

Microwave-assisted extraction of pectin from jackfruit rinds using different power levels

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Abstract

Pectin is a heterogeneous branched polysaccharide with complex structure. Microwave-assisted extraction (MAE) is more efficient in extracting pectin compared to conventional method. The objective of this study was to compare the efficiency of microwave-assisted pectin extraction against conventional extraction method. This study was also to investigate the effect of power level on yield and quality of extracted pectin from jackfruit rinds. Water-based extraction method was performed with the extraction duration for conventional extraction and MAE were 1 h and 10 min, respectively. The temperature of conventional extraction was set at 90°C and the power levels of MAE were 450 W, 600 W and 800 W. High yield of pectin was obtained from conventional extraction (14.59%) and MAE (16.72-17.63%). All quality characteristics determined were found to be insignificant different for pectin extracted from both conventional extraction and MAE except moisture and ash content. Increase in microwave power did not affect yield and quality characteristics of pectin from jackfruit rinds significantly. In conclusion, MAE requires shorter time than conventional extraction in extracting comparable amount and quality of pectin from jackfruit rinds. Microwave-assisted extraction at 450 W was the most effective and economic extraction condition among the different power levels tested.

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Introduction

Jackfruit rinds are normally disposed as wastes by food industries and vendors. Based on report from the Ministry of Agricultural and Agro-Based Industry Malaysia, production of 56,631 MT of jackfruit in year of 2011 had led to production of 33,979 MT of jackfruit rinds as byproducts (Foo and Hameed, 2012). The disposal of jackfruit rinds may burden environment protection. However, proper utilization of jackfruit rinds can increase economic value of the jackfruit rinds and reduce cost of waste disposal. In order to reduce the wastage and negative effect to the environment, beneficial compound such as pectin in jackfruit rinds can be extracted.

Pectin is a complex heteropolysaccharide that can be found in cell wall and middle lamella of plant tissues (Bagherian *et al.*, 2011). It serves as an important role in the structure of plant cells and junction between the cells (Ridley *et al.*, 2001). The structure of pectin comprises of α -(1,4)-linked D-galacturonic acid units where methyl ester partially substituted to this backbone (Canteri *et al.*, 2012). In the food industry, pectin is used as gelling agent, thickener and stabilizer to produce foods such as jams, jellies, confectionery and fruit juice (Bagherian

et al., 2011).

Pectin is obtained commercially using extraction method in the presence of acid and heat treatment by direct boiling (Li *et al.*, 2012). However, this extraction is time-consuming which commonly takes more than one hour to obtain a certain amount of pectin. In addition, pectin extracted using traditional method is not only poor in term of quantity but also quality in which prolonged exposure of pectin to the heat treatment during extraction leads to pectin degradation (Liu *et al.*, 2006). Thus, undesired alteration in physicochemical and functional properties of pectin can occur. Extraction of pectin from different sources using MAE method had been investigated for the purpose of reducing extraction time and energy (Fishman *et al.*, 2006; Kratchanova *et al.*, 2004). Since conventional extraction of pectin is time-consuming, it is essential to investigate alternate potential extraction method such as MAE method in extracting pectin in order to obtain higher yield of pectin with comparable or better quality compared to pectin extracted using conventional extraction method. The objective of this study was to compare the efficiency of MAE method on pectin extraction against a conventional extraction method. The effect of microwave power on yield and quality

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of extracted pectin from jackfruit rinds was also investigated.

Materials and Methods

Experimental design

Pectin was extracted from jackfruit rinds using a conventional extraction and MAE. Effect of extraction power (450 W, 600 W and 800 W) on MAE of pectin was further studied. Yield and quality characteristics of extracted pectin were investigated.

Jackfruit rind powder preparation

Jackfruit rinds of cultivar JF 6 were collected from a local vendor in Serdang, Selangor. Figure 1 (a) is the image of jackfruit. The jackfruit rinds were then washed and the fibrous strips on the rinds were removed. Only the white and green parts of the rinds were included as shown in Figures 1 (b) and (c). The washed rinds were cut into smaller pieces and treated according to method reported by Mohamed and Hasan (1995) with slight modification in which ethanol was used instead of blanching in boiling water and drying temperature was changed to 55°C. The rinds were treated with equal amount of 95% ethanol at 80°C for 15 min followed by drying of rinds at 55°C until the weight of the rinds was constant. The dried rinds were ground into powder.

Alcohol insoluble solid (AIS) preparation

The alcohol insoluble solids (AIS) were prepared according to methods of Yapo and Koffi (2006) with some modifications where acetone was not used and drying temperature was set at 55°C. The rind powder were treated with 80% ethanol at 80°C for 45 min with the ratio of 1:4 (rind powder:ethanol, w/v). Following that, the sample was washed with 75 ml 60% ethanol for three times and then 75 ml 95% ethanol for one time to remove impurities, pigments, and free sugars. The treated jackfruit rind powder was dried at 55°C until constant weight. This dried sample is known as AIS.

