderness values of biscuits measured as a distance unit (Mm) which represented the depth of the hole formed by the metal cone when goes down over the piece of biscuit. Measurements were followed during four weeks of storage. Results showed three trends: the first: reduction of tenderness during storage (Rows). The second: reduction of tenderness as substituted level of (L.f) increased. The third: its increase as substituted level of (L.P.C) increased. Also it was noticed that values at second and third trends had the same tendency of sensory evaluation of texture scores (Table 5). These results agreed to the conclusion of **Pop et al. (2017) & Feyera(2020)** but didn't agree with results of, which indicated that hardness measured with Instronuniversal testing machine was opposite panelists opinion. This difference between current study and that research may be due to the variance of raw materials tested in biscuit preparation. Anyhow, the physical texture of biscuit is related to its moisture content and functional properties. Such as oil binding, water binding capacity, gelation properties, and fat content in the basic formulation (**Xing et al. (2021)**). Decreasing of biscuits tenderness during storage as illustrated by previous studies as well as current study- render the products losing brittleness, a desirable property of biscuits.

**Table (5):** Tenderness of biscuit during storage for four weeks.

Weeks/Treats	0 (mm)	1 (mm)	2 (mm)	3 (mm)	4 (mm)
C	20	20	16	12	10
T1	16	12	12	10	8
T2	21	15	12	9	6
T3	13	13	10	8	3
T4	25	19	15	9	6
T5	28	19	15	7	7
T6	30	14	14	8	7

Zero time: (after 2 h of baking).

### CONCLUSIONS

Biscuits of high dietary fiber and protein can be produced by using lupine flour, while high protein biscuit with good quality can be made by incorporation of lupine protein concentrate. This study showed that lupine flour can be substituted 20% of wheat patent flour in biscuit without adverse effects. Also it indicated that lupine protein concentrate can be substituted 30% of wheat patent flour successfully. This study emphasized that lupine flour, its protein concentrated is lake had superior attributes rendered them a promising alternative of low-cost protein, with nutritional quality comparing with other sources. Further research will be conducted to improve quality of biscuits and then promote the use of lupine to alleviate protein malnutrition problems in world .

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## SYNTHESIS OF NEW LEVOFLOXACIN SELECTIVE MEMBRANE SENSOR BASED ON MOLECULARLY IMPRINTED POLYMERS.

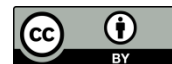
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### ABSTRACT

Two molecular imprinted polymer (MIP) membranes for Levofloxacin (LEV) were prepared based on PVC matrix. The imprinted polymers were prepared by polymerization of styrene (STY) as monomer, N,N methylene di acrylamide as a cross linker, benzoyl peroxide (BPO) as an initiator and levofloxacin as a template. Di methyl adepate (DMA) and acetophenone (AOPH) were used as plasticizers, the molecular imprinted membranes and the non molecular imprinted membranes were prepared. The slopes and detection limits of the liquid electrodes ranged from -21.96 to -19.38 mV/decade and  $2 \times 10^{-4}$  M to  $4 \times 10^{-4}$  M, and its response time was around 1 minute, respectively. The liquid electrodes were packed with 0.1 M standard drug solution and its response were stable at pH ranges from 1.0 to 11.0 and with good selectivity for more than several type. The electrodes produced have been successfully applied in preparation of the pharmaceutical sample for the determination of the analyte without any time consuming pretreatment steps.

**Keywords:** Molecularly impressed electrodes, levofloxacin, potentiometric method, styrene (STY) monomer.



## تخليق مستشعر الغشاء الانتقائي الليفوفلوكساسين الجديد على اساس بوليمرات مطبوعة جزيئياً

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## الخلاصة

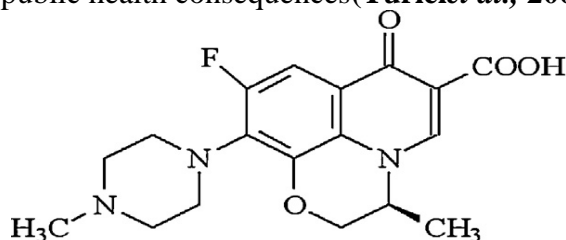
تم تحضير غشاءين من البوليمر المطبوع جزيئياً (MIP) للـ Levofloxacin بناءً على مصفوفة PVC. تم تحضير البوليمرات المطبوعة بواسطة بلمرة الستايرين (STY) كمواد مورو N، N-ميثيلين داي أكريلاميد كموصل متشابك، البنزويل بيروكسيد (BPO) كبادئ وليفوفلوكساسين كقالب. تم تحضير ثنائي ميثيل أديبات (DMA) وأستوفينون (AOPH) كمواد ملدنة، وتم تحضير الأغشية المطبوعة جزيئياً والأغشية غير المطبوعة. تراوحت حدود المنحدرات وحد الكشف للأقطاب السائلة من -19.38 - 21.96 مللي فولت/ عقد و  $4 \times 10^{-4} M$  -  $2 \times 10^{-4} M$ ، وكان وقت الاستجابة حوالي دقيقة واحدة على التوالي. تمت تعبئة الأقطاب الكهربائية السائلة بمحلول دوائي قياسي 0.1 M وكانت استجابتها مستقرة عند درجة الحموضة من 1.0 إلى 11.0 وبناتقانية جيدة لأكثر من عدة أنواع. تم تطبيق الأقطاب الكهربائية المنتجة بنجاح في تحضير العينة الصيدلانية لتقدير المادة التحليلية بدون أي خطوات معالجة مسبقة تستغرق وقتاً طويلاً. الكلمات المفتاحية: الأقطاب المطبوعة جزيئياً، الليفوفلوكساسين، طريقة قياس الجهد، المونمرستائرين (STY).

## INTRODUCTION

The first documented in the molecular imprinting technique in 1970 Nishide & Tsuchida (1976), molecular imprinting is a commonly emerging technique for producing polymers with different molecular properties for a given compound, its analogs, or an enantiomer (Whitcombe *et al.*, 1998; Yan & Row, 2006). MIPs are the sensing elements of a molecular imprinting electrochemical sensor (MIECS) which are molecularly imprinted. MIECS has many characteristics include: high selectivity, simplest techniques, lower costs, low D.L, highly stable. The combination of both the template molecules and the cavities can also be easily achieved and is carefully applicable even with harmful chemical materials. Thus, MIECS was used for optical and electrochemical application (Tadi & Motghare, 2016; Al-Bayati & Abd, 2017; Al-Bayati & Al-Safi, 2018; Momenh & Gholivand, 2018; D'Aurelio *et al.*, 2020). Molecularly imprint polymers (MIPs) are prepared By combining template molecule (LEV) with functional monomers styrene (STY), a cross-linker N,N-methylene diacrylamide (N,N MDAM) and initiator benzoyl peroxide (BPO) in appropriate solvent. Most of the time, Aprotic and unipolar solvents. So after polymerization, template molecule extraction reveals recognition cavities that complement the template molecule's shape, height, and chemical functionality, allowing the resulting polymers to selectively rebind the template molecule from a mixture of closely related compounds (Lavignacet *et al.*, 2004). In additional, the MIP have easily synthesis and have good mechanical properties, reliability for pressures and temperatures, and thus are cost-effectively and suitable to applicable for harmful chemicals (Li *et al.*, 2012, Al-Bayati & Aljabari, 2016; Zhang *et al.*, 2019). Interactions between template and monomer have been studied through spectral and computer simulation research. The utilization of molecular imprinting techniques have increased significantly in recent years, thus illustrating magnificently the ability of MIP model for detection toward the target molecules. (Bateset *et al.*, 2017; Al-Bayati, 2018; Marćet *et al.*, 2018). There Several analytical methods such as High performance liquid chromatography (HPLC) (Santoro *et al.*, 2006; Mahajanet *et al.*, 2007), chromatography (Mishal & Sober, 2005), fluorescence (Pratet *et al.*, 2006), mass spectrometry (Boxallet *et al.*, 2006) and potentiometric method were used for LEV determination in contrast. Such techniques are distinguished by a lengthy and complicated preparation of the sample for analysis, as well as costly instrumental equipment. In this study, a

new levofloxacin ion-selective membrane electrode (lev-MIP+DMA or AOPH) in the PVC membrane process was prepared.

LEV is a broad-spectrum racemic fluoroquinolone antimicrobial (Figure 1). It is the ofloxacin's active L-isomer, and its antibacterial efficacy is around two times that of ofloxacin, and are widely used today in both human and veterinary applications. Medicine as antibacterial agents of different diseases (Turiolet *et al.*, 2007). Over the past few years, the research on antibiotic residues in the multiple environmental compartments has been of great importance due to the biodiversity and public health consequences (Turiolet *et al.*, 2007).



**Figure (1):** Chemical structure of the levofloxacin.

## MATERIALS AND METHODS

Levofloxacin was purchased from company of drug manufacturing and medical supplies (IRAQ-SID-Samara). generic levofloxacin tablets purchased from local pharmacies are; Levoneer 10 tablets 500 mg from (Pioneer-Iraq) levofloxacin 7 tablets 750 mg from (Iliflox-Turkey). Di methyl adipate (DMA) and acetophenone (AOPH) also metal salts, they were obtained by Sigma-Aldrich and used as provided. Styrene (99%), monomer N,N-methylenediacrylamide (N,N-MDAM) (99%), and benzoyl peroxide (BPO) (78%) from Sigma-Aldrich is obtained. the chemical used was the largest purity reagent and was used when obtained without further purification.

### Apparatus

A digital voltmeter (HANA pH211 instrument Microprocessor pH meter) was used to perform potentiometric measurements. Digital pH meters (wissenschaftlich- Technische Werkstätten GmbH WTW pHmeter in lab pH720-Germany) were used for pH measurements. Electrode efficiency was investigated by calculating the potential of levofloxacin solutions at ambient temperature with a variety of concentrations from  $5 \times 10^{-4}$  M to  $10^{-1}$  M. Every solution was stirred, and the reading potential was recorded at equilibrium. The calibration curves obtained by plotting the response against the levofloxacin concentration logarithmic functions.

### Preparing of Standard Solutions for ISEs studies

For the preparation of a standard solution of 0.1 M levofloxacin by dissolving 3.614 g of standard levofloxacin in the methanol and completed to 100 mL in the volumetric flask. In the same procedure, the other solutions were prepared in 100 mL ranging from ( $5 \times 10^{-5}$  –  $10^{-1}$ ) M. The stock standard solution of  $1 \times 10^{-3}$  M  $1 \times 10^{-4}$  M phosphomolybdic acid was prepared by dissolving 1.1288 g, 0.11288 g from phosphomolybdic acid respectively in distilled water and completed for 100 mL. All interfering ions ( $K^{+}$ ,  $Ca^{+2}$  and  $Al^{+3}$ ) are preparing of 0.1 M. The other solutions at the range from ( $5 \times 10^{-5}$  –  $10^{-1}$ ) M were prepared in 100 mL.

### Synthesis of the Imprinted Polymer (MIP)

For the preparation of levofloxacin molecularly imprinted polymer (LEV-MIP), 1 mmol (0.34 g) of levofloxacin was mixed with 2 mmol (0.208 g) styrene as the monomer. After this, 10.6 mmol (1.634 g) N, N-methylene diacrylamide was added to the solution as the cross linker, followed by (0.3 g) benzoyl peroxide as the initiator. All these materials were subsequently dissolved in 5 mL mixture of methanol and chloroform, and the mixture was stirred for 5 minutes to obtain a homogenous solution. Afterwards, the gas  $N_2$  was passed through the

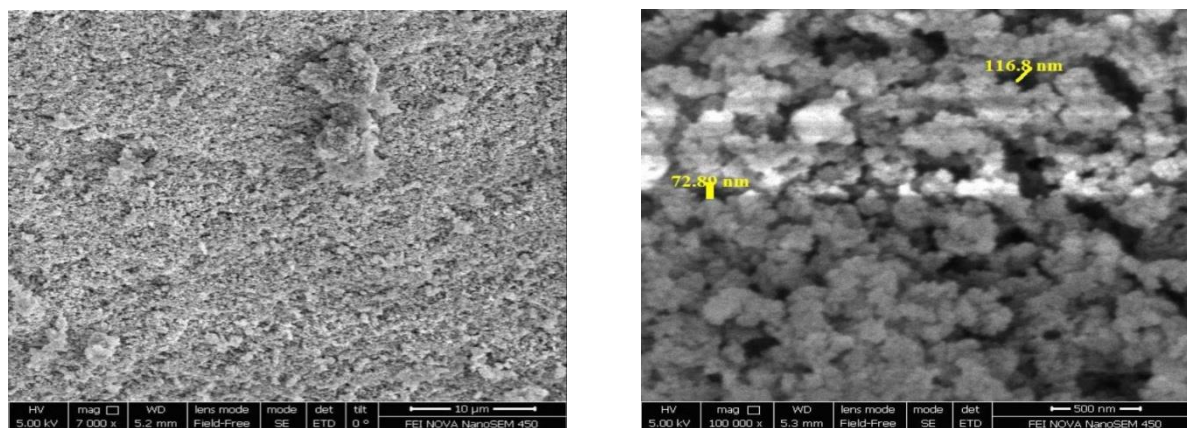
solution for 30 minutes to remove oxygen from it, and the solution was placed in a water bath at 65°C. When the reaction was complete, the molecularly imprinted polymer became hard, and, after the polymerization process, the polymer was dried and crushed to obtain it as particles. Finally, these particles were sonicated in CHCl<sub>3</sub>/ CH<sub>3</sub>OH / CH<sub>3</sub>COOH (7 : 2 : 1) (v/v/v) to remove the template from the MIP. The particles size of LEV- MIP (125 μm).

The preparation of non-molecularly imprinted polymers was carried out using the same method, using the same compounds and under the same conditions as in LEV-MIP preparation, but without levofloxacin. A PVC tube (1-2 cm long) was flattened and polished by placing it on a glass plate and soaking it with THF. Similar to the actual diameter of the PVC tubing, the membrane was then sliced and pasted onto the polished end. The other end of that was attached to an electrode of Ag-AgCl.

### Scanning Electron Microscope (SEM)

The SEM is used to get an understanding of the pore's membrane thickness, composition, and surface distribution. SEM analysis showed that molecular imprinted polymer has a strongly ordered and normal pore structure in surface and cross-section which serves as interface sites. Several papers have shown that due to the shape and function of the porous structures, the amolecular imprinted membrane of this type recognizes that the molecule of the template is quickly transported with good efficacy.

The morphology of MIP and non- imprinted polymer NIP membranes before and after washing showed by electron microscope in (Figure2).



(A)

(B)

**Figure (2):** SEM photograph of the surface of MIP, a) before washing b) after washing.

### Construction of Ion-Selective Electrodes

Construction of Ion-Selective Electrodes As shown by **Mahajanet al. (2007)**, electrode body building and immobilization have been achieved. The solution of Levofloxacin 0.1 M was filled as an internal solution in the glass tube. Membrane was preferred to be immersed in a standard solution of 0.1M levofloxacin for at least three hours before measurements representing membrane electrode stipulations.

### Preparation of Pharmaceutical Samples

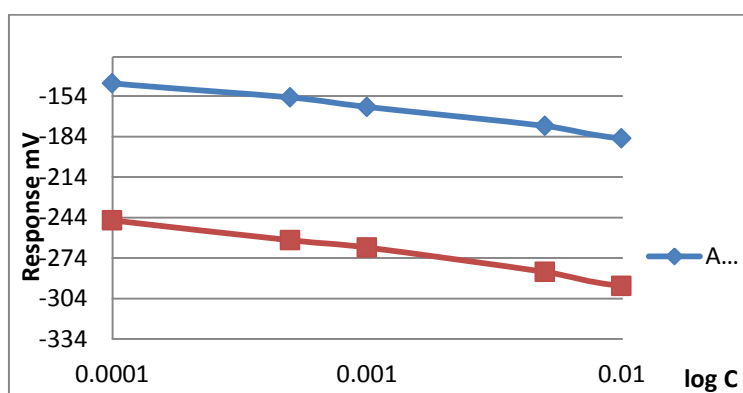
The drug tablets were ground to powder by using pestle and mortar. Subsequently, a required weight of the powder was used to prepare 100 mL solutions. Here, a certain amount of powder was dissolved in methanol (CH<sub>3</sub>OH) and stirred by magnetic stirrer for 30 minutes to completely dissolve the powder. The solution was completed to 100 mL by methanol to prepare 1×10<sup>-3</sup> M and 1×10<sup>-4</sup> M levofloxacin solutions.

## RESULTS AND DISCUSSION

MIP based liquid electrodes, their concentrations range and slopes response to Nernstian equation has been investigated. The membranes of MIP made of the monomer styrene with a PVC matrix using two plasticizers DMA and AOPH. The internal solution was used 0.1M aqueous standard solution of drug for all liquid electrodes. Experimental results of synthesis of molecularly imprinted (MIP) and non-imprinted polymers (NIP) based on monomer Styrene indicate that monomer can be used for the preparation of effective MIP for Levofloxacin. The plasticizer is an essential part of the sensing membrane which have important role as a solvent for the different components and determines the mobility of the analyte in membrane. Both of the plasticizers that are used, DMA and AOPH, are suitable for the fabrication of MIP-based LEV electrodes. (Table 1) show the parameters of the fabricated and tested electrodes, Two membranes of the different compositions were prepared using two different plasticizers Di methyl adepate (DMA) and acetophenone (AOPH). The results of electrode specification were obtained from the calibration curves that listed in (Table 1). The slopes of the electrodes ranged between  $-21.96 - -19.38$  mV/decade (Figure 3). In generally the preparation electrodes have a short response time (about 60 second) mostly at high concentrations.

**Table(1):** Levofloxacin-MIP electrode properties dependent on various functional monomers and plasticizers.

Membrane composition	LEV-MIP (DMA)	LEV-MIP (AOPH)
Slop (MV/decade)	-21.96	-19.38
correlation coefficients	0.9819	0.9872
detection limit (M)	$2 \times 10^{-4}$	$4 \times 10^{-4}$
range of linearity(M)	$5 \times 10^{-5} - 1 \times 10^{-1}$	$5 \times 10^{-5} - 1 \times 10^{-1}$
lifetime (day)	13days	14days



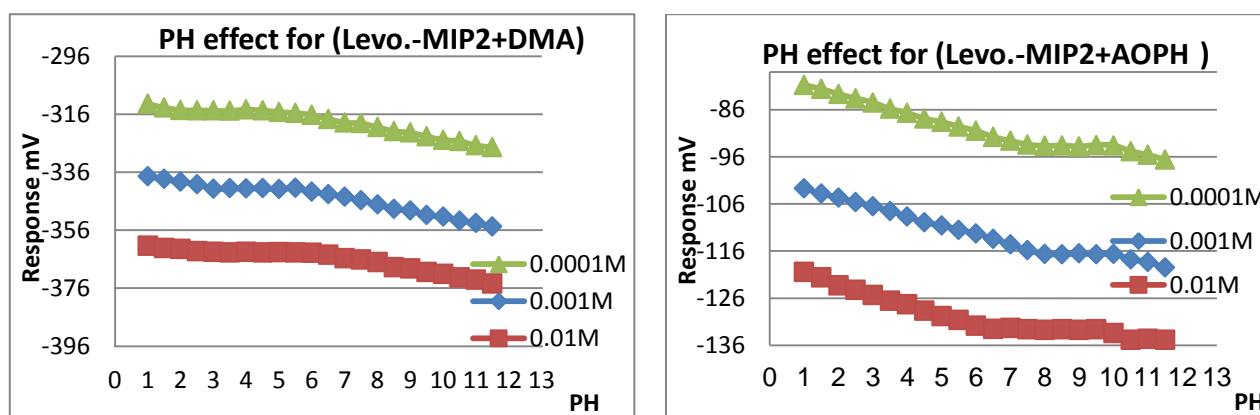
**Figure (3):** Calibration curve for Lev-MIP membrane electrodes.

### Effect of pH on electrode response

The pH effect of the two electrode potential values for pH varied from 2.5 to 10 was studied and the pH effect was modified by adding drops of 0.1 N HCl and 0.1 M NaOH to aqueous drug solutions and then the potentials obtained for each value were recorded. With three concentrations of standard drug solutions  $1 \times 10^{-2}$ ,  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  M, the pH effect on the electrode potential has been reported. The results obtained are shown in (Table 2) and the typical pattern of electrode potential versus pH for electrode M1 and M2 as seen in (Figure 4).

**Table(2):** Working pH range for levofloxacin selective electrode.

Number and composition of MIPs	Membranes	Composition of the membrane	pH range		
			$1 \times 10^{-2}$	$1 \times 10^{-3}$	$1 \times 10^{-4}$
MIP Lev+STY+N,NMDAM	M1	Lev-MIP+DMA	2.5-6.0	3.0-5.5	2.0-4.5
	M2	Lev-MIP+AOPH	6.5-9.5	8.0-10	7.5-10



**Figure (4):** Effect of pH on levofloxacin (Lev-MIP+DMA(M1))and(Lev-MIP+AOPH(M2)) at  $1 \times 10^{-2}$ ,  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  concentrations.

### Response time and life time

Response time for both MIP.DMA and MIP.AOPH electrodes was obtained from dynamic potential reaction at range of concentration  $5 \times 10^{-5}$  –  $1 \times 10^{-1}$  M by calculating the time needed to achieve 95 percent of the equilibrium potential. The findings show that the electrode reaction time was approximately 15 seconds for the solution of levofloxacin at a high concentration of  $10^{-1}$  M and approximately 46 seconds at a low concentration of  $10^{-5}$ M. The electrode lifetime was obtained by measuring the slope periodically from calibration curves for Lev. MIP. short life time was noticed for electrodes M1 and M2 (Table-1)

### Interference Studies

The separate Solution Method (SSM) has been used to test potentiometric sensor selectivity coefficients for different species. In the SSM, two separate solutions are used to evaluate the potential of a cell comprising an active electrode and a reference electrode: one containing drug ions, E1, and the other containing interference ion potential (E2), respectively. The coefficient of selectivity was determined using the following equation:

$$\text{Log } K_{\text{pot}} = (E_2 - E_1) / Z_1 F / 2.303 RT (1 - Z_1 / Z_2) \log a_1.$$

E1, E1; z1, z2; and a1, represents the potentials, charge numbers, and activities for the primary 1 and interfering 2 ions, respectively at a1=a2. Selectivity coefficient of the electrodes L1 and L2 were studied toward several different ions like ( $K^+$ ,  $Ca^{+2}$ ,  $Al^{+3}$ ). (Table3 and 4) (Figure5 and

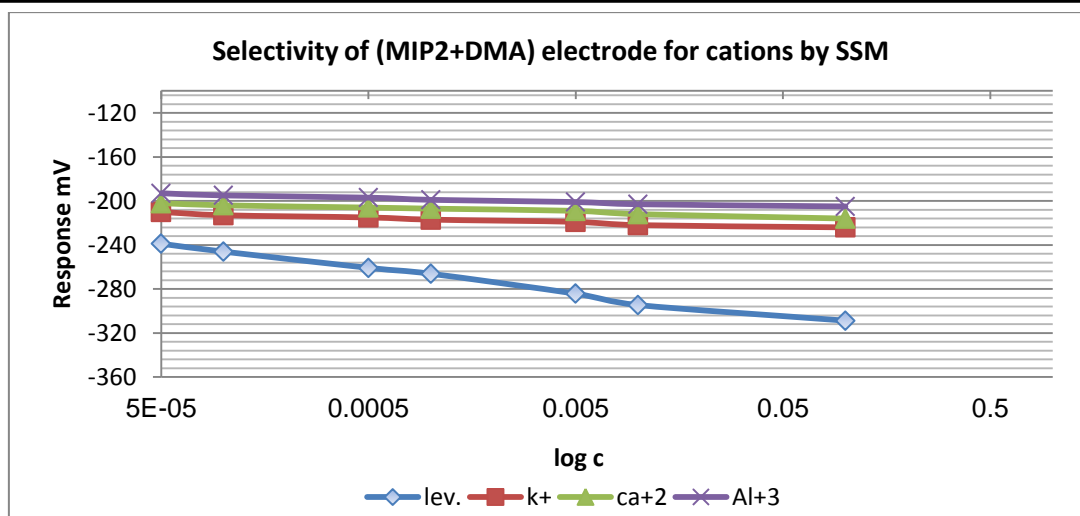
6) have provided both values for selectivity coefficients.

