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Original Article

Dragon fruit peel pectin: Microwave-assisted extraction and fuzzy assessment

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ABSTRACT

Dragon fruit peels were used as a material for pectin extraction. Microwave-assisted extraction (MAE) using powers of 300, 450 or 600 W and heating times of 5 or 10 min were investigated. Compared to the conventional method, the MAE method produced a higher yield of pectin, with the highest pectin yield (23.11%) being obtained using a microwave power of 600 W and a heating time of 10 min. However, during the MAE extraction, the degradation of pectin may have occurred which resulted in a drop in viscosity. The fuzzy assessment method (FAM) was applied to determine suitable conditions for MAE. The highest overall performance index obtained from FAM indicated that a microwave power of 450 W and an extraction time of 5 min were suitable conditions to produce a high pectin quantity with less degradation. Moreover, the anhydrouronic acid content, degree of esterification and the Fourier-transform infrared spectrum of MAE pectin did not differ from the conventional pectin and therefore, the extract could be categorized as a high methoxyl pectin.

Introduction

Dragon fruit (*Hylocereus* spp.) is one of the famous fruits found in Thailand; having either white flesh (*H. undatus*), red flesh (*H. polyrhizus*) or yellow flesh (*H. megalanthus*), with many black seeds dispersed through it (Ariffin et al., 2008). Dragon fruit is abundant in nutrients, such as beta-carotene, lycopene, and vitamin E, and it contains essential fatty acids (Ariffin et al., 2008; Charoensiri et al., 2009). The dragon fruit peel is usually treated as waste; however, it has been reported that the peel of dragon fruit can be used as a raw material for pectin extraction (Yuagfang et al., 2007; Ismail et al., 2012; Muhammad et al., 2014; Thirugnanasambandham et al., 2014).

Pectin is an important plant cell wall component which is composed of α-(1, 4)-linked D-galacturonic acid residues in which a part of the galacturonic acid is methyl esterified or acetylated or both (Voragen et al., 1995). The degree of methyl esterification (DE) classifies pectin into two main types, which can strongly influence their functionality: high DE pectin (a DE of more than 50%) and low DE pectin (Voragen et al., 1995). Pectin is used in the food industry as a thickener, an emulsifier, a gelling agent, a stabilizer, and also as a fat substitute (Maran et al., 2013).

Pectin extraction is normally performed using solvent extraction from raw materials where the extraction conditions, such as the extraction temperature, extraction time, pH and the type of extraction solvent, affect the yield of pectin and ultimately the quality of the pectin (Yeoh et al., 2008; Ismail et al., 2012; Seixas et al., 2014). On an industrial scale, the extraction of pectin is usually obtained by using hot acidified water under the following conditions: pH of 1.3–3, temperatures of 60–100 °C and a duration of between 20 min and 360 min (Koubala et al., 2008). However, after a long period of heating, thermal degradation of the pectin may occur resulting in changes that affect the quality of the pectin, such as its physicochemical and functional properties (Koubala et al., 2008). Many techniques have been developed to reduce this thermal degradation, for example, microwave heating, ultrasonication and a super high frequency electromagnetic field (Guo et al., 2012).

Microwave-assisted extraction (MAE) has been reported to have great potential and to be a powerful technique (Eskilsson and Björklund, 2000). Compared to the conventional method, MAE can reduce the extraction time and the consumption of the solvent, as well as increasing the extraction rate (Maran et al., 2013). Moreover, it is possible that the process can be scaled up even

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further (Mandal et al., 2007). Maran et al. (2014) used MAE for pectin extraction from the waste of *Citrullus lanatus* fruit rinds and reported that the optimum MAE conditions, which gave the highest pectin yield (25.79%), were a microwave power of 477 W, an extraction time of 128 s, a pH of 1.52, and a solid-to-liquid ratio of 1:20.3. Seixas et al. (2014) showed that the highest pectin yield from passion fruit peel was achieved using a microwave power of 628 W and an extraction time of 9 min. However, it has been reported that longer periods of radiation time can degrade the pectin chains (Maran et al., 2014).