Extraction of pectin

Pectin extraction was done using the methods mentioned by Wai *et al.* (2010) and Kratchanova *et al.* (2004) with some modifications for conventional extraction and MAE, respectively. For both conventional extraction and MAE, the solid to solvent ratio was adjusted to be 1:25 (w/v) and no acid was added. For modification of MAE, microwave treatment was applied during extraction instead of being used for pretreatment. Pectin was extracted by immersing the AIS in distilled water without

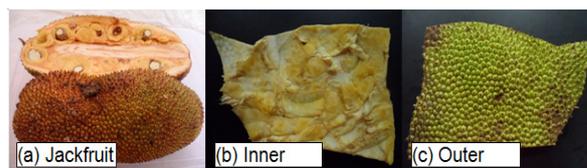


Figure 1. (a) Jackfruit, (b) inner part and (c) outer part of jackfruit rind

pH adjustment. For MAE, the mixture in a closed transparent glass vessel was then placed at the center of microwave oven (Samsung, Model ME86V-BBH) with different powers of 450 W, 600 W and 800 W for exposure duration of 10 min. For conventional extraction, the mixture was placed in water bath shaker (Protech 903) with temperature set at 90°C for 1 h. The extracted solid was filtered from solvent. The filtered solid material was used for a second extraction with the same procedure as mentioned above while the filtrate was concentrated 2/5 of initial volume using rotary evaporator at temperature of 55°C.

Purification of pectin

Purification of pectin was conducted using methods of Kratchanova *et al.* (2004) with slight alterations. Precipitation was carried out by adding 95% ethanol to the filtrate in ratio of 1:2 (filtrate:ethanol, v/v). The mixture was then stirred for 30 min at room temperature followed by keeping it under refrigeration at 5°C for 1.5 h. Then, coagulated pectin was separated by filtration and washed with 75 ml 70% ethanol for two times and 75 ml 95% ethanol until the filtrate becomes colorless. The washed pectin was then dried at 30°C overnight then followed by grinding into powder.

Methods of analysis

Determination of yield of pectin

$$\text{Yield of pectin (\%, w/w)} = \frac{\text{pectin weight}}{\text{AIS weight}} \times 100 \quad (\text{Li et al., 2012})$$

Proximate analysis

Proximate analysis of moisture content, ash content, crude lipid content, crude protein content and crude fiber content for jackfruit rinds and moisture content, ash content and crude protein content of pectin were conducted according to AOAC method (2000).

Determination of galacturonic acid content

Acid hydrolysis of pectin was carried out based on method of Ahmed and Labavitch (1977). Uronic acid content of pectin was estimated using a colorimetric assay according to Filisetti-Cozzi and Carpita (1991) with modification according to Ibarz

et al. (2006). Glass tubes for sample and sample control were prepared. Aliquot of 100 μL from hydrolysate supernatant was mixed with 300 μL of distilled water in all glass tubes. A 40 μL of a 4 M sulfamic acid-potassium sulfamate solution with pH 1.6 was added to all glass tubes and mixed thoroughly with the mixture by using Vortex. Following that, 2.4 mL of 75 mM sodium tetraborate in the presence of analytical grade (96.4%) H_2SO_4 was added and the solution was stirred vigorously using Vortex. The solution was placed in 100°C water bath (boiling) for 20 min of incubation then cooled by plunging tubes in ice bath for 10 min. After cooling, 80 μL of 0.15% (w/v) m-hydroxydiphenyl in NaOH 0.5% (w/v) was added to glass tubes of sample while 40 μL 0.5% (w/v) NaOH was added to glass tubes of sample control. The mixture was stirred vigorously with Vortex. Absorbance of sample was read at 525 nm against sample control and galacturonic acid content were determined using a standard curve of galacturonic acid.

Determination of gel strength

Pectin gel strength was measured according to method of Jiang et al. (2012) with slight modification in term of probe diameter. In the preparation of pectin gel, pectin solution at 1% (w/v) was made with the pH adjusted to 2.5 using citrate buffer solution. Sucrose was added at temperature of 90°C under magnetic stirring until total solid content becomes 63°Brix. The temperature of the mixture was then increased until boiling. The gel in flowing condition before solidification was immediately transferred from beaker to testing container and allowed to stand overnight at 4°C for 12 h before textural analysis. Pectin gel strength was analyzed using TX-XT2i texture analyzer. The gel was deformed by compression at a constant speed of 1.0 mm/s to a distance of 4 mm from the gel surface using a cylindrical probe with diameter of 10 mm.

Colour analysis

Colour of pectin gel was analyzed using Hunter Lab colorimeter. The results were reported as values of L (lightness), a (redness and greenness), and b (yellowness and blueness).

