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Some Valuable Compounds from Prickly Pear Seeds and Peels

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CHEMICAL properties of crude prickly pear seed oil were: acid value 0.63% (mg KOH/g oil), peroxide values 1.65 (meq O_2/kg oil); saponification value 188.41, unsaponifiable matter 1.36%, and iodine value 108.26 (g I_2/100 g oil). Moreover, palmitic acid was the major saturated fatty acid, while lenoleic acid was the major unsaturated fatty acid in crude oil of prickly pear seed. The biological value (BV) of prickly pear seed was 49.16 and the PER 0.07.

The obtained results showed that pectin extracted from peels of ripe prickly pear had higher ash content (2.61 and 2.12%) than that extracted from peels of mature prickly pear (1.59 and 1.05%). Anhydrogalacturonic acid content was higher in pectin extracted from fresh and dried peels of ripe prickly pear (50.93 and 51.12) than that extracted from fresh and dried peels of mature prickly pear (48.98 and 48.30), respectively. Methoxyl percentage of pectin extracted from peels of ripe prickly pear had higher value than the methoxyl percentage of pectin extracted from peels of mature prickly pear. Acetyl content of pectin extracted from peels of ripe prickly pear was slightly higher than that extracted from peel of mature prickly pear. Degrees of estrification(DE) of pectin extracted from ripe prickly pear peels were 31.49% for fresh peels and 34.93% for dried peels. Meanwhile, it was 25.67% for pectin extracted from fresh peels and 26.84% for dried peels of mature prickly pear.

The results indicated that there were no significant differences in the viscosity values (at 10 rpm) between pectin extracted at different maturity stages (mature and ripe) or pectin obtained by different extraction methods (fresh and dry).

Keywords: Prickly pear seeds, Prickly pear peels, Chemical composition, Pectin.

Nowadays Egypt is considered an important producer for Prickly pear (*cactus* pear) fruits, (EL-Rashidy, 1997). The cultivated area reached more than 3000 Fadden with an annual production of 29442 Ton prickly pear fruits. The production is mainly located in three governorates (Kalubia, Giza and Nobareia, Alexandria) which represents 92.76 % of the total production (MALR, 2003).

Modification of wastes and, or recovery of nutrients or valuable substances from by-products of food industry as well as their use as foods or feed after their treatment, are the main trends of proper utilization.

The chemical composition, including amino acid content, of prickly pear seeds (*Opuntia ficus-indica*) was studied by Radwan (1978); Shaheen *et al.* (1980_a) and Sawaya *et al.* (1983) to determine its suitability for human consumption. Forni *et al.* (1994) studied the preliminary characterization of hot acid extracted pectin from prickly pear peel, whereas EL-Rashidy (1997) studied the chemical composition of raw prickly pear peel before blanching and drying. There are also many studies on some physical and chemical properties of prickly pear seeds oil (Shaheen *et al.* 1980_b; Sawaya & Khan (1982); Sepulveda & Saenz (1988); Krifa *et al.* (1993) and Abd EL-Nabey (2001). The studies included, refractive index, specific gravity, free fatty acids, acid value, peroxide value, iodine value, saponification value and unsaponifiable matter, as well as fatty acid composition of prickly pear seeds oil .

There is a wide scope for obtaining by-products from cactus pear; edible oil can be obtained from its seeds with yields of between 5.8 and 13.6 percent (Sawaya & Khan, 1982 and Sepulveda & Saenz, 1988). The oil shows a high grade of instauration, with high linoleic acid content of 57.7-73.4% . These and other physical and chemical characteristics (such as the refractive index, iodine number, and saponification number) make it similar to some other edible vegetable oils (corn or grape seed oils). In another study, Sawaya *et al.* (1983) found that the protein, fat and fiber contribution of the seeds were 16.6, 17.2 and 49.6%, respectively.

Flores *et al.* (1994) concluded that citric acid production by solid-state fermentation of prickly pear peel by *Asparagillus niger* might be economically feasible.

