



Utilization of food processing wastes of eggplant as a high potential pectin source and characterization of extracted pectin

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ABSTRACT

In the present study, the various properties of pectin extracted using microwave-assisted extraction (MAE) from eggplant peel and eggplant calyx (as food processing wastes of eggplant) were compared with each other. The eggplant peel pectin (EPP) exhibited higher extraction yield (29.17%) than eggplant calyx pectin (ECP; 18.36%). Both of EPP and ECP were high in methoxyl and rich in galacturonic acid. HPLC analysis showed that EPP was high in HG (homogalacturonan) (58.6%), while ECP was high in RG-I (rhamnogalacturonan-I) (44.9%). Also, higher phenolic contents were observed for EPP in comparing with ECP. Approximately in all of the functionalities (WHC (water holding capacity) and OHC (oil holding capacity), emulsifying and foaming properties, and antioxidant activity), EPP showed higher value rather than ECP. ^1H NMR (hydrogen-1 nuclear magnetic resonance), FT-IR (Fourier transform-infrared) and XRD (x-ray diffraction) spectra confirmed the presence of high methylated crystalline pectin in both EPP and ECP.

1. Introduction

The food processing sector generates a large amount of waste and by-product mostly composed of the rejected and inedible plant tissues such as peel, seed, husk and etc. This large amount of waste represents a high loss of valuable materials, and also increases environmental and economic issues (Philippi, Tsamandouras, Grigorakis, & Makris, 2016).

Eggplant is one of the common vegetables grown all around the world with a global production quantity of 52.3 million tonnes in 2016 based on FAO reports. The waste of this vegetable mostly consists of peel and green calyx that have no commercial value, while are great sources of phenolic contents and polysaccharides such as pectin (Philippi et al., 2016; Todaro et al., 2009; Dranca & Oroian, 2016; Boulekbache-Makhlouf, Medouni, Medouni-Adrar, Arkoub, & Madani, 2013; Kazemi, Khodaiyan, & Hosseini, 2019).

Pectin is a heteropolysaccharide widely used in food systems as emulsifying, stabilizing and thickening agent. Besides of its technological applications, this polysaccharide has numerous health benefits, which lead to an increase in global demand for it (4–5% growth every

year) (Bayar, Friji, & Kammoun, 2018).

The chemical compositions and other features of pectin are extremely affected by its source and extraction process. Generally, pectin, as a very complex polysaccharide, is composed of two main polymers: HG (homogalacturonan) and RG-I (rhamnogalacturonan-I), which are covalently joined to each other. However, RG-II (rhamnogalacturonan-II) and XG (xylogalacturonan) sections are also observed in lower amounts in its structure (Mohnen, 2008). HG is a linear homopolymer of galacturonic acid units covalently linked to each other at the O-1 and O-4 positions. This homopolymer is partially esterified in the carboxyl groups of galacturonic acid (defined as the degree of esterification or DE) and based on it, pectin is classified in two classes: HM (high methoxyl, DE > 50%) and LM (low methoxyl, DE < 50%). DE is one of the most important features of pectin due to its effect on other properties (Mohnen, 2008). On the other hand, RG-I, as the most abundant side chain, consists of α -(1,4)-D-galacturonic acid- α -(1,2)-L-rhamnose unit repeats. The RG-I polymer is substituted with the neutral sugars side chains, especially arabinan, galactan, and arabinogalactan. It should be stated that the esterification can be also found in

Abbreviations: MAE, microwave-assisted extraction; EPP, eggplant peel pectin; ECP, eggplant calyx pectin; DE, degree of esterification; HG, homogalacturonan; RG-I, rhamnogalacturonan-I; RG-II, rhamnogalacturonan-II; XG, xylogalacturonan; DE, degree of esterification; LM, low methoxyl; HM, high methoxyl; UAE, ultrasound-assisted extraction; SFE, supercritical fluid extraction; GalA, galacturonic acid; Ara, arabinose; Gal, galactose; Rha, rhamnose; Xyl, xylose; Glu, glucose; Fru, fructose; TFA, trifluoroacetic acid; DPPH, 2,2-Diphenyl-1-Picrylhydrazyl; HPLC, high performance liquid chromatography; TPC, total phenolic content; AA, ascorbic acid; GAE, gallic acid equivalent; WHC, water holding capacity; OHC, oil holding capacity; EA, emulsifying activity; ES, emulsion stability; FC, foaming capacity; FS, foam stability; ^1H NMR, hydrogen-1 nuclear magnetic resonance; FT-IR, Fourier transform-infrared; XRD, x-ray diffraction; SEM, scanning electron microscope

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galacturonic acid units of RG-I (Petkowicz, Vriesmann, & Williams, 2017).

Commercially, pectin is produced by the thermal method using high temperature in an acidic medium including nitric acid, hydrochloric acid and etc., from apple pomace and citrus peel. However, the produced wastewater of this process is not eco-friendly and increases environmental issues. Thus, many green chemistry techniques such as microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE) and supercritical fluid extraction (SFE) have been developed to reduce these problems. Among these techniques, MAE is a suitable method for pectin extraction due to its advantages such as shorter time, less solvent, lower consumed power and energy, and higher extraction rate (Maran, Sivakumar, Thirugnanasambandham, & Sridhar, 2013). For this reason, so far, many researchers used from this method for pectin extraction from various raw materials such as apple pomace (Wang et al., 2007), sour orange peel (Hosseini, Khodaiyan, & Yarmand, 2016), orange peel (Maran et al., 2013), dragon fruit peel (Thirugnanasambandham, Sivakumar, & Maran, 2014) and pistachio green hull (Kazemi, Khodaiyan, Labbafi, Hosseini, & Hojjati, 2019) and reported that MAE was a high potential method for pectin production.

To the best of our knowledge, there is no study on the pectin extraction from food processing wastes of eggplant. Therefore, considering the abovementioned notes and also the importance of maximum use of the agricultural and industrial wastes, the present study was focused on the production and characterization of the eggplant peel pectin (EPP) and eggplant calyx pectin (ECP) extracted using MAE method.

2. Materials and methods

2.1. Materials

Eggplant wastes (peels and calyxes) were provided from a restaurant located in Karaj, Alborz, Iran. The peels and calyxes were cut separately into small pieces and placed in an oven to dry (45 °C, 16 h). The dried peels and calyxes were powdered separately using a lab grinder and sieved (40-mesh). The obtained fine powders were packed and stored in a dark dry place for use in the extraction process.

Citric acid (99%), hydrochloric acid (37%), sulfuric acid (95–97%), sodium hydroxide ($\geq 99\%$), TFA (trifluoroacetic acid) ($\geq 99\%$), Folin-Ciocalteu reagent, DPPH (2,2-Diphenyl-1-Picrylhydrazyl) and Standard monosaccharides (galacturonic acid (GalA), arabinose (Ara), galactose (Gal), rhamnose (Rha), xylose (Xyl), glucose (Glu), and fructose (Fru)) were purchased from Merck Chemical Co. (Darmstadt, Germany).

2.2. Pectin recovery process

Pectin was extracted from eggplant wastes (peels and calyxes) using a microwave device (Butane industry Co., Tehran, Iran) in microwave power of 700 W, irradiation time of 2 min, pH of 1.5 and liquid to solid ratio of 20 (v/w). After extraction, the extracts were purified by centrifugation ($10000 \times g$, 20 min), treated with ethanol (96%) in the ratio of 1:1 and placed in 4 °C for 16 h. Then, the precipitated pectin was isolated by centrifugation ($10000 \times g$, 20 min), washed for three times with ethanol and dried in an oven (45 °C, 16 h). The yield of extracted pectin from eggplant peel and eggplant calyx was calculated using the following equation (Eq. (1)):

$$\text{Yield of pectin (\%)} = \frac{\text{weight of dried pectin}}{\text{weight of dried powder}} \times 100 \quad (1)$$

2.3. Chemical and physicochemical properties

In this stage, the chemical composition of the extracted EPP and ECP was evaluated. For this purpose, the moisture and ash content of

pectin was measured based on the described method by Kazemi, Khodaiyan, and Hosseini (2019). The protein content was determined using the Kjeldahl method ($N \times 6.25$).

For evaluation of the monosaccharide composition of EPP and ECP, high performance liquid chromatography (HPLC) method was applied. Briefly, pectin was hydrolyzed by TFA (2 M) at 85 °C for 3 h, then the hydrolysates were purified by centrifugation ($3000 \times g$, 15 min) and freeze-dried to remove TFA. The dried remnants were dissolved in deionized water and purified by centrifugation ($3000 \times g$, 15 min). The obtained solution was injected to an HPLC device using a Eurokat H column (300×8 mm, $10 \mu\text{m}$) and a smartline refractive index detector 2300 (Knauer, Berlin, Germany). The flow rate and temperature of the mobile phase (0.005 M sulfuric acid) were 0.8 ml/min and 70 °C, respectively. The monosaccharide composition was evaluated using the retention time of standard monosaccharides (GalA, Ara, Gal, Rha, Xyl, Glu and Fru).

The DE of GalA units was calculated using the titrimetric method as described by Hosseini, Khodaiyan, Kazemi, and Najari (2019).

The total phenolic content (TPC) of EPP and ECP was evaluated using the Folin-Ciocalteu method (Sharma, Kamboj, Khurana, Singh, & Rana, 2015). Briefly, 0.5 ml of pectin solution (1% w/v), 2.5 ml of Folin-Ciocalteu solution (10%) and 2 ml of sodium carbonate (Na_2CO_3) solution (7.5% w/v) were mixed and incubated for 1 h in room temperature. The absorbance of the obtained mixture was measured by a UV-Vis spectrophotometer (Spectrum SP-UV500DB) at 750 nm. The gallic acid was used as the standard sample (R^2 of the gallic acid curve was 0.9937). The TPC of pectin was defined as mg gallic acid equivalent per 1 g of pectin (mg GAE/g pectin).

The surface tension of EPP and ECP solutions (0.1 and 0.5% w/v) were measured using the method as described by Yapo, Robert, Etienne, Wathelet, and Paquot (2007). For this purpose, a tensiometer device (Nanometric-pazhoh Co. Tehran, Iran) with the Wilhelmy plate method and a mica plate was used.

2.4. Functional features

The functionalities are important features for pectin to be used in food systems. Thus, functional properties of extracted pectin (EPP and ECP) were evaluated using five methods including WHC (water holding capacity), OHC (oil holding capacity), emulsifying properties, foam properties, and DPPH radical scavenging (antioxidant activity). The experiments were repeated three times for each sample.

WHC and OHC were evaluated using the method described by Bayar et al. (2018). The WHC and OHC of each sample were expressed as the amount of water or oil (gram) retained by 1 g of pectin (g/g).

The emulsifying properties were measured using the method described by Yapo et al. (2007). The EA (emulsifying activity) was calculated at room temperature and ES (emulsion stability) was measured after storing the emulsions for 1 and 30 days at 4 °C and 24 °C.

Foaming properties for pectin solutions (2 and 4% w/v) were determined by the method as previously described (Bayar et al., 2017). For this purpose, FC (foaming capacity) and FS (foam stability) were calculated just after vortex and 30 min after vortex, respectively.

Antioxidant activity of pectin solutions was evaluated using DPPH radical scavenging method. Briefly, 1 ml of the pectin solution (1–50 mg/ml) was introduced to the DPPH methanolic solution (0.1 mM) and then the prepared mixture was incubated in a dark place for 30 min. The absorbance of the mixture was recorded using a UV-Vis spectrophotometer (Spectrum SP-UV500DB) at 517 nm. AA solution as a standard sample in the same concentrations (1–50 mg/ml) was used for comparison. The antioxidant activity of pectin solution was calculated by the following formula (Eq. (2)):

$$\text{Antioxidant activity (\%)} = \left[1 - \left(\frac{A_{\text{sample}}}{A_{\text{control}}} \right) \right] \times 100 \quad (2)$$

where A_{sample} and A_{control} were the absorbance of the pectin or AA solutions and control solution (DPPH solution without sample).

2.5. Structural analysis

Structural analysis of EPP and ECP were carried out through ^1H NMR (hydrogen-1 nuclear magnetic resonance), FT-IR (Fourier transform-infrared) and XRD (x-ray diffraction) spectroscopy.

^1H NMR spectra were recorded by a Varian Unity Inova 500 MHz spectrometer (Palo Alto, CA, United States). The internal temperature relaxation delay and acquisition time was 23 °C, 1 s and 4.00 s, respectively.

FT-IR spectra were obtained using a Thermo Avatar 370 spectrometer (Waltham, MA, USA) with KBr method. The range of wave-number was 500–4000 cm^{-1} with a resolution of 4 cm^{-1} .

XRD patterns were recorded using an X-ray diffractometer (PHILIPS, Amsterdam, Netherlands). The range of diffraction angle (2θ), step size and time per point were 10°–80° (2θ), 0.05° (2θ) and 1 s, respectively.

2.6. Morphological analysis

Scanning electron microscope (Vega3, Tescan Co. Ltd., Brno, Czech Republic) was applied for visualizing microwave-assisted extraction process and the morphological properties of extracted pectin. For this purpose, the dried powder of eggplant peel and eggplant calyx (before and after MAE), and the obtained pectin for each raw material (EPP and ECP) were coated with gold by a gold sputter and photographed with the accelerating voltage of 15 KV.

2.7. Statistical analysis

The obtained data for experiments were analyzed using SPSS software v.16 (Chicago, IL, USA) and the data were shown as mean values $\pm$ standard deviation (SD) ($n = 3$). The differences between mean values were evaluated by one-way ANOVA and using Duncan's test at 0.05 significance level ($p < 0.05$).

3. Results and discussion

3.1. Yield and chemical composition

We used a similar method (MAE) for pectin extraction from eggplant peel and eggplant calyx. For this purpose, many pre-tests were carried out with different extraction conditions and among them, the extraction conditions (microwave power of 700 W, irradiation time of 2 min, pH of 1.5 and liquid to solid ratio of 20 w/v) with maximum extraction yield were selected. According to Table 1, under mentioned conditions, the extraction yield of EPP and ECP were 29.17 ± 0.97 and $18.36 \pm 1.34\%$, which showed EPP yield was significantly higher than ECP. Also, the extraction yield of EPP and ECP (especially EPP) was higher than the reported data for banana peel pectin (12.2%; Oliveira et al., 2016), passion fruit peel pectin (18.2%; Seixas et al., 2014), pistachio green hull pectin (18.13%; Kazemi, Khodaiyan, Labbafi et al., 2019), prickly pear peel pectin (9.8%; Lira-Ortiz et al., 2014), apple pomace pectin (15.2%; Ziari, Ashtiani, & Mohtashamy, 2010) and orange peel pectin (19.24%; Maran et al., 2013). However, both of the obtained yields were lower than grape pomace pectin (32.3%; Minjares-Fuentes et al., 2014) and Ubá mango peel pectin (32.1%; do Nascimento Oliveira et al., 2018).

According to Table 1, EPP and ECP had approximately similar value

for moisture content, while ash and protein content of EPP were higher than ECP. Regarding the monosaccharide composition of EPP and ECP, GalA (galacturonic acid) was the most abundant in both samples. GalA content of EPP was 67.4%, which was in agreement with FAO and EU rules (GalA > 65%), while this parameter for ECP was lower (60.2%). In the case of neutral sugars, Gal (galactose), Rha (rhamnose) and Ara (arabinose) were the main neutral monosaccharides for both samples. Same observations were reported for grapefruit peel pectin (Wang et al., 2016). However, Gal and Ara were dominant among neutral sugars of EPP and ECP samples, respectively. Thus, this can be concluded that galactans and arabinans are the main side chains in the structure of EPP and ECP. Besides, Xyl (xylose) and Fru (fructose) were detected in a small percentage in both of pectin, which can be due to the presence of RG-II in the structure of these by-products pectin. The presences of Glu (glucose) in small amounts for both of the extracted pectin (EPP and ECP) may be due to the remnants of non-pectic polysaccharides such as cellulose or hemicellulose (Wang et al., 2016). In addition, the molar ratio of monosaccharides showed that EPP was more linear than ECP, because the HG and RG-I portions in EPP (58.6 and 38.5%) were higher and lower than ECP (52.8 and 44.9%), respectively. Thus, this can be stated that EPP and ECP were rich in the smooth portions (homogalacturonan portions with no side chains) and hairy regions (portions of pectin structure with side chains), respectively. The lower value of (Ara + Gal)/Rha for EPP (2.375) suggested that shorter side chains (mostly galactans) were attached to the RG-I of EPP structure, while the value of this ratio was higher for ECP (4.067) (Petkiewicz et al., 2017).

As can be seen in Table 1, the DE value for the EPP and ECP was approximately 68.18 and 60.74%, respectively. Therefore, these two pectin types should be classified as HM pectin. However, DE of EPP was higher than ECP, which was confirmed by FT-IR and ^1H NMR spectroscopy (see to Section 3.3).

The results of TPC measurement for both EPP and ECP were listed in Table 1. According to the results, EPP was significantly rich in phenolic compounds (161.10 mg GAE/g pectin), while this parameter was low in ECP sample (15.59 GAE/g pectin). This observation was expected because many researchers reported the presence of the high amount of phenolic compounds in eggplant peel (Philippi et al., 2016; Todaro et al., 2009; Dranca & Oroian, 2016; Boulekbache-Makhlouf et al., 2013). The TPC value of EPP was higher than the obtained result from pistachio green hull pectin (18.18 mg GAE/g pectin; Kazemi, Khodaiyan, Labbafi et al., 2019) and tamarind pectin (79.66 mg GAE/g pectin; Sharma et al., 2015). This higher value could be effective on the antioxidant activity of EPP, because the phenolic contents were known as antioxidant agents. Also, higher phenolic contents could have an effect on some other properties such as surface tension and foam properties of pectin solution (Di Mattia, Sacchetti, Mastrocola, Sarker, & Pittia, 2010).

In this stage, surface tension of EPP and ECP solutions (0.1 and 0.5 %w/v) were measured and the obtained results were shown in Table 1. According to the results, the surface tension of EPP was decreased with an increase in the pectin concentration from 0.1 to 0.5% w/v, while in ECP solution, this parameter was not significantly changed with an increase in pectin concentration. This observation maybe due to the TPC, because higher phenolic contents could decrease the surface tension of the solution (Di Mattia et al., 2010). Thus, it seems that higher TPC of EPP solution in 0.5 %w/v is the reason of reducing the surface tension, while in ECP solution, the effect of TPC is not dominant and leads to no changes in surface tension. It should be noted that foam properties have proven the mentioned results.

Table 1

Yield and chemical properties of EPP (eggplant peel pectin) and ECP (eggplant calyx pectin).

	Pectin sample	
	EPP	ECP
Yield (%)	29.17 ± 0.97 ^a	18.36 ± 1.34 ^b
Chemical composition (%)		
Moisture content	5.85 ± 0.27 ^a	5.02 ± 0.12 ^b
Ash content	9.03 ± 1.09 ^a	5.21 ± 0.23 ^b
Protein content	9.13 ± 0.17 ^a	7.68 ± 0.29 ^b
Monosaccharide composition (%) ^A		
GalA	67.4	60.2
Gal	18.1	2.3
Rha	8.8	7.4
Ara	2.8	27.8
Xyl	1.3	0.7
Fru	0.7	0.4
Glu	0.9	1.2
Molar ratio (%) ^B		
HG	58.6	52.8
RG-I	38.5	44.9
(Ara + Gal)/Rha	2.375	4.067
DE (%) ^C	68.18 ± 1.19 ^a	60.74 ± 1.47 ^b
TPC (mg GAE/g pectin) ^D	161.10 ± 2.47 ^a	15.59 ± 3.39 ^b
Surface tension (mN/m)		
0.1% (w/v)	54.36 ± 0.11 ^a	50.07 ± 0.31 ^b
0.5% (w/v)	46.94 ± 0.24 ^b	49.89 ± 0.27 ^a

^A GalA, Gal, Rha, Ara, Xyl, Fru and Glu were galacturonic acid, galactose, rhamnose, arabinose, xylose, fructose and glucose, respectively.

^B HG and RG-I were homogalacturonan and rhamnogalacturonan-I, respectively.

^C DE was degree of esterification.

^D TPC was total phenolic content.

^{a,b} Different letters in the same row indicate significant differences (p < 0.05).

3.2. Functional properties

Water holding capacity (WHC) is one of the important functionalities that refers to the capability of 1 g of moist material to hold water. The retained water is the sum of bound water, hydrodynamic water and physically trapped water (Ghribi et al., 2015). In fact, pectin with higher WHC could be used to retain water and improve sensory properties in the food systems (such as yogurt). The WHC of EPP and ECP were listed in Table 2. The results showed that although both of the pectin types showed good values for WHC, this parameter for EPP (6.02 g/g) was significantly higher than ECP (4.62 g/g). The studies showed that the different factors such as chemical composition, structure, porosity, and ionic strength of extracted pectin could be effective on this parameter (Elleuch et al., 2011).