Statistical analysis

All experiments were performed in triplicates. The data was analyzed using MINITAB v.14 Statistical Package (Minitab Inc., State College, Pennsylvania) based on analysis of variance and expressed as mean value \pm standard deviation. The confidence level for statistical significance was set at a probability value of 0.05. Tukey's test was used to determine significant

Table 1. Proximate composition of jackfruit rinds

Analysis	Jackfruit rinds
Moisture (% w/w) ^a	76.33 \pm 0.29
Ash (% w/w) ^b	0.99 \pm 0.10
Crude lipid (% w/w) ^b	1.71 \pm 0.24
Crude protein (% w/w) ^b	1.54 \pm 0.16
Crude fiber (% w/w) ^b	13.51 \pm 0.25
Carbohydrate (% w/w)	5.92

^a All values are expressed as mean \pm standard deviation of triplicate.

^b Proximate composition is given in percentage of wet basis.

^c Proximate composition is given in percentage of dry basis.

Table 2. Chemical composition and physical properties of pectin extracted from jackfruit rinds

Analysis	MAE				
	Conventional	450 W	600 W	800 W	
Chemical composition					
Yield (% w/w)	14.59 ^a \pm 0.44	16.72 ^a \pm 1.40	17.63 ^a \pm 1.79	17.21 ^a \pm 1.68	
Moisture (% w/w)	9.98 ^a \pm 0.16	5.18 ^b \pm 0.33	5.26 ^b \pm 0.60	5.19 ^b \pm 0.39	
Ash (% w/w)	8.18 ^a \pm 0.10	7.23 ^b \pm 0.02	7.48 ^b \pm 0.20	7.30 ^b \pm 0.13	
Crude protein (% w/w)	1.53 ^a \pm 0.10	1.67 ^a \pm 0.10	1.67 ^a \pm 0.10	1.74 ^a \pm 0.00	
Galacturonic acid (% w/w)	72.62 ^a \pm 0.85	75.34 ^a \pm 4.56	70.29 ^a \pm 5.32	69.47 ^a \pm 2.08	
Physical properties					
Gel strength (g)	5.43 ^a \pm 0.08	5.53 ^a \pm 0.05	5.47 ^a \pm 0.08	5.49 ^a \pm 0.07	
Gel colour	L	19.37 ^a \pm 1.02	18.22 ^a \pm 1.04	19.11 ^a \pm 0.30	18.26 ^a \pm 1.08
	a	0.76 ^a \pm 0.80	1.27 ^a \pm 0.78	0.93 ^a \pm 0.54	0.99 ^a \pm 0.54
	b	2.05 ^a \pm 0.29	2.19 ^a \pm 0.09	1.89 ^a \pm 0.47	2.13 ^a \pm 0.09

^aMean and standard deviation are shown.

^bValues superscripted with different letters are statistically different ($p \leq 0.05$).

difference of the data.

Results and Discussion

Proximate composition of jackfruit rinds

The proximate composition of raw jackfruit rinds is presented in Table 1. Raw jackfruit rinds contained low ash (0.99%), crude lipid (1.71%) and crude protein (1.54%). The crude lipid content of jackfruit rinds was lower than 3% and similar to that of cacao pod husks (1.5%). Ash and crude protein content of jackfruit rinds were lower than *Akebia trifoliata* var. *australis* peels, cacao pod husks and peach pomace as reported by Jiang et al. (2012), Vriesmann et al. (2011) and Pagán and Ibarz (1999) respectively. According to Yapó (2009), low ash and protein content leads to better gelling properties of pectin. Therefore, jackfruit rinds are potential source of pectin with good gelling properties.

Chemical composition and physical properties of pectin

The chemical composition and physical properties of pectin is presented in Table 2. Without pH adjustment using acid, the extraction condition was weakly acidic (pH 5.96). Based on Table 2, among the four treatment conditions tested using MAE, there was no significant difference ($p > 0.05$) in term of pectin yield. However, the yield of pectin for MAE was higher than conventional extraction method (14.59%). Higher efficiency of MAE against conventional extraction was confirmed by Bagherian et al. (2011) and Guo et al. (2012) as well with pectin

yield of 27.81% and 18.13% from grapefruit and navel orange peel, respectively. Theoretically, application of microwave reacts on cell wall matrix and causes severing of parenchymal cells which results in opening of skin tissues by microwave (Bagherian *et al.*, 2011; Kratchanova *et al.*, 2004). As a consequence, the distilled water was able to penetrate into the skin tissues leading to increased interaction between solvent and tissues (Bagherian *et al.*, 2011). It results in increase of pectin yield for MAE compared to conventional extraction. In addition, Guolin *et al.* (2012) claimed that direct interaction of microwave with solvent allows release of intracellular products into the solvent. Hence, extraction using less solvent at a shorter extraction time can be carried out for MAE to achieve high extraction efficiency (Guolin *et al.*, 2012). Moreover, since solubility of pectin will be reduced due to interaction of pectinesterase with pectic substances, increase in microwave power leads to complete inactivation of pectinesterase activity in peels and thus increase extractability of pectin (Kratchanova *et al.*, 2004). The yields of pectin extracted from jackfruit rinds using MAE at 450 W and 600 W were 16.72% and 17.63%, respectively. Conversely, the pectