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EXTRACTION AND EVALUATION OF PECTIN FROM SUNFLOWER HEAD RESIDUE USING ULTRASONIC

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ABSTRACT

Sunflower heads remaining after seed removal are a potential source of low-methoxyl pectin (35% dry weight basis). Ultrasonic was used during extraction of pectin from alcohol insoluble solids of sunflower heads using 0.75% sodium hexametaphosphate (1:20, w:v). The extraction was carried out using different ultrasonic power levels (20, 40, 60 and 80% of maximal power) at 70, 80 and 90°C for 15, 30 and 60 min. The obtained pectin was physically and chemically evaluated compared with commercial citrus low methoxyl pectin type 104 A/S of Hercules Copenhagen.

The highest yield (23.20%) and recovery (66.4%) of pectin were obtained by using ultrasonic at 60% power at 80°C for 15 min of extraction. These selected conditions yielded pectin with methoxyl content and degree of esterification of 4.55 and 31.97%, respectively. On the other hand, viscosity, gel strength, setting temperature and setting time of this pectin representing 80% of those of the commercial citrus low methoxyl pectin. Results showed that viscosity of the extracted pectin was gradually decreased with the increase of ultrasonic power, extraction temperature and extraction time.

INTRODUCTION

Wastes of farms and industries play an important role in environment contamination. Pectin is one of the famous by-products of citrus fruits peel and apple pomace residues. It is important in food processing as a food additive to modify the texture of jams, jellies, confectionery and in low fat dairy products.

Sunflower (*Helianthus annus L.),* the fourth source of oil-seeds worldwide with a production of 23851 thousand tons of seeds in 2002, representing around 18934 hectare of cultivated land is currently produced for its seeds. The annual production of sunflower seeds in Egypt was 44 thousand tons in 2002 (FAO, 2002).

The process of the traditional method of pectin extraction includes degradation by acid and deposition. The process lastes more than one hour at 80 – 100° C at pH 2. These conditions lead to pectin degradation, so this traditional method is not good for either quantity or quality of the extracted pectin Zhongdong *et al*. (2006)... Therefore, it is necessary to establish a new method by which the pectin could be extracted in a shorter time and with better quality.

Panchev *et al*. (1988) studied extraction of pectin from apple pomace with 0.5% nitric acid at a ratio of 1:20 (w/v). The pectic substances were extracted at 70 or 80°C for specified times under continuous or intermittent sonication. They found that continuous sonication at 80°C for 30 min

increased yield of pectin but decreased gel strength. On the other hand, when intermittent sonication was used the yield of pectin increased to 13.3% after 45 min of extraction.

Therefore, the effect of ultrasonic power, extraction time and extraction temperature on the yield and quality of extracted pectin from sunflower head residue were studied.

MATERIALS AND METHODS

Materials:

Sunflower head residues (vidoc variety) were collected from farms at New Valley Governorate, Egypt. Commercial citrus low methoxyl pectin (150 grade) type 104 A/S was obtained from Hercules Copenhagen DK-4623 Lille Skensved, Denmark. D (+) galacturonic acid monohydrate was purchased from Fluka Biochemika Co. (Slovakia).

Methods:

1- Production methods of pectin

a- Washing process:

Sunflower head residues without seeds were cleaned and sun dried before grinding and sieving (60 mesh).Ground sunflower head residues were suspended in ethanol 80% at a ratio of 1:15 (w/v) and heated to boiling for 15 and 30 min. Finally, the samples were filtered through cheese cloth and the filtered cake was dried at 40^{o}C overnight and stored at room temperature.

b- Extraction process:

Methods of extraction could be summarized as follows:-

1- Traditional method

Pectin was extracted from the dried washed material using the method of Iglesias and Lozano (2004).

2- Ultrasonic method

Ultrasound generator (Fisher sonic dismemberator, Model 300, 50 Hz, USA), equipped with a 19 mm diameter tip, was used in the extraction process of pectin from the washed material. The washed material was dispersed in 0.75% sodium hexametaphosphate at a ratio of 1:20 (w/v) and pH 4. The ultrasound probe was immersed into the mixture at a depth of approximately 5 cm. The extraction process was carried out using different ultrasound intensities (20, 40, 60 and 80% of the maximal power), different temperatures (70, 80 and 90^{o}C) and different times (15, 30 and 60 min) then the mixture was filtered through double cheese cloth.

c- Precipitation and purification of extracted pectin

Extract was weighed before addition of an equal weight of ethanol 95%. The mixture was then stirred and allowed to stand for 1 h in a refrigerator. The precipitated pectin was filtered through cheese cloth and washed three times with 95, 60 and 95% ethyl alcohol, respectively before washing with acetone. The purified pectin was dried at room temperature overnight until constant weight was attained and the dried pectin was ground to a fine powder (80 mesh) and kept at room temperature in polyethylene bags until analysis.

d- Pectin yield and recovery

The yield and the recovery of sunflower head residue pectins extracted by different techniques were calculated on dry weight basis using the following equations.

$$\% \text{ pectin yield} = \frac{\text{Wt. of produced pectin (g)}}{\text{Wt. of raw materials (g)}} \times 100$$

$$\% \text{ Pectin recovery} = \frac{\text{Wt. of produced pectin (g)}}{\text{Wt. of pectin in raw materials (g)}} \times 100$$

2-Methods of analyses: -

Moisture, ash, total sugars, fat and protein contents of raw samples were determined according to the AOAC (2000). Total pectin content, methoxyl content of extracted pectin and color intensity were determined as described in the methods of Ranganna (1978). Anhydrogalacturonic acid content was determined according to the colorimetric method reported by McComb and McCready (1952). Degree of esterification of pectin was determined according to the method described by Mizote and Odagiri (1975). The absolute viscosity of pectin solution was determined using a Brookfield rotational viscometer (Model RV-TDV-II) according to the method reported by Miyamoto and Chang (1992). Turbidity of pectin solution was determined as described in Stoll *et al*. (2003). Gel strength, setting time and setting temperature of pectin samples were determined according to the methods of Ranganna (1978). Jelly grade of pectin samples was determined according to the method described by Kertesz (1951).

RESULTS AND DISCUSSION

Chemical composition of sunflower head residue:

Data in Table (1) show that the moisture content of sunflower head residues was 10.47% which is in agreement with those reported by Wiesenborn *et al.* (1999). Ash, protein and fat contents of sunflower head residues were 16.28, 6.79 and 3.72% on dry weight basis, respectively. These results are in good agreement with those reported by Miyamoto and Chang (1992); Ahmed (1993) and Marechal and Rigal (1999). Also, the results reveal that the total sugars of sunflower head residues were 3.77% on dry weight basis.

Data in Table (1) illustrat that total pectin of sunflower head residues was 35.58% as calcium pectate and 25.28% as AGA on dry weight basis. Miyamoto and Chang (1992); Marechal and Rigal (1999) and Wiesenborn *et al.* (1999) found that total pectin of sunflower head residues as AGA ranged from 19.8 to 24% on dry weight basis.

Table (1): Chemical composition of sunflower head residue (Dry weight basis)

Constituents (%)	Sunflower head residues
Moisture	10.47
Ash	16.28
Protein	6.79
Crude fat	3.72
Total sugars	3.77
Total pectin (as calcium pectate)	35.58
Total pectin (as AGA*)	25.28

***AGA: Anhydrogalacturonic acid**

Effect of alcohol washing process on chemical composition of sunflower head residues:

The results in Table (2) show that the yield of sunflower head residues after 15 and 30 min of alcohol washing process was 91 and 87.5%, respectively. From the results in the same table, it could be deduced that alcohol washing process of sunflower head residues for 15 and 30 min caused reduction in ash content by 3.6 and 5.16%, respectively; in sugar content by 41.64 and 72.15%, respectively and in pectin content by 9.97 and 14.18%, respectively. Also, the results in Table (2) show that washing with alcohol 80% for 30 min caused sharp decrease in the absorbance at 420 nm of 60% alcohol sunflower head residue extract from 0.287 to 0.029 indicating sharp decrease in yellow pigments during the washing process. These results are in agreement with those reported by Sarhan (1975) and Marry *et al*. (2000).

Table (2): Effect of alcohol washing process on yield and chemical composition of sunflower head residues (Dry weight basis).

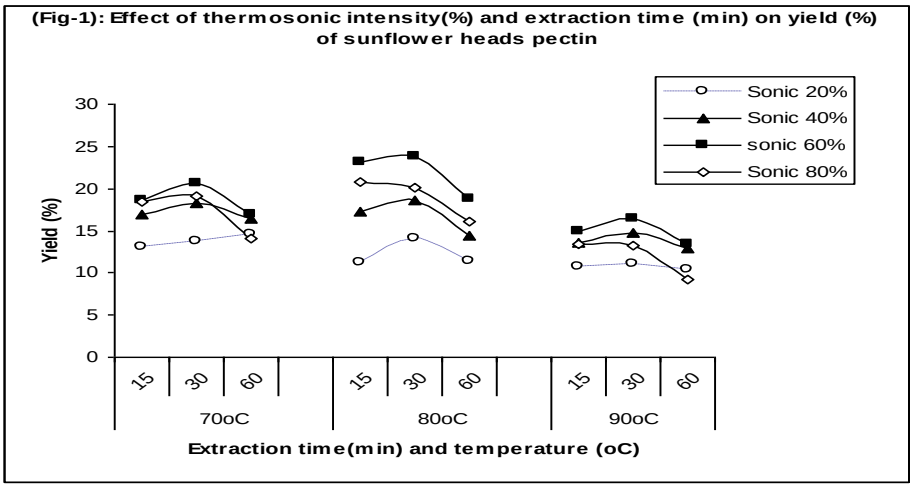
Properties (%)	Before washing	Washing time (min)	
		15	30
Yield*	-	91	87.5
Moisture	10.47	11.20	11.34
Ash	16.28	17.25	17.65
Total sugars	3.77	2.42	1.20
Absorbance at 420nm	0.287	0.179	0.029
Total pectin(as calcium pectate)	35.58	35.20	34.94

$$\text{* Yield} = \frac{\text{Quantity of material before washing - Quantity of material after washing}}{\text{Quantity of material before washing}} \times 100$$

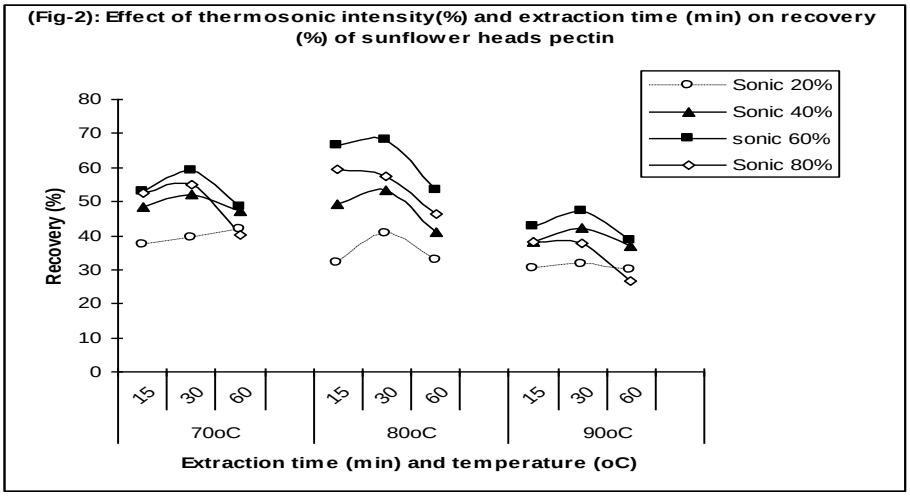
From the results in Table (2) it could be conclude that the optimal time for washing process of sunflower head residues was 30 min that led to remarkable reduction in the absorbance at 420 nm and sugar content with the lowest possible loss of pectin.

Effect of thermosonic intensity and extraction time on yield and recovery of extracted sunflower head pectin

From the results in Figs. (1and 2) it could be noticed that the highest yield (23.20 and 23.80%) and recovery (66.4 and 68.12%) of pectin were obtained when ultrasonic was used at 60% of its maximal power at 80°C for 15 and 30 min of extraction, respectively. These results exceeded that were obtained by the traditional method (14.52 and 41.56%) by about 60%. Panchev *et al*. (1988) mentioned that sonication of apple pomace using 0.5% nitric acid at a ratio of 1:20 (w/v) at 80°C for 30 min increased the yield of pectin from 10.8% for the unsonicated sample to 13.1%.