Fuzzy set theory is a mathematical calculation of the criteria function as set by a membership function whose values range from 0 to 1 (Perrot et al., 2006). This theory gives imprecise or vague results in the form of numerical data which can be easily compared. Therefore, it is particularly applied in assessment systems in which there is not a statistical difference or when it is necessary to make a relatively difficult decision in order to choose the suitable condition from the assessment. The application of fuzzy set theory is widely studied, and over the last 10 years, it has been developed as an evaluation tool (Perrot et al., 2006). Its concepts have been applied to food product quality control (Perrot et al., 2006) and to sensory evaluation (Kaushik et al., 2015; Mukhopadhyay et al., 2015). It was used to rank the preference of different samples such as commercially available jam (Shinde and Pardeshi, 2014) and different drinks formulated from dahi (Indian yogurt) powder (Routray and Mishra, 2012). Moreover, it has been applied in the supply chain performance measurement method (Chan et al., 2003; Chan and Qi, 2003; Theeranuphattana and Tang, 2007).

The objective of the current work was to use MAE for pectin extraction from dragon fruit peel (red flesh, *H. polyrhizus* or *H. costaricensis*). In order to gain the suitable conditions in which a higher yield and a less degradation of pectin could be obtained, the MAE conditions were subsequently evaluated using the fuzzy assessment method. Furthermore, the physicochemical properties of the obtained pectin were investigated and were then compared to conventional pectin and to commercial pectin.

Material and methods

Dragon fruit peel powder

A sample of dragon fruit with red-flesh was collected from a local farm in Nong Khai province, Thailand. The peels were removed and were then cleaned with tap water. First, the cleaned peels were cut into pieces (1 cm wide and 5 cm long) and then they were blanched in hot water for 1 min at about 95 °C. Next, they were cooled in cold water (about 2 °C). After that, the blanched peels were dried at 60 °C in a hot air oven, and finally, they were ground. The dragon fruit peel powder was kept in a plastic bag and stored in a desiccator before use.

Microwave-assisted extraction

The pectin extraction using MAE was performed according to Seixas et al. (2014) with some modifications. The dragon fruit peel powder (2.5 g) was added to 100 mL of distilled water and 100 mL of 0.05 M nitric acid. After that, the pH of the solution was once again adjusted to 2.0 using 0.05 M nitric acid. The solution was then heated in a household microwave oven at a working frequency of 2450 MHz. In order to evaluate the effects of the MAE conditions, microwave powers of 300, 450 or 600 W and extraction times of 5 min or 10 min were applied along with a temperature range of 70–100 °C. The conventional (CV) method was performed at 85 °C (about the middle of the temperature range using MAE) for 1 h (Ismail et al., 2012). After heating (for MAE and CV samples), each solution was centrifuged, then the supernatant was precipitated using 95% ethanol. The precipitate was later rinsed three times using 75% ethanol. Subsequently, it was dried at 40 °C using a vacuum drying oven, and afterward, the pectin yield was calculated.

Viscosity of pectin solution

The obtained pectin was dissolved in distilled water at concentration of 1.0% (weight per volume). The viscosity of the solution was determined using a viscometer (Model RVDV-II with spindle ULA00; Brookfield; Middleboro, MA, USA) at 100 rpm and at approximately 22 °C.

Fuzzy assessment method

The fuzzy assessment method (FAM) was applied to evaluate the MAE conditions. The pectin yield and pectin viscosity under each set of conditions were combined using the FAM into an overall performance index in which the major steps were as follows:

1. The pectin yield and pectin viscosity criteria were calculated in the form of a performance score, μ, in the same closed interval from 0 to 10 by using the greatest lower bound, l, and the least upper bound, u, for each of the criteria, as shown in Eq. (1):

$$\mu = P(x) = \frac{x-l}{u-l} \times (10-0) \tag{1}$$

where x is the value of pectin yield or pectin viscosity.

2. The performance score was converted into a fuzzy performance grade set, $F_g(\mu)$, using six grades: *A, B, C, D, E* and *F* (ranging from the perfect to the worst) using the triangular fuzzy number by which the membership function are represented by two end points l and μ and a peak point m and can be defined as shown in Fig. 1.

So that, the fuzzy performance grade set can be represented in Eq. (2).

$$F_g(\mu) = [f_A(\mu) \quad f_B(\mu) \quad f_C(\mu) \quad f_D(\mu) \quad f_E(\mu) \quad f_F(\mu)]^t \tag{2}$$

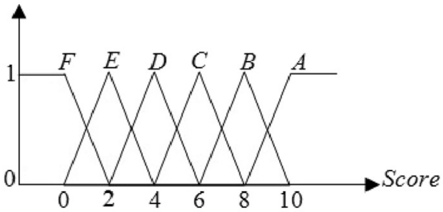
Then, the fuzzy grade sets for the pectin yield and pectin viscosity criteria in Equation 2were composed into the fuzzy performance grade matrix, PG, as $\left[F_g(\mu_{yield}) \quad F_g(\mu_{viscosity})\right]$.