**Table (3):** Selectivity coefficients for (lev –MIP +DMA) electrode at different concentrations of levofloxacin.

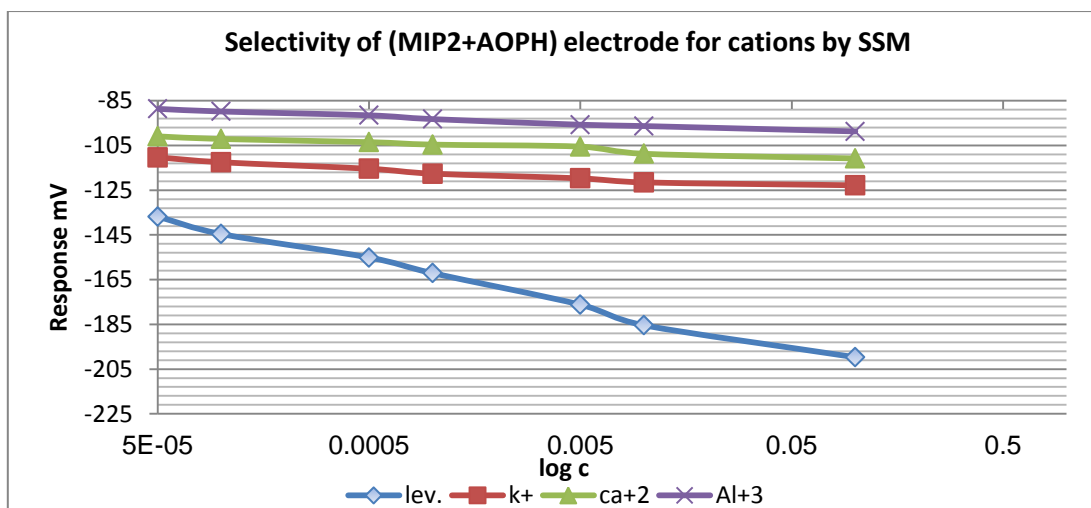
Con.	Concentrations of levofloxacin(M):concentrations of interference ions (M)					
	Interfering ions					
	K <sup>+1</sup>		Ca <sup>+2</sup>		Al <sup>+3</sup>	
	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>
10 <sup>-1</sup>	-224	1.3754×10 <sup>-4</sup>	-216	1.8799×10 <sup>-5</sup>	-205	4.0413×10 <sup>-6</sup>
10 <sup>-2</sup>	-222	4.9429×10 <sup>-4</sup>	-212	1.7323×10 <sup>-5</sup>	-203	3.1288×10 <sup>-6</sup>
5×10 <sup>-3</sup>	-219	1.0627×10 <sup>-3</sup>	-209	2.6366×10 <sup>-5</sup>	-201	4.7069×10 <sup>-6</sup>
1×10 <sup>-3</sup>	-217	5.7069×10 <sup>-3</sup>	-207	6.3707×10 <sup>-5</sup>	-199	8.7074×10 <sup>-6</sup>
5×10 <sup>-4</sup>	-215	8.2109×10 <sup>-3</sup>	-206	7.1541×10 <sup>-5</sup>	-197	7.8292×10 <sup>-6</sup>
1×10 <sup>-4</sup>	-213	3.1097×10 <sup>-2</sup>	-204	1.2103×10 <sup>-3</sup>	-195	1.0147×10 <sup>-5</sup>
5×10 <sup>-5</sup>	-210	4.8303×10 <sup>-2</sup>	-202	1.4776×10 <sup>-4</sup>	-193	1.1029×10 <sup>-5</sup>

**Table(4):**Selectivity coefficients for (lev– MIP+ AOPH) electrodeat differen tconcentration soflevofloxacin.

Con.	Concentrations of levofloxacin (M):concentrations of interference ions (M)					
	Interfering ions					
	K <sup>+1</sup>		Ca <sup>+2</sup>		Al <sup>+3</sup>	
	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>
10 <sup>-1</sup>	-122.8	1.0764×10 <sup>-4</sup>	-110.8	8.1808×10 <sup>-5</sup>	-98.7	1.3236×10 <sup>-6</sup>
10 <sup>-2</sup>	-121.5	5.0441×10 <sup>-4</sup>	-108.7	1.1023×10 <sup>-5</sup>	-96.3	1.1724×10 <sup>-6</sup>
5×10 <sup>-3</sup>	-119.7	1.2151×10 <sup>-3</sup>	-105.5	1.5918×10 <sup>-5</sup>	-95.7	2.0552×10 <sup>-6</sup>
1×10 <sup>-3</sup>	-117.6	5.0563×10 <sup>-3</sup>	-104.6	3.4121×10 <sup>-5</sup>	-93.2	2.7848×10 <sup>-6</sup>
5×10 <sup>-4</sup>	-115.4	8.9433×10 <sup>-3</sup>	-103.5	4.8692×10 <sup>-5</sup>	-91.5	3.2905×10 <sup>-6</sup>
1×10 <sup>-4</sup>	-112.5	2.2062×10 <sup>-2</sup>	-102.1	6.4124×10 <sup>-3</sup>	-89.8	3.2036×10 <sup>-5</sup>
5×10 <sup>-5</sup>	-110.3	4.2915×10 <sup>-2</sup>	-100.9	9.9443×10 <sup>-4</sup>	-88.6	4.4218×10 <sup>-5</sup>



**Figure(5):** Selectivity of (Lev-MIP + DMA) electrodes with ions via separation solution method.

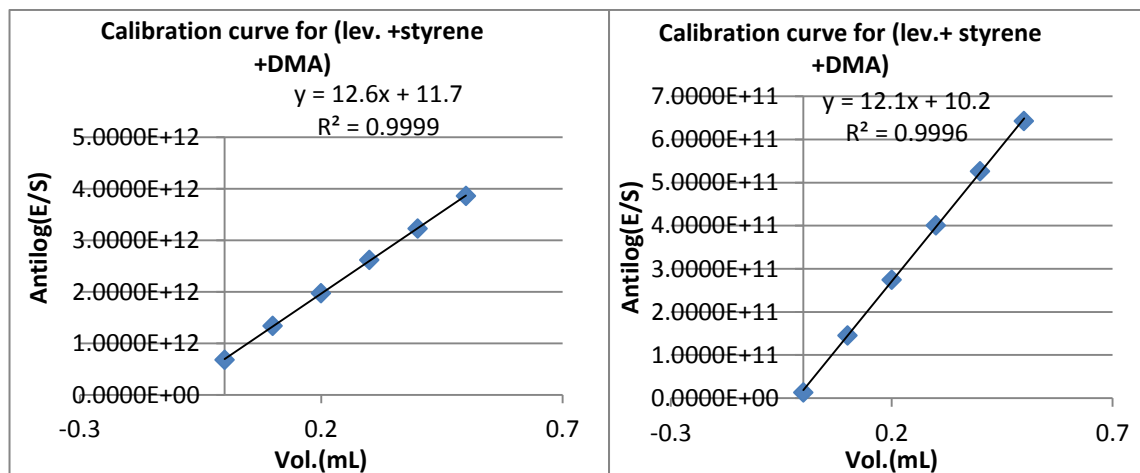


**Figure (6):** Selectivity of (Lev-MIP + AOPH) electrodes with ions via separation solution method.

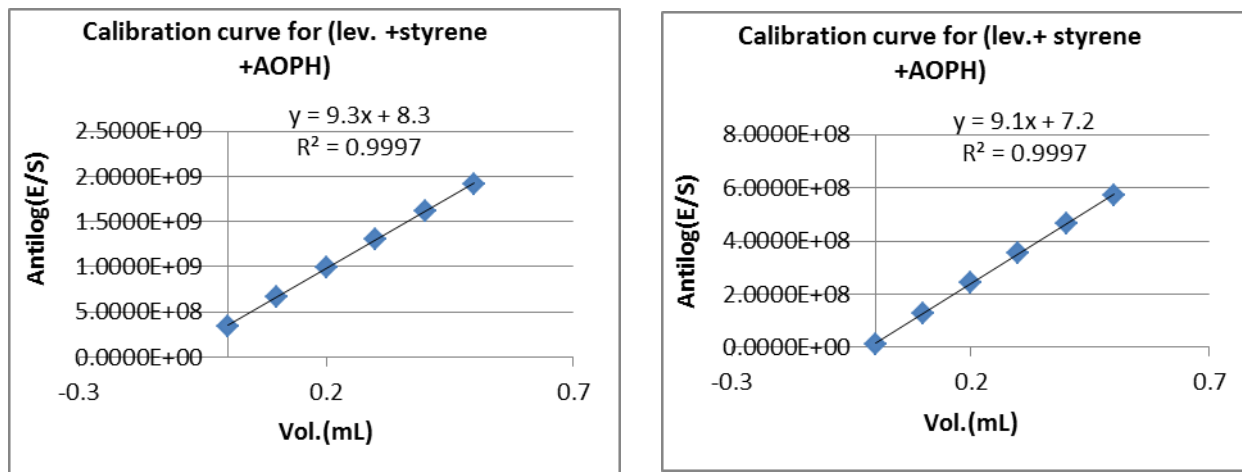
### Calculation using the Multiple Standard Addition Method (MSA)

The concentrations used in this process ( $1 \times 10^{-3}$  &  $1 \times 10^{-4}$ ) for two solutions of levofloxacin for plotting the antilog E / S (Y-) against the amount of regular levofloxacin (X-). (Figure 7 and 8) reflects the effects of the concentrations of levofloxacin determined by means of electrodes centered on Lev-MIP+ DMA, Lev-MIP+AOPH.





**Figure (7):** Antilog(E/S) against the volume of the added standard for the determination of Levofloxacin solution ( $1 \times 10^{-3}$  and  $1 \times 10^{-4}$ ) by MSA using (Lev-MIP+DMA) electrode.



**Figure (8):** Antilog(E/S) against the volume of the added standard for the determination of Levofloxacin solution ( $1 \times 10^{-3}$  and  $1 \times 10^{-4}$ ) by MSA using (Lev-MIP+AOPH) electrode.

### Titration Methods (Titrimetry)

These methods relied on the identification of the end point of the titration. Such techniques have been using volumetric analysis between the concentrations and the reactants often make certain shifts slowly, resulting in a significant shift in the electrode reaction. Titration between the levofloxacin and ligand phosphomolybic acid (PMA). The findings for parameters RSD percent, RC percent and RE percent for all electrodes are shown in (Table 5).

**Table (5):** Levofloxacin sample analyses by using titration method for Lev electrodes.

Electrode NO.	Concentration (M)		
	Measured using PMA as titrant		
Pure (LEV)	Parameter	DMA	AOPH
	$1 \times 10^{-3}$	$1.0198 \times 10^{-3}$	$1.012 \times 10^{-3}$
	RSD%	2.74	2.96
	RC%	101.98	102.14
	RE%	1.98	2.14
	$1 \times 10^{-4}$	$1.0208 \times 10^{-4}$	$1.022 \times 10^{-4}$
	RSD%	2.89	3.11
	RC%	102.08	102.25
	RE%	2.08	2.25
ILFLOX(IL KO- TURKEY )	$1 \times 10^{-3}$	$1.0302 \times 10^{-3}$	$1.025 \times 10^{-3}$
	RSD%	3.8	3.48
	RC%	103.02	102.52
	RE%	3.02	2.52
	$1 \times 10^{-4}$	$1.0230 \times 10^{-4}$	$1.027 \times 10^{-4}$
	RSD%	3.18	3.78
	RC%	102.30	102.74
	RE%	2.30	2.74

### Applications of pharmaceuticals

Ion sensitive electrodes based on molecularly imprinted polymers have been used for the determination of levofloxacin in pharmaceuticals. This ISE tests including: direct, standard addition, multiple standard and Gran plot. Levofloxacin preparation solutions at concentrations of  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  M. The RE percent, RC percent and RSD percent were measured for Levofloxacin in pharmaceuticals. The results obtained represented in the (Table 6 and 7).

### Table

**(6):** Determination of levofloxacin samples by ion selective electrodes (ISEs) techniques based on PVC membranes.

Electrode NO. and composition	Measurement by using ISEs methods				
LEV-MIP+ DMA (I)	Standard sample $1 \times 10^{-3}$				
	Parameter	RSD%	RC%	RE%	Con. found
	Direct	1.64	101.70	1.70	$1.0169 \times 10^{-3}$
	SAM	1.05	101.40	1.40	$1.003 \times 10^{-3}$
	MSA	-	100.71	0.71	$1.007 \times 10^{-3}$
	Standard sample $1 \times 10^{-4}$				
	Parameter	RSD%	%RC	RE%	Con. found
	Direct	0.43	101.62	1.62	$1.016 \times 10^{-4}$
	SAM	1.07	101.55	1.55	$1.0046 \times 10^{-4}$
	MSA	-	100.80	0.80	$1.0080 \times 10^{-4}$
LEV-MIP + AOPH (II)	Standard sample $1 \times 10^{-3}$				
	Parameter	RSD%	RC%	RE%	Con. found
	Direct	2.8	101.60	1.60	$1.01598 \times 10^{-3}$
	SAM	1.49	101.75	1.75	$1.0024 \times 10^{-3}$

	MSA	-	100.84	0.84	$1.0084 \times 10^{-3}$
	<b>Standard sample <math>1 \times 10^{-4}</math></b>				
	<b>Parameter</b>	<b>%RSD</b>	<b>%RC</b>	<b>%RE</b>	<b>Con. found</b>
	<b>Direct</b>	1.2	101.93	1.93	$1.019 \times 10^{-4}$
	<b>SAM</b>	1.22	101.55	1.55	$1.0031 \times 10^{-4}$
	<b>MSA</b>	-	100.80	0.80	$1.0080 \times 10^{-4}$

Table (7): Sample analysis of pharmaceuticals Levofloxacin by using ISE.

Membrane composition	LEV-MIP+DMA (ILFLOX -TURKEY)		
Pharmaceutical	D M	SA M	MSA M
Concentration ( prepared ) M	<b><math>1 \times 10^{-3}</math></b>		
Value founded	$1.0294 \times 10^{-3}$	$1.0055 \times 10^{-3}$	$1.0236 \times 10^{-3}$
Recovery %	102.94	101.98	101.23
RE%	2.94	1.98	1.23
RSD%	1.37	1.41	-
Concentration ( prepared ) M	<b><math>1 \times 10^{-4}</math></b>		
Value founded	$1.019 \times 10^{-4}$	$1.0276 \times 10^{-4}$	$1.0147 \times 10^{-4}$
Recovery %	101.85	101.64	101.47
RE%	1.85	1.64	1.47
RSD%	0.73	1.10	-
Membrane composition	LEV-MIP+AOPH (ILFLOX - TURKEY)		
Pharmaceutical	D M	SA M	MSA M
Concentration ( prepared ) M	<b><math>1 \times 10^{-3}</math></b>		
Value founded	$1.0229 \times 10^{-3}$	$1.0174 \times 10^{-3}$	$1.0146 \times 10^{-3}$
Recovery %	102.29	101.75	101.46
RE%	2.29	1.75	1.46
RSD%	3.1	1.49	-
Concentration ( prepared ) M	<b><math>1 \times 10^{-4}</math></b>		
Value founded	$1.023 \times 10^{-4}$	$1.0031 \times 10^{-4}$	$1.0129 \times 10^{-4}$
Recovery %	102.27	101.88	101.29
RE%	2.27	1.88	1.29
RSD%	3.7	1.34	-

\*Each calculation was repeated three times.

## CONCLUSION

Installation of molecularly imprinted electrode sensors (MIP) use levofloxacin as a template and Styrene as a monomer in various plasticizers. Excellent MIP tests with high sensitivity, reasonable selectivity, strong static reaction, long-term stability and applicability over a wide pH range have been obtained by using a DMA and AOPH plasticizer based electrode. The purpose of the construction electrodes to be used for the determination of Levofloxacin in the pharmaceutical analysis.

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## SYNTHESIS OF NEW LEVOFLOXACIN SELECTIVE MEMBRANE SENSOR BASED ON MOLECULARLY IMPRINTED POLYMERS.

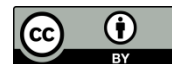
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### ABSTRACT

Two molecular imprinted polymer (MIP) membranes for Levofloxacin (LEV) were prepared based on PVC matrix. The imprinted polymers were prepared by polymerization of styrene (STY) as monomer, N,N methylene di acrylamide as a cross linker, benzoyl peroxide (BPO) as an initiator and levofloxacin as a template. Di methyl adepate (DMA) and acetophenone (AOPH) were used as plasticizers, the molecular imprinted membranes and the non molecular imprinted membranes were prepared. The slopes and detection limits of the liquid electrodes ranged from -21.96 to -19.38 mV/decade and  $2 \times 10^{-4}$  M to  $4 \times 10^{-4}$  M, and its response time was around 1 minute, respectively. The liquid electrodes were packed with 0.1 M standard drug solution and its response were stable at pH ranges from 1.0 to 11.0 and with good selectivity for more than several type. The electrodes produced have been successfully applied in preparation of the pharmaceutical sample for the determination of the analyte without any time consuming pretreatment steps.

**Keywords:** Molecularly impressed electrodes, levofloxacin, potentiometric method, styrene (STY) monomer.



## تخليق مستشعر الغشاء الانتقائي الليفوفلوكساسين الجديد على اساس بوليمرات مطبوعة جزيئياً

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## الخلاصة

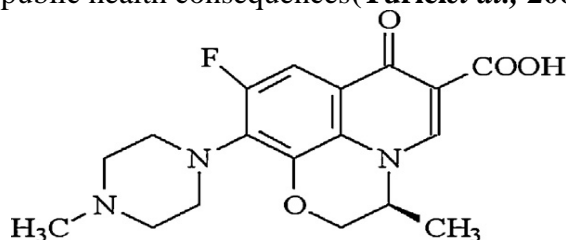
تم تحضير غشاءين من البوليمر المطبوع جزيئياً (MIP) للـ Levofloxacin بناءً على مصفوفة PVC. تم تحضير البوليمرات المطبوعة بواسطة بلمرة الستايرين (STY) كمواد مورو N، N-ميثيلين داي أكريلاميد كموصل متشابك، البنزويل بيروكسيد (BPO) كبادئ وليفوفلوكساسين كقالب. تم تحضير ثنائي ميثيل أديبات (DMA) وأستوفينون (AOPH) كمواد ملدنة، وتم تحضير الأغشية المطبوعة جزيئياً والأغشية غير المطبوعة. تراوحت حدود المنحدرات وحد الكشف للأقطاب السائلة من -19.38 - 21.96 مللي فولت/ عقد و  $4 \times 10^{-4} M$  -  $2 \times 10^{-4} M$ ، وكان وقت الاستجابة حوالي دقيقة واحدة على التوالي. تمت تعبئة الأقطاب الكهربائية السائلة بمحلول دوائي قياسي 0.1 M وكانت استجابتها مستقرة عند درجة الحموضة من 1.0 إلى 11.0 وابتقانية جيدة لأكثر من عدة أنواع. تم تطبيق الأقطاب الكهربائية المنتجة بنجاح في تحضير العينة الصيدلانية لتقدير المادة التحليلية بدون أي خطوات معالجة مسبقة تستغرق وقتاً طويلاً. الكلمات المفتاحية: الأقطاب المطبوعة جزيئياً، الليفوفلوكساسين، طريقة قياس الجهد، المونمر ستايرين (STY).

## INTRODUCTION

The first documented in the molecular imprinting technique in 1970 Nishide & Tsuchida (1976), molecular imprinting is a commonly emerging technique for producing polymers with different molecular properties for a given compound, its analogs, or an enantiomer (Whitcombe *et al.*, 1998; Yan & Row, 2006). MIPs are the sensing elements of a molecular imprinting electrochemical sensor (MIECS) which are molecularly imprinted. MIECS has many characteristics include: high selectivity, simplest techniques, lower costs, low D.L, highly stable. The combination of both the template molecules and the cavities can also be easily achieved and is carefully applicable even with harmful chemical materials. Thus, MIECS was used for optical and electrochemical application (Tadi & Motghare, 2016; Al-Bayati & Abd, 2017; Al-Bayati & Al-Safi, 2018; Momenh & Gholivand, 2018; D'Aurelio *et al.*, 2020). Molecularly imprint polymers (MIPs) are prepared By combining template molecule (LEV) with functional monomers styrene (STY), a cross-linker N,N-methylene diacrylamide (N,N MDAM) and initiator benzoyl peroxide (BPO) in appropriate solvent. Most of the time, Aprotic and unipolar solvents. So after polymerization, template molecule extraction reveals recognition cavities that complement the template molecule's shape, height, and chemical functionality, allowing the resulting polymers to selectively rebind the template molecule from a mixture of closely related compounds (Lavignacet *et al.*, 2004). In additional, the MIP have easily synthesis and have good mechanical properties, reliability for pressures and temperatures, and thus are cost-effectively and suitable to applicable for harmful chemicals (Li *et al.*, 2012, Al-Bayati & Aljabari, 2016; Zhang *et al.*, 2019). Interactions between template and monomer have been studied through spectral and computer simulation research. The utilization of molecular imprinting techniques have increased significantly in recent years, thus illustrating magnificently the ability of MIP model for detection toward the target molecules. (Bateset *et al.*, 2017; Al-Bayati, 2018; Marćet *et al.*, 2018). There Several analytical methods such as High performance liquid chromatography (HPLC) (Santoro *et al.*, 2006; Mahajanet *et al.*, 2007), chromatography (Mishal & Sober, 2005), fluorescence (Pratet *et al.*, 2006), mass spectrometry (Boxallet *et al.*, 2006) and potentiometric method were used for LEV determination in contrast. Such techniques are distinguished by a lengthy and complicated preparation of the sample for analysis, as well as costly instrumental equipment. In this study, a

new levofloxacin ion-selective membrane electrode (lev-MIP+DMA or AOPH) in the PVC membrane process was prepared.

LEV is a broad-spectrum racemic fluoroquinolone antimicrobial (Figure 1). It is the ofloxacin's active L-isomer, and its antibacterial efficacy is around two times that of ofloxacin, and are widely used today in both human and veterinary applications. Medicine as antibacterial agents of different diseases (Turiolet *et al.*, 2007). Over the past few years, the research on antibiotic residues in the multiple environmental compartments has been of great importance due to the biodiversity and public health consequences (Turiolet *et al.*, 2007).



**Figure (1):** Chemical structure of the levofloxacin.

## MATERIALS AND METHODS

Levofloxacin was purchased from company of drug manufacturing and medical supplies (IRAQ-SID-Samara). generic levofloxacin tablets purchased from local pharmacies are; Levoneer 10 tablets 500 mg from (Pioneer-Iraq) levofloxacin 7 tablets 750 mg from (Ilflox-Turkey). Di methyl adipate (DMA) and acetophenone (AOPH) also metal salts, they were obtained by Sigma-Aldrich and used as provided. Styrene (99%), monomer N,N-methylenediacrylamide (N,N-MDAM) (99%), and benzoyl peroxide (BPO) (78%) from Sigma-Aldrich is obtained. the chemical used was the largest purity reagent and was used when obtained without further purification.

