



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 rhamnase linked to glucose units by β , 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.

RESULTSAND DISCUSION

FTIR Analysis

FTLR spectrum of the different gums are shown in (Table 1) and (Figures 1, 2 and 3) characteristic band at 3010.88 and3410.15 cm⁻¹referred to the OH bending of water, The boardpeak round 2343.51 and 2925 cm⁻¹ 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⁻¹ 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 ⁻¹			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⁻¹ attributed to the carbonyl, the finger print region of mustard spectra contain of many board bands in the region 677.01-493.27cm⁻¹ which the finger print region of standard gums spectra consist of 2-5 peaks at range 970.19-493.27cm⁻¹for Arabic gum, 808.17-709.80cm⁻¹ 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 3634.01cm^{-1} was assigned to the OH while the peaks at $2851.35, 2914.16$ and 2671.98cm^{-1} attributed to the alkane CH group, the band in 1663.66cm^{-1} was belong to C=O indicated to glucuronic acid.

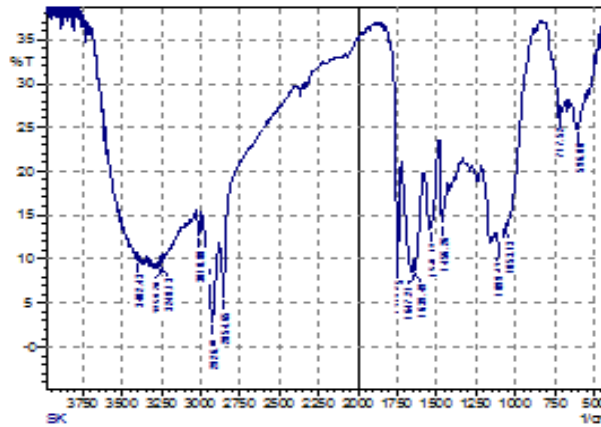


Figure (1):FTIR spectra of mustard seed gum.

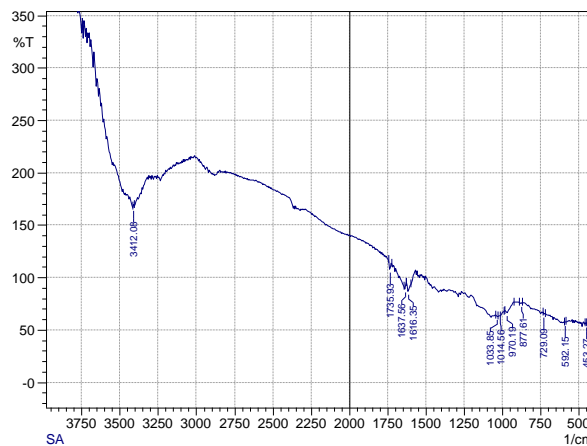


Figure (2): FTIR spectra of Arabic gum.

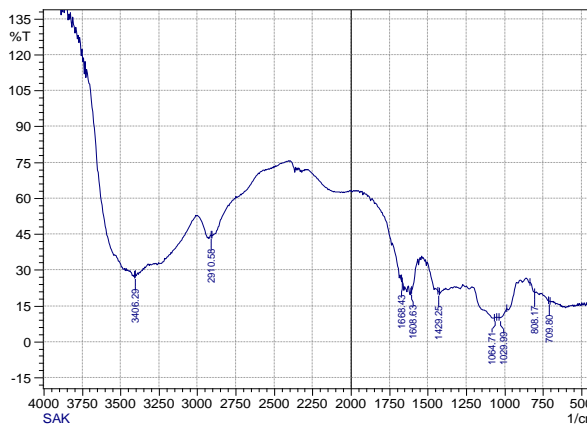


Figure (3):FTIR spectra of acacia gum.

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° in highest 53.35 cts .This indicates that mustard mucilage was a partially crystalline.

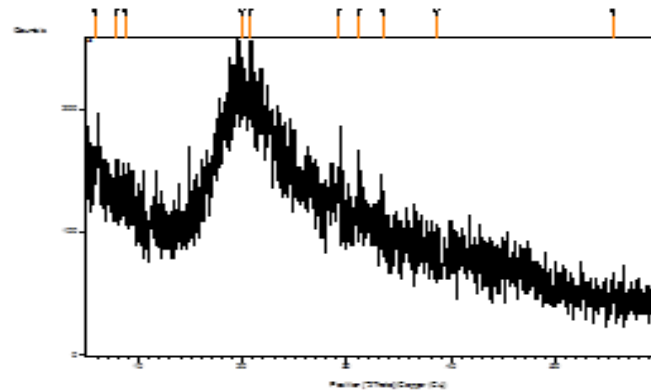


Figure (4): X-ray spectra of mustard mucilage.

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.

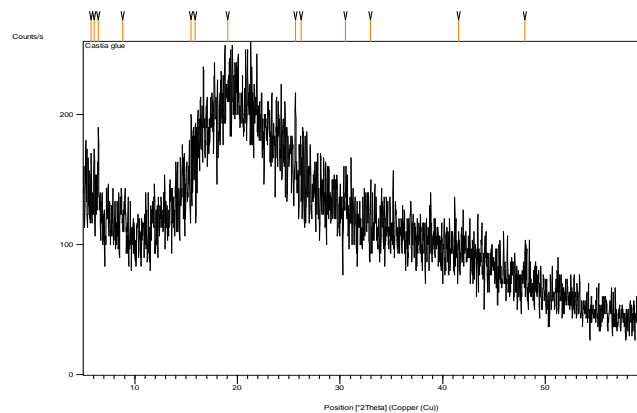


Figure (5):X-ray of Arabic gum.

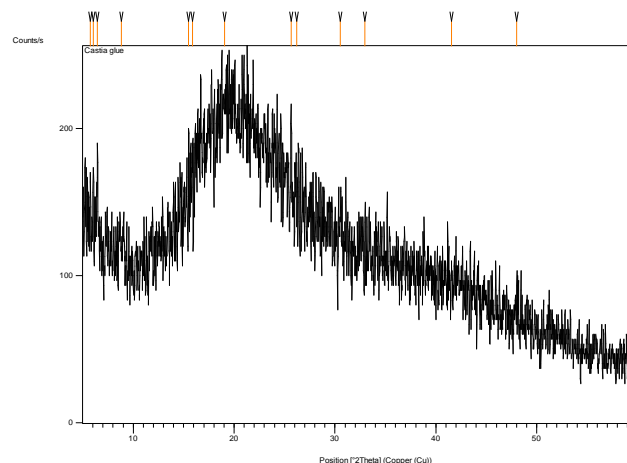


Figure (6): X-ray of Acacia gum

The result were in agreement with the finding by **Zaharuddin et al.(2014)**that okra gum had one weak peak, also resultswere 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%.

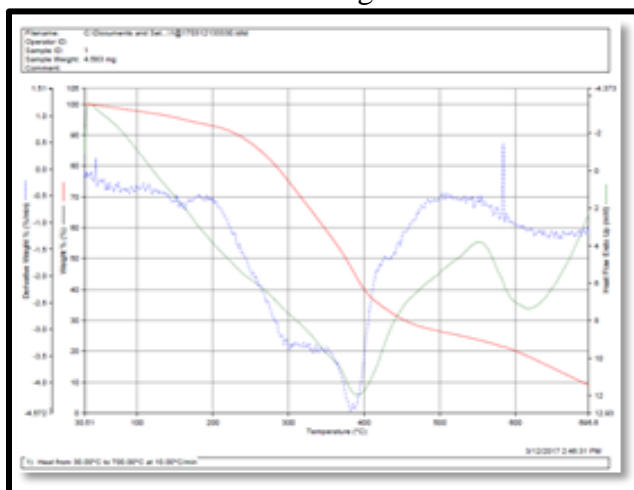


Figure (7): The rmogram of mustard mucilage.

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%.

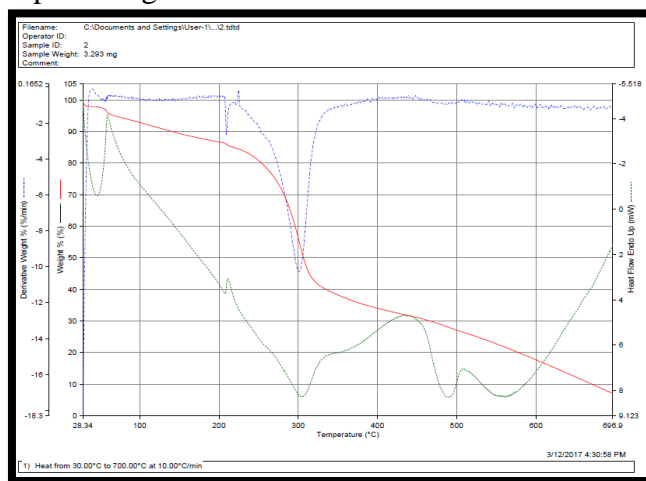


Figure (8): The rmogram of Arabic gum.