3. The fuzzy performance grade matrix, with the relative weights was integrated as the index score, I. Since the main advantage of MAE is its higher rate of pectin extraction, it is important to ensure that there is the least degree of degradation in the pectin chain. Therefore, with respect to the pectin yield to the pectin viscosity, the relative weights were 55 to 45 for this work. This relative weight clearly indicates the overall performance index which can be used to make precise calculations. The index score for each of the conditions was determined using Eq. (3):

$$I = PG \cdot \begin{bmatrix} 0.55 \\ 0.45 \end{bmatrix} = [I_A \quad I_B \quad I_C \quad I_D \quad I_E \quad I_F]^t \tag{3}$$

4. The overall performance index, PI, was calculated using Eq. (4):

$$PI = \frac{(10 \times I_A) + (8 \times I_B) + (6 \times I_C) + (4 \times I_D) + (2 \times I_E) + (0 \times I_F)}{I_A + I_B + I_C + I_D + I_E + I_F} \tag{4}$$



$$Triangular(x;l,m,u) = \begin{cases} 1 & : \quad x = m \\ \dfrac{x-l}{m-l} & : \quad l \le x < m \\ \dfrac{u-x}{u-m} & : \quad m < x \le u \\ 0 & : \quad x < l, x > u \end{cases}$$

Fig. 1. Triangular fuzzy grade and its membership function and parameters are defined in Eq. (1).

Determination of the anhydrouronic acid contents and the degree of esterification

The percentages of contents for anhydrouronic acid (AUA) and the degree of esterification were determined in accordance with Ismail et al. (2012).

Structural analysis

The structures of the pectin samples were evaluated using Fourier-transform infrared spectroscopy (Spectrum One; Perkin-Elmer; Waltham, MA, USA) with wavelengths in the range 4000–400/cm. The data were then compared to commercial pectin (HIMEDIA, RM396) sourced from HiMedia Laboratories Pvt. Ltd., Mumbai, India.

Statistical analysis

All measurements were analyzed in triplicate using analysis of variance and Duncan's multiple range test ($p < 0.05$) was used to detect differences among the mean values.

Results and discussion

Microwave-assisted extraction

In this study, MAE was used to obtain a higher extraction rate. The dried dragon fruit (red flesh) peel powder was extracted using MAE with heating power of 300 W, 450 W or 600 W for heating times of 5 min or 10 min. The results are shown in Fig. 2A. When compared to the conventional (CV) method (85 °C, 1 h), the results showed that the MAE method mostly produced a higher pectin yield. Moreover, the higher microwave power and the longer extraction produced a greater pectin yield. The highest pectin yield was 23.11% from a microwave power of 600 W and a time of 10 min. This can be explained by the fact that the microwave unit consists of an electric field and a magnetic field which can vibrate the polar molecules and can conduct ionic molecules resulting in a quick generation of heat (Chan et al., 2011) and can allow for a high temperature to be reached within a few minutes. This process of quickly heating the material reinforces the loosening of plant tissue by the vapor inside the capillary porous structure of the plant material (Kratchanova et al., 2004; Maran et al., 2013, 2014; Seixas et al., 2014). When more microwave energy is used in the process, there is enhancement of rupturing of the cell material. This heating phenomenon also enhances the solvent penetration into the plant material (Maran and Prakash, 2015). As a consequence, there is an increase in the release of the pectin from the plant material to the solvent. Conversely, during a longer time of irradiation, there is a greater accumulation of heat within the extraction solution, and this enhances the dissolution of pectin (Maran et al., 2013, 2014). This phenomenon was also found in the pectin extraction of orange peels using MAE (Maran et al., 2013), of waste *Citrullus lanatus* fruit rinds (Maran et al., 2014), and in the pectin extraction of waste *Carcia papaya* L. peels (Maran and Prakash, 2015). This could indicate that the MAE extraction can improve the pectin extraction rate compared to the CV method. The yield, which was found to be higher than that of dragon fruit peel in research conducted by Thirugnanasambandham et al. (2014)