### Apparatus

A digital voltmeter (HANA pH211 instrument Microprocessor pH meter) was used to perform potentiometric measurements. Digital pH meters (wissenschaftlich- Technische Werkstätten GmbH WTW pHmeter in lab pH720-Germany) were used for pH measurements. Electrode efficiency was investigated by calculating the potential of levofloxacin solutions at ambient temperature with a variety of concentrations from  $5 \times 10^{-4}$  M to  $10^{-1}$  M. Every solution was stirred, and the reading potential was recorded at equilibrium. The calibration curves obtained by plotting the response against the levofloxacin concentration logarithmic functions.

### Preparing of Standard Solutions for ISEs studies

For the preparation of a standard solution of 0.1 M levofloxacin by dissolving 3.614 g of standard levofloxacin in the methanol and completed to 100 mL in the volumetric flask. In the same procedure, the other solutions were prepared in 100 mL ranging from ( $5 \times 10^{-5}$  –  $10^{-1}$ ) M. The stock standard solution of  $1 \times 10^{-3}$  M  $1 \times 10^{-4}$  M phosphomolybdic acid was prepared by dissolving 1.1288 g, 0.11288 g from phosphomolybdic acid respectively in distilled water and completed for 100 mL. All interfering ions ( $K^{+}$ ,  $Ca^{+2}$  and  $Al^{+3}$ ) are preparing of 0.1 M. The other solutions at the range from ( $5 \times 10^{-5}$  –  $10^{-1}$ ) M were prepared in 100 mL.

### Synthesis of the Imprinted Polymer (MIP)

For the preparation of levofloxacin molecularly imprinted polymer (LEV-MIP), 1 mmol (0.34 g) of levofloxacin was mixed with 2 mmol (0.208 g) styrene as the monomer. After this, 10.6 mmol (1.634 g) N, N-methylene diacrylamide was added to the solution as the cross linker, followed by (0.3 g) benzoyl peroxide as the initiator. All these materials were subsequently dissolved in 5 mL mixture of methanol and chloroform, and the mixture was stirred for 5 minutes to obtain a homogenous solution. Afterwards, the gas  $N_2$  was passed through the



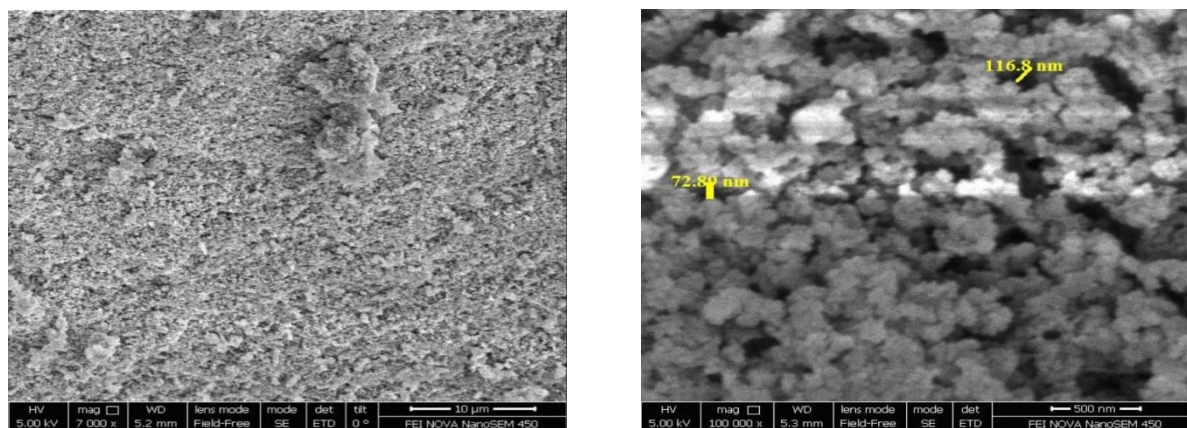
solution for 30 minutes to remove oxygen from it, and the solution was placed in a water bath at 65°C. When the reaction was complete, the molecularly imprinted polymer became hard, and, after the polymerization process, the polymer was dried and crushed to obtain it as particles. Finally, these particles were sonicated in CHCl<sub>3</sub>/ CH<sub>3</sub>OH / CH<sub>3</sub>COOH (7 : 2 : 1) (v/v/v) to remove the template from the MIP. The particles size of LEV- MIP (125 μm).

The preparation of non-molecularly imprinted polymers was carried out using the same method, using the same compounds and under the same conditions as in LEV-MIP preparation, but without levofloxacin. A PVC tube (1-2 cm long) was flattened and polished by placing it on a glass plate and soaking it with THF. Similar to the actual diameter of the PVC tubing, the membrane was then sliced and pasted onto the polished end. The other end of that was attached to an electrode of Ag-AgCl.

### Scanning Electron Microscope (SEM)

The SEM is used to get an understanding of the pore's membrane thickness, composition, and surface distribution. SEM analysis showed that molecular imprinted polymer has a strongly ordered and normal pore structure in surface and cross-section which serves as interface sites. Several papers have shown that due to the shape and function of the porous structures, the amolecular imprinted membrane of this type recognizes that the molecule of the template is quickly transported with good efficacy.

The morphology of MIP and non- imprinted polymer NIP membranes before and after washing showed by electron microscope in (Figure2).



(A)

(B)

**Figure (2):** SEM photograph of the surface of MIP, a) before washing b) after washing.

### Construction of Ion-Selective Electrodes

Construction of Ion-Selective Electrodes As shown by **Mahajanet al. (2007)**, electrode body building and immobilization have been achieved. The solution of Levofloxacin 0.1 M was filled as an internal solution in the glass tube. Membrane was preferred to be immersed in a standard solution of 0.1M levofloxacin for at least three hours before measurements representing membrane electrode stipulations.

### Preparation of Pharmaceutical Samples

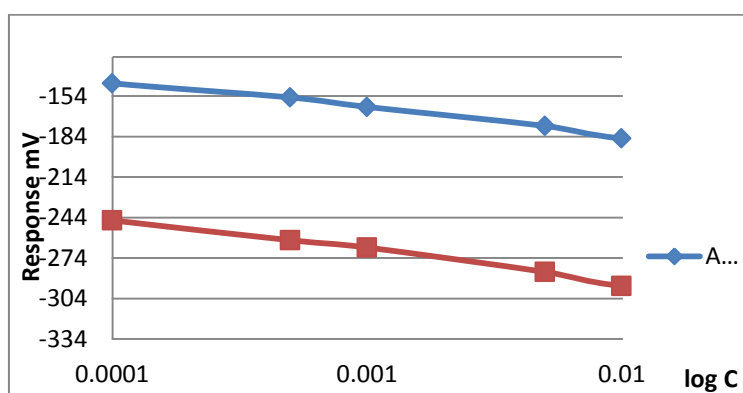
The drug tablets were ground to powder by using pestle and mortar. Subsequently, a required weight of the powder was used to prepare 100 mL solutions. Here, a certain amount of powder was dissolved in methanol (CH<sub>3</sub>OH) and stirred by magnetic stirrer for 30 minutes to completely dissolve the powder. The solution was completed to 100 mL by methanol to prepare 1×10<sup>-3</sup> M and 1×10<sup>-4</sup> M levofloxacin solutions.

## RESULTS AND DISCUSSION

MIP based liquid electrodes, their concentrations range and slopes response to Nernstian equation has been investigated. The membranes of MIP made of the monomer styrene with a PVC matrix using two plasticizers DMA and AOPH. The internal solution was used 0.1M aqueous standard solution of drug for all liquid electrodes. Experimental results of synthesis of molecularly imprinted (MIP) and non-imprinted polymers (NIP) based on monomer Styrene indicate that monomer can be used for the preparation of effective MIP for Levofloxacin. The plasticizer is an essential part of the sensing membrane which have important role as a solvent for the different components and determines the mobility of the analyte in membrane. Both of the plasticizers that are used, DMA and AOPH, are suitable for the fabrication of MIP-based LEV electrodes. (Table 1) show the parameters of the fabricated and tested electrodes, Two membranes of the different compositions were prepared using two different plasticizers Di methyl adepate (DMA) and acetophenone (AOPH). The results of electrode specification were obtained from the calibration curves that listed in (Table 1). The slopes of the electrodes ranged between  $-21.96 - -19.38$  mV/decade (Figure 3). In generally the preparation electrodes have a short response time (about 60 second) mostly at high concentrations.

**Table(1):** Levofloxacin-MIP electrode properties dependent on various functional monomers and plasticizers.

Membrane composition	LEV-MIP (DMA)	LEV-MIP (AOPH)
Slop (MV/decade)	-21.96	-19.38
correlation coefficients	0.9819	0.9872
detection limit (M)	$2 \times 10^{-4}$	$4 \times 10^{-4}$
range of linearity(M)	$5 \times 10^{-5} - 1 \times 10^{-1}$	$5 \times 10^{-5} - 1 \times 10^{-1}$
lifetime (day)	13days	14days



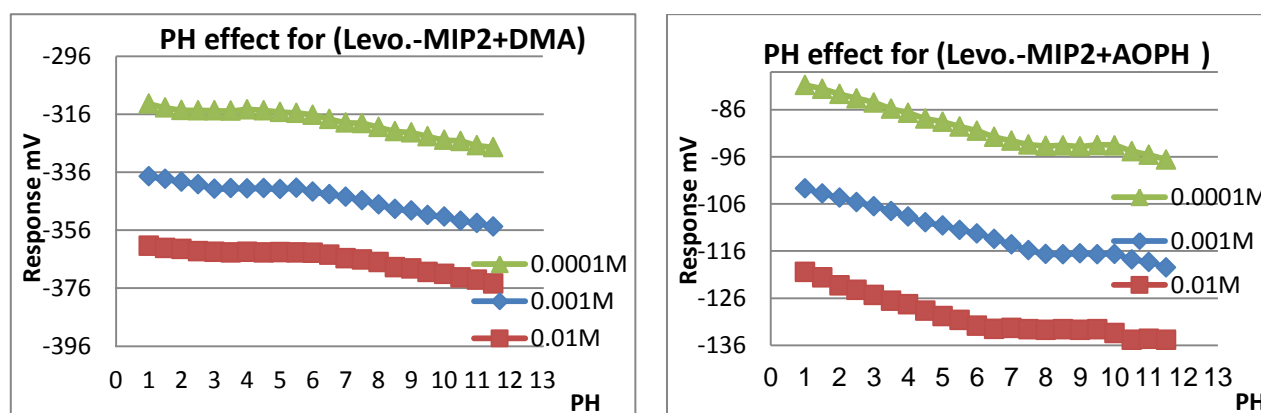
**Figure (3):** Calibration curve for Lev-MIP membrane electrodes.

### Effect of pH on electrode response

The pH effect of the two electrode potential values for pH varied from 2.5 to 10 was studied and the pH effect was modified by adding drops of 0.1 N HCl and 0.1 M NaOH to aqueous drug solutions and then the potentials obtained for each value were recorded. With three concentrations of standard drug solutions  $1 \times 10^{-2}$ ,  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  M, the pH effect on the electrode potential has been reported. The results obtained are shown in (Table 2) and the typical pattern of electrode potential versus pH for electrode M1 and M2 as seen in (Figure 4).

**Table(2):** Working pH range for levofloxacin selective electrode.

Number and composition of MIPs	Membranes	Composition of the membrane	pH range		
			$1 \times 10^{-2}$	$1 \times 10^{-3}$	$1 \times 10^{-4}$
MIP Lev+STY+N,NMDAM	M1	Lev-MIP+DMA	2.5-6.0	3.0-5.5	2.0-4.5
	M2	Lev-MIP+AOPH	6.5-9.5	8.0-10	7.5-10



**Figure (4):** Effect of pH on levofloxacin (Lev-MIP+DMA(M1))and(Lev-MIP+AOPH(M2)) at  $1 \times 10^{-2}$ ,  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  concentrations.

### Response time and life time

Response time for both MIP.DMA and MIP.AOPH electrodes was obtained from dynamic potential reaction at range of concentration  $5 \times 10^{-5}$  –  $1 \times 10^{-1}$  M by calculating the time needed to achieve 95 percent of the equilibrium potential. The findings show that the electrode reaction time was approximately 15 seconds for the solution of levofloxacin at a high concentration of  $10^{-1}$  M and approximately 46 seconds at a low concentration of  $10^{-5}$ M. The electrode lifetime was obtained by measuring the slope periodically from calibration curves for Lev. MIP. short life time was noticed for electrodes M1 and M2 (Table-1)

### Interference Studies

The separate Solution Method (SSM) has been used to test potentiometric sensor selectivity coefficients for different species. In the SSM, two separate solutions are used to evaluate the potential of a cell comprising an active electrode and a reference electrode: one containing drug ions, E1, and the other containing interference ion potential (E2), respectively. The coefficient of selectivity was determined using the following equation:

$$\text{Log } K_{\text{pot}} = (E_2 - E_1) / Z_1 F / 2.303 RT (1 - Z_1 / Z_2) \log a_1.$$

E1, E1; z1, z2; and a1, represents the potentials, charge numbers, and activities for the primary 1 and interfering 2 ions, respectively at a1=a2. Selectivity coefficient of the electrodes L1 and L2 were studied toward several different ions like ( $K^+$ ,  $Ca^{+2}$ ,  $Al^{+3}$ ). (Table3 and 4) (Figure5 and

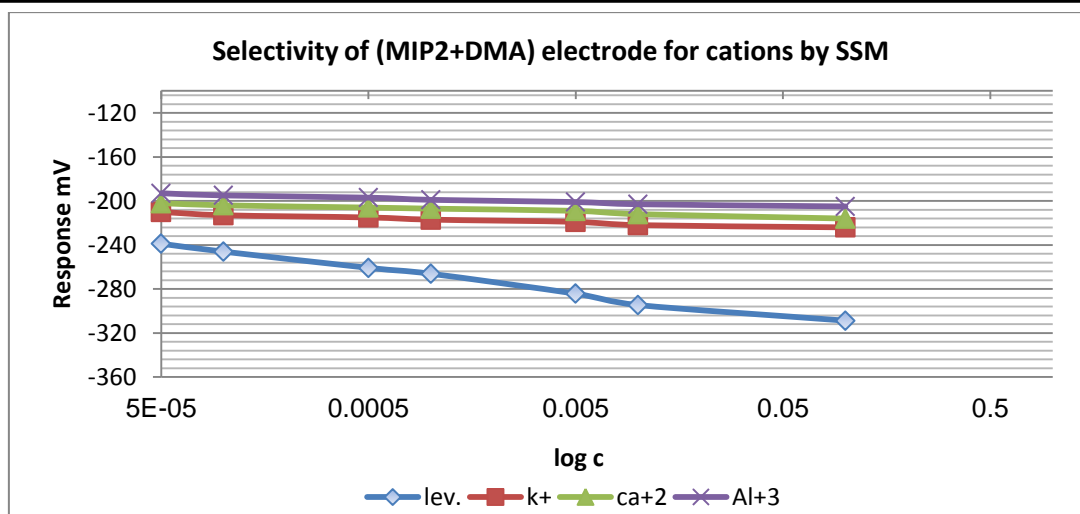
6) have provided both values for selectivity coefficients.

**Table (3):** Selectivity coefficients for (lev –MIP +DMA) electrode at different concentrations of levofloxacin.

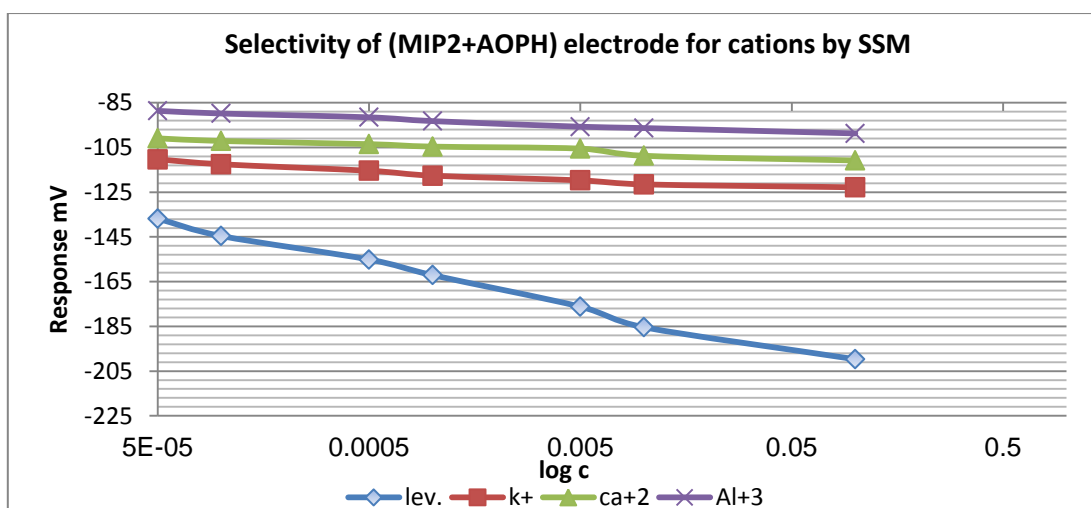
Con.	Concentrations of levofloxacin(M):concentrations of interference ions (M)					
	Interfering ions					
	K <sup>+1</sup>		Ca <sup>+2</sup>		Al <sup>+3</sup>	
	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>
10 <sup>-1</sup>	-224	1.3754×10 <sup>-4</sup>	-216	1.8799×10 <sup>-5</sup>	-205	4.0413×10 <sup>-6</sup>
10 <sup>-2</sup>	-222	4.9429×10 <sup>-4</sup>	-212	1.7323×10 <sup>-5</sup>	-203	3.1288×10 <sup>-6</sup>
5×10 <sup>-3</sup>	-219	1.0627×10 <sup>-3</sup>	-209	2.6366×10 <sup>-5</sup>	-201	4.7069×10 <sup>-6</sup>
1×10 <sup>-3</sup>	-217	5.7069×10 <sup>-3</sup>	-207	6.3707×10 <sup>-5</sup>	-199	8.7074×10 <sup>-6</sup>
5×10 <sup>-4</sup>	-215	8.2109×10 <sup>-3</sup>	-206	7.1541×10 <sup>-5</sup>	-197	7.8292×10 <sup>-6</sup>
1×10 <sup>-4</sup>	-213	3.1097×10 <sup>-2</sup>	-204	1.2103×10 <sup>-3</sup>	-195	1.0147×10 <sup>-5</sup>
5×10 <sup>-5</sup>	-210	4.8303×10 <sup>-2</sup>	-202	1.4776×10 <sup>-4</sup>	-193	1.1029×10 <sup>-5</sup>

**Table(4):**Selectivity coefficients for (lev– MIP+ AOPH) electrodeat differen tconcentration soflevofloxacin.

Con.	Concentrations of levofloxacin (M):concentrations of interference ions (M)					
	Interfering ions					
	K <sup>+1</sup>		Ca <sup>+2</sup>		Al <sup>+3</sup>	
	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>	E <sub>B</sub> (mv)	K <sub>A,B</sub>
10 <sup>-1</sup>	-122.8	1.0764×10 <sup>-4</sup>	-110.8	8.1808×10 <sup>-5</sup>	-98.7	1.3236×10 <sup>-6</sup>
10 <sup>-2</sup>	-121.5	5.0441×10 <sup>-4</sup>	-108.7	1.1023×10 <sup>-5</sup>	-96.3	1.1724×10 <sup>-6</sup>
5×10 <sup>-3</sup>	-119.7	1.2151×10 <sup>-3</sup>	-105.5	1.5918×10 <sup>-5</sup>	-95.7	2.0552×10 <sup>-6</sup>
1×10 <sup>-3</sup>	-117.6	5.0563×10 <sup>-3</sup>	-104.6	3.4121×10 <sup>-5</sup>	-93.2	2.7848×10 <sup>-6</sup>
5×10 <sup>-4</sup>	-115.4	8.9433×10 <sup>-3</sup>	-103.5	4.8692×10 <sup>-5</sup>	-91.5	3.2905×10 <sup>-6</sup>
1×10 <sup>-4</sup>	-112.5	2.2062×10 <sup>-2</sup>	-102.1	6.4124×10 <sup>-3</sup>	-89.8	3.2036×10 <sup>-5</sup>
5×10 <sup>-5</sup>	-110.3	4.2915×10 <sup>-2</sup>	-100.9	9.9443×10 <sup>-4</sup>	-88.6	4.4218×10 <sup>-5</sup>



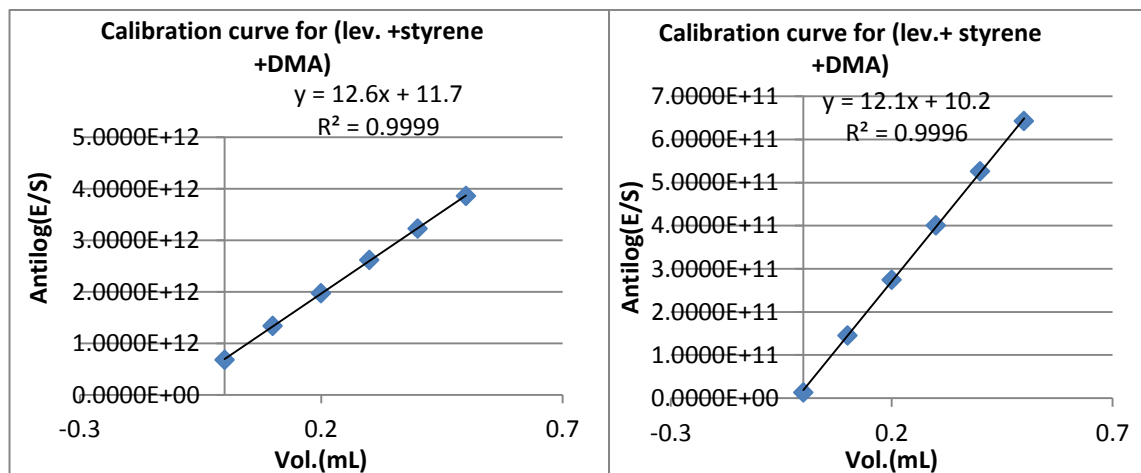
**Figure(5):** Selectivity of (Lev-MIP + DMA) electrodes with ions via separation solution method.



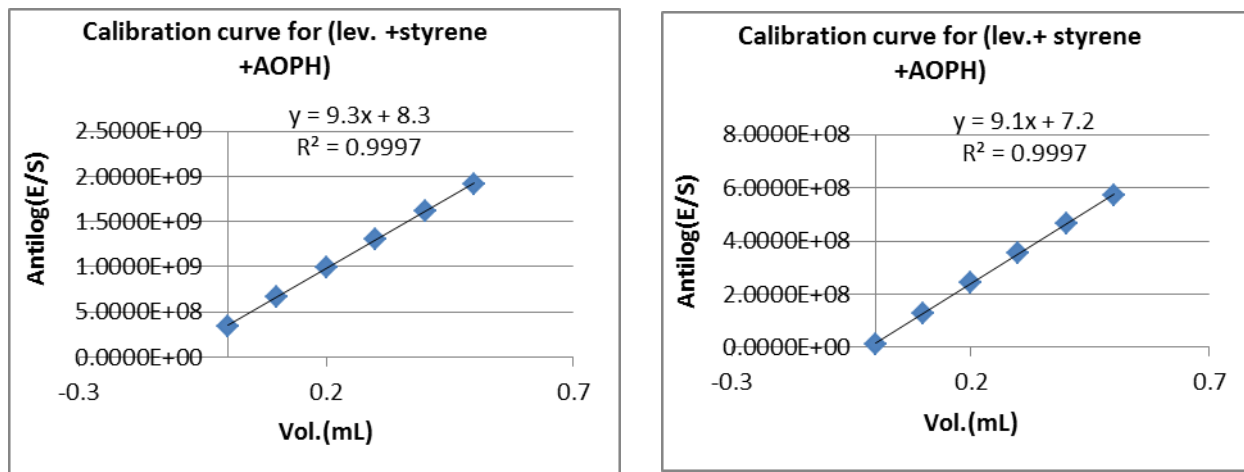
**Figure (6):** Selectivity of (Lev-MIP + AOPH) electrodes with ions via separation solution method.

### Calculation using the Multiple Standard Addition Method (MSA)

The concentrations used in this process ( $1 \times 10^{-3}$  &  $1 \times 10^{-4}$ ) for two solutions of levofloxacin for plotting the antilog E / S (Y-) against the amount of regular levofloxacin (X-). (Figure 7 and 8) reflects the effects of the concentrations of levofloxacin determined by means of electrodes centered on Lev-MIP+ DMA, Lev-MIP+AOPH.



**Figure (7):**Antilog(E/S)againstthevolumeoftheaddestandardforthedeterminationofLevofloxacin solution( $1 \times 10^{-3}$ and $1 \times 10^{-4}$ )by MSAusing(Lev–MIP+DMA)electrode.



**Figure (8):**Antilog(E/S)againstthevolumeoftheaddestandardforthedeterminationofLevofloxacin solution( $1 \times 10^{-3}$ and $1 \times 10^{-4}$ )by MSAusing(Lev–MIP+AOPH)electrode.

### Titration Methods (Titrimetry)

These methods relied on the identification of the end point of the titration. Such techniques have been using volumetric analysis between the concentrations and the reactants often make certain shifts slowly, resulting in a significant shift in the electrode reaction. Titration between the levofloxacin and ligand phosphomolybic acid (PMA). The findings for parameters RSD percent, RC percent and RE percent for all electrodes are shown in (Table 5).

**Table (5):** Levofloxacin sample analyses by using titration method for Lev electrodes.

Electrode NO.	Concentration (M)		
	Measured using PMA as titrant		
Pure (LEV)	Parameter	DMA	AOPH
	$1 \times 10^{-3}$	$1.0198 \times 10^{-3}$	$1.012 \times 10^{-3}$
	RSD%	2.74	2.96
	RC%	101.98	102.14
	RE%	1.98	2.14
	$1 \times 10^{-4}$	$1.0208 \times 10^{-4}$	$1.022 \times 10^{-4}$
	RSD%	2.89	3.11
	RC%	102.08	102.25
	RE%	2.08	2.25
ILFLOX(IL KO- TURKEY )	$1 \times 10^{-3}$	$1.0302 \times 10^{-3}$	$1.025 \times 10^{-3}$
	RSD%	3.8	3.48
	RC%	103.02	102.52
	RE%	3.02	2.52
	$1 \times 10^{-4}$	$1.0230 \times 10^{-4}$	$1.027 \times 10^{-4}$
	RSD%	3.18	3.78
	RC%	102.30	102.74
	RE%	2.30	2.74

### Applications of pharmaceuticals

Ion sensitive electrodes based on molecularly imprinted polymers have been used for the determination of levofloxacin in pharmaceuticals. This ISE tests including: direct, standard addition, multiple standard and Gran plot. Levofloxacin preparation solutions at concentrations of  $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  M. The RE percent, RC percent and RSD percent were measured for Levofloxacin in pharmaceuticals. The results obtained represented in the (Table 6 and 7).

### Table

**(6):** Determination of levofloxacin samples by ion selective electrodes (ISEs) techniques based on PVC membranes.

Electrode NO. and composition	Measurement by using ISEs methods				
LEV-MIP+ DMA (I)	Standard sample $1 \times 10^{-3}$				
	Parameter	RSD%	RC%	RE%	Con. found
	Direct	1.64	101.70	1.70	$1.0169 \times 10^{-3}$
	SAM	1.05	101.40	1.40	$1.003 \times 10^{-3}$
	MSA	-	100.71	0.71	$1.007 \times 10^{-3}$
	Standard sample $1 \times 10^{-4}$				
	Parameter	RSD%	%RC	RE%	Con. found
	Direct	0.43	101.62	1.62	$1.016 \times 10^{-4}$
	SAM	1.07	101.55	1.55	$1.0046 \times 10^{-4}$
	MSA	-	100.80	0.80	$1.0080 \times 10^{-4}$
LEV-MIP + AOPH (II)	Standard sample $1 \times 10^{-3}$				
	Parameter	RSD%	RC%	RE%	Con. found
	Direct	2.8	101.60	1.60	$1.01598 \times 10^{-3}$
	SAM	1.49	101.75	1.75	$1.0024 \times 10^{-3}$

	MSA	-	100.84	0.84	$1.0084 \times 10^{-3}$
	<b>Standard sample <math>1 \times 10^{-4}</math></b>				
	<b>Parameter</b>	<b>%RSD</b>	<b>%RC</b>	<b>%RE</b>	<b>Con. found</b>
	<b>Direct</b>	1.2	101.93	1.93	$1.019 \times 10^{-4}$
	<b>SAM</b>	1.22	101.55	1.55	$1.0031 \times 10^{-4}$
	<b>MSA</b>	-	100.80	0.80	$1.0080 \times 10^{-4}$

Table (7): Sample analysis of pharmaceuticals Levofloxacin by using ISE.

Membrane composition	LEV-MIP+DMA (ILFLOX -TURKEY)		
Pharmaceutical	D M	SA M	MSA M
Concentration ( prepared ) M	<b><math>1 \times 10^{-3}</math></b>		
Value founded	$1.0294 \times 10^{-3}$	$1.0055 \times 10^{-3}$	$1.0236 \times 10^{-3}$
Recovery %	102.94	101.98	101.23
RE%	2.94	1.98	1.23
RSD%	1.37	1.41	-
Concentration ( prepared ) M	<b><math>1 \times 10^{-4}</math></b>		
Value founded	$1.019 \times 10^{-4}$	$1.0276 \times 10^{-4}$	$1.0147 \times 10^{-4}$
Recovery %	101.85	101.64	101.47
RE%	1.85	1.64	1.47
RSD%	0.73	1.10	-
Membrane composition	LEV-MIP+AOPH (ILFLOX - TURKEY)		
Pharmaceutical	D M	SA M	MSA M
Concentration ( prepared ) M	<b><math>1 \times 10^{-3}</math></b>		
Value founded	$1.0229 \times 10^{-3}$	$1.0174 \times 10^{-3}$	$1.0146 \times 10^{-3}$
Recovery %	102.29	101.75	101.46
RE%	2.29	1.75	1.46
RSD%	3.1	1.49	-
Concentration ( prepared ) M	<b><math>1 \times 10^{-4}</math></b>		
Value founded	$1.023 \times 10^{-4}$	$1.0031 \times 10^{-4}$	$1.0129 \times 10^{-4}$
Recovery %	102.27	101.88	101.29
RE%	2.27	1.88	1.29
RSD%	3.7	1.34	-

\*Each calculation was repeated three times.

## CONCLUSION

Installation of molecularly imprinted electrode sensors (MIP) use levofloxacin as a template and Styrene as a monomer in various plasticizers. Excellent MIP tests with high sensitivity, reasonable selectivity, strong static reaction, long-term stability and applicability over a wide pH range have been obtained by using a DMA and AOPH plasticizer based electrode. The purpose of the construction electrodes to be used for the determination of Levofloxacin in the pharmaceutical analysis.

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## VISUALCONTAMINATION WITHRANDOMADVERTISEMENTS AND THE POSSIBILITY OF APPLYING FINES TO THEIR OWNERS

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### ABSTRACT

Baghdad governorate has many areas of distinctive architectural and architectural character, which are heritage and valuable areas that we must preserve and take care of. But we see many manifestations of it, which have a negative impact on buildings, areas and roads, so that they distort the view and thus lead to visual pollution in general. The research examined the visual pollution from random advertising, which stretched buildings, walls, electricity poles and sidewalks. The study covered different areas of al-Karkh and al-Rassafa (Jadreja Bridge, Nation Square, Jordan Square, alkindy Street) Most of the distortions were the result of non-removable posters, Handwriting, election candidate advertisements or repeated advertisements. Then we could be reached to the advertis by the phone shown in the advertisement, the location of the advertiser or the advertiser's character and the imposition of financial fines for the purpose of removing the advertisement and not repeating it in the future.

**Keywords:** Political, paper, medical, deformity.



## التلوث البصري بالإعلانات العشوائية وإمكانية تطبيق الغرامات على أصحابها

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## الخلاصة

تمتلك محافظة بغداد العديد من المناطق التي تتميز بالطابع المعماري والعمراني المميز والتي تعد من المناطق التراثية وذات القيمة التي يجب أن نحافظ عليها ونعتني بها، ولكننا نرى العديد من مظاهر التجاوزات عليها والتي تنعكس سلباً على المباني والمناطق والطرق بحيث تؤدي إلى تشويه المنظر وبالتالي تؤدي إلى التلوث البصري بشكل عام وتم في البحث دراسة التلوث البصري الناتج من الإعلانات العشوائية والذي طال المباني والجدران وأعمدة الكهرباء وأرصفة الطرقات حيث شملت الدراسة مناطق مختلفة من جانبي الكرخ والرصافة (جسر الجادرية، ساحة الأمة، ساحة الأردن، شارع الكندي) وكانت أغلب التشوهات ناتجة عن ملصقات غير قابلة للإزالة وكتابات بقتاني الأصباغ وإعلانات لمرشحي الانتخابات أو إعلانات متكررة ومن الممكن الوصول إلى الجهة المعلنة عن طريق الهاتف الموضح في الإعلان ومكان المعلن أو شخصية المعلن وفرض الغرامات المالية لغرض إزالة الإعلان وعدم تكرار ذلك مستقبلاً.

الكلمات المفتاحية: سياسي، ورقي، طبي، تشوه.

## INTRODECTION

## 1. METHODOLOGICAL SIDE

**The problem of research**

Study of visual pollution as a type of environmental pollution resulting from random declarations in Baghdad province that extended all of the streets from power poles, sidewalks, bus stop, etc.

**The importance of research**

The importance of research lies in preserving the architectural and architectural character of the city of Baghdad, which is a heritage area when advertising.

**The goal**

The possibility of applying fines to advertisers through the phone registered in the advertisement, the location of the advertiser and the character of the advertiser.

**Sample Study**

The study covered different areas of the Karkh and Al-Rassfa (Jadreya Bridge, Nation Square, Jordan Square, alkindy Street).

## 2. THEORETICAL SIDE

**What is advertising and what types of advertising**

The advertisement is one of the most important means used by producers and market people to reach the buyer or consumer in the simplest and most common way. One of the best ways to influence a person's attitude and change direction is to communicate the information and concepts you want to them to the hearing and vision, because they have a magical effect on the mind of the audience and the speed with which they change. As we see it, the media, both visual and audio, are creating different views and orientations for the public at a speed that has not been preceded by previous methods used to influence the target audience (Azmmour, 2011).

The declaration can be defined simply as the various visual and audible activities and methods used to spread the sold product or service offered. To communicate information and features to the customer in a simplified and easy way that is consistent with their trends, trends and purposes and motivates them to buy this product or get service and the promotion and advertising process is supposed to be preceded by several other things:

1. Selection the item to be sold to the consumer: Before you start doing anything, and before making any decision about the marketing or sales process, you must first identify and

advertise the goods or service that is to be offered to the consumer and then proceed to market.

2. Product promotion: Once we have selected and identified the goods that will be offered to consumers, we will move to the second stage, which is how this product will be marketed and what means it will depend on to promote the product and stimulate consumer buying, so we must take a number of steps:

1. Public road survey and surveys to learn about trends and trends in consumers and what they like and what they like about the product to be launched, and the companies used to do this survey by roving delegates on the roads to hold dialogs with people. However, this method has recently been replaced by the work of online surveys, as they have received more ease and speed of reaching the target audience as well as providing good money. This method helps the commodity producer determine what aspects and things the audience likes and dislikes in advertising products and processes to avoid negative and set pros and product advantages so that the audience will be very impressed and therefore drive sales to this product, Not only is the buyer's quality to be known at this stage, but what is the daily consumer's needs, income level, and purchasing motivation to identify a consumer value product that is consistent with its purposes and objectives and one of its basic needs to ensure that this product achieves high sales. Individual behavior plays an important role in understanding it as the basis for the right marketing of a successful product, and it should be considered for the effects that affect the consumer in it, which is expressed in its hobby, and also what the primary consumer's motives for buying mean what motivates the person to make a purchase any brand or store in general, selective buying motives are the drivers that lead the consumer to buy from a particular brand or to select a particular shop from other stores or brands that sell the same product. When the buyer arrives at this stage, the buyer has trusted that brand or shop or is emotionally associated with it.

2. Research indicates that the senses in the human body are raised faster and better influenced in order to determine the type of advertising to be published for the product. Will it be audio or visual propaganda or Monday, and research and psychological studies at this point indicate that the visual effects greatly affect the emotions of man, his response to and the excitement with him, as well as the sound effects that create an atmosphere of sympathy, especially if sound is familiar to the target. Advertisers may use celebrities or football players in advertising work for their popular public outlandish intimacy and encourage consumers to decide and buy this product because of the confidence they have gained from seeing its favorite star plus the emotional atmosphere they have gained toward the product when his favorite character view appears in it (Al-Hwerini,2018).

### **Media means of advertising**

At this stage, emphasis is placed on the media through which advertising for the product will be disseminated, with multiple media tools and methods that define the product to the consumer, and the best advertising method, appropriate and appropriate for the product, should be used to reach the target audience with the lowest costs, expenses, the most cost-effective methods, and the most return possible, and the ad will be divided according to the target To:

1. an educational announcement: The kind of advertising associated with marketing new products that have never been on the market before, or old and used goods, but for which new uses and purposes have appeared and have never been mentioned before.
2. News announcement: This type of advertising is linked to new services or goods that everyone knows, without sufficient information to allow them to apply for or purchase. It is a demonstration and relies on the explanation of product information in the ad in a concise,

concise and concise manner that identifies the consumer with respect to the product or service, but in a simplified and concise manner.

3. Media advertising: This type of advertising is intended to promote a product or commodity in order to strengthen the industry, produce the product, and create a popular consumer for this product, in which words or things are placed in the advertising that creates a kind of passion between the consumer and the producer (Tarek, 2017), the advertising is for the target audience divided into:
  1. This is intended for traders and wholesalers who make and direct the advertising to those distributors who then put it in a final form at stores and other outlets.
  2. an agricultural declaration: This declaration is for farmers and landowners, where seeds and fertilizers are presented in this and other declarations.
  3. introductory statement: The advertising for the product is directed to the end-user and the normal consumer who buys the product to satisfy his or her needs and desires, the primary consumer for which the advertising is made and for which the product is made (Tarek, 2017).

### Visual pollution

Visual pollution is a term called unattractive optical elements. They are landscapes or anything else that a person wants not to look at as examples of bad paintings, rubbish, some walls, unstudied buildings, unstructured architecture, signs, grass, random advertisements, or in other words, a distortion of any eye view that is perceived to be uneasy to myself and to us he also described it as a kind of artistic insecurity or the disappearance of the esthetic image of everything surrounding us from buildings to roads, sidewalks, streets, public squares and others (Abdulrazaaq, 2017). One of the most important sources of visual pollution in general, which is part of environmental pollution, is the lack of cleanliness and the proliferation of empty cans in the streets, paper and plastic waste, as well as damaged and damaged cars scattered in the streets, in front of houses, workshops and repair shops, which have expired and need to be recycled and remanufactured, street excavations and abandoned dumps the unneeded, random bumps that destroyed cars and caused alarm for passengers, as well as incomplete governmental and non-governmental installations, the neglected construction that turned into containers for big waste and houses for random housing. Also, the debris and debris of the construction were scattered randomly, and the swamps were found in some quarters because of the leakage of water from homes, as well as the spread of internet towers and mobile phones between one house and another and other visual pollution. What we mean in our research is signs and posters (repeated ads are remarkably distorted) also on the walls, which are exuberant, vibrant and distorted, as well as the paintings, full of misspellings, and the signs of old and worn shops, which constitute a visual affinity and slate billboards in the streets with their conflicting colors and contents, which may sometimes offend public taste with their images, sayings and a recent display of election ads that remain for a while long time after election ends and candidates win or loss see (Figure 1, 2, 3 and 4).



**Figure (1):**Random distribution of electrical generator wires.



**Figure (2):** Random distribution of electrical generator wires and significance for places.



**Figure (3):** random advertisements significance for doctors



**Figure (4):** Random trash in the street.

The aim of the study is to address the impact of visual contamination with random advertisements spread in Baghdad governorate, where some main ways were studied, how advertisements were published and distributed, ways and methods of writing and others, and the possibility of imposing sanctions on their owners.

#### **Problems caused by advertisements and banner spam**

1. distorting sites, buildings and private properties of heritage and physical value that adversely affect tourism resources.
2. Ads conflict with pedestrian movement and the negative impact on traffic.
3. the interference of ceremonial and commercial declarations with the visual restriction of the historical, architectural or esthetic features of the city.
4. Environmental pollution as a result of leaving advertising waste without safe disposal.
5. Accidents and material losses are the result of advertising, especially large and poorly secured.
6. Unsustainable consumption patterns and their negative economic, environmental and social impacts.
7. Waste of electrical power and photosynthesis due to unregulated lighting that may cause road accidents (EA/Ems,2010).

#### **The role of advertising in societies**

There is no doubt that the declaration as a communications activity has had a major impact on the lives of contemporary societies. Just as it affects the promotion of goods, it does in practice contribute to the dissemination of new values and trends and changes in people's habits and tastes. Today, ads are influenced by dietary habits in terms of food quality and eating style, and new generations are raising their taste of sandwiches, sweets, and soft drinks, and are influenced by the different kinds of fashion the world needs. The declaration effectively contributes to the expansion of consumption and creates a pattern of consumer behavior in society (Khalil,1995).

#### **The important aspect of advertising**

It changes human thinking and then develops its behavior and comprehensiveness, making it a civilization role in promoting, sensitizing, and convinced society that the only solution to the survival of life on this planet in a way that guarantees human dignity is to act seriously, in order to rationalize the exploitation of natural resources and preserve the environmental balance (Almalik,1999).

#### **The dangers of visual contamination**

The danger of visual contamination is mainly related to the loss of beauty and the collapse of esthetic considerations, dissatisfaction, acceptance of the ugly image and its spread so that the visual scale of the eye becomes an existing custom and law, and visual contamination affects the human soul. The effects of pollution start with psychological problems such as tension, tight, excessive nervousness, disturbed behavior and psychological pressure, and become worse as physical diseases such as high pressure, heart, diabetes, swollen joints, colon, difficulty in breathing. He also said that some of the specialists returned the nature of the emotions resulting from feeling a negative visual effect to increase the release of adrenaline, the hormonal material produced by the human pituitary gland, in turn, translated into what the eyes showed and sent the brain to produce the hormone, which in turn increases the stomach's load. It increases the heart rate and thus the speed of the emotion and may lead to a positive visual effect of feeling beauty and thereby increase the secretion of cortisone in the body which reduces pain sensation of the body or its parts, especially those suffering from rheumatic diseases, thus resulting in a feeling of comfort and psychological calm. This explains why our societies have become more aggressive and aggressive, especially in the densely



populated urban slums and populace, with negative influences in the planned and new areas, which have some beautiful, i.e., ripe visual effects(Qassim,2016).

### **The Sources of Visual Pollution**

1. Excessive and overcrowded advertisements.
2. Telecommunication and electric wires and poles, mobile towers.
3. Signboards, billboards, posters and hoardings.
4. unproductive lands and deforestation.
5. Bad designed buildings and huge constructions.
6. Smoke spewing chimneys of factories.
7. Graffiti.
8. Open storage trashes(Sudeepta,2015).

### **How it is affecting human health**

1. Distraction.
2. Eye fatigue.
3. Decrease in opinion diversity.
4. Loss of identity.
5. Accidents.
6. Harming mental health: Unpleasant visuals can cause exhaustion, depression, stress and anxiety due to bad views.
7. Rheumatic diseases.
8. Negative and chaotic visuals can reduce decision making power of human mind especially in kids.
9. Dark dangerous color combinations can change human perception and human psychological mood and behavior(Shafi,2015).

### **3. PRACTICAL SIDE**

It includes written and display ads on walls, building roofs, road walls, pavements, etc. collected through the researcher's daily views, all of which are within the practical framework of the research.

#### **Benefits from advertising in ways**

As an important aspect of the economic educational process, the use of road ads can be made of the color picture as a big effect on the child's self, as the child is by nature photographing, so they should be used as an effective element in the field of economic education, by showing pictures showing the profligacy of water and electricity and its risk to society. With easy and simple phrases, or with two-sided pictures: As a picture of a child with a lot of food and no food to eat, half of the second picture is a child from Africa who is starved, his lips cracked from thirst, and so on... the pictures with two contrasting dimensions are too many. The importance of the media replication of the image has been made clear to the human soul, so on public roads we see short-distance and double-sided propaganda: On the road to travel and public roads, these advertisements are not used in a meaningful educational way, rather than displaying pictures of semi-naked women, or pictures of Western cuisine, in a way that plays well and arouses lust (Al-Turkayi,2017).

#### **The role of the Capital municipality**

The Baghdad Capital municipality declared setting controls, instructions and models that define to advertisers the sizes and measurements of billboards and the specified places to be set in the streets. The department of relations and information quoted Baghdad's under-secretary for municipal affairs as saying that the design department in the Baghdad secretariat has set regulations, instructions and models that specify to advertisers the size and

measurements of billboards and the specified positions to avoid their spread randomly to preserve the capital Baghdad's beauty, not affect traffic and traffic, blocking vision, and called companies and advertisers Those who wish to promote their goods through the use of large-size billboards to review the relevant municipal districts for the purpose of obtaining original approvals and to know where to set them up and otherwise would be subject to legal liability as well as financial fines .The Baghdad Capital municipality pointed out that the municipal districts on both sides of al-Karkh and al-Rassafa completed the lifting of a large number of billboards promoting goods and services that were randomly installed in the capital's streets, areas and important provinces, which caused the distortion of the capital's esthetics and appearance and impeded the view of road users. In addition to violating the controls and instructions established by the secretariat, the secretariat explained that this urgent action is carried out in the light of the unlimited proliferation of these paintings in an unorganized and indiscriminate manner, contrary to what is being done in neighboring countries and other countries of the world. In addition, the owners of these billboards did not have licenses from the Baghdad Capital municipality to install and install these advertisements (Ain,2016).

## RESULTS AND DISCUSSIN

Random advertising is we see on the walls to indicate a particular subject, columns of electricity, or sideways, and finally campaign advertising have been added see in (Figure5,6,7,8,9,10,11,12,13,14,15 and 16).



Figure (5): Random advertisements in the street.



Figure (6): Random advertisements at bus stops.



Figure (7): advertisements that are affixed to the posts of the electric lights.



Figure (8): advertisements that are affixed to the posts of the electric lights.



Figure (9):The random advertisements on walls.



Figure (10): The random advertisements for politicians.



Figure (11): The random advertisements for politicians on al-ferdows square.



Figure (12): The random advertisements for politicians on one of the squares.



Figure (13): The random advertisements in al-jadiriya bridge.



Figure (14): Random advertisements in adin square.



Figure (15): Random advertisements down al-liqaa bridge.



Figure (16): Random advertisements in adin square on the electrical board.

### Advertising types

Road advertising are that consumers can see in roads and fields. Its purpose is to be seen by the public passing through the road, by displaying advertising messages in specific locations specifically created for this purpose, or being carried out and placed directly on the walls of buildings.

### Types of road advertising

Advertisements with traditional formats and advertising with modern formats are different and are described as follows:

1. Posters: Prints the advertising on one side of the papers or on multiple sides, then pastes it next to the wood. This is a traditional method, used to advertise movies, theater shows and conferences, and is the most widely used method in small cities.
2. Advertising plotted: Includes advertising plotted on boards and ads plotted on walls. They are fixed and mobile.
3. Flashlights advertising: Placed in the main squares in the facades of buildings, the roofs of buildings set up in strategic locations or places of clear vision. These ads are large in size to get the public's attention.
4. The billboards on a stand and in the form of a metal cylinder: This type of ad is placed at a high height on a stand that is mounted as a cylinder and mounted on the ground. This type of advertising is spread both in travel routes and within cities.
5. Mobile stationary ads: This type of ad is in the form of vertical, triangular, prism columns. It consists of three faces with three ads, with these columns revolving around themselves over a certain period of time. The advertiser can choose three goods or products to advertise, or choose one and advertise it in three different designs.
6. The bright figures: The luminous box, the laser and the TV screen are included.



7. a circular announcement on a column: This is a circular advertising and is mounted on a stand, carrying a guide sign to go to a street. The advertising message of this type of ad is limited (<https://doi.org/10.3390/ijmr13010118>,2020).

### Street signage

Street signage has been a formidable place among all kinds of advertising, and has been highly suited to consumer culture and behavior, but as the culture of societies evolves, it has become a particular urban and community pollution when used without license and without regard to its standards. However, this type of advertising is still being used strongly today between streets, squares, subway stations and other transportation stations, mainly to raise awareness of the brand and its products or services rather than to achieve a sale or profit rate (<https://doi.org/10.3390/ijmr13010118>,2020).

### Reasons for the spread of random advertisements

Specialists believe that advertisements on buildings and surfaces are chaotic and that some governmental institutions are involved in this. Because of the lack of people specialized in advertising marketing and choosing the right places to promote products and other propaganda matters that concern the company and the citizen, specialists pointed out that it is difficult now to control this for a number of reasons, including the absence of law in particular and the culture of some in this vital matter Some of the forms and types of advertisements that go over the law distort the city's face and affect the building's esthetics, and some are using the night to raise and suspend its own advertisement even if it is at the expense of another paid advertisement with one of the relevant companies Some advertisements also spread in a great way in the middle carrots that lack the simplest right elements for the declaration, including the Arabic language. Commercialization is not a simple concept but an important tool in a large, broad world that extends from product to recipient ([Al-Mada,2016](https://doi.org/10.3390/ijmr13010118)).

### Solutions for random advertising

Financial sanctions are imposed on its publishers by chasing them through the mobile phone pre-placed in the advertisement or by means of spatial significance, and by forcing them to remove it, with a written undertaking signed to do so and limiting the subject to the authorities responsible.

## CONCLUSION AND RECOMMENDATION

1. Accountability for the public taste-distorting advertising owners, most of whom have set a telephone number or contact address such as (crane for car drag, house for sale or rent, etc.) so that they are easily accessible and fined.
2. Stop selling the stingers used in writing, especially on the walls of houses.
3. The billboards must be on and off the driver's horizon and not sideways so that they do not get the driver's attention and distract the road.
4. Advertising content must not be animated but static and does not contain video, animations, or animations so as not to distract the driver.
5. Two consecutive ads are prohibited, as each ad must be given a period of time.
6. The display panel is not positioned on the intersection and signal limits for driver distraction and no obscuration of visibility.
7. The light intensity sensors must be placed on the street so that the light intensity of the panel matches the intensity of the light in the street so that it does not cause light glare at night and thus distract the driver and lose attention.

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## PREPARATION OF POLYMERIC COMPOSITES FROM SATURATED POLYESTERS GLASS POWDER (FLOURSCENT) AND STUDY OF ITS MECHANICAL PROPERTIES

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### ABSTRACT

In this study, polymeric composites were prepared from unsaturated polyester as a base material with glass powder (fluorescent) in different weight ratios (4, 6, 8, 10, and 11%) as a support material and after comparison before and after reinforcement of the prepared composites, an increase was found. In the values of mechanical properties (hardness, compressive strength), the shock resistance values decreased, but an increase in temperature leads to an increase in the values of shock resistance, as well as the values of compressive strength And it reduces the hardness value.

**Keywords:** Unsaturated polyester, fluorescent powder, mechanical properties.





## تحضير متراكبات بوليمرية من البولي استر غير المشبع ومسحوق الزجاج (الفلورسنت) ودراسة خواصها الميكانيكية

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## الخلاصة

تم في هذا البحث تحضير متراكبات بوليمرية من البولي استر غير المشبع كمادة أساس مع مسحوق الزجاج (الفلورسنت) بنسب وزنية مختلفة (4 و 6 و 8 و 10 و 11%) كمواد مدعمة وبعد المقارنة قبل وبعد التدعيم للمترابكات المحضرة وجد زيادة في قيم الخواص الميكانيكية (الصلادة، مقاومة الانضغاط)، اما قيم مقاومة الصدمة قد انخفضت لكن زيادة درجة الحرارة تؤدي الى زيادة قيم مقاومة الصدمة وكذلك قيم مقاومة الانضغاط وتقلل من قيمة الصلادة. الكلمات المفتاحية: البولي استر غير المشبع، مسحوق الزجاج (الفلورسنت)، الخواص الميكانيكية.

## INTRODUCTION

Composite materials possess some properties that are suitable for a number of industrial applications, so they have gained an important position among the engineering materials (Ives *et al.*, 1971). Composite materials generally are formed by combining two or more materials to obtain a material with engineering and physical properties that has better properties than the materials included in its composition. The composite material consists of three phases; the matrix phase and the Reinforcing phase the intermediate phase between them (the interphase) (Akovali, 2001). The interest in polymer-based composites increased due to their engineering capacities, especially in applications that requires high-quality resistance. The most important materials that are used as basic materials are the three types of polymeric materials that are thermoplastics, thermosetting and elastomers (Al Adam 1983; Mouzakis, 2008). Plastic composite materials reinforced with different types of supporting materials include two types of base materials, which are a thermoplastic base material and a thermo set base material. It should be noted that thermostats are stronger than thermoplastic materials which are widely used such today such as low-density polyethylene, high-density polyethylene, and polypropylene (Akovali, 2001). Unsaturated polyester resin is a thermally hardening resin and cannot be used alone in engineering applications, because it is a brittle material and does not have the appropriate efficiency, however, it can be strengthened with some strengthening materials such as glass fiber, carbon fiber, and glass waste powder particles affect some of the mechanical properties of the brittle unsaturated polyester. They also affect the fracture toughness. (Varga, *et al.*, 2010). Waste glass powders are a non-metallic and inorganic substance. This material is not burnt and does not decompose, so it is difficult to recover (Malik, 2000). Among the applications of these materials in corrosive media is their use in chemical industries such as tanks, pipelines, cleaning devices, because of their resistance to corrosive acid media such as (H<sub>2</sub>SO<sub>4</sub>) used in space and the automobile industries (Callister, 2005). The aim of the study: This study aims to prepare composite materials that show good resistance to shocks and high compressive strength, identify the behavior of polymeric materials under standard conditions, make a comparison between them and the polymeric material before reinforcement, and then study the mechanical properties (impact strength, hardness, compressive strength) for all samples before and after the reinforcement with fluorescent glass powder.

## MATERIALS AND METHODS

### The matrix

The unsaturated polyester made by (B-CHEM SRL) company was used as the matrix, which is in the form of yellowish glue at room temperature, it is a type of Thermosetting polymers, that turns from the semi-solid state to the solid state by the addition of the hardener (di benzoyl peroxide), which is at the form of white cream that is added to the unsaturated polyester resin in a ratio of 2:1 at room temperature (Mark,1999). Waste glass powder was also used in the form of powder, the powder was obtained after grinding and sifting by a 200-mesh sieve in the laboratory to obtain the granular size  $75\mu\text{m}$ . Suitable samples could be obtained for molding at room temperature.

### Reinforcing materials

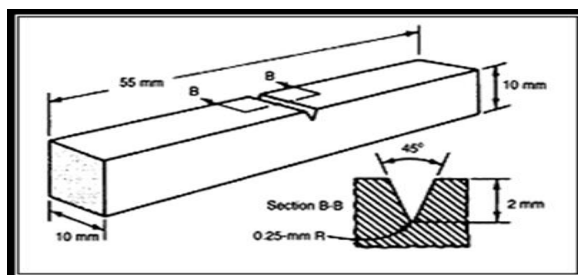
Reinforcing materials (Glass Powder that was sifted with a 200-mesh sieve) were used to reinforce the unsaturated polyester resin. The (damaged glass of candles) of the type (PARS LAMP 18W) from Iran was used.

### Preparation of samples

Three types of samples were prepared for the mechanical tests:

#### Impact test samples:

Impact test samples were prepared according to American standard (256-87ASTM-D) with dimensions  $(10 \times 55 \text{ mm} \times 10^3)$  for the examination. The grooving depth in the models is (2mm) with a notch angle of  $(45^\circ)$ . The absorbed energy required for the occurrence of the breakage was obtained directly from the Charpy Impact Instrument provided by (Tokyo Koki Seizosho, LTD) (Figure1), Illustrates the shape and dimensions of the sample prepared for the impact strength testing (Al-Bakri,2012).



**Figure (1):**Shape and dimensions of the sample.

#### Hardness testing samples

Non-standard models were used. Any model of composite material would be suitable for this test. The hardening device of the (Rockwell) hardness type was used, the penetration tool is a hardened steel ball with a diameter of (12.7mm) for the weight applied (60kg), equipped from the company (WOLPERT-Germany) for measuring the hardness of thermosetting polymer. The method of examination is to place the device perpendicular to the sample for which the hardness is to be measured in order for the needle to be inserted into the surface of the material and then wait for a period of three seconds after which the hardness value is taken from the device, and no less than three readings were taken from different places of the sample and then the average was taken (Al-Mosawi, 2009).

#### Samples of compressive resistance test:

The compressive resistance test models were prepared according to the specification (618ASTM-D) it is a cylindrical device using a hydraulic press of the type (Testing Machine Co.LTD) supplied by (WOLPERT - Germany) to determine the maximum compressive load the sample can bear, and by dividing the load by the cross-sectional area of the model before the deformation

occurs Compression strength calculation for all models was made, (Figure 2) shows the shape of the sample prepared for compressive resistance testing.



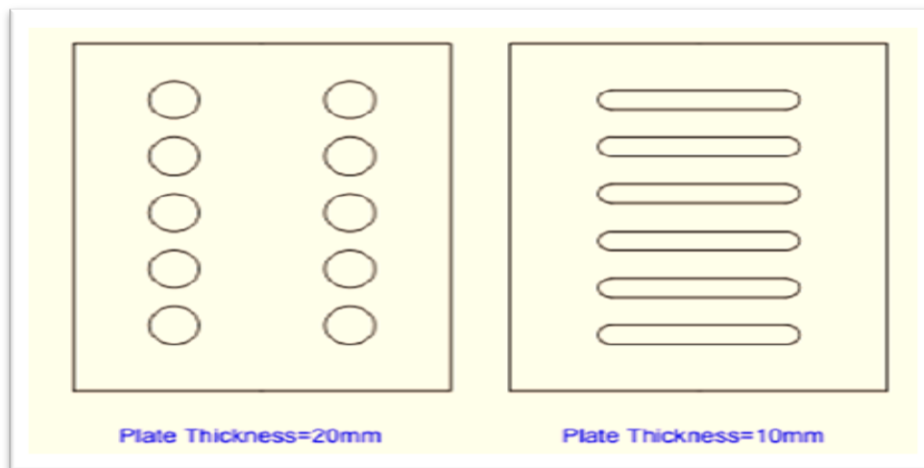
**Figure (2):**The shape of the sample prepared for compressive resistance testing.

**Preparation method**

An unsaturated polyester resin was used with a hardener (di benzoyl peroxide) in a ratio of 2:1 to turn it into a solid material that solidifies at room temperature. Then samples were prepared of composite materials of unsaturated polyester reinforced with glass powder with weight ratios of (4, 6, 8, 10, and 11%).

Hand-Layup Molding was followed in the process of preparing samples before and after using the powder in reinforcement (**Sulyman et al., 2019**).

The resin was prepared by adding a hardener to the unsaturated polyester in a ratio of (1:2), then the resin was added to the hardeners, and then the mixture was placed in the molds designated for measurements, which were made according to the standard specifications for each examination, and after the completion of the pouring process, it was left for 24 hours at a temperature of (23°C) and (53°C) to complete the curing process, overlap, homogeneity between the particles, and reduce the internal stresses formed during the pouring process, (Figure3) shows a schematic diagram of the templates used in preparing samples.



**Figure(3):** Schematic diagram of the templates used in preparing the samples.

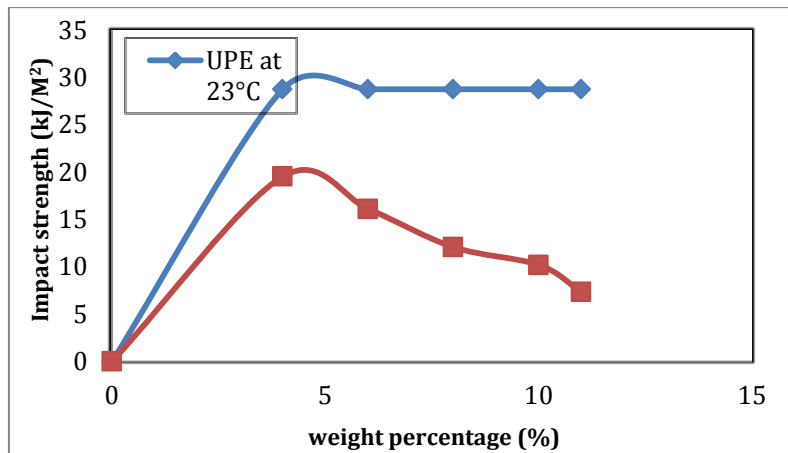
**RESULTS AND DISCUSSION**

**Impact strength**

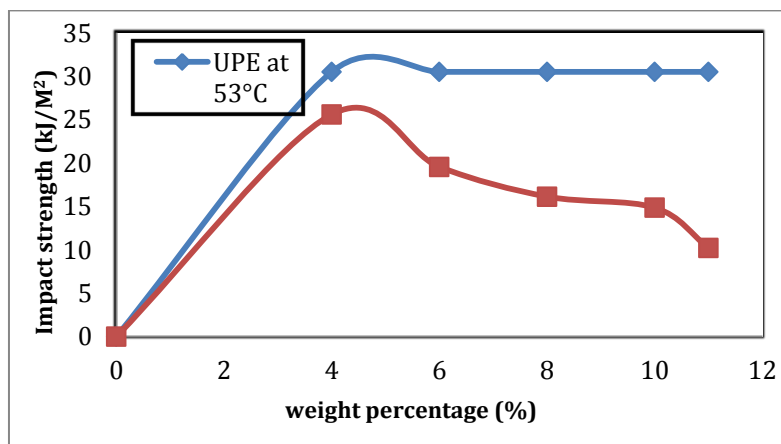
Examination of impact strength is one of the practical methods that give a correct indication of materials strength and resistance to fracture under stress at high velocity (Alrawi&AlHalim,2019). The impact strength is calculated using the equation:

$$I \cdot S = \frac{\text{energy of fracture (J)}}{\text{area of cross section (m}^2\text{)}} \dots\dots\dots(1)$$

From (Figure4) we notice that the impact strength decreases with the increase in the percentage of addition of damaged candle glass powder, this is an expected result because the glass is a brittle ceramic material, (Figure 5) shows the effect of the weight ratios of the unsaturated polyester support material on the impact resistance of polymeric composites at the processing temperature (23-53°C), (Table1) shows the effect of the weight ratios of the reinforcement material added to the unsaturated polyester on the impact resistance of polymeric composites at the processing temperature (23-53°C).



**Figure (4):** The relationship between impact strength (I.S) and the weight percentage of unsaturated polyesters before and after the reinforcement at 23°C.



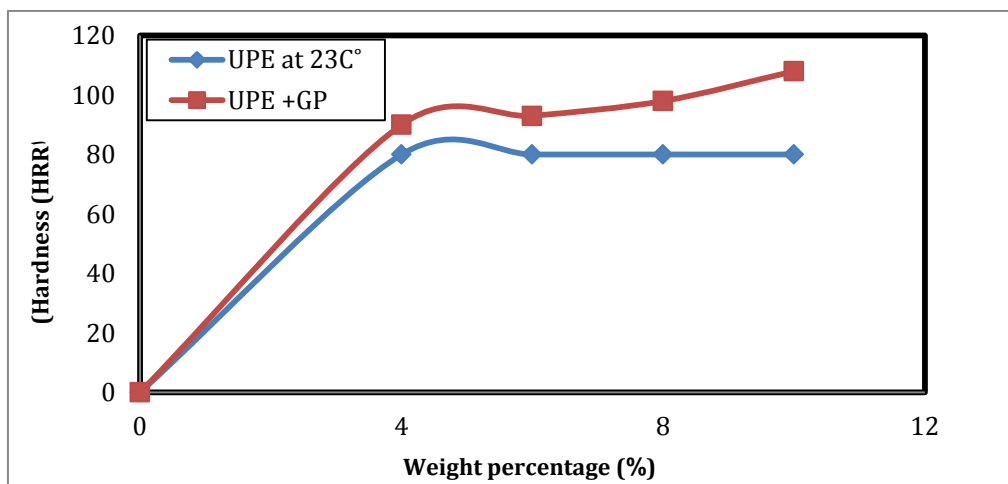
**Figure (5):** The relationship between impact strength (I.S) and the weight percentage of unsaturated polyesters Before and after the reinforcement at 53°C.

**Table (1):** Impact strength values (I.S) for unsaturated polyester before and after reinforcement with different weight ratios at temperatures of 23 and 53°C.

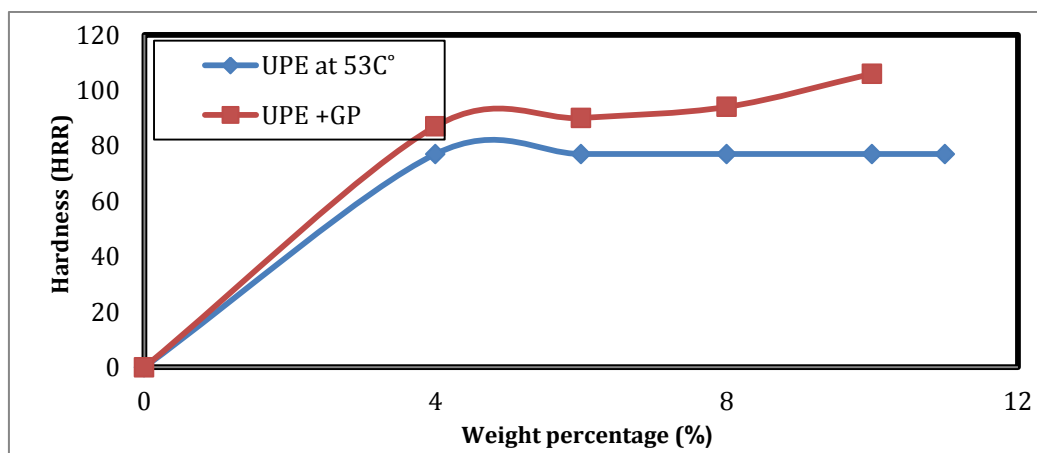
Composite at 23°C	Impact strength (KJ/m <sup>2</sup> )	Composite at 53°C	Impact strength (KJ/m <sup>2</sup> )
UPE	28.76	UPE	30.5
UPE+ GP 4%	19.54	UPE+ GP 4%	25.6
UPE+ GP 6%	16.12	UPE+ GP 6%	19.54
UPE+ GP 8%	12.08	UPE+ GP 8%	16.12
UPE+ GP 10%	10.2	UPE+ GP 10%	14.84
UPE+ GP 11%	7.34	UPE+ GP 11%	10.2

### HARDNESS

The hardness property is one of the most important mechanical properties of the material, it's considered as a measurement of plastic deformation, in which the material suffer from an influence of an external stress, imposed on it by its exposure to scratching and penetration with tools that are stiffer than it during its use in various applications (Anothony&David, 1983), through research It was found that the higher the percentage of addition of damaged candle glass powder, the higher the hardness value. The reason behind that is that the glass is a ceramic material that gives hardness to the base material (polyester). As a result of the physical contact between the glass particles and the polymeric chains, the penetration resistance of the composite material is decreased. (Figure 6 and 7) shows the effect of the weight ratios of the support material added to the unsaturated polyester on the hardness value of polymeric composites at the processing temperature (23-53°C), (Table 2) hardness values of unsaturated polyesters before and after the reinforcement with deferent weight ratios the temperature varies between 23 and 53°C.



**Figure(6):** The relationship between Rockwell hardness (RHH) values and the weight percentage for the unsaturated polyesters before and after reinforcing at a temperature of 23°C.



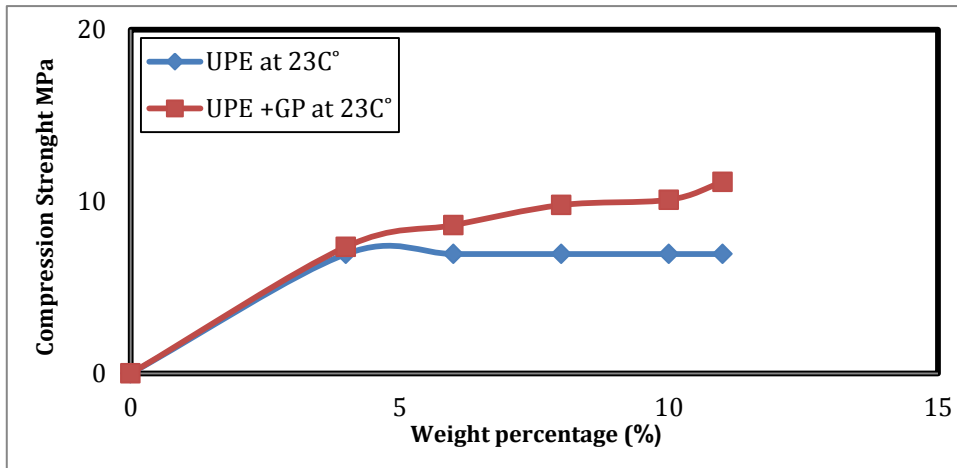
**Figure(7):** The relationship between Rockwell hardness (RHH) values and the weight percentage for the unsaturated polyesters before and after reinforcing at a temperature of 53°C.

**Table (2):** Hardness values of unsaturated polyesters before and after the reinforcement with deferent weight ratios the temperature varies between 23and 53°C.

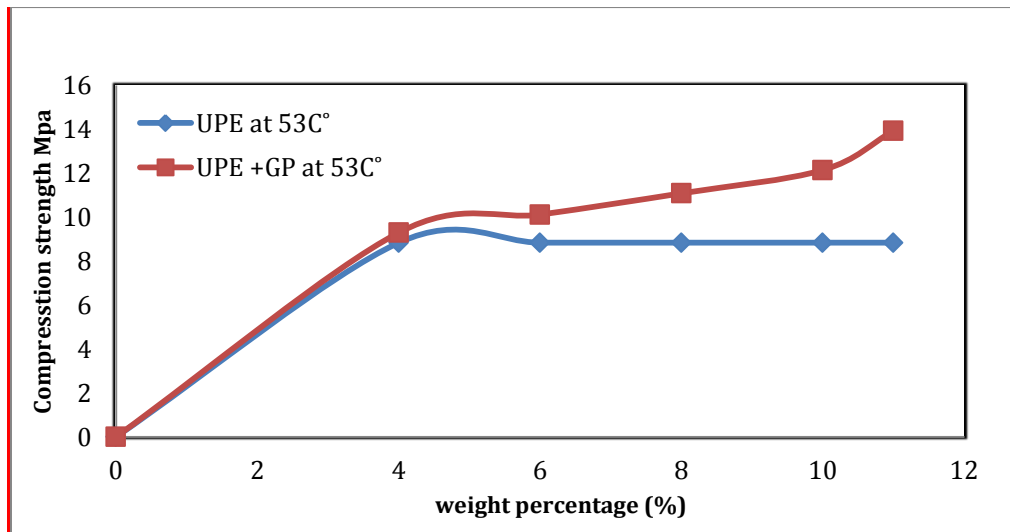
Composite at 23°C	Hardness Rockwell(HRR)	Composite at 53°C	Hardness Rockwell (HRR)
UPE 23°C	80	UPE 53°C	77
UPE+GP 4%	90	UPE+ GP 4%	87
UPE+ GP 6%	93	UPE+ GP 6%	90
UPE+ GP 8%	98	UPE+ GP 8%	94
UPE+ GP 10%	108	UPE+ GP 10%	106
UPE+ GP 11%	113	UPE+ GP 11%	109

### Compressive resistance test

It is one of the most important tests to know the durability and strength of the polymer. There are number of materials that may be brittle in tension condition, but appear ductile in compression. Therefore, compressive resistance testing is used to determine the submissive strength as well as the compressive strength (Sulyman *et al.*, 2019). It is defined as the amount of mechanical stress that a solid material endures under perpendicular stress. Without breaking or shattering or changing in its shape after the stress is removed. This test is widely used to examine fragile materials such as glass, concrete pieces, rocks, mastic iron and heat-hardened polymers due to the fact that these materials have more strength in compression than in tension condition, test samples used are cubic or cylindrical (Sulyman & Sulyman, 2020), (Figure 9 and 8) shows the effect of the weight ratios of the support material added to the unsaturated polyester on the value of the compressive strength of polymeric composites at the processing temperature (23-53°C), (Table 3) Compression strength of unsaturated polyester before and after reinforcing in different proportion weights at 23 and 53°C.



**Figure(8):** The relationship between compression strength (C.S) and the weight percentage of the unsaturated polyesters before and after reinforcing at a temperature of 23°C.



**Figure(9):** The relationship between compression strength (C.S) and the weight percentage of the unsaturated polyesters before and after reinforcing at a temperature of 53°C.

**Table (3):** Compression strength of unsaturated polyester before and after reinforcing in different proportion weights at 23 and 53°C.

Composite at 23°C	Compression strength Mpa	Composite at 53°C	compression strength Mpa
UPE 23°C	6.96	UPE 53°C	8.84
UPE+GP 4%	7.37	UPE+GP 4%	9.3
UPE+GP6%	8.63	UPE+GP6%	10.12
UPE+GP 8%	9.8	UPE+GP 8%	11.09
UPE+GP 10%	10.11	UPE+GP 10%	12.15
UPE+GP 11%	11.15	UPE+GP 11%	13.94

## CONCLUSIONS

The hardness increases with the increase in the addition of damaged candle glass powder. Impact resistance decreases with the increase in the addition of damaged candle glass powder. The compression strength increases with the addition of the damaged glass powder.

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## IRAQI CONSUMER OPINION OF THE QUALITY AND SAFETY OF LOCAL FOOD PRODUCTS

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### ABSTRACT

The aim of this research was to indicate the opinion of the Iraqi consumer about the quality and safety of local food products, the questionnaire was included 19 questions for product quality, price, distribution and promotion as a tool to survey the opinions of 128 consumers in Baghdad, the data was analyzed by using percentage, weighted mean, and weight percent, the results obtained showed that the Iraqi consumer prefer local food products for their high quality and appropriate price, however they need attention to packaging, promotion and distribution.

**Keywords:** Consumer, quality, food safety, local food products.



## رأي المستهلك العراقي في جودة وسلامة منتجات الأغذية المحلية

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## الخلاصة

هدف هذا البحث إلى بيان رأي المستهلك العراقي حول جودة وسلامة المنتجات الغذائية المحلية، وشمل الاستبيان المعد لهذا الغرض 19 سؤالا لجودة المنتج وسعره وتوزيعه والترويج له كأداة لاستطلاع آراء 128 مستهلك في مدينة بغداد، وجرى تحليل البيانات باستخدام النسب المئوية والوسط الحسابي المرجح والوزن المنوي، وأظهرت النتائج المستحصل عليها أن المستهلك العراقي يفضل المنتجات الغذائية المحلية لجودتها العالية وسعرها المناسب، إلا أنها تحتاج إلى الاهتمام بالتغليف والترويج والتوزيع.

الكلمات المفتاحية: المستهلك، الجودة، سلامة الغذاء، منتجات الأغذية المحلية.

## INTRODUCTION

The food industry in all countries around the world represents one of the most important sources of income and the most important of economic and productive activities on which the economy is based, given what it represents as a fundamental necessity in providing the continuing needs of the consumer and its ability for achieve self-sufficiency and food security as well as its importance in moving various economic sectors such as Agriculture, industry, trade, and providing job opportunities for the available human energies. This industry generally aims to change the form of raw materials from one state to another in order to provide the needs and wish of the consumer, as in the manufacture of milk, meat, flour, sugar, dates, etc., as well as the role of this industry in extending the shelf life of food products through packaging that aim to preserve the product for the longest possible period as well as the ease of delivery to the consumer in sizes that suit his daily needs, and in all cases, this industry must preserve of food quality in accordance with the standards adopted in this field. The quality is considered as the essential criterion of success of food industry, and the consumer represents the most important link in this industry due he is the ultimate beneficiary and his opinion represents the decisive factor that determines of acceptability of these products, so, this article came to know the opinion of the Iraqi consumer about the quality and safety of local food products Through four topics, including research methodology, theoretical framing, analysis and discussion of research results, conclusions and recommendations.

## FIRST TOPIC: RESEARCH METHODOLOGY

## First: Research problem

As a result of the opening of the Iraqi markets and their flooding with imported food products, in contrast to a significant decrease in local products, the research problem is embodied in the following questions:

1. What is the Iraqi consumer opinion of local food products in terms of quality, safety, price, distribution and advertising?
2. Is there a refusal to consume local food products?
3. Does the Iraqi consumer prefer imported food commodities over their local counterparts?

## Second: Importance research

The concern of local food manufacturers with the quality and safety of their products that are marketed through the local markets, as the approved standards in quality and safety have become the basic requirements for any company in this field, which is confirmed by the

standard specifications and good manufacturing practices, which will provide the high ability of these products in competition with The importer product and obtaining the satisfaction of the Iraqi consumer, which will reflect positively on the growth and development of the Iraqi industry and economy in this field, Therefore, the importance of the research is as follows:

1. Knowledge addition in field of quality and safety of local food products due they play an important role in moving the economy sectors and providing consumer needs in preparation for achieving self-sufficiency, diversifying sources of income and reducing unemployment rates.
2. Providing a new horizon for researchers to delve into further studies and research in the field of local food products.
3. The importance of the added value that will be achieved in the event of the development of the local food products sector.

### Third: Objectives research

1. Knowing the opinion of the Iraqi consumer regarding the quality and safety of local food products.
2. Determine the reasons that lead to reluctance of Iraqi consumer to consume local products.
3. Developing appropriate proposals and recommendations for the development of the local food industries, achieving its capacity in market competition and achieving consumer satisfaction.

### Fourth: Method research

Analytical approach descriptive for information was adopted related to the research variables by conducting an opinion poll for a specific sample and then interpreting the data obtained and drawing conclusions from it.

### Fifth: Limits research

1. Time limits: The period for completing the research extended from January 1, 2020, to December 1, 2020.
2. Place limits: the questionnaire was applied in the city of Baghdad, Iraq.
3. Scientific limits: The research focused on the elements of marketing mix (**Pourdehghan 2015**) and its relationship to the quality of local food products.

### Sixth: Sample Research

The study sample was 128 persons was randomly selected from city of Baghdad, they represented the opinion of the Iraqi consumer regarding the quality and safety of local food products in the markets (Table 1).

**Table (1):** Characteristics of the research sample.

Details	Category	Number	Percentage (%)
Gender	Male	56	44.8
	Female	72	56.2
Educational	Intermediate	14	10.9
	Secondary	12	9.4
	Diploma	15	11.7
	Bachelor	65	50.8
	Master	10	7.8
	PhD	12	9.4
Age	20-29	9	7
	30-39	29	22.7
	40-49	38	29.7
	50-59	32	25
	> 60	20	15.6

**Seventh: Research tool**

The questionnaire was adopted as a tool to collect data related to the research variables. It consisted of 19 questions, 8 for product quality, 4 for price, 3 for distribution and 4 questions for promotion. The fifth Likert Scale was used (strongly agree, agree, neutral, disagree, strongly disagree) by weights (5, 4, 3, 2 and 1) respectively, the answer to the questionnaire questions was as shown in (Table 2).

**Eighth: Methods of statistical Methods**

Percentage, arithmetic mean, hypothetical mean and weight percent were used to interpret the results

**SECOND TOPIC: THEORETICAL FRAMING****First: Products identification**

The products are known according to **Kowalskaet al. (2018)** that everything that leads to the satisfaction of a need or a wish can be presented in the market to attract attention, appetite or consumption.

**Second: Research Variables**

The research variables included the elements of the marketing mix that include product quality, price, distribution, and promotion, as indicated by **Ali et al. (2016)**. As follows:

**1. Product quality**

**Alshikhi& Abdullah (2018)** defined quality in general as a set of physical and formal characteristics of the product, which includes functional, social and psychological benefits according to which the purchase decision for these products is determined by the consumer, the quality of the product achieved when the level of defects in it falls to the minimum found in the standard specifications (**Carrascosaet al., 2016**), as for the quality of the food product, it is a set of characteristics by which it is possible to determine the acceptability of the product to the consumer or that it awards to fulfill the consumer's utmost wishes in the food product (**Grunert&Aachmann, 2016; Yormirzoevet al., 2019**), so, food companies compete with regard to developing the best quality policy in its factories in order to maintain the quality of its products and develop them by following an appropriate quality system for them, continuous improvement of manufacturing methods, and following all procedures that would ensure the quality of their products and gain consumer satisfaction and confidence through a number of requirements including product quality, appearance, size, shelf-life and product stability during period of storage and marketing (**Gutierrez-Gutierrez et al., 2018; Panghalet al., 2018; Tutu &Anfu, 2019**) and due to the increasing wish of the Iraqi consumer to obtain local food products and not dependence on imported food products present in the local markets (**Fahed 2016; Alkhafaji, 2018**), which is maybe a low quality for various reasons, including the adoption by exporting countries of a commodity dumping policy (**Hamadet al., 2012; Lafta, 2016**). So, this studying for the Iraqi consumer's opinion on the quality of local food products is be necessity to upgrade this industry and enhance Iraqi consumer confidence in it.

**2. Price**

**Bellemare (2015)**refer that it's represents the monetary value that the consumer pays to obtain the good or service required.

**3. Distribution**

**Kotler& Keller (2006)** showed that it's means all activities and events that lead to the provision of required products in time, quantity and suitable place for the purpose of consumption.

#### 4. Promotion

**Bognanno&Melero (2015)** defined it's as all activities that aimed to introducing individuals and organizations to essential information of the required products in order to encourage them to buy by using various methods such as advertising, publicity and public relations.

### THIRD TOPIC: ANALYZE AND DISCUSS RESEARCH RESULTS

#### Percentage, arithmetic mean, hypothetical mean and weight percent Analysis of Research Variables

The results (Table 2) refer to:

##### 1. Quality

The average of this element showed that the arithmetic mean and weight percent (%) was 3.3 and 66.5%, respectively, that ensured samples agree (>3) with paragraphs of quality.

The degree of samples response was strongly agreed (>3) with the quality of the local product is influence the consumer's purchasing decision (Question No. 6), the degree of samples response was agreeing (>3) with preferring of Iraqi consumer to consume local products over imported products (Question No. 1), the local product has good quality and specifications (Question No. 2) and The producing companies seek to pay attention to the quality of the product according to the standard specifications (Question No. 7), while they are Neutral (=3) with is found a clear informational label at local product containing all nutritional and warning information (Question No. 3), similarly, neutral ( $\geq 3$ ) with available of local products in various sizes that cover consumer demand (Question No. 5) and it was ( $\leq 3$ ) for work of the producing companies to make continuous improvement to the products provided to maintain their quality (Question No. 8). On the other hand, the degree of samples response was don't agree (<3) with the packaging of local product was attracts the consumer (Question No. 4).

Table (2): Analysis of research variables.

Research variables	Question No.	Question	Degree of response					Arithmetic mean	Arithmetic mean relation with		Weight percent (%)
			Strongly agree	Agree	Neutral	Don't agree	Strongly don't agree		Degree of response	hypothetical mean	
			5	4	3	2	1				
Quality	1	In general, Iraqi consumers prefer to consume local products over imported products	35	45	23	22	3	3.7	Agree	>3	74
	2	The local product has good quality and specifications	12	61	29	25	1	3.5	Agree	>3	70
	3	The local product has a clear informational label containing all nutritional and warning information	6	37	38	43	4	3.0	Neutral	=3	60
	4	The local product has packaging that attracts the consumer	2	25	41	53	7	2.7	Don't agree	<3	54
	5	The local product is available in various sizes that cover consumer demand	8	43	38	37	2	3.1	Neutral	$\geq 3$	62
	6	The quality of the local product influences the consumer's purchasing decision	64	53	7	3	1	4.4	Strongly agree	>3	88
	7	The producing companies seek to pay attention to the quality of the product according to the standard specifications	10	45	41	31	1	3.3	Agree	>3	66
	8	The producing companies work to make continuous improvement to the products provided to maintain their quality	10	48	43	23	4	2.9	Neutral	$\leq 3$	58
		Average						3.3	Agree	>3	66.5
Price	9	The prices of local products are appropriate to the consumer	24	66	28	10	0	3.8	Agree	>3	76
	10	Government agencies monitor the prices of local products to be uniform in all markets	6	18	20	56	28	2.4	Don't agree	<3	48
	11	Governmental agencies enforce laws to protect local products and a consumer protection law to prevent price hikes	7	11	25	58	27	2.3	Don't agree	<3	46
	12	The producing companies offer different purchasing offers to encourage the consumer to acquire their products	10	31	40	41	6	3.0	Neutral	=3	60
		Average						2.9	Neutral	$\leq 3$	57.5
Distribution	13	Local product is available in quantities that meet consumer need	7	33	23	60	5	2.8	Neutral	$\leq 3$	56
	14	The producing companies use multiple methods to distribute and deliver the product to the consumer across different regions	8	42	38	36	4	3.1	Neutral	$\geq 3$	62
	15	The availability of direct and effective distribution outlets contributes to increasing the proportions of distribution to different places	35	60	17	15	1	3.9	Agree	>3	78
		Average						3.3	Agree	>3	65
Promotion	16	Advertising has a prominent role in promoting local products	65	48	6	8	1	4.3	Strongly agree	>3	86
	17	Advertising campaigns for local products greatly affect the consumer's purchasing decision	48	60	12	6	2	4.1	Agree	>3	82
	18	Local companies advertise their products through various advertising media	16	36	39	33	4	3.2	Neutral	$\geq 3$	64
	19	Advertising styles of local products influence consumer behavior towards buying	34	76	10	7	1	4.1	Agree	>3	82
		Average						3.9	Agree	>3	78.5
Category length $4 \leq 0.8$			4.20-5	3.40-4.19	2.60-3.39	1.80-2.59	1-1.79				
hypothetical mean = 3											

It is noted through the results of the quality element that sample strongly agreed that the quality of the product is the main factor in determining the consumer's purchase decision, thus the producing companies must pay attention to the quality of the products, the sample also agreed that the Iraqi consumer prefers to consume local products over imported products due to the good quality and specifications of these products, therefore this represents an added strength for the local product that helps producers develop their local products by applying standard specifications to ensure successful marketing to the consumer, while the sample was neutral with regard to the presence of a clear informational label that containing all nutritional and warning information on the local product and availability of these products in different sizes that meet the consumer demand as well as the work of the companies for quality improvement, thus the consumer has expressed his desire to provide local products in different sizes that meet the consumer's need and that these products contain a clear informational label and a procedure continuous improvement of the product, and with regard to packaging the local product, the sample showed its disagreement, so its needs a wide range of attention to ensure that local products are widely promoted.

## 2. Price

The average of arithmetic mean, weight percent (%) and degree of response was 2.9, 57.5% and neutral ( $\leq 3$ ), respectively for Price element.

The degree of samples response was agreed ( $>3$ ) for appropriate of price for local products to the consumer (Question No. 9), and they are Neutral ( $=3$ ) for different purchasing offers which offer by producing companies to encourage the consumer to acquire their products (Question No. 12), while they are not agreed ( $<3$ ) for the role of government agencies through monitor the prices of local products in the markets (Question No. 10) and enforce laws to protect local products and a consumer protection law to prevent price hikes (Question No. 11).

The results showed that the sample agreed that the prices of local products available in the local markets were appropriate for the consumer, while it was neutral for the role of companies that providing different purchasing offers to encourage the consumer to obtain of their products, and this indicates the need for producers to maintain the level of prices and not increase them, as well as to provide offers and advantages for encourages the consumer to buy, on the other hand, the sample showed disagreement of the role of government agencies in controlling of prices and implementing laws that related to the protection of national products and consumer, this matter requires that the authorities implement the legislation effectively.

## 3. Distribution

The degree of samples response was agreed ( $>3$ ) with the contribution of direct distribution outlets to increasing the marketing proportions (Question No. 15), While, they are Neutral ( $\leq 3$ ) for availability of local product with a quantity that consumer need (Question No. 13), also they are Neutral ( $\geq 3$ ) for methods of products distribute by companies (Question No. 14).

It is observed from the results that the sample agreed that the presence of direct marketing outlets to the consumer will contribute to increasing the distribution ratios, which is the thing that companies must do to achieve this and provide their products to the consumer, and in return, the sample was neutral in its assertion of the availability of local products in enough quantities, and using companies multiple methods to distribute and deliver to the consumer, so, to achieve this, companies must work to increase production by increasing working hours and using different methods to deliver their products to the consumer through

traditional marketing outlets or electronic marketing which has become a common occurrence recent in order to increase the percentage of sales and profits.

#### 4. Promotion

The average of this element showed that the arithmetic mean and weight percent (%) and degree of response was 3.9, 78.5% and agree (>3), respectively.

The degree of samples response was strongly agreed (>3) Advertising role for promoting local products (Question No. 16), the degree of samples response was agreeing (>3) for effect of Advertising campaigns (Question No. 17) and styles (Question No. 19) at the consumer's purchasing local products decision, while they are neutral ( $\geq 3$ ) for using various media by companies for advertise of their products (Question No. 18).

The results showed the strongly agreed of sample that advertising has a prominent role in promoting local products, and they are agreed that advertising campaigns for the local product greatly effect at consumer's purchasing decision, and the methods of advertising for local products have a significant effect on consumer behavior towards buying, while the sample was neutral for local companies advertise their products through various advertising media, and from this, the importance of advertising is in promoting local products and the importance of companies intensive advertising campaigns for display their products and marketing them to the consumer and follow various advertising methods and invest social media in reaching the largest target group of consumers.

#### CONCLUSIONS AND RECOMMENDATIONS

The Iraqi consumer prefers to consume local products that available with appropriate price and good quality, while, local product need to attention of packaging and availability of these products in different sizes from producers and providing different purchasing offers to encourage the consumer to obtain of their products through presence of direct marketing outlets to the consumer and attention to advertising and promoting for local products to contributed of increase of sales, also, local product need to role government authorities controlling of prices and implementing laws that related to the protection of national products and consumer.

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## RATE CONSTANT OF SOME AMINO DERIVATIVES DISSOCIATION

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### ABSTRACT

Amino glycoside derivation including, Neomycin, Streptomycin, Kanamycin and Gentamycin with special reagents, which are benzoylchloride; benzene sulfonyl chloride and phthalic anhydride were made to enhance Uv-detectability for HPLC analysis. But there are many problems facing pre column derivation and in order to solve this, the conductivity of antibiotic derivatives were used to calculate the dissociation constant and the hydrolysis rate which determined concern type reaction. In addition the characteristics those controlling the hydrolysis of antibiotic-derivatives were investigated.

**Keywords:** Aminoglycosides, derivative reagents, conduct metric measurement, dissociation constant calculation.



## ثابت سرعة تحلل بعض مشتقات الامينوكلايكوسيدات

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## الخلاصة

تتضمن مركبات الأمينوكلايكوسيد صيغ متعددة منها النيومايسينو الاستربتومايسينو الكاناميسينو الجنتاميسينو التي يتم معالجتها بكواشف معينة لتحسين تحديد امتصاصيتها بواسطة الأشعة فوقالنفسيجية لمركبات الكواشف كالبنزويل كلوريد والبنزين باراسلفونيل كلوريد والتلوين بارا سلفونيل كلوريد والفتاليكاتهدريد من خلال الحصول على معقدات لتحديد كيميائيا بواسطة تقنية كروماتوغرافيا السائل عالي الكفاءة، الا انه هناك مشكلة تواجه تلك الدراسة وهي تفكك المعقدات التي يتم حقنها في العمود، ولهذا وجد من الضروري معرفة استقراره تلك المعقدات من خلال تعيين ثابت التفكك ومعدل التحلل لهما وذلك بدراسة توصيلية تلك المعقدات المتكونة، وكذلك تم تحديد العوامل التي تتحكم في عملية تحلل كل منهما.

الكلمات المفتاحية: الامينوكلايكوسيدات، العوامل الكاشفة، مقياس التوصيلية، حساب ثابت التحلل.

## INTRODUCTION

Aminoglycoside are groups of antibiotics that are used to treat certain bacterial infection (Macdonal, 1978). This group of antibiotics includes, Gentamycin, Kanamycin, Neomycin and Streptomycin. Some of these antibiotics are complex and consists of two major isomeric components. Streptomycin the first amino glycoside was isolated from streptomyces griseous in mid-1940S (Macdonal,1978). This antibiotic effective against tuberculosis. Neomycin is analogous to streptomycin and isolated from *Streptomyces faradiae* (Tsuji & Jenkins,1986), also Kanamycin produced by *Streptomyces kanamyceticusanti* microbial spectrum is similar to neomycin(Sybil,1998) Gentamycin's. Neomycin's, Kanamycin and streptomycin's are antibiotic belonging to the amino glycoside group and they are effective against wide variety of microorganisms(Delyet *al.*, 1975). Because these antibiotics are not Uv-absorbent therefore we need to introduce a suitable organic reagent as chromosphere in pre column technique for high performance liquid chromatography (HPLC) to enhance solubility, separation and detectability. Its bactericidal power derives from the binding of the molecule to the protein of the bacterial subunit 30S, which disturbs protein synthesis (Roberts *et al.*,2001).

The chemical modification has been made in order to increase sensitivity and selectivity for a variety of pharmaceutical compounds to some or all of the functional groups because no Uv-absorbance between 212-360 nm and chromatic characteristic of these compound can be improved (Leach *et al.*,1951).

Some antibiotics have hydroxyl and amino functional groups (i.e, poly functional with two types of groups) which effected by reaction conditions (2010; David, 2009).For trace analysis acid chloride the suitable reagent for quantification by HPLC.The reaction of amino sugar with acid chloride is effected by base as catalyst which enhance the reaction and neutralized the liberated acid (Snyder & Kirkaland,1979; Furniss *et al.*,1984).

The effects of solvents and/ or catalysts play important role in types of products. i.e. complete or incomplete (Ziadan, 1989).The problems facing amino characterization of glycosides (antibiotics) and their derivatives.

- The quantitative determination of antibiotics is one of the most difficult areas of pharmaceutical analysis (Snyder & Kirkaland, 1979).
- Derivatization products recovery from solvent shows difficulty specially that used for recrystallization.



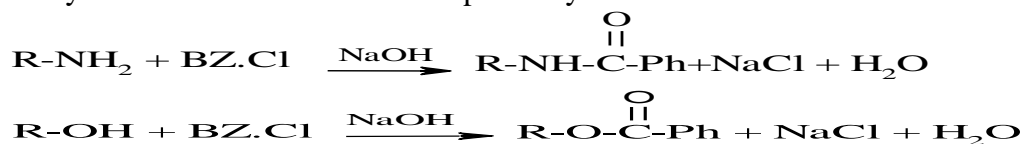
- The derivatives of aminoglycoside under investigation which gives more than one type of products (ester and/ or amide)(Ziadan,1989), and this made difficulty to find a suitable solvent to give precise Uv- result(Delyet *al.*, 1975).
- Most amino glycoside derivatives have not sharp melting point (Furnisset *al.*, 1984).

### Derivation Reagents

Benzoyl chloride was suitable reagent for aliphatic derivatives and polyhydric alcohol as benzoate also for derivatives primary and secondary amine as Benz amide benzene sulfonyl chloride and P-toluene sulfonyl chloride used for primary and secondary amine gives benzene sulfonamide and P-Toluene sulfonamide respectively (Furnisset *al.*, 1984). Phthalic anhydride used for primary amine and resolution of racemic alcohol as phthalate(Delyet *al.*, 1975;Furnisset *al.*,1984). alcohol reacts with benzene sulfonyl chloride to form ester (Morrison&Boyd,1995).The product called sulfonate(Graham&Craig,2002).

### Physical properties of antibiotic derivation

Neomycin, Kanamycin, Gentamycin and Streptomycin have OH-Groups and NH<sub>2</sub>-groups, and according to these functional groups; they have amine and alcohol character. In general, the reaction of amine and alcohol with acid chloride (Benzoyl chloride, BZ.Cl) using catalysis to form amide and ester respectively in "Schotten-Baumann" reacts as follows:-



These are simple molecules, but for complex molecule such as Neomycin, Kanamycin, Gentamycin and Streptomycin the product is dependent on reaction conditions(Morrison & Mosher,1971).

### Benzoylation

An acid chloride is widely used as a derivatizing reagent to enhance detectability for isolation and quantification analysis(Brooks *et al.*, 1953). The (BZ.Cl) used for separation of some carbohydrates by HPLC with pre benzoylation(Lehrfeld,1976).Some polyhydric alcohols were separated as -P-NO<sub>2</sub>- benzoate (SchwerZenback, 1977). p-NO<sub>2</sub>-benzoate used as a derivative from neomycin complex for separation by HPLC (Ziadan,1989).

### Amidation

Acid chlorides were used also for amidation of amino groups to produce polyamine, it is a simple and rapid procedure and also(Linskens&Jakson,1978).benzoyl chloride used for derivatization of amine in liquid chromatography (LC)(Clark & Wells,1978).The reaction of acid chloride with primary and secondary amines also used(Iwamori *et al.*, 1979).The BZ.Cl used for analysis of aminoglycoside antibiotics as benzoyl derivatives by HPLC and its application to quantitation of Neomycin in perilymph.The benzoyl chloride used for pre-column derivatization of neomycin complex (Ziadan,1989). The molar conductivity of strong electrolytes vary linearly with square root of concentration. (Crow,1994).

$$\Lambda = \Lambda_{\infty} - B\sqrt{C} \quad \dots\dots\dots(1)$$

This relation is called Kohlrausch's law (Bokris & Reddy, 1977).

Λ - equivalent conductance.

Λ<sub>∞</sub> - equivalent conductance at infinity dilution .

C-concentration.



**Weak electrolytes**

Weak electrolytes are partially ionized in solution. They include weak Bronsted acids and bases. The molar conductivities arise from the displacement of equilibrium toward products at low molar.



The conductivity depends on the number of ions in solution and therefore on the degree of ionization  $\alpha$  of electrolyte.

$$K_a = \frac{[H_3O^+][A^-]}{[HA][H_2O]} \dots\dots\dots (3)$$

$$[H_3O^+] = \alpha C, [A^-] = \alpha C, [HA] = (1 - \alpha) C$$

$$K_a = \frac{\alpha^2 C}{1 - \alpha} \dots\dots\dots (4)$$

The electrolyte is fully ionized at infinite dilution, and its molar conductivity,  $\Lambda_\infty$ . (Paul, 2001).

$$\alpha = \frac{\Lambda}{\Lambda_\infty} \dots\dots\dots (5)$$

Substituting eq. (5) in eq. (4) we get eq. (6)

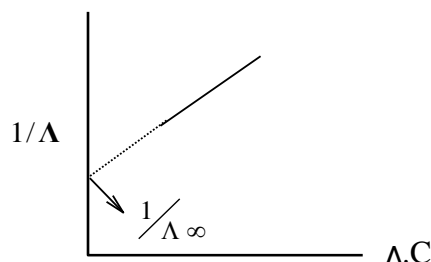
$$K_a = \frac{C \Lambda^2}{\Lambda_\infty (\Lambda_\infty - \Lambda)} \dots\dots\dots (6)$$

Which can be rearranged to give :

$$\frac{1}{\Lambda K_a \Lambda_\infty^2} = \frac{1}{\Lambda_\infty} (\Lambda C) + \frac{1}{\Lambda_\infty} \quad \text{(Ostwald dilution law)}$$

Plotting of  $(1/\Lambda)$  versus  $(\Lambda C)$  give straight line with a slope of  $(1/ K_a \Lambda_\infty^2)$   $(1/\Lambda_\infty)$  can be determined by extrapolation to zero.

$$\text{Slope} = 1/ K_a \Lambda_\infty^2, \text{ intercept} = 1/\Lambda_\infty$$

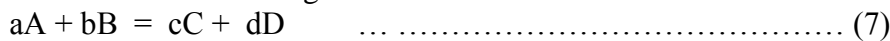


**Chemical Kinetics**

Chemical kinetics concerns with study of reaction rates, the changes in concentrations of reactants (or products) as a function of time. Rate laws and rate constants.

The rate is a change in some variable per unit of time.

Consider the general reaction :



Rate change of [C] is defined as rate =  $\frac{1}{c} \frac{d[C]}{dt}$ .....(8)

The rate varies with time and is equal to some function of concentration (Paul, 2001).

$$\frac{1}{c} \frac{d[C]}{dt} = f[A], [B], [D]. \dots \dots \dots (9)$$

## MATERIALS AND METHODS

### Instruments

The main instruments used in the study were:

1. Conductivity meter. HANNA Instruments. HI 8733. Made Portugal.
2. Melting point apparatus. Philip Harris, Shinnston-England, serial No. B/A-211.
3. Weight balance; Sartorius analytic type. A200S. Made in Germany.

### Experimentation of Drugs

Benzoyl chloride derivatization procedure(Furnissset al.,1989).

#### Sodium hydroxide (NaOH)

Dissolve 1.0g of drugs (*Gentamycin, Kanamycin, Neomycin* and *Streptomycin*) in 50mL of D.W in 150mL conical flask then add 12 mL of benzoylchloride and 30 mL of 10% NaOH solution. Stopper the flask and shake vigorously at frequent intervals until the odor of benzoylchloride disappear (about 5 minutes) and crystalline product precipitates out. Collect the crystals by suction filtration and wash well with water. The crude product was recrystallized from ethanol and dried in oven. Benzensulfonyl chloride derivation procedure (Furnissset al., 1989).

#### Sodium hydroxide (NaOH)

Dissolve 1.0g of drugs in 30mL of 10% aqueous NaOH solution In 150mL conical flask and then add (3.0 mL of *benzene sulfonyl chloride* (toluene-p-sulfonyl chloride) in 13mL cold acetone, cork the flask securely and shake the flask frequently for duration 15-20 minutes. Cool the flask in running water from the tap and then pour its contents into about 150mL water. Stir the aqueous mixture well and wash the crystal with H<sub>2</sub>O and drain. Recrystallize the product by methylated spirit and dry on filter paper in the air.

### Determination of OH groups

Put 1M phallic anhydride, 10mL equivalent D-glucose. 12.5mL of reagent and 1.8g D-glucose all in round bottom flask for 1hr on steam bath. 12.5mL of reagent without sample as blank, heated on steam bath for 1hr, and 5 mL of distilled water to both and heat for 5min. Cooling then titrated both blank and sample with 0.5N NaOH using pH indicators.

Occupation method: (Smith & March, 2001).

Using the following equation:  $1/\bar{v} = 1/\bar{v}^0 - nK[A]/\bar{v}^0$  .....(10)

[A] reagent concentration need for antibiotic substitution.

$\bar{v}^0$ - average number of reagent molecule bonded to one antibiotic molecule., n. number of position available for occupation on antibiotic.

and K-equilibrium constant.

### Kinetic study procedure

1. prepared 100 mL of 0.05M ester in methanol and 0.05M NaOH.
2. Take 25mL of NaOH and dilute to 50mL and measure the conductivity,  $C_0$ .
3. Add 25mL of 0.05M ester (form from reagents) and 25mL of 0.05M NaOH, start to measure conductivity after 2min. for first 10min and then 5min,  $C_t$ .
4. Take solution of ester and NaOH; after 1hr. to complete reaction, then measure the conductivity,  $C_\infty$ .

### Conductivity measurement

1. prepared 0.1M of reagents benzoyl chloride (BZ.Cl), benzene sulphonyl chloride (BZ.S.Cl) Para toluene sulfonyl chloride (T.P.S.Cl) and phthalic anhydride (ph.A).
2. Prepared stock solution for antibiotic-derivatives ( $g.L^{-1}$ ) as in (Tables 5-16).
3. Makes different solution by dilution for (1,2) and measure the conductivity.

### Determination of rate constant of ester hydrolysis catalyzed by sodium hydroxide.

x

The second order reaction when ( $a = b$ ) is  $k_2t = \frac{x}{a(a-x)}$ , using

conductivity instead of concentration as:

$$C_0 \text{ at } t=0, C_t \text{ at } t=t, \text{ and } C_\infty \text{ at } t=\infty$$

$$\text{Therefore } a = C_0 - C_\infty \text{ and } x = C_t - C_\infty.$$

$$k_2 = \frac{1}{a \cdot t} \cdot \frac{C_t - C_\infty}{C_0 - C_t} \dots\dots\dots(11)$$

$$C_t = \frac{1}{a \cdot K_2} \cdot \frac{C_0 - C_t}{t} + C_\infty \dots\dots(12)$$

Plotting of  $\frac{C_0 - C_t}{t}$  against  $C_t$  gives a straight line with slope  $\frac{1}{a \cdot k_2}$  and intercept at  $C_\infty$  (John & Ralph, 1981).

## RESULTS AND DISCUSSION

### Conductivity measurement

(Tables 1-4) and (Figure 1-4) show dissociation constant ( $K_a$ ) for several concentration for reagents (BZCl), (BZ.S.Cl), (T.P.S.Cl) and (Ph.A), while the (Tables 5-16) and (Figure 5-11) gives the antibiotic-derivatives results.

**Table (1):** Dissociation constant of *BZ.CL* at several concentration from conductivity measurements (Solvent MeOH).

C (M.L <sup>-1</sup> )	k (ms.cm <sup>-1</sup> )	Λ (ms.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.100	9.85	98.5000	0.001015	9.850
0.065	6.97	107.231	0.009330	6.970
0.048	5.68	118.333	0.008450	5.679
0.038	4.72	124.211	0.008051	4.720
0.032	4.27	133.4375	0.007490	4.270
Cal.      Λ ∞=176.923      K <sub>d</sub> =0.0661				

**Table (2):** Dissociation constant of *BZ.S.CL* at several concentration from conductivity measurements (Solvent MeOH).

C (M.L <sup>-1</sup> )	k (ms.cm <sup>-1</sup> )	Λ (ms.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.100	9.01	90.1000	0.011100	9.01
0.065	5.68	87.3850	0.011440	5.68
0.048	4.49	93.5542	0.010690	4.49
0.038	3.71	97.6320	0.010243	3.71
0.032	3.16	98.7500	0.010130	3.16
Cal.      Λ ∞= 111.44      K <sub>d</sub> = 0.2423				

**Table (3):** Dissociation constant of *T.P.S.Cl* at several concentration from conductivity measurements (Solvent MeOH).

C (M.L <sup>-1</sup> )	k (ms.cm <sup>-1</sup> )	Λ (ms.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.100	6.01	60.100	0.01664	6.01
0.065	4.32	66.462	0.01505	4.32
0.048	3.49	72.708	0.01375	3.49
0.038	2.93	77.105	0.01297	2.93
0.032	2.61	81.563	0.01226	2.61
Cal.      Λ ∞= 109.61      K <sub>d</sub> = 0.0643				

**Table (4):** Dissociation constant of (*Ph.A*) at several concentration from conductivity measurements (Solvent MeOH).

C (M.L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.100	32.0	320.000	0.003125	32.0
0.065	20.5	315.385	0.003171	20.5
0.048	16.5	343.75	0.00291	16.5
0.038	14.1	371.053	0.002695	14.1
0.032	12.1	378.125	0.002645	12.1
Cal.      Λ ∞ = 461.32      K <sub>d</sub> = 0.124				

**Table (5):** Dissociation constant of *Kan.BZ.* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.031000	11.0	354.8390	0.002818	11.0
0.002000	7.9	3950.000	0.0002532	7.9
0.001490	6.1	4093.960	0.0002443	6.1
0.001185	5.3	4472.574	0.0002236	5.3
0.000980	4.5	4577.820	0.0002184	4.5
Cal.      Λ ∞ = 357.73      K <sub>d</sub> = 0.0154				

**Table (6):** Dissociation constant of *Neo.BZ.* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.026	5.4	207.692	0.00481	5.4
0.017	3.9	229.41	0.00436	3.9
0.012	3.1	258.34	0.00387	3.1
0.0098	2.9	295.92	0.00338	2.9
0.00813	2.6	325.0	0.00308	2.6
Cal.      Λ ∞ = 652.13      K <sub>d</sub> = 0.00353				

**Table (7):** Dissociation constant of *Gent.BZ* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.0288	7.5	260.417	0.00384	7.5
0.01872	5.7	304.487	0.00328	5.7
0.0139	4.6	330.935	0.00302	4.6
0.0110	4.2	381.818	0.00262	4.2
0.009	3.6	400.0	0.0025	3.6
Cal.      Λ ∞ = 1222.67      K <sub>d</sub> = 0.0015				

**Table (8):** Dissociation constant of *Gent.T.P.S.* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.069	64	927.54	0.0011	64
0.045	49	1088.9	0.00092	49
0.033	37.5	136.4	0.00088	37.5
0.027	29.3	1085.2	0.00092	29.3
0.022	25.2	1145.5	0.000873	25.2
Cal.      Λ ∞ = 1456.7      K <sub>d</sub> = 0.069				

**Table (9):** Dissociation constant of *Streptomycin* at several concentration from conductivity measurements (Solvent MeOH).

C (M. L <sup>-1</sup> )	k (ms.cm <sup>-1</sup> )	Λ (mS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.01	2.89	289.0	0.00346	2.89
0.0087	2.48	285.1	0.003508	2.48
0.007	2.16	308.57	0.003241	2.16
0.0061	1.92	314.75	0.003177	1.92
0.0053	1.76	332.075	0.003011	1.76

**Table (10):** Dissociation constant of *Strept.BZ.* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.018	2.4	133.33	0.0075	2.4
0.014	2.2	157.14	0.00636	2.2
0.011	2.1	190.91	0.00524	2.1
0.009	2.1	233.33	0.00429	2.1
0.008	2	250.00	0.0040	2
Cal.      Λ ∞ = 66.49      K <sub>d</sub> = 0.024				

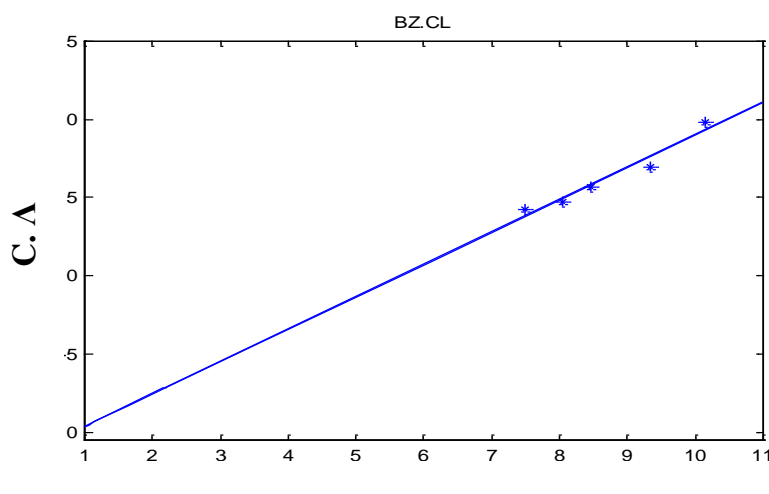


**Table (11):** Dissociation constant of *Strept.BZ.S.* at several concentration from conductivity measurements (Solvent MeOH).

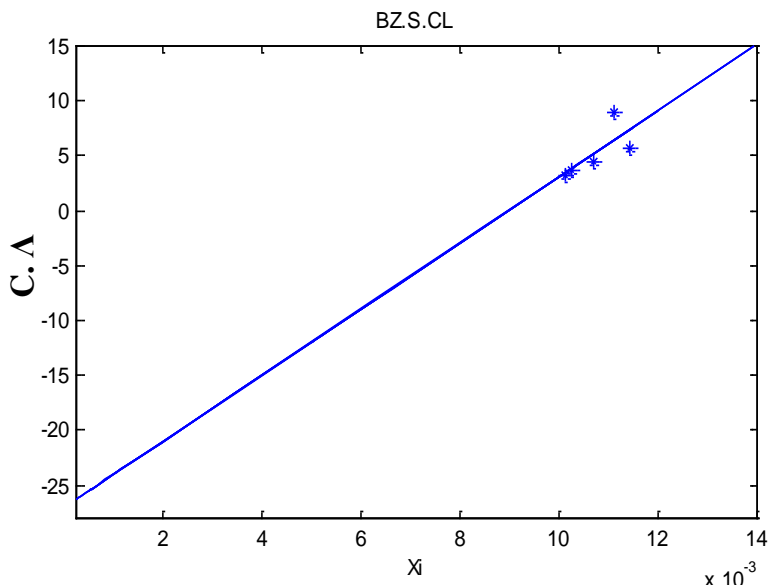
C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.026	9.2	353.85	0.00283	9.2
0.02	7.9	395.0	0.00253	7.9
0.016	6.9	431.25	0.00232	6.9
0.013	6.2	476.92	0.0021	6.2
0.011	5.7	518.18	0.0019	5.7
Cal.		Λ ∞= 2125.056	K <sub>d</sub> = 0.000485	

**Table (12):** Dissociation constant of *Strept.T.P.S.* at several concentration from conductivity measurements (Solvent MeOH).

C (g. L <sup>-1</sup> )	k (μs.cm <sup>-1</sup> )	Λ (μS.cm <sup>2</sup> .mol <sup>-1</sup> )	1 / Λ	C. Λ
0.021	9.2	438.095	0.00228	9.2
0.016	7.5	468.750	0.00213	7.5
0.013	6.2	476.923	0.00210	6.2
0.011	5.2	472.727	0.00212	5.2
0.009	4.4	488.89	0.00205	4.4
Cal.		Λ ∞=556.1	K <sub>d</sub> = 0.0065	



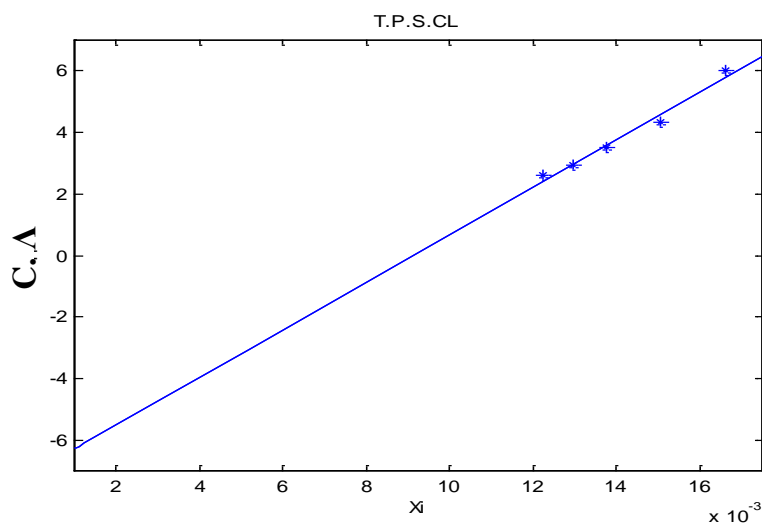
**Figure (1):** Determination of dissociation constant (K<sub>d</sub>) from plotting (1/Λ) against (C. Λ) for *BZ.CL*.



$1 / \Lambda$

Slope = 3008.9      b = -27.0

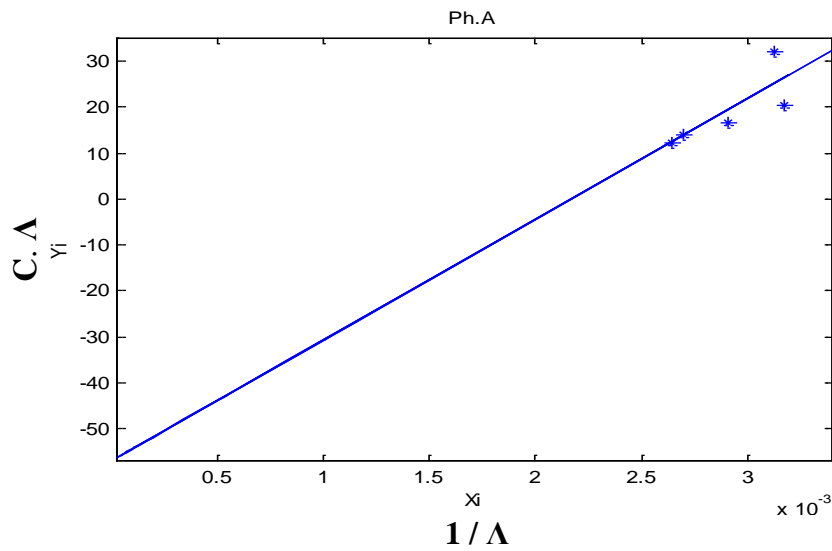
**Figure (2):** Determination of dissociation constant ( $K_d$ ) from plotting ( $1/\Lambda$ ) against ( $C. \Lambda$ ) for *BZ.S.CL*.



$1 / \Lambda$

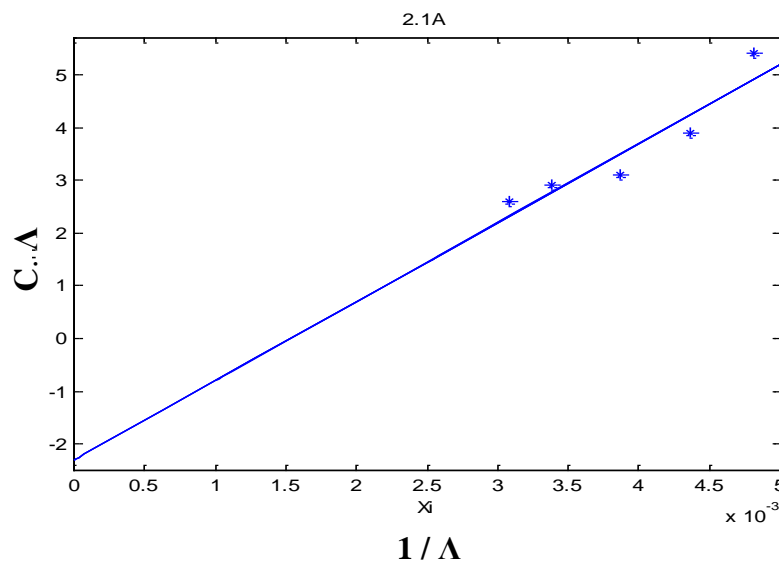
Slope = 772.778      b = -7.0504

**Figure (3):** Determination of dissociation constant ( $K_d$ ) from plotting ( $1/\Lambda$ ) against ( $C. \Lambda$ ) for *T.P.S.CL*.



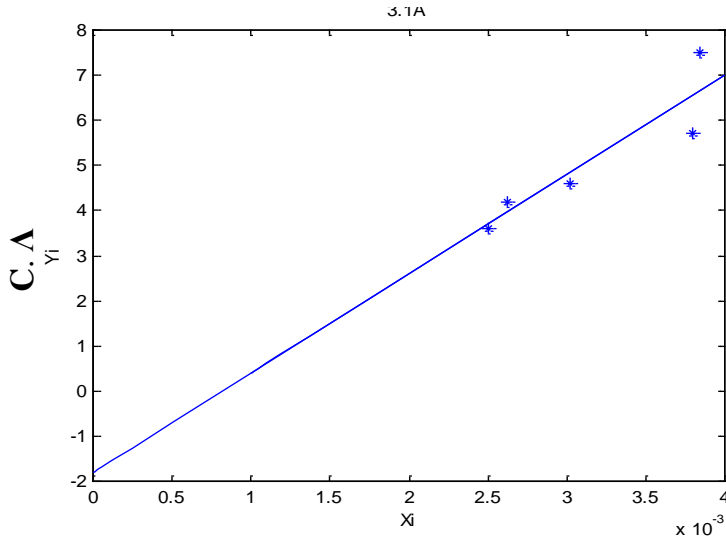
Slope=26295.0      b=-57.0

**Figure (4):** Determination of dissociation constant ( $K_d$ ) from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ) for *Ph.A.*



Slope = 1499.9      B = -2.3

**Figure (5):** Determination of dissociation constant ( $K_d$ ) for *Neo.BZ. /A* from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ).

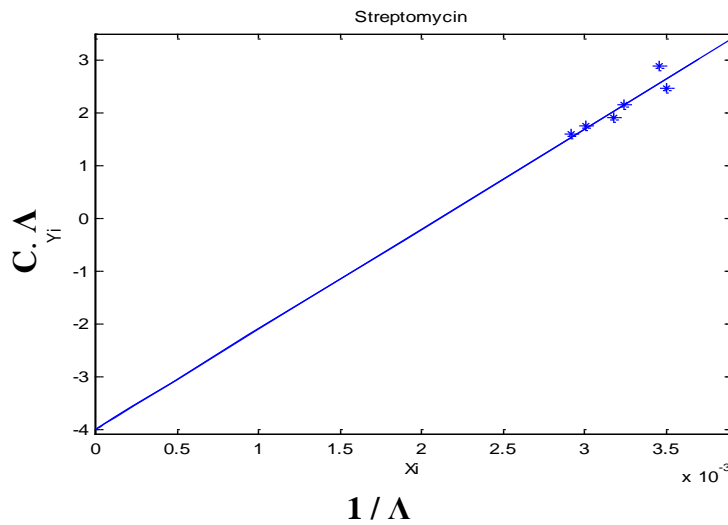


$1 / \Lambda$

Slope = 2200.8

$b = -1.8$

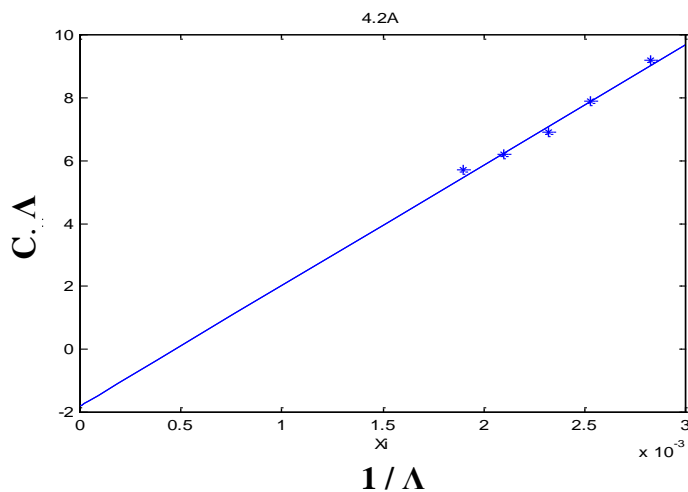
**Figure (6):** Determination of dissociation constant ( $K_d$ ) for *Gent.BZ.* from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ).



Slope = 1900.8

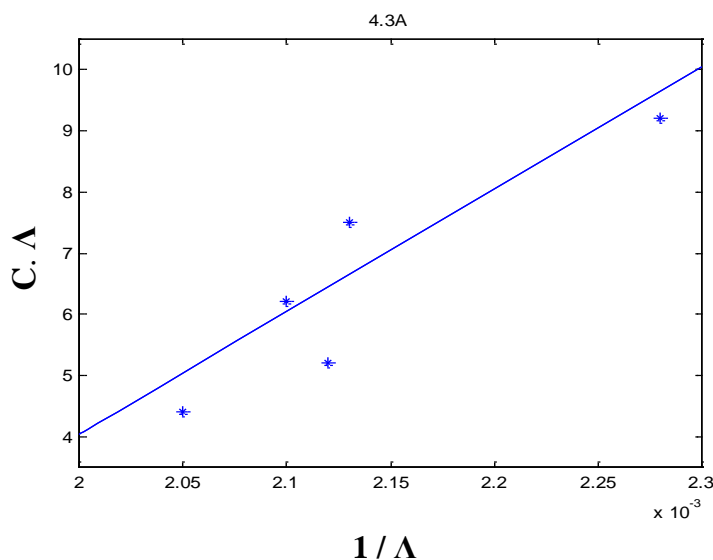
$b = -4.0$

**Figure (7):** Determination of dissociation constant ( $K_d$ ) for *Streptomycin* from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ).



Slope = 3825.1       $b = -1.8$

**Figure (8):** Determination of dissociation constant ( $K_d$ ) for *Strept.BZ.S.* from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ).



Slope = 20020       $b = -36.0$

**Figure (9):** Determination of dissociation constant( $K_d$ ) for *Strept.T.P.S* from plotting ( $1 / \Lambda$ ) against ( $C. \Lambda$ ).

### Conductivity measurements

#### Dissociation constant( $K_d$ )

The conductivity measurements to calculate dissociation constant( $K_d$ ) through dilution for reagents as in (Tables1-4) and for antibiotic-derivatives as in (Tables4-12).The dissociation constant( $K_d$ ) for reagents follow the order T.P.S.Cl (0.0643)<BZ.Cl(0.0661)<Ph.A(0.124) <BZ.S.Cl (0.2423).

Substituted reagent on the Kanamycin not the same process to give antibiotic-derivative product. In the (Figures 1-9) the molar conductivity at infinite dilution ( $\Lambda_{\infty}$ ), is differ for antibiotic-derivatives which leads to give various values of dissociation degree. From (Table9) the value of  $\Lambda_{\infty}$ , for Gen.BZ.S. is ( $1601 \mu\text{S}.\text{cm}^2 .\text{mol}^{-1}$ ) which give dissociation degree ( $\alpha$ ) is equal to (0.212).

**Table (13):**Kinetic study of *D-glucose-phthalic anhydride* derivatives.

Time (min.)	Vol. Of NaOH (mL)	$\bar{v}$	1/ $\bar{v}$	[A]/ $\bar{v}$
5	15.0	0.300	3.34 0	0.9000
10	17.4	0.348	2.870	0.7760
15	20.0	0.400	2.500	0.6750
20	21.5	0.430	2.326	0.6280
30	21.7	0.434	2.304	0.6220
40	23.7	0.462	2.165	0.5840
50	42.8	0.856	1.168	0.3154
60	44.0	0.880	1.136	0.3068

$\bar{v}$ - average number of reagent molecule bonded to one antibiotic olecule.

**Table (14):** Determiation of rate constant *Neo.BZ.* by conductivity Measurement (MeOH).

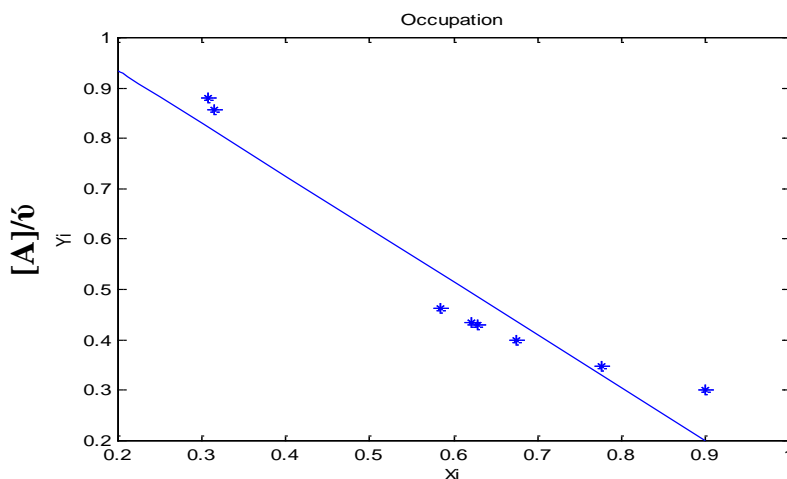
t (min).	$(C_0 - C_t)/t$	C t ( $\mu\text{S}.\text{cm}^{-1}$ )	K ( $\mu\text{S}.\text{cm}^{-1} .\text{min}^{-1}$ )
0	$\infty$	69.3	-
2	20.85	27.6	926.67
4	10.4	27.7	416.0
6	6.92	27.8	251.52
8	5.175	27.9	172.5
13	3.185	27.9	106.154
18	2.3	27.9	76.852
34	0.99	26.7	-
Cal.	Rate constant (k)=4301.08		Average=350.59

**Table (15):**Determiation of rate constant *Strept.BZ.S.* by conductivity easurement (MeOH).

t (min)	$(C_0 - C_t)/t$	C t ( $\mu\text{S}.\text{cm}^{-1}$ )	K ( $\mu\text{S}.\text{cm}^{-1} .\text{min}^{-1}$ )
0	$\infty$	69.3	-
4	-6.4	94.9	54.074
6	-4.58	96.8	21.695
8	-3.55	97.7	13.382
13	-2.2	98.3	9.73
18	-1.63	98.6	5.87
23	-1.27	98.5	4.2
28	-1.03	98.1	3.298
33	-0.87	98.1	2.298
38	-0.74	97.4	2.068
43	-0.32	83.1	-
Cal.	Rate constant (k)=111.02		Average=13.033

**Table (16):**Determination of rate constant *Strept.T.P.S* by conductivity measurement (MeOH).

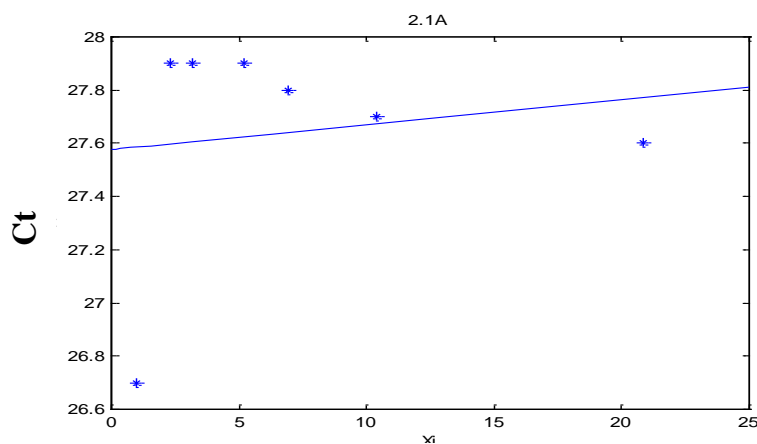
t (min).	(C <sub>0</sub> - C <sub>t</sub> )/t	C t (μs.cm <sup>-1</sup> )	K (μs.cm <sup>-1</sup> . min <sup>-1</sup> )
0	∞	69.3	-
2	17.9	33.5	397.78
4	8.975	33.4	211.176
6	5.95	33.6	125.263
8	4.463	33.6	93.95
13	2.76	33.4	64.796
18	2.0	33.3	50.0
23	1.57	33.2	41.86
50	0.752	31.7	-
Cal.	Rate constant (k)=863.93		Average=140.689



$1/u$

Slope = -1.0502       $[A]/u = 1.1448$

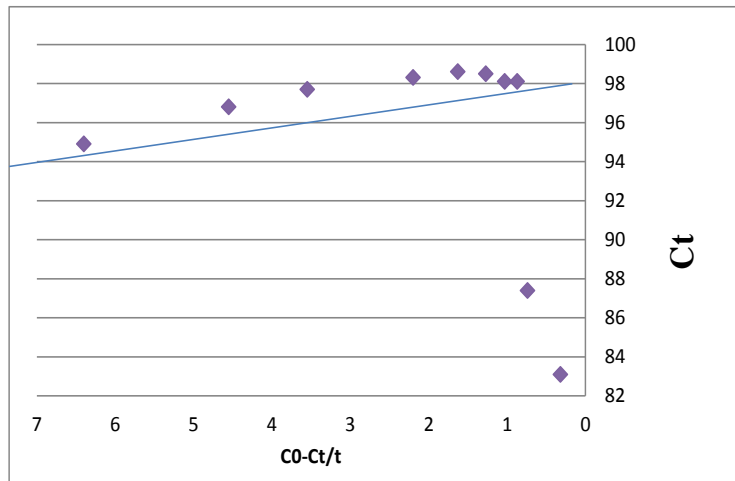
**Figure (10):**The relation between  $(1/u)$  and  $[A]/u$ .



$(C_0 - C_t)/t$

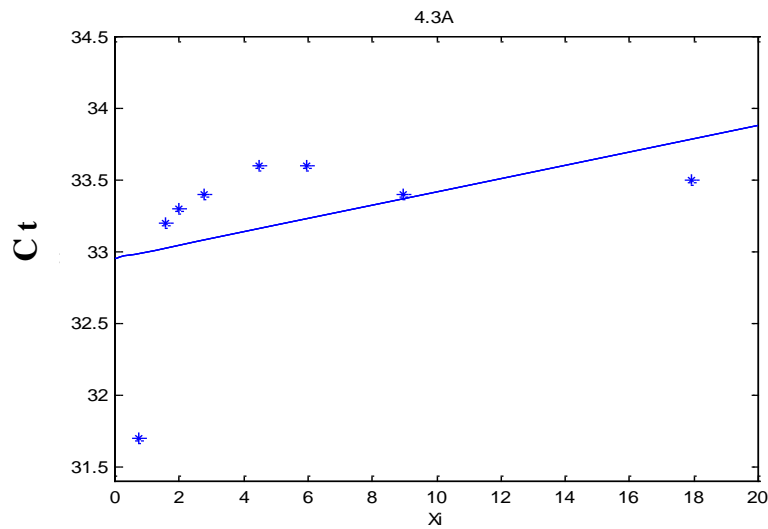
Slope = 0.0093       $C_\infty = 27.5767$

**Figure (12):**Curve of  $(C_0 - C_t)/t$  vs.  $C_t$  for *Neo.BZ. /A*.



$(C_0 - C_t)/t$   
Slope = 0.3603       $C_\infty = 95.3361$

Figure (13): Curve of  $(C_0 - C_t)/t$  vs.  $C_t$  for *Strept.BZ.S. /A*.



$(C_0 - C_t)/t$   
Slope = 0.0463       $C_\infty = 32.956$

Figure (14): Curve of  $(C_0 - C_t)/t$  vs.  $C_t$  for *Strept.T.P.S. /A*.



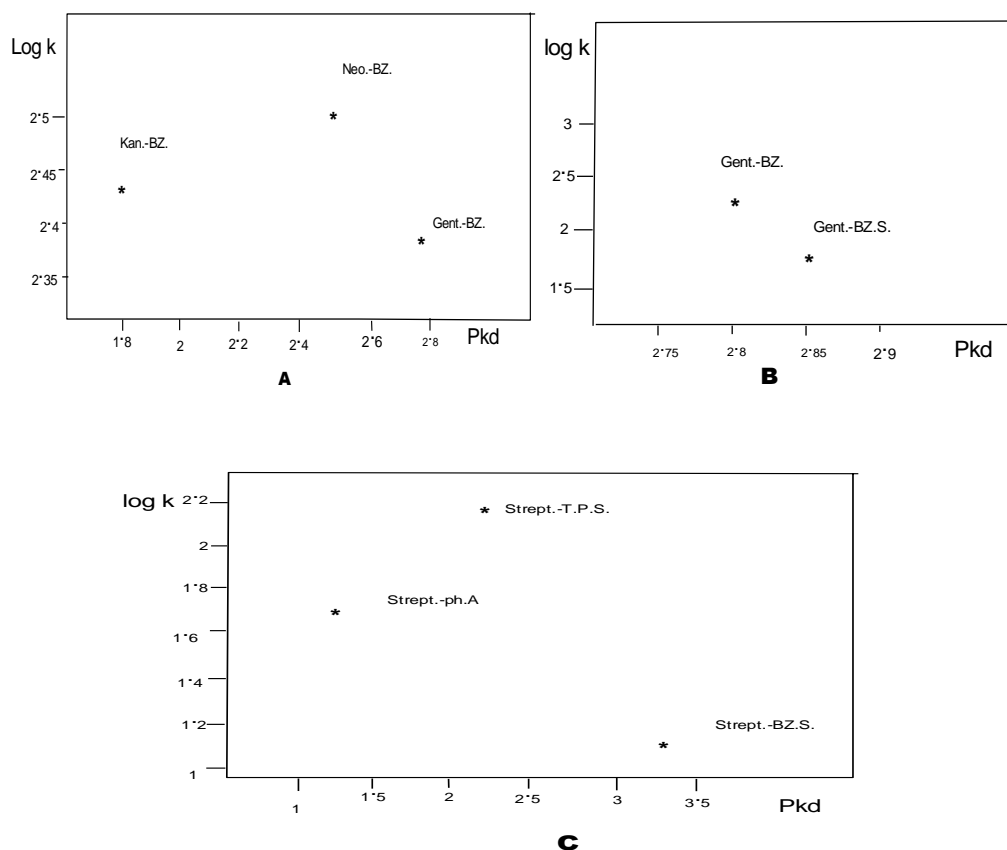
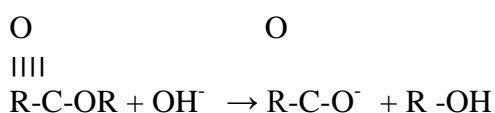


Figure (15):A, B, C plots log k against pkd.

### Rate constant (k)

Determination the rate constant(k) of alkaline (NaOH)hydrolysis of antibiotic-derivatives (as ester), the formation of benzoate or sulfonate ion and alcohol, as in general:



The rate formation of acetate ion proportional both to the concentration of ester and to the concentration of hydroxide ion (Frost & Ralph,1963).The rate constant represented in (Tables13-16) for antibiotic-derivatives, from theoretical view of these types of reaction should be follow second-order reaction, but the experimental values (Tables and Figures11-14) of derivatives not follow second-order rule because it gives different values for rate constant(k) when integrated rate.

This means that this type of hydrolysis for antibiotic-derivatives are not simple but it follow complex type reaction specially concurrent reaction type of different reactions produce a common product. The experimental results of antibiotic-derivatives, look like to be more convenient with theoretical concept and expression of thistype of reaction(Gorden,1978).This hydrolysis reaction for antibiotic derivatives is composite reaction due to presence of more than one type of functional groups(ester and amide) as well as the position of the same and functional groups where differ (Bluesone& Yan,1995).There are three ways in which structure

of the reacting ester can influence the rate of attack by hydroxide ions. These three ways relate to:

Electrophilic character of carbonyl carbon atom. Steric hindrance and stabilization of the carbonyl group by conjugation. The steric hindrance around the carbon site antibiotic molecule make difference in the rate of the  $SN^2$  reaction. The reaction of the antibiotic-derivatives strongly depended on stability of the central carbon (electrophilic) and on nucleophile reagent (Jack, 1962), (Figures 15-A, B and C) show the relation between rate constant,

$\log K$  and dissociation constant reagent  $pK_a$  in (Figure 15-A) reagents for the derivatization are constant while for antibiotic-derivatives are variant, the points are seen to deviate markedly from any possible straight line. This give indication that the antibiotic (Kanamycin, Gentamycin, Neomycin) are structural character control hydrolysis of (Kan.-BZ., Gen.-BZ. and Neo.-BZ.), while in (Figure 15-B and C) the predominant factor control the hydrolysis (Gen.-BZ and Gen.-BZ.S.) also (Strept.-BZ.S.), (strept.-ph.A.) and (strept. T.P.S.) are reagent character (Smith & March, 2001; Martin, 2003).

## CONCLUSIONS

The reactivity in the formation of products and hydrolysis of antibiotic-derivatives esters and/ or amides. The central carbon atom in reactant and product is tetrahedral, whereas carbon in the transition state is bonded to five atoms or groups, therefore, there will be an increase in crowding on going from starting substrate to the transition state. The more crowded the transition state relative to substrate the higher its energy will be, and the slower it will be formed. The rate of  $SN^2$  reaction is strongly dependent on the nature of nucleophilic reagent used, it increases with nucleophilic strength of the incoming. The rate of reaction is dependent on the nature of the solvent. The rate of reaction increases with increase solvent due to stabilized transition state of the reaction. The crystal form for some of these antibiotic-derivatives, does not melt directly to a liquid phase but first passes through an intermediate stage (the Para-crystalline state) which only at higher temperature undergoes transition to the liquid state. These intermediate states have been called liquid crystals, since they, display some properties of both liquid and crystals. Liquid crystals tend to occur when molecules are markedly unsymmetrical in shape. Also liquid crystal polymer does not melt, it decomposed. The chemical kinetics of antibiotic-derivatives hydrolysis follow parallel type of reaction (different reactants amide and ester produce a common product).



Our experimental result indicate this phenomena, the curvature of most curve of antibiotic-derivatives during the initial part of the reaction and after a sufficient length of time the curve becomes linear which means the reaction mechanisms are proceed through different reaction order.

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