Control: Traditional method= 0.75% SHMP (1:20)/75°C/1 hr
Control: Yield: 14.52%
Yield = Wt of produced pectin / Wt of raw material X 100

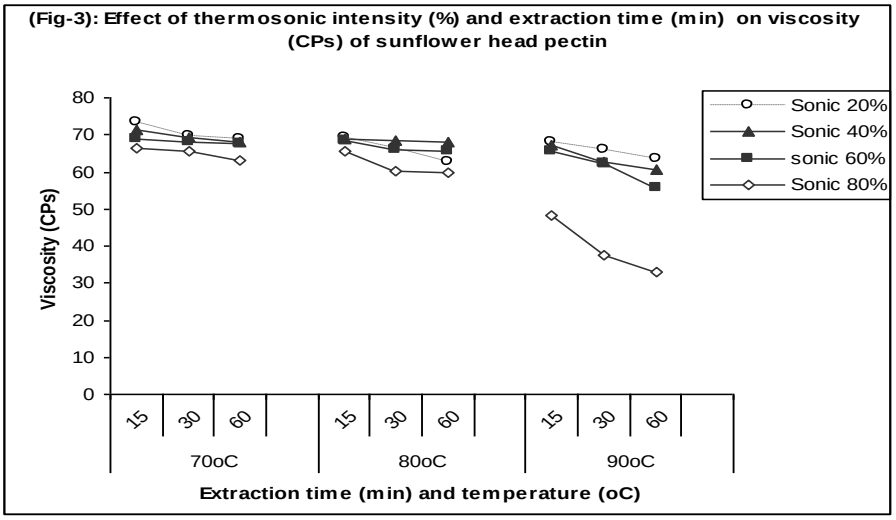


Control: Traditional method= 0.75% SHMP (1:20)/75°C/1 hr
Control: Recovery: 41.56%
Recovery = Wt of final pectin / Wt of original pectin X 100

Effect of thermosonic intensity and extraction time on viscosity of extracted sunflower head pectin solution (1%)

The results shown in Fig. (3) reveal that increase of ultrasonic power from 20% to 80% of its maximal power during the extraction caused remarkable decrease in the viscosity of sunflower head pectin solution (1%). These results are in agreement with those reported by Seshadri *et al*. (2003) who reported that the viscosity of the high methoxyl pectin solution (1.15%) decreased as the sonication intensity increased. They attributed this decrement to degradation of pectin molecules.

From the results in Fig. (3), it could be noticed that the viscosity of sunflower head pectin solution (1%) gradually decreased with the increase of pectin extraction temperature from 70 to 90°C and extending of extraction time from 15 to 60 min in all treatments at the same sonic power used. These results are in agreement with those reported by Zafiris and Oreopoulou (1992).



Control= Traditional method: 0.75% SHMP (1:20) /75°C/1 hr
Control: 66.54 (CPs)

From the results in the Figs. (1, 2 and 3) it could be concluded that treatments that gave the highest yield (18.56 – 23.80%) and recovery (53.12 – 68.12%) of pectin with the highest viscosity (66.15 – 69.04 CPs of 1% solution) were those where extraction of pectin was carried out with ultrasonic (at 60% of the maximal sonic power) at 70 or 80°C for 15 and 30 min. Therefore, these treatments were selected for further physical and chemical studies.

Physical and chemical properties of produced pectin:

The results in Table (3) indicate that the viscosity (solution 1%) of the thermosonic extracted sunflower head pectin and that of pectin resulted from traditional method of extraction was somewhat lower (66.15 – 69.04 CPs) than that of the commercial LMP (70.50 CPs). Tabekha *et al*. (1994) reported

that the viscosity of (solution 2%) pectin extracted from lime peel dehydrated with oven or sun drying process varied from 28.18 to 37.57 CPs.

Results in the Table (3) reveale that the gel strength of extracted sunflower head pectin gel (1% pectin, 30% sucrose, 25mg Ca^{++}/g pectin at pH 5.4) extracted with thermosonic and traditional methods ranged from 20 to 25 g/cm^2 while the gel strength of the commercial LMP gel under same conditions was 30 g/cm^2. These results are in agreement with those reported by Shi *et al*. (1995). They reported that gel strength of 1% sunflower head pectin containing 20% sucrose and 26mg Ca^{++}/g pectin at pH 3 ranged from 21.38 to 27.35 g/cm^2. Also, results in the same table show that the setting time of thermosonic extracted pectin gel was less than that extracted with the traditional method but slightly higher than that of the commercial LMP. On the other hand, the setting temperature of the sunflower head pectin gel extracted with thermosonic and traditional methods ranged from 35 to 45°C. Also, the setting temperature of commercial LMP gel was 47°C. Moreover, the results show that the setting time of all treatments was higher than 10 min and the setting temperature was less than 85°C. So, the produced pectin could be classified as slow set pectin according to Ranganna (1978).

Table (3):Physical and chemical properties of produced sunflower head pectin

Treatments / Properties(%)	Control	T_1	T_2	T_3	T_4	Commercial LMP***
Viscosity (CPs)	66.54	69.04	67.88	68.27	66.15	70.50
Gel strength (g / cm^2)	20	25	25	25	20	30
Setting temperature (C)	36	42	45	45	35	47
Setting time (min)	30	27	26	27	31	25
Ash*	5.50	4.10	4.15	3.80	4.18	3.90
Total neutral sugars*	4.71	4.01	4.09	5.26	5.26	5.61
Methoxyl content (MC)*	5.10	4.70	4.60	4.55	4.50	5.60
Degree of esterification (DE)*	35.02	32.09	31.00	31.97	30.04	38.03
Anhydrogalacturonic acid (AGA)*	86.30	86.73	86.43	88.48	87.10	88.68
Recovery (AGA)**	49.57	63.67	70.57	81.20	82.00	-
Moisture	5.85	6.15	5.70	5.35	6.05	5.84

*** Dry weight basis**
**** Recovery (AGA) = Wt of final AGA / Wt of original AGA X 100**
*****LMP = Low methoxyl pectin**
Control: Traditional method= 0.75% SHMP (1:20)/75°C/1 h
T_1=60% thermosonic intensity + 70°C + 15 min
T_2=60% thermosonic intensity + 70°C + 30 min
T_3=60% thermosonic intensity + 80°C + 15 min
T_4=60% thermosonic intensity + 80°C + 30 min

The results in Table (3) reveal that the ash content of the thermosonic extracted sunflower head pectin ranged from 3.80 to 4.18% on dry weight basis while it was 5.50 for sunflower head pectin extracted with the traditional method. Also, the results show that the ash content of commercial LMP was 3.90%. Shi *et al*. (1995) found that the ash content of sunflower head pectin purified with different ethanol: pectin ratios (1.5:1, 1.8:1 and 2:1) ranged from 4.53% to 5.44% on dry weight basis. According to The Egyptian

Standard (1992) and FCC (1981) the ash content of pectin used in food has to be < 10%. Also, the results in Table (3) reported that the total neutral sugars of extracted sunflower head pectins ranged from 4.01 to 5.26% but it was 5.61% for the commercial LMP. These results are in agreement with those reported by Miyamoto and Chang (1992).

The results presented in Table (3) illustrate that the methoxyl content (MC) of the thermosonic extracted sunflower head pectin ranged from 4.50 to 4.70% on dry weight basis and it was somewhat less than that of the pectin extracted with tradittional method (5.10%). The methoxyl content (MC) of commercial LMP was 5.60% on dry weight basis. Sabir *et al*. (1976) reported that methoxyl content of sunflower head pectin ranged from 6.4 to 7.6% on dry weight basis. Also, the results in the same Table show that degree of esterification (DE) of extracted sunflower head pectin with thermosonic method (30.04 – 32.09%) was slightly lower than that extracted with tradittional method (35.02%) and commercial LMP (38.03%). Panchev *et al*. (1994) reported that maceration of apple pressings with periodical ultrasound treatment led to decrease of DE compared to the untreated samples. Also, they reported that the same effect was obtained with extending time of sonication. These results are in agreement with those mentioned by Sahari *et al*. (2003) who found that DE of sunflower head pectin ranged from 29.63 to 40.91% on dry weight basis. The results of MC and DE show that the type of pectin extracted from sunflower head residues was low methoxyl pectin according to Ranganna (1978).