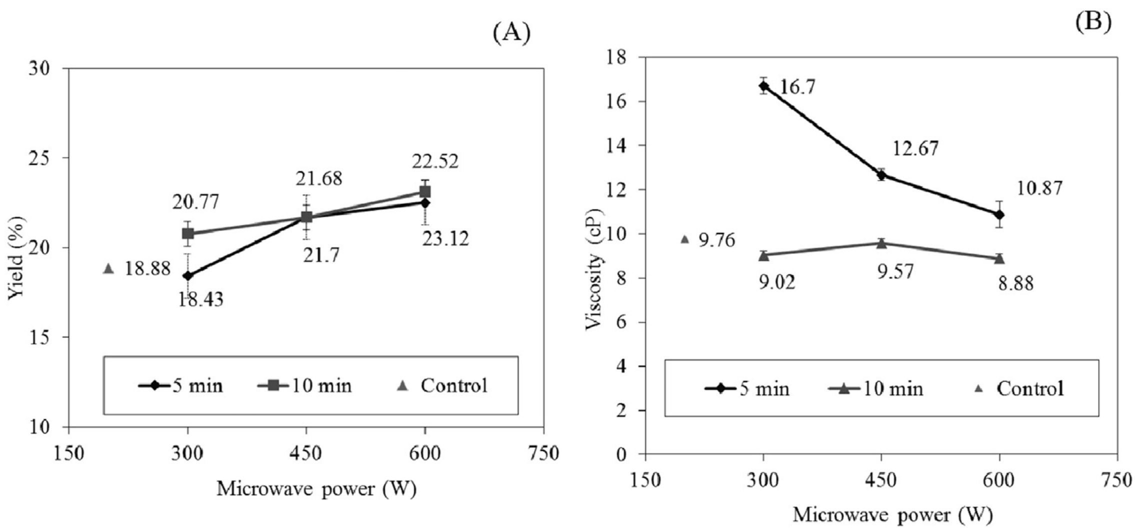


Fig. 2. Effects of microwave power and extraction time on: (A) pectin yield; (B) viscosity of the pectin solution compared to control conditions (85 °C, 1 h) (Error bars are mean ± SD).

which had been extracted using a microwave power of 400 W, at 45 °C, with an extraction time of 20 min and a solid-to-liquid ratio of 24 g/mL. These results most likely depended upon the dragon fruit cultivars, the plant growing environment and the type of acid used for extraction. In the current work, the dragon fruit with red flesh and grown in Nong Khai province was used as the raw material and the nitric acid was used to adjust the pH.

The viscosity of the pectin solution obtained was determined to study its degradation which is shown in Fig. 2B. The results showed that the viscosity of the pectin obtained from 5 min of MAE extraction was higher than the pectin that had been obtained from the CV extraction. This can be explained by the long period of heating using the CV extraction which might affect the degradation of the pectin chain (Koubala et al., 2008). However, pectin degradation was also found following using MAE. By increasing the microwave power, the pectin viscosity decreased. Moreover, at the same microwave power, the pectin viscosity decreased with increased extraction time. An extraction time of 10 min at a microwave power of 600 W produced the lowest viscosity. This result was in agreement with Maran et al. (2014) who reported that a longer microwave radiation time might degrade the pectin chain.

Fuzzy assessment method

Even though the MAE extraction produced the highest pectin yield, the best conditions may have degraded the pectin chain. Therefore, to evaluate the MAE conditions that produced a high yield and resulted in a lesser degree of degradation to the pectin chain, fuzzy set theory was applied. This theory included two criteria results (pectin yield and pectin viscosity) in the form of numerical data (Perrot et al., 2006).

First, in order to obtain the performance score, the lower bound and upper bound for each of the criteria was evaluated. In this work, the greatest integer lower bound and the least integer upper bound for each of the criteria were: a) 18 and 24 for pectin yield and b) 8 and 17 for pectin viscosity. Then the performance scores (with respect to a ten-point scale) for each of the criteria were calculated using Eq. (1). For example, the performance scores in Condition 1 (300 W, 5 min) were calculated in the following manner; by using Eq. (1), the performance score of the pectin yield (18.43) was

$$\mu_{yield} = \frac{x-l}{u-l} \times (10-0) = \frac{18.43-18}{24-18} \times (10-0) = 0.716$$

and for the pectin viscosity (16.70) was

$$\mu_{viscosity} = \frac{x-l}{u-l} \times (10-0) = \frac{16.70-8}{17-8} \times (10-0) = 9.667$$

Second, from the performance scores of pectin yield (0.716) and pectin viscosity (9.667), the fuzzy performance grade set was calculated using Eq. (2):

$$\text{Pectin yield } (0.716)\text{: } f_A(0.716) = \begin{array}{l} f_B(0.716) = f_C(0.716) = f_D(0.716) = 0 \\ f_E(0.716) = \dfrac{0.716-0}{2-0} = 0.36 \\ f_F(0.716) = \dfrac{2-0.716}{4-2} = 0.64 \end{array}$$

$$\text{Pectin viscosity } (9.667)\text{: } \begin{array}{l} f_A(9.667) = \dfrac{9.667-8}{10-8} = 0.83 \\ f_B(9.667) = \dfrac{10-9.667}{10-8} = 0.17 \\ f_C(9.667) = f_D(9.667) = f_E(9.667) = f_F(9.667) = 0 \end{array}$$

The calculations determined that the fuzzy performance grade sets of pectin yield and pectin viscosity were $F_g(\mu_{yield}) = (0,\ 0,\ 0,\ 0,\ 0.36,\ 0.64)^t$ and $F_g(\mu_{viscosity}) = (0.83,\ 0.17,\ 0,\ 0,\ 0,\ 0)^t$, respectively.

Finally, in order to calculate the overall performance index, the index score was calculated by integrating the fuzzy grade matrix with the relative weights. Since, the main advantage of MAE was to obtain a high pectin extraction rate, reducing degradation of the pectin chain should be of primary importance. Therefore, the relative weights in this work for pectin yield to pectin viscosity were 0.55–0.45. This relative weight range gave a clear overall performance index. The overall PI was calculated using Eqs. (3) and (4). For example, in Condition 1, the fuzzy performance grade matrix was integrated by giving weight to the criteria as shown in Eq. (3):

$$I = \begin{bmatrix} 0 & 0.83 \\ 0 & 0.17 \\ 0 & 0 \\ 0 & 0 \\ 0.36 & 0 \\ 0.64 & 0 \end{bmatrix} \cdot \begin{bmatrix} 0.55 \\ 0.45 \end{bmatrix} = \begin{bmatrix} 0.37 \\ 0.08 \\ 0 \\ 0 \\ 0.2 \\ 0.35 \end{bmatrix}$$

As a result, the following was found to be the overall *PI* from Eq. (4) of this condition:

$$PI = \frac{(10 \times 0.37) + (8 \times 0.08) + (6 \times 0) + (4 \times 0) + (2 \times 0.2) + (0 \times 0.35)}{0.37 + 0.08 + 0 + 0 + 0.2 + 0.35} = 4.740$$

Similarly, the overall *PI* values of the other conditions are shown in Table 1.

Therefore, for this work, the best conditions for MAE were a microwave power of 450 W and an extraction time of 5 min which produced the highest overall *PI* (5.703). For the obtained pectin, these conditions gave the best quantity (a yield of 21.68%) and less degradation (12.66 cPs). This microwave power was also close to the optimum MAE conditions for the extraction of pectin from orange peels with a microwave power of 422 W for 169 s (Maran et al., 2013), from the waste of *C. lanatus* fruit rinds with a microwave power of 477 W for 128 s (Maran et al., 2014); and from dragon fruit peels with a microwave power of 400 W for 20 min (Thirugnanasambandham et al., 2014). However, the extraction time for the current study was faster.

Characterization of pectin

Pectin is composed of an α-(1, 4)-linked D-galacturonic acid residue in which some parts can be methyl esterified and expressed as the degree of methyl esterification (DE) which affects its functionality (Voragen et al., 1995). To determine the primary chemical characteristics of the obtained pectin, the anhydrouronic acid (AUA) content and the DE of the obtained pectin were determined using a titration method and compared to commercial pectin. The results are shown in Table 2.

The AUA content in all obtained pectin samples was not significantly different from that of CV pectin (control) which was in the range 60–72%. This result was higher than pectin from dragon fruit (*Hylocereus polyrhizus)* peel of 45.25% reported by Ismail et al. (2012) and of 39.11% reported by Muhammad et al. (2014) most likely due to the growing environments and also to the varieties used. Moreover, the differences in the acids used in the extractions may have affected the AUA content. Seixas et al. (2014) reported that pectin from passion fruit which was extracted using nitric acid had the highest uronic acid content compared to citric acid and acetic acid. Furthermore, the Food and Agriculture Organization (FAO) and the European Union (EU) recommend that the minimum amount of galacturonic acid in pectin should be 65% (Willats et al., 2006).

Moreover, similar to the AUA content, the DE of the MAE pectin showed no significant differences from that of CV pectin (control) which was in range 57–60%. This DE value was higher than the value reported by Ismail et al. (2012) which could be accounted for by the differences in acid extraction and in the plant varieties, as well as their growing environments. In the present study, the obtained pectin can, therefore, be categorized as a high methoxyl pectin, and this finding is in agreement with Muhammad et al. (2014).

Structural analysis

The structure of pectin obtained under suitable MAE conditions (450 W, 5 min) was determined using Fourier-transform infrared spectroscopy (FT-IR) in order to compare the CV pectin and commercial pectin as shown in Fig. 3. The absorption bands, which were in the wavelengths 800–1200/cm, are the finger-print regions of the typical pectin polymers (Kačuráková et al., 2000). It was observed that both of the pectins derived from MAE extraction and from CV extraction, showed the finger-print of a typical pectin which was found to be similar to the high methoxyl commercial pectin. In addition, the absorption bands at 1630–1650/cm and 1740–1760/cm indicated the free and esterified carboxyl groups, respectively (Gnanasambandam and Proctor, 1999). Furthermore, a higher absorption band at 1745/cm and a lower absorption band at 1630/cm indicated the presence of high methoxyl pectins (DE > 50%) as shown in the FT-IR spectra of high methoxyl commercial pectins (Muhammad et al., 2014). However, the absorption bands at 1745 cm of MAE pectin and the control pectin were slightly higher than the absorption band at 1630/cm. This might have been due to the fact that the DE of both pectin samples was not that much higher than 50% compared to the high methoxyl commercial pectin which contained approximately 73% DE. However, the FT-IR spectrum of the MAE pectin was similar to that of the CV control pectin. Thus, the MAE method was a potential method for pectin extraction with the obtained pectin not differing from convectional pectin but requiring a shorter extraction time.

In conclusion, dragon fruit peel, which is usually treated as a waste product, can now be used as a material for pectin extraction. In the present work, MAE was applied for pectin extraction. The results of the study showed that MAE improved the rate of pectin extraction by giving a higher pectin yield in a shorter time compared to the CV method. However, the MAE might degrade the pectin chain, and a resulting drop in the viscosity may occur. In order to obtain the best conditions for this study, the fuzzy assessment method was used and by including the two criteria of pectin yield and pectin viscosity in the fuzzy grade set, as well as the relative weight as the pectin yield to the pectin viscosity (55:45), the overall performance index illustrated that the best MAE conditions in this study were a microwave power of 450 W and an extraction time of 5 min. This MAE condition gave a high pectin quantity (21.68% yield) with less product degradation. Furthermore, compared to the pectin obtained from the CV

Table 2
Anhydrouronic acid (AUA) content and degree of esterification (DE) of pectin obtained in the study compared to commercial pectin.

Condition	AUA (%)^{ns}	DE (%)^{ns}
Control (85 °C, 1 h)	71.63 ± 6.97	59.16 ± 2.91
300 W, 5 min	71.90 ± 0.97	60.14 ± 1.99
300 W, 10 min	60.28 ± 12.29	59.97 ± 1.82
450 W, 5 min	66.16 ± 2.03	57.50 ± 1.33
450 W, 10 min	69.61 ± 4.47	59.56 ± 2.54
600 W, 5 min	66.05 ± 1.02	59.30 ± 0.90
600 W, 10 min	63.67 ± 0.16	58.72 ± 0.00
Commercial pectin	62.00 ± 1.88	73.60 ± 1.04

^{ns} = not significantly different ($p < 0.05$) in the same column. Values are means ± SD.

Table 1
Index score and overall performance index (*PI*) of each condition.