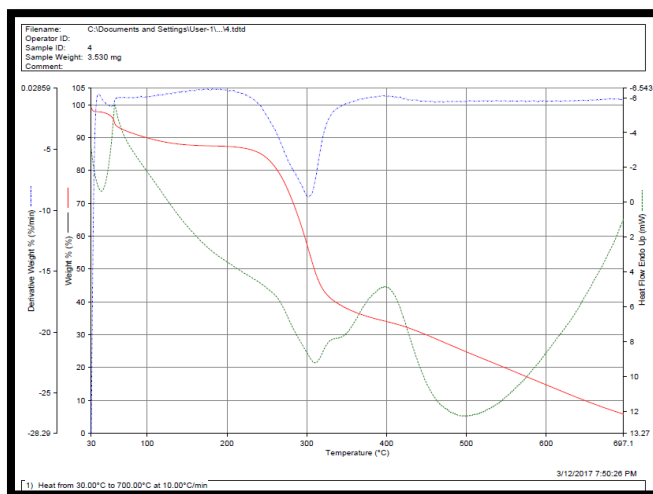


Figure (9):The rmogram of acacia gum.

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 CH₃ of rhamnose, indicators around at 3.98-3.087 ppm in mustard mucilage assigned to CH₂ 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 ₃
	2.761- 2.493			CH ₂ O
	4.187	4.03		C=O
	3.98 – 3.087			CH ₂
				CH

The results were agreed with **Daoub et al. (2016)** who indicated the H-NMR spectrum of acacia gum contain CH₃of rhamnose and C=O group of galactronic acid.

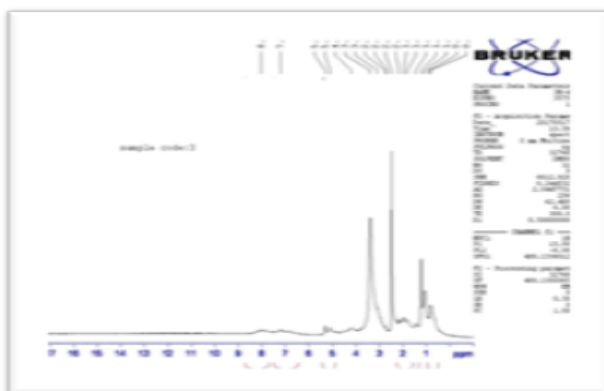


Figure (10): Nuclear magnetic resonance of mustard mucilage.

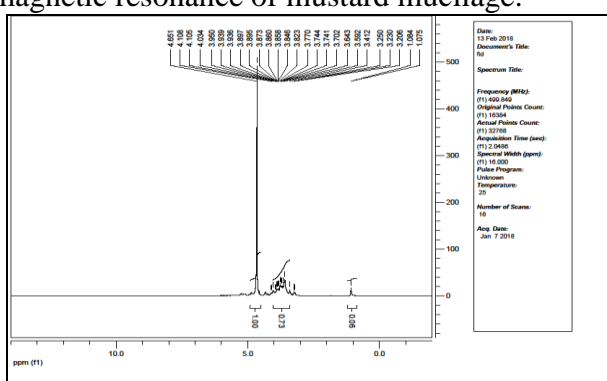


Figure (11): Nuclear magnetic resonance of Arabic gum.

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 CH₃ of rhamnose, indicators round at 3.73-3.85 ppm belong to CH₂OH of galactose.

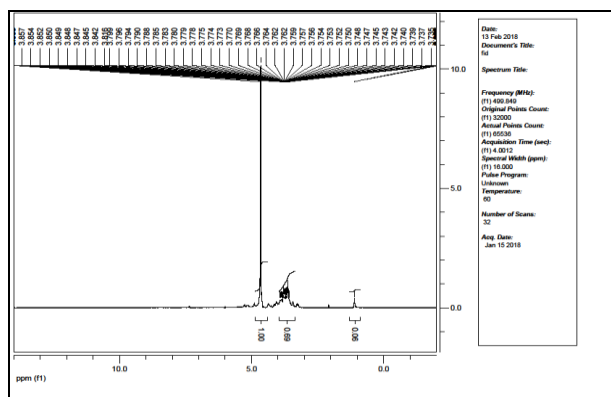


Figure (12): Nuclear magnetic resonance of acacia gum.

The H-NMR spectrum of the studied gums showed the presence of main sugars spectrum of acacia gum contain CH₃ 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.

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