The obtaining of mucilage's from the fruit peel and the cladodes is another interesting possibility, for alimentary, medical and cosmetic uses, The mucilage's complex polysaccharides, are capable of forming viscous or gelatinous colloids in water. The mucilage is composed of arabinose, galactose, rhamnose and galacturonic acid, (Barbera *et al.*, 2002).

Therefore this work was planned to study the following points: The chemical composition, mineral content, amino acids pattern, protein quality and physico-chemical characteristic and fatty acids profile of crude of prickly pear seeds oils.

Studying the possibility of extracting pectin from prickly pear peels and evaluation of its chemical and rheological properties.

Material and Methods

Prickly pear (*Opuntia ficus-indica*) fruits of two cultivars, red and yellow-orange, from Belbis region (the local commercial names are Frawla and Shamiea) were purchased from the local market. The fruits were stored, washed, drained and hand peeled. They were then passed through a pulper finisher (Langsen kamp, Indianapolis, IN, Model 18) for the separation of seeds from the pulp. The obtained seeds were washed in water several times and air-dried at ambient temperature. The seeds were next ground to fine powder using a Wiley Mill (Model 4, Philadelphia, PA).

Preparation of pectin from the peels

Prickly pear fruits at two maturity stages (mature and ripe) were brought. In each stage of maturity the fruits were hand peeled, then the peels were shredded and blanched in hot water at 80°C for 10 min to inactivate pectin esterase and remove juices, then pressed to release excess water. The peels were divided into two parts. The first part was dried in an oven at 50°C over night, then milled to a fine powder and stored for extraction of pectin. The second part was used directly (fresh) for extraction of pectin. The peels were extracted by 0.5% ammonium oxalate solution (the ratio was 1:25 w/v in case of dry peels and 1:3 in case of fresh peels) and stirred. The mixture was heated at 90 °C for 90 min. The macerate was immediately cooled, filtered and clarified then the pectic substances were precipitated by one volume chilled acidified ethyl alcohol (60 ml. conc. hydrochloric acid in 100 ml. alcohol) with stirring rapidly. The precipitate was washed several times by chilled ethyl alcohol to be free from chloride ions and treated with acetone then with ether and left to be dried at 40 °C overnight or under vacuum and weighted. The dried pectin was ground to pass through a 60-mesh screen.

Preparation of oil from prickly pear fruit wastes

The prepared ground samples (prickly pear seeds and prickly pear peel) were covered with n-hexane for 48 hr at room temperature, and then filtered through filter paper. This process was repeated 3 times using fresh solvent each time to extract most of oil from samples. The miscella was collected, mixed and evaporated at 60 °C under vacuum. The crude oils obtained was separated and immediately analyzed .

Methods of analysis

Ether extract and protein contents were determined according to the standard methods of A.O.A.C. (1990). Pectin content was measured as described by Lees (1975). Anhydrogalacturonic acid was determined according to the method described by Furutani and Osajima (1965). Methoxyl content was performed by saponification of pectin and titration of liberated carboxyl group according to the method described

described by Pippen *et al.* (1950). Esterification percentage was determined according to National Formulatory Committee (1965).

The refractive index, peroxide number, acid value, saponification number, unsaponifiable matter and iodine value were determined according to the methods described in A.O.C.S. (1990).

Lipids were extracted according to Bligh and Dyer (1959). Fatty acids profile was determined quantitatively using a Gas Chromatograph-Mass selective detector instrument "GC-MS" type HP 6890 series. Condition of analysis: Column: capillary HP-Innowax column; 30 m length; 250 µm diameter; 0.25 µm film thickness. Oven: programmable with initial temperature of 150 °C for one minute, then raised in three ramps to reach 255° C after 6 min. and Injector at 260 °C with a split ratio 1:30, Gas: Helium 0.8 ml/min

Amino acid determination was carried out according to the method described by Baxter (1996). The system used for the analysis was high performance Amino Acid Analyzer, Biochrom 20 (Auto sample version) Pharmacia Biotch constructed at NCRRT. Data analysis of chromatogram was done by EZChrom™ chromatography data system Tutorial and user's Guide-Version 6.7.

All results were the mean of three replicates of each analysis.

Computation of protein nutritional values

Chemical Score (CS) was calculated according to FAO (1973). Protein Efficiency Ratio (PER) was calculated using the equation suggested by Alsmeyer *et al.* (1974). Biological Value (B.V) was calculated according to equation of Oser (1959). Pectin yield was calculated according to Abd EL-Fattah and EL-Din (1970). Viscosity measurements for the pectin extracted from prickly pear peels were carried out by using the Brookfield Digital Rheometer model DV-III +. Data were analyzed by using IPC paste math models provide a numerically analyze data sets.

Statistical analyses

The analysis of variance (ANOVA) was carried out to test the possibility of significance treatments effect. LSD as described by Ott (1984) was used to perform all possible pair comparisons (between means of different treatments).

Results and Discussion

Physico-chemical characteristics of prickly pear seeds and peels oil

From the results shown in Table 1 it could be noticed that the Chemical characteristics of oil of prickly pear seeds or peels were in accordance with those results of other authors, *e.g.* Sawaya & Khan (1982), Sepulveda & Saenz (1988) and Abd EL-Nabey (2001) who mentioned that prickly pear seed oil is liquid at ambient temperature and had low acid value and free fatty acids as well as peroxide value which indicates its high stability to deterioration. On the other

hand, refractive index of prickly pear seed oil was higher than that reported by Sawaya & Khan (1982) and Abd EL-Nabey (2001) who found that refractive indeces of prickly pear seeds oil were 1.4596 and 1.4624, respectively.

TABLE 1. Chemical characteristics of prickly pear seed and peel oils.

Characters	Seed of prickly pear fruits		Prickly pear peels
	Variety red	Variety yellow	
Acid value (mg KOH/g oil)	0.55	0.63	1.02
Peroxid value (meq O_2/Kg oil)	1.48	1.65	1.38
Saponification value	186.24	188.41	187.41
Unsapenifiable Matter (%)	1.35	1.36	1.34
Iodine value (g I_2/ 100 g oil)	104.23	108.26	105.05
Refractive index (at 25 °C)	1.4753	1.4724	1.4623

Fatty acids content of prickly pear seed and peel oils

Table 2 illustrates the fatty acids content in crude oil of prickly pear seeds and peel. Palmitic acid was the major saturated fatty acid in the tested crude oil .Saturated fatty acids content of crude prickly pear seeds oil was similar to that reported by Krifa *et al.* (1993) and Abd EL-Nabey (2001). The content of total unsaturated fatty acids in crude prickly pear seeds oil were in agreement with those determined by Sawaya and Khan (1982). They reported that the amount of linoleic acid was higher than the amount of oleic acid.

TABLE 2. Fatty acid content of prickly pear seed and peel oils .

Fatty acid		Prickly pear seeds		Prickly pear peels oil
		Variety red	Variety yellow	
Saturated				
Lauric	12:0	ND*	ND*	ND*
Myristic	14:0	0.19	ND	5.95
Palmitic	16:0	12.91	8.17	27.31
Stearic	18:0	3.49	3.88	24.77
Arachidic	20:0	0.32	ND	0.53
Behenic	22:0	0.28	ND	ND
Total S.F.A.		**17.19**	**12.05**	**58.78**
Unsaturated				
Plamitoleic	16:1	0.76	ND	2.73
Oleic	18:1	16.88	9.11	33.95
Linoleic	18:2	60.19	72.26	1.01
Linolenic	18:3	4.56	6.58	2.79
Eicosenoic	20:1	0.32	ND	0.74
Total U.S.F.A.		**82.84**	**87.95**	**41.22**
TS/ TUS ratio		**0.208**	**0.137**	**2.081**

N.D = Not detected.

Generally, the oil of prickly pear seeds may be used as an edible cooking oil, or as a salad oil and / or for the manufacture of margarines. This is because its Physico-chemical characteristics and its fatty acid composition are quite similar to corn oil (Krifa *et al.*, 1993 and Abd EL-Nabey, 2001).

In this study, fatty acid composition appeared similar to that of other edible vegetable oils, with a large percentage of polyunsaturated fatty acids.

Amino acid profile of protein from prickly pear seed and peel

Regarding the protein content the results of this study were higher than those reported by EL-Rashidy (1997) who found that the protein content of dehydrated prickly pear peels was 2.49 %.

From the results shown in Table 3 it could be noticed that leucine was the predominant essential amino acid of prickly pear seeds and peels. On the other hand, the predominant non-essential amino acids in prickly pear peel was proline . Prickly pear seeds were rich in total non essential amino acids when compared with prickly pear peels. These results agreed with that mentioned by Radwan (1978) who reported that amino acids content of prickly pear seed were deficient in the essential amino acids. Glutamic acid, threonine and alanine seem to contribute the major fractions of amino acids, whereas the amino acids lysine, proline, hydroxproline and tyrosine were deficient.

TABLE 3. Amino acid content of protein from prickly pear seeds and peels (g/ 100 g protein).

Amino acids	Prickly pear seeds protein		Prickly pear peels protein
	Variety red	Variety yellow	
Essential Amino Acid (EAA)			
Threonine	0.8	0 9	0 5
Valine	0.9	1.0	0.6
Methionine	0 2	0.2	0.2
Isoleucine	0.6	0.7	0.48
Leucine	1.4	1.5	0.82
Phenylalanine	0.9	1.0	0.54
Histidine	0.6	0.7	0.28
Lysine	0.6	0.7	0 38
Total EAA	**6.0**	**6.7**	**3.8**
Non-EAA			
Aspartic acid	1.7	1.9	1.22
Serine	0.9	1.0	0.60
Glutamic acid	4 2	4.9	1.48
Proline	0.0	1.4	2.46
Glycine	1.5	1 7	0.66
Alanine	0.9	1.0	0.80
Cystine	0.4	0.4	0.10
Tyrosine	0.4	0.5	0 28
Arginine	2 6	3.0	0.52
Total N.E. A.A.	**12.6**	**15 8**	**8.12**
Ratio EAA/Non EAA	**0.48**	**0.42**	**0.47**

Protein quality

Chemical score (C. S)

Concerning the chemical protein score of the studied prickly pear seeds protein and prickly pear peels protein compared with the FAO (1973) reference patterns the date listed in Table 4 clarified the highest score for threonine, it recorded 20.0, 22.5 and 12.5 for prickly pear seeds variety red, prickly pear, seeds variety yellow and prickly pear peels, respectively.

TABLE 4. Chemical score (C. S) of protein extracted from prickly pear seeds and peels .

Amino acids	FAO (1973) requirement pattern (gm/gm protein)	Prickly pear seed protein (variety red)	Prickly pear seed protein (variety yellow)	Prickly pear peel protein
Iso leucine	4.0	15	17.5	12
Leucine	7.0	20	21.4	11.7
Lysine	5.5	10.9	12.7	6.9
Methionine + cystine	3.5	(5.7)	(8)	(5.7)
Phenylalanine+ tyrosine	6.0	15	16.2	9
Threonine	4.0	20	22.5	12.5
Valine	5.0	18	20	12

Limiting amino acids are shown between brackets.

Furthermore, sulfur-containing amino acids (methionine + cystine) was the first limiting amino acid and lysine was the second limiting amino acid in prickly pear seed protein variety red, prickly pear seed protein variety yellow and prickly pear peel protein.

Protein efficiency ratio (PER) and biological value (B.V.)

From the results recorded in Table 5 it could be noticed that, PER and B.V. of prickly pear seeds protein variety red were higher than prickly pear seed protein variety yellow and prickly pear peels protein. In general PER of either prickly pear seeds or peels protein were lower than that of casein stander (2.5) (Satterlee *et al.*, 1979).

TABLE 5. Protein Efficiency Ratio (PER) and Biological Value (B.V.) of protein from prickly pear seeds and peels.

Parameter	Prickly pear seeds protein		Prickly pear peels protein.
	Variety red	Variety yellow	
PER	0.05	0.07	0.43
B.V.	49.38	49.16	45.37

Some properties of extracted pectin from prickly pear peels

The prickly pear seeds were free from pectin, meanwhile pectin content of prickly pear peels was 15.96 %. This value was higher than that reported by Radwan (1978) and EL-Rashidy (1997).

Table 6 represents some of the parameters generally used for quality evaluation of pectin. It could be noticed that peels of ripe prickly pear gave higher yield of pectin than the mature one. In addition drying the peels before pectin extraction helped to increase the yield of the extracted pectin.

Moisture content of pectin is necessary to be determined for commercial consideration. Data presented in Table 6 showed the moisture content of the pectins .These results were suitable for the recommendations stated by FAO/WHO which mentioned that the moisture content was not more than 12 %.

TABLE 6. Some properties of extracted pectin from prickly pear peels.

Physico- chemical properties (%)	Prickly pear peel			
	At mature stage		At ripening stage	
	Fresh	Dried	Fresh	Dried
Yield	11.04	11.90	14.30	15.50
Moisture	9.91	9.52	10.71	10.42
Ash	1.05	1.59	2.12	2.61
Anhydrogalacturonic acid	48.98	48.30	50.93	51.12
Methoxyl content	4.19	4.338	5.14	5.70
Acetyl value	0.52	0.58	0.63	0.65
Estrification degree	25.67	26.84	31.49	34.93

Data from the same table showed that, pectin extracted from peels of ripe prickly pear had higher ash content, than that extracted from peels of mature one .

Anhydrogalacturonic acid content was higher in pectin extracted from fresh and dried peels of ripe prickly pear, than that extracted from fresh and dried peels of mature stage. According to IFT specification (1959) pectin should contain not less than 65 % galacturonic acid.

The results showed also that the methoxyl content of pectin extracted from peels of ripe prickly pear had higher value than that extracted from peels of mature prickly pear. Methoxyl content of pectin is one of the most important factors affecting pectin quality as it influences gel formation and its properties as well as its sensitivity to changes in hydrogen ion concentration. In this respect, Joseph (1955) reported that low methoxyl pectin (LMP) contains below 7 % methoyxl, while high methoxyl pectin (HMP) contains more than 7 to 12 % methoxyl content. Our pectin samples extracted from prickly pear peel wastes could be grouped under (LMP).

Acetyl content affects the gel formation for pectin solutions because of the presence of these blocking groups that prevents the close approach of molecule necessary for gel network formation. The results from Table 6 showed that the acetyl content of pectin extracted from peels of ripe prickly pear was slightly higher than that extracted from peels of the mature one.

Rheological behavior of the extracted pectin

Varietal characteristics had important influence on the viscosity of pectin. Shear stress, shear rate and viscosity data of pectin at two concentrations (0.25 and 0.5 %), two states of extraction (fresh and dried) and at two maturity stages were obtained using Brookfield viscometer at 20, 30, 40, 50, 60 and 70 °C and demonstrated in Table 7.

TABLE 7. Viscosity (m Pa.s) of the extracted pectin solution.

Temp. °C	Mature				Ripe			
	Fresh		Dry		Fresh		Dry	
	0.25	0.50	0.25	0.50	0.25	0.50	0.25	0.50
20	0.66	2.00	1.62	1.69	0.69	1.97	1.71	3.89
30	1.94	2.01	1.08	1.79	1.71	2.08	0.71	2.98
40	0.54	1.74	0.78	1.28	0.72	1.18	1.40	2.36
50	0.23	0.45	0.24	0.82	0.12	0.73	0.13	0.50
60	0.18	0.51	0.16	0.50	0.04	0.42	0.13	0.77
70	0.02	0.33	0.04	0.25	0.04	0.27	0.03	0.14

Statistical analysis

Statistical analysis was applied for the obtained data and analysis of variance (ANOVA) was carried out. The obtained data were considered as data of factorial experiment 2 x 2 x 6 in complete randomized block design (2 blocks). There were no significant differences ($p > 0.05$) between either concentration, maturity stages (mature or ripe) or state of raw material (fresh and dry). Meanwhile there were significant differences between temperatures of pectin solution. That means increasing temperature from 30 °C to 70 °C was accompanied with decreasing 10 rpm viscosity.

Conclusion

The oil of prickly pear seeds may be used as an edible cooking oil, or as a salad oil and / or for the manufacture of margarines. Pectin samples extracted from prickly pear peel wastes could be grouped under low methoxyl pectin (LMP). The results indicated that there were no significant differences in the viscosity values (at 10 rpm) between pectin extracted at different maturity stages (mature and ripe) or pectin obtained by different extraction methods (fresh and dry).

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بعض المكونات المفيدة من قشور وبذور التين الشوكى

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معهد بحوث تكنولوجيا الأغذية- مركز البحوث الزراعية، *قسم الميكروبيولوجي- المركز القومي لبحوث وتكنولوجيا الاشعاع- هيئة الطاقة الذرية – القاهرة و **قسم تكنولوجيا الأغذية-كلية الزراعة-جامعة قناة السويس- الاسماعيلية .

أجرى هذا البحث بهدف دراسة التركيب الكيماوى التفصيلى لبذور وقشر التين الشوكى واستخلاص بعض المركبات الهامة اقتصاديا منها.

وكانت خصائص زيت بذور التين الشوكي هي: رقم الحامض ٠,٦٣ مجم بو ايدروكسيد بوتاسيوم/جم زيت، رقم البيروكسيد ١,٦٥ ملليمكافئ أكسجين/كجم زيت، رقم التصبن ١٨٨,٤١ مجم/جم المواد غير المتصبنة ١,٣٦% والرقم اليودي ١٠٨,١٦جم يود/١٠٠ جم زيت. وكان حمض البالمتيك هو الحمض الدهني المشبع السائد، بينما اللينوليك هو الحمض الدهني غير المشبع السائد. كما كانت القيمة الحيوية لبروتين ٠,٠٧. وقد تم استخلاص البكتين من القشر وأوضحت النتائج المتحصل عليها أن البكتين المستخلص من قشر التين الشوكي الناضج (الطازج والجاف) أعلى من المحتوى من السماني (٢,١٢، ١,٦١%) عن البكتين المستخلص من قشر التين الشوكي مكتمل النمو (الطازج والجاف) (١,٠٥ ، ١,٥%). محتوى البكتين من حمض انهيدروجلاكتيورونيك Anhydrogalacturonic المستخلص من قشر التين الشوكي المكتمل النمو الطازج والجاف (٥٠,٩٣ و٥١,١٢) كان أعلى من المستخلص من قشر التين الشوكي الناضج والجاف (٤٨,٩٨ و٤٨,٣٠) على التوالي. كما أن نسبة الميثوكسيل Methoxyl percentage للبكتين المستخلص من قشور التين الشوكي مكتمل النمو كانت أعلى من المستخلص من قشور التين الشوكي الناضجة، محتوى الأسيتايل Acetyl content للبكتين المستخلص من قشور التين الشوكي مكتمل النمو أعلى بدرجة طفيفة من البكتين المستخلص من قشور التين الشوكي الناضجة، درجة الاستر للبكتين المستخلص من قشور التين الشوكي مكتمل النمو كانت ٣١,٤٩% للقشور الطازجة و٣٤,٩٣% للقشور الجافة، بينما المستخلصة من التين الشوكي الناضج كانت ٢٥,٦٧% للقشور الطازجة و٢٦,٤٨% للقشور الجافة. لم تظهر النتائج أي فروق معنوية في قيم لزوجة البكتين عند ١٠ الفة/دقيقة بين المستخلص من قشور الثمار مكتملة النمو أو الناضجة، أو لزوجة البكتين المستخلص بالطرق المختلفة.