Results in Table (3) show that the AGA content of extracted sunflower head pectin with traditional or thermosonic ranged from 86.30 to 88.48% on dry weight basis. The AGA content of the commercial LMP was 88.68%. These results are in agreement with those reported by Chang *et al*. (1994) and Miyamoto and Chang (1992). The results in the same Table reveal that the AGA content of all treatments was within limits of The Egyptian Standard (2006) for pectin used in food products. According to this standard AGA content of pectin has to be higher than 65% on free ash and dry weight basis. Results indicate that the highest AGA recovery (~ 82%) was recovered for pectin obtained by T_3 (60% ultrasonic power at 80^{o}C for 15 min) and T_4 (60% ultrasonic power at 80^{o}C for 30 min).

From the results in Table (3) it could be noticed that pectin obtained by T_3 (60% ultrasonic power at 80^{o}C for 15 min), had properties (setting time, setting temperature and AGA content) closely related to that of the commercials LMP. Therefore, pectin resulted from this treatment was selected for further investigations.

Effect of sunflower head pectin concentration on physical properties of its solution and gel compared with those of commercial low methoxyl pectin

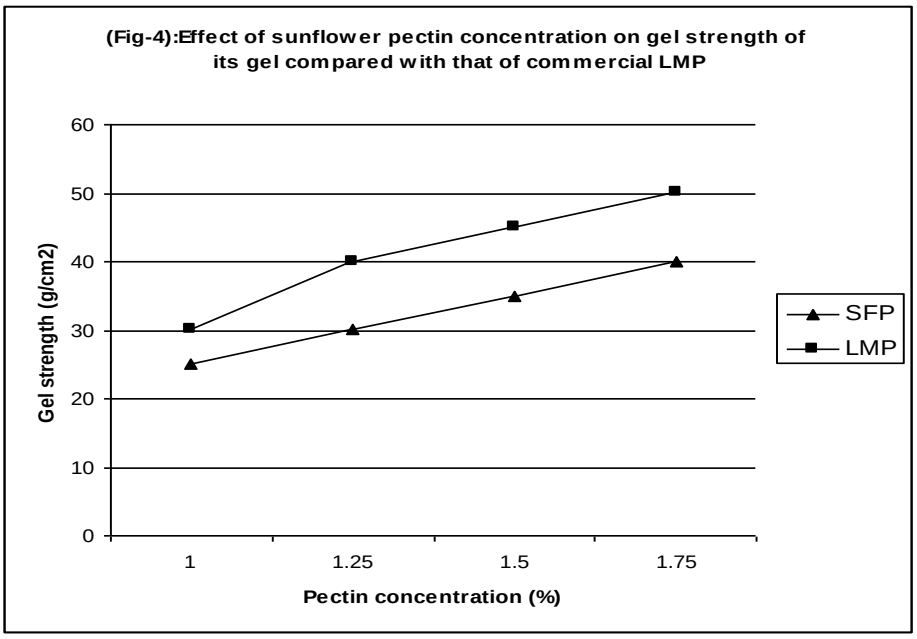
Results illustrated in Table (4) indicate that there was a gradual increase in turbidity of solution with the increase of pectin concentration. Turbidity value of sunflower heads pectin solution was higher than that of the commercial low methoxyl pectin. These results are in agreement with those reported by Lopez and Li (1968). Also, data in the same Table show that

increasing of pectin concentration from 1% to 1.75% led to a gradual increase in viscosity of all samples. These results are in agreement with those reported by Ismail *et al*. (1995) and Arslan and Togrul (1996). The results revealed that the gel strength increased linearly as the pectin concentration increased from 1.25 to 1.75% as shown in Fig. (4). These results are in agreement with those reported by Lopez and Li (1968). Gel strength (g/cm^2) of the sunflower heads pectin gel ranged from 25 to 40 (g/cm^2) compared to 30 to 50 (g/cm^2) for the commercial citrus low-methoxyl pectin gel at pectin concentration ranged from 1 to 1.75%..