Condition	Performance score		Fuzzy performance grade		PI
	Yield	Viscosity	Yield	Viscosity	
(1) 300 W, 5 min	0.72	9.67	(0, 0, 0, 0, 0.36, 0.64)	(0.83, 0.17, 0, 0, 0, 0)	4.740
(2) 300 W, 10 min	4.62	1.13	(0, 0, 0.31, 0.69, 0, 0)	(0, 0, 0, 0, 0.57, 0.43)	3.049
(3) 450 W, 5 min	6.13	5.18	(0, 0.07, 0.93, 0, 0, 0)	(0, 0, 0.59, 0.41, 0, 0)	5.703*
(4) 450 W, 10 min	6.23	1.74	(0, 0.12, 0.88, 0, 0, 0)	(0, 0, 0, 0, 0.87, 0.13)	4.213
(5) 600 W, 5 min	7.52	3.19	(0, 0.76, 0.24, 0, 0, 0)	(0, 0, 0, 0.60, 0.40, 0)	5.569
(6) 600 W, 10 min	8.52	0.98	(0.26, 0.74, 0, 0, 0, 0)	(0, 0, 0, 0, 0.49, 0.51)	5.124

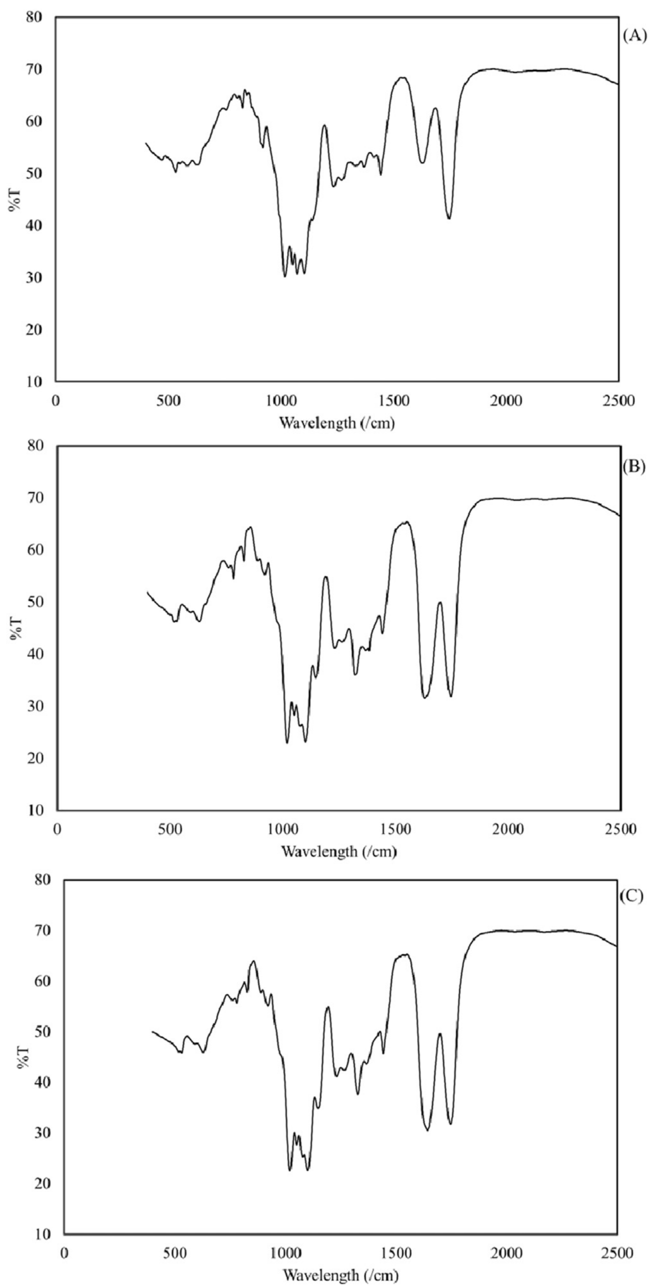


Fig. 3. FT-IR spectra of: (A) high methoxyl commercial pectin; (B) control pectin; (C) MAE pectin (C).

method, the pectin obtained from MAE exhibited similar chemical characteristics. The DE of the obtained pectin was in the range 57–60% which was classified as high methoxyl pectin. Moreover, its anhydrouronic acid content was in the range 60–72% which was within the minimum range recommended by the FAO and EU. In this study, the FT-IR spectrum of the obtained pectin which was extracted under the best MAE conditions was similar to that of the CV pectin. It can be noted that this pectin has potential commercial applications. However, the functionality of the other pectins should be further investigated.

Conflict of interest

The authors declare that there are no conflicts of interest related to the publication of this article.

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