Oil holding capacity (OHC) is a very important technological property of polysaccharides (Ghribi et al., 2015). Pectin with high OHC value could be known as a good stabilizer and emulsifier in food systems with high fat (Yuan et al., 2018). The obtained OHC values for EPP and ECP were 2.60 and 1.46 g/g, respectively. This result showed that OHC value of EPP is higher than ECP, while both of them were higher than the obtained data for *Opuntia ficus indica* cladodes pectin (1.23 g/g; Bayar et al., 2018). It should also be noted that the effective factors on this parameter are the pectin source, chemical composition, structure and affinity of pectin to oil (Yuan et al., 2018).

Considering their strong hydrophilic nature, most polysaccharides are known as non-surface active materials, while proteins were

Table 2

Functional properties of EPP (eggplant peel pectin) and ECP (eggplant calyx pectin).

Parameter ^A	Pectin sample	
	EPP	ECP
WHC (g water/g pectin)	6.02 ± 0.16 ^a	4.62 ± 0.34 ^b
OHC (g oil/g pectin)	2.60 ± 0.24 ^a	1.46 ± 0.13 ^b
EA (%)	–	24 °C
ES (%)	After 1 day	4 °C
		24 °C
	After 30 days	4 °C
		24 °C
FC (%)	2% (w/v)	17.13 ± 2.14 ^a
	4% (w/v)	30.34 ± 1.49 ^a
FS (%)	2% (w/v)	4.61 ± 2.78 ^a
	4% (w/v)	17.64 ± 2.37 ^a

^A WHC, OHC, EA, ES, FC and FS were water holding capacity, oil holding capacity, emulsifying activity, emulsion stability, foaming capacity and foam stability, respectively.

^{a,b} Different letters in the same row indicate significant differences (p < 0.05).

considered as surface active agents due to the hydrophilic and hydrophobic nature of their side chains. Thus, the presence of small fractions of protein linked with some polysaccharides (which is reported in most of the polysaccharides) could offer observable emulsifying properties. In fact, surface active polysaccharides such as EPP and ECP could be used as both stabilizer and emulsifier (Ghribi et al., 2015). In this study, emulsifying activity (EA) of EPP and ECP was determined in 24 °C. Also, the emulsion stability (ES) of both pectin samples was evaluated in 4 and 24 °C after 1 and 30 days (Table 2). The results showed that the EA of EPP was significantly higher than ECP, which could be due to the higher amounts of protein fractions in EPP (Ghribi et al., 2015). Both of the EA values were higher than the value obtained for citron peel pectin (46.2%; Pasandide, Khodaiyan, Mousavi, & Hosseini, 2018) and sugar beet pulp pectin (43–47%; Yapo et al., 2007). In the case of ES, both of the EPP and ECP showed higher stability in low temperature (4 °C) which was in line with previous reports (Yapo et al., 2007; Ma et al., 2013). Also, in all of the storage conditions, the emulsions obtained from EPP was more stable than ECP.

Foam is a two phase system of air bubbles (dispersed phase) trapped in the liquid (continuous phase). Polysaccharides with high foaming capacity (FC) and foam stability (FS) could improve foaming properties and sensory properties of aerated food systems (Ghribi et al., 2015). FC of EPP and ECP were shown in Table 2. The results showed that the FC in EPP was significantly higher than ECP in the concentration of 2 and 4% w/v. FC in the EPP (30.34%) was approximately similar to *Opuntia ficus indica* cladodes pectin (30%; Bayar et al., 2017). Also in both of the pectin solutions, FC was increased with increase in pectin concentration from 2 to 4%, which was probably due to the higher TPC and protein content in the higher concentration of pectin (4% w/v). In the case of FS, the EPP solution in the concentration of 4% w/v showed the highest stability (17.64%) after 30 min storing in room temperature.

DPPH is a free radical widely used in measuring antioxidant activity of the natural material. Basically, its mechanism is dependent on transferring the hydrogen atoms of the OH groups from the antioxidant to the free radical of DPPH and forming a stable form of the DPPH molecule (DPPH-H), and in consequence, decreasing the free radical concentration and the solution absorbance. The antioxidant activity of

EPP and ECP were shown in Fig. 1. The results showed that the antioxidant activity of EPP solution was much higher than ECP, which could be due to the higher GalA content and TPC of EPP solution (Bayar et al., 2018). Also, the data indicated that although the antioxidant activity of EPP solution was very close to AA (ascorbic acid), none of them has reached to it.

3.3. Structural analysis

According to Fig. 2 (A and B), the ^1H NMR spectra confirmed the dominant presence of GalA units of pectin structure in both of the pectin samples (EPP and ECP). The sharp and strong chemical shift at 3.7 ppm (in both spectrum) was probably derived from the methoxy

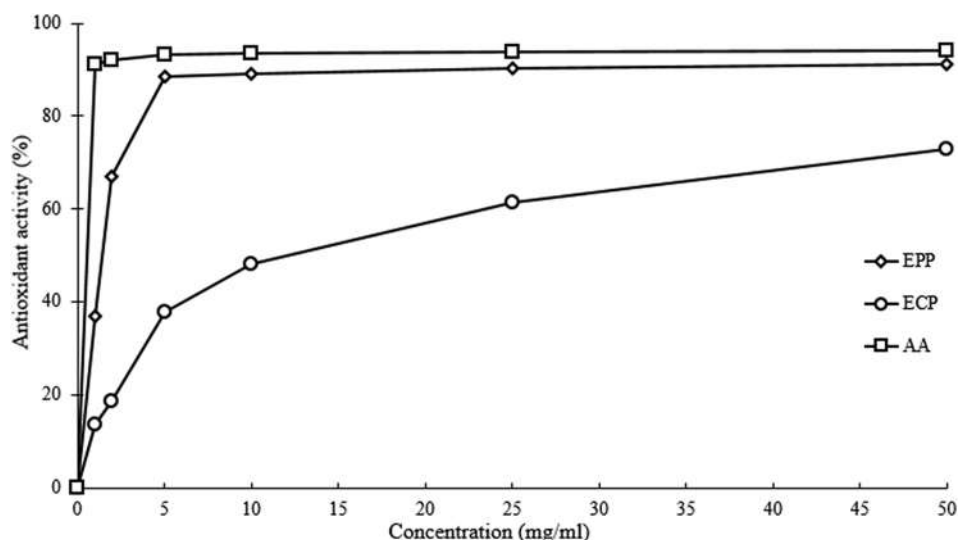


Fig. 1. Comparison of DPPH radical scavenging activity of EPP (eggplant peel pectin), ECP (eggplant calyx pectin) and AA (ascorbic acid).

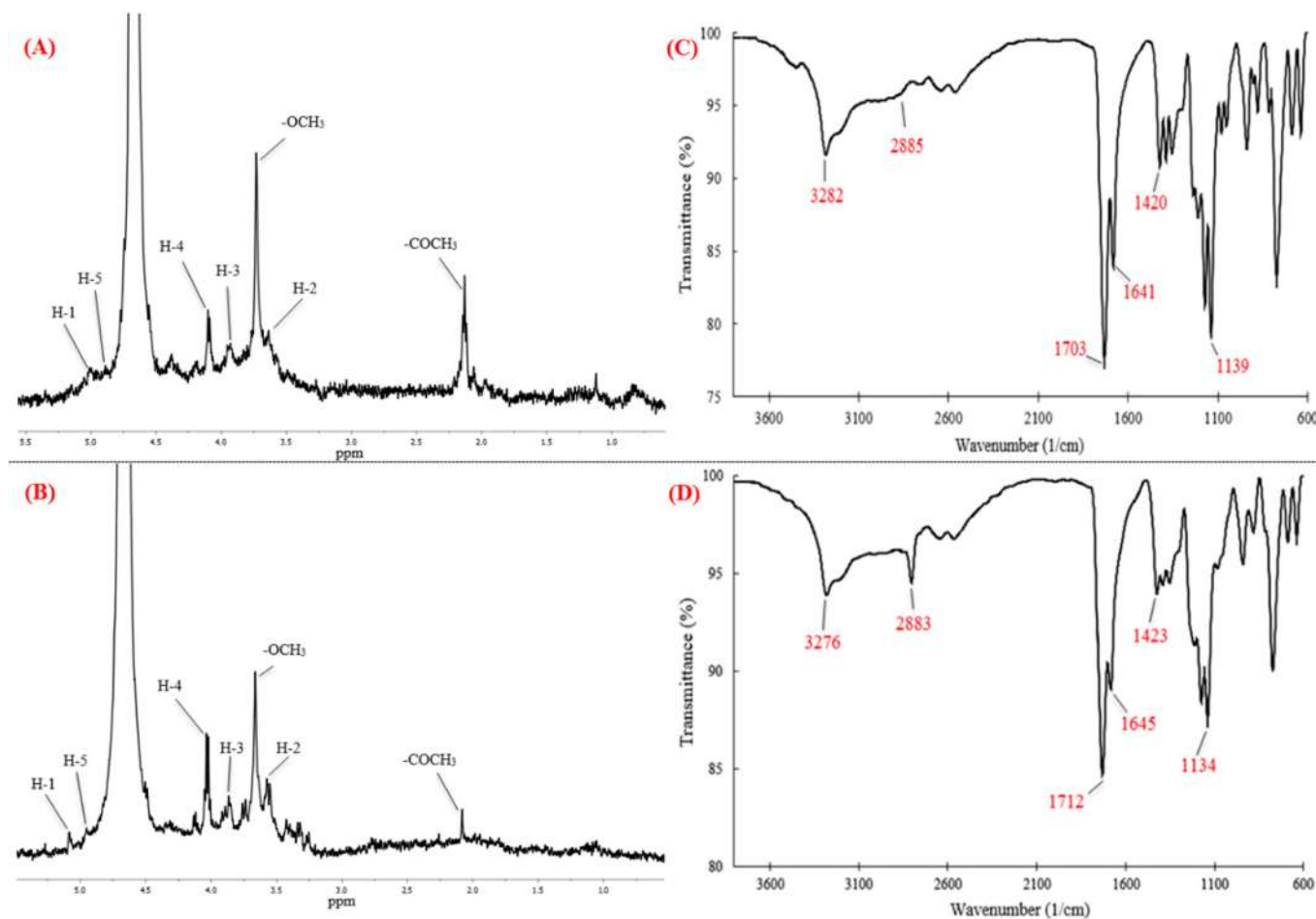


Fig. 2. ^1H NMR and FT-IR spectra of eggplant peel pectin (A and C) and eggplant calyx pectin (B and D).

group ($-\text{OCH}_3$) of esterified GalA units. Also, the signal around 2.1 ppm was due to the acetyl groups ($-\text{COCH}_3$) of esterified GalA units. In the both of obtained spectra for EPP (Fig. 2A) and ECP (Fig. 2B), the protons of C-1, C-2, C-3, C-4 and C-5 of GalA, were observed at 5.1, 3.6, 3.9, 4.1 and 4.9 ppm, respectively. Besides, the high intensity of chemical shifts of methoxy and acetyl groups (3.7 and 2.1, respectively) represents high DE value (Khatib et al., 2017; Talekar, Patti, Vijayraghavan, & Arora, 2018). Thus, it could be concluded that although the both of the extracted pectin samples from eggplant wastes (eggplant peel and eggplant calyx) showed high value of DE ($> 50\%$), the EPP showed higher value, which was in accordance with the results of titrimetric DE measurement. It should

(EPP and ECP) by MAE were shown in Fig. 3. The presence of sharp signals at $13.9, 16.4, 17.7, 19.2, 23.5, 25.7, 28.5, 30.9, 35.9$ and 44.8° (2θ) for EPP and $13.4, 17.1, 25.6, 26.5, 32.9, 40.2, 42.4$ and 44.6° (2θ) for ECP suggested that the crystalline portions in both of pectin samples were dominant (Sharma et al., 2015). Besides, according to the intensity of the mentioned diffraction peaks, the crystallinity of EPP was higher than ECP. Previously, the crystallinity of apple pomace pectin (Kumar & Chauhan, 2010), *Musa sapientum* L. pectin (Suvakanta, Narsimha, Pulak, Joshabir, & Biswajit, 2014), *Tamarindus indica* L. pulp pectin (Sharma et al., 2015), citrus peel pectin (Jiang et al., 2012) and grapefruit peel pectin (Wang et al., 2016) were also reported.

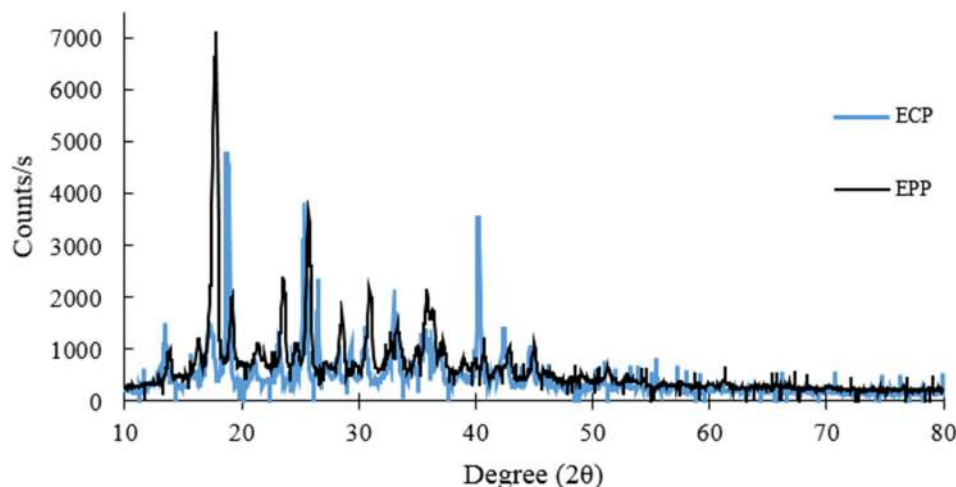


Fig. 3. XRD patterns of EPP (eggplant peel pectin) and ECP (eggplant calyx pectin).

be noted that all of the chemical shifts were in line with previously published data (Khatib et al., 2017; Talekar et al., 2018; Grassino et al., 2016; Wang et al., 2016).

The FT-IR spectra of pectin samples (EPP and ECP) were shown in Fig. 2 (C and D, respectively). The signals in the range of $3000\text{--}3500\text{ cm}^{-1}$ were due to the inter- and intra-molecular hydrogen links of GalA units. The absorption peaks ranged from 2800 to 3000 cm^{-1} were attributed to C–H of CH_2 and CH_3 of carbohydrates and GalA units. The intensive signals in the range of $1700\text{--}1750$ and $1600\text{--}1650\text{ cm}^{-1}$ were related to the esterified and free carboxyl groups of GalA units, respectively. The absorptions ranged from 1000 to 1200 cm^{-1} were attributed to the C–O of glycosides (Pasandide, Khodaiyan, Mousavi, & Hosseini, 2017; Lefsihi et al., 2017). Considering the mentioned absorptions, the presence of pectin structure was confirmed in both of the samples (EPP and ECP). Besides, the high intensity of characteristic peak at 1703 (for EPP) and 1712 cm^{-1} (for ECP) represents high DE value (higher than 50%) for both of the pectin samples (Manrique & Lajolo, 2002). Also, higher intensity of the mentioned peak in comparison with the related peak to free carboxyl groups for EPP showed that the DE in this pectin was higher than ECP, which was in line with the results of DE measurement.

XRD (X-ray diffraction) patterns for the extracted pectin samples

3.4. Morphological analysis

SEM (scanning electron microscopy) images were used to observe the effect of microwave irradiation on the physical structure of eggplant peel (Fig. 4A and B) and eggplant calyx (Fig. 4D and E) before and after microwave treatment. Also, the extracted pectin samples (EPP and ECP) from these materials were scanned and the results were provided in Fig. 4C and F. According to the mentioned figure, the destructive effect of microwave treatment on the eggplant peel (Fig. 4B) and eggplant calyx (Fig. 4E) structure could be observed. Previously, Zhongdong, Guohua, Yunchang, and Kennedy (2006) studied the effect of MAE on the pectin extraction from orange skin and stated that the microwave irradiation splits the orange skin cells and causes a huge disintegration in the physical structure of the raw material, which leads to increase in extraction yield. Besides, the images of EPP (Fig. 4C) and ECP (Fig. 4F) suggested a rough, ruptured and wrinkled surface which could be due to the sudden increase of temperature in MAE process. In another study, Liew, Ngoh, Yusoff, and Teoh (2016) reported similar results and suggested that the coarse surface of the extracted pectin by MAE could be due to the quick raise in temperature.