Table (4): Effect of sunflower head pectin concentration on physical properties of its solution and gel compared with those of commearcial low methoxyl pectin

Pectin concentrations (%)	Sunflower head pectin (T_3)				Commercial LMP***			
Properties	**1**	**1.25**	**1. 5**	**1.75**	**1**	**1.25**	**1. 5**	**1.75**
Turbidity (NTU)*	1.65	2.08	2.58	2.88	0.78	0.96	1.44	1.92
Viscosity (Cps)*	68.3	80.5	93.8	105	70.5	87.4	94.6	110
Gel strength (g/Cm^2)**	25	30	35	40	30	40	45	50
Setting temp.(C)**	45	50	57	60	47	53	60	65
Setting time (min)**	27	26	23	20	25	23	20	18
Jelly grade**	-	120	-	-	150	-	-	-

*** Determined in pectin solution**
**** Determined in pectin gel**
*****LMP = Low methoxyl pectin**



From the results in Table (4), it could be observed that the increase of setting temperature of gel was accompanied with the increase of pectin concentration in sunflower heads pectin gel as well as in commercial citrus

low- methoxyl pectin gel. On the other hand, increase of pectin concentration led to a remarkable decrease of setting time of all samples. Also, results indicate that the jelly grade of the commercial citrus low-methoxyl pectin gel (1% pectin) was 150 as obtained in data sheet. According to Kertesz (1951) one can calculate the jelly grade of the prepared sunflower heads pectin gel and find that it reached 120.

From the results in Table (4) the gel strength, setting time and setting temperature of 1.25% sunflower head pectin had the same properties of those of 1% commercial citrus low methoxyl pectin, indicating that its efficiency was about 80% of that of the commercial low methoxyl pectin.

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استخلاص وتقييم البكتين من مخلف رؤوس عباد الشمس باستخدام الموجات فوق الصوتية

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١- وحدة التصنيع الزراعى- مركز بحوث الصحراء
٢- قسم الصناعات الغذائية- كلية الزراعة- جامعة القاهرة

تعتبر رؤوس عباد الشمس المتبقية بعد ازالة البذور مصدر جيد للحصول على البكتين منخفض المحتوى الميثوكسيلى. تم استخدام الموجات فوق الصوتية فى استخلاص البكتين من المواد الصلبة غير الذائبة فى الكحول لرؤوس عباد الشمس باستخدام محلول ٠,٧٥ % هكسا ميتافوسفات الصوديوم بنسبة خلط ١:٢٠ وزن:حجم وتم الاستخلاص تحت تاثير قوى مختلفة من الموجات فوق الصوتية (٢٠، ٤٠ ، ٦٠ ، ٨٠% من اقصى قوة) ودرجات حرارة ٧٠ ، ٨٠ ، ٩٠° م لمدة ١٥ ، ٣٠ ، ٦٠ دقيقة. تم تقييم البكتين المتحصل عليه من الناحية الطبيعية والكيميائية مقارنة" بالبكتين التجارى منخفض المحتوى الميثوكسيلى.

اوضحت النتائج ان اعلى عائد من البكتين تم الحصول عليه (٦٦,٤%) تحت ظروف استخلاص ٦٠% من اقصى قوة للموجات فوق الصوتية على درجة حرارة ٨٠ ° م لمدة ١٥ دقيقة حيث ادت تلك الظروف الى الحصول على محتوى ميثوكسيلى ودرجة استرة للبكتين الناتج ٤,٥٥ ، ٣١,٩٧ % على التوالى. ايضا اظهرت النتائج ان اللزوجة وقوة الجل ودرجة حرارة وزمن تكوين الجل للبكتين المستخلص تحت هذه الظروف كان يمثل حوالى ٨٠% من تلك الخصائص للبكتين التجارى منخفض المحتوى الميثوكسيلى. كما اوضحت النتائج حدوث انخفاض تدريجى فى لزوجة البكتين المستخلص بزيادة قوة الموجات فوق الصوتية ودرجة حرارة الاستخلاص وزمن الاستخلاص