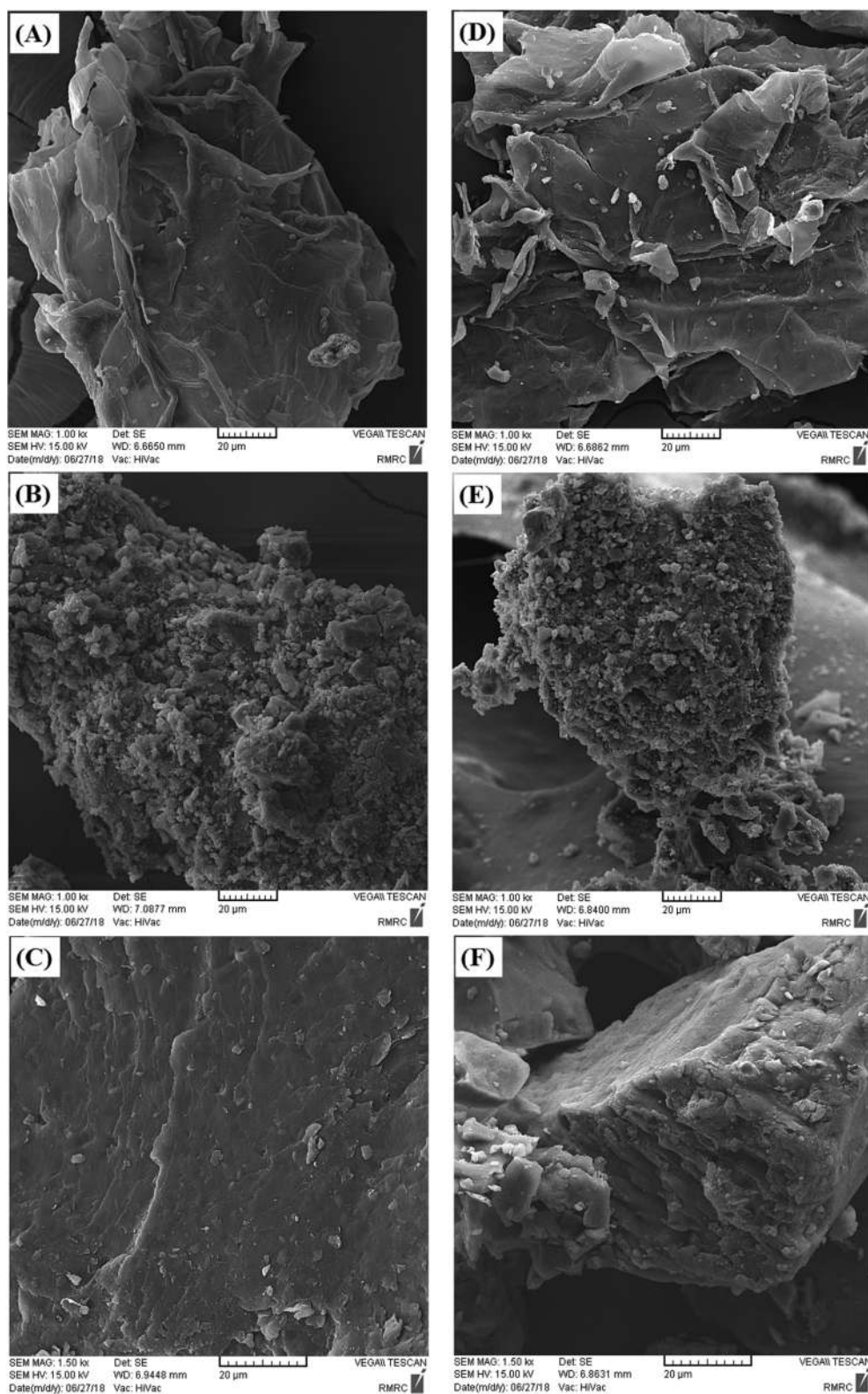


Fig. 4. SEM images of untreated and microwave treated powder of dried eggplant peel (A and B) and eggplant calyx (D and E), eggplant peel pectin (C) and eggplant calyx pectin (F).

4. Conclusion

In the current study, two different pectin samples were extracted from food processing wastes of eggplant (eggplant peel and eggplant calyx) using MAE. The various properties of the extracted pectin

samples (EPP and ECP) were investigated and compared with each other. The results showed that the EPP had a higher extraction yield and GalA content than ECP. Also, ECP was rich in hairy regions, while EPP was rich in smooth portions. Besides, TPC of EPP was much higher than ECP, while in both of them, DE was higher than 50%. In addition,

the functionalities of EPP were better than ECP (WHC, OHC, emulsifying and foaming properties and antioxidant activity). The existence of high methylated pectin in both of the samples was confirmed by ^1H NMR and FT-IR spectra. XRD patterns suggested that the EPP and ECP were crystalline in their nature. With respect to the high extraction yield and great functionalities, pectin recovery from eggplant wastes could be a promising solution for food processing wastes of eggplant and also the resulting pectin could be used as a food ingredient in the formulation of various food systems.

Declaration of Competing Interest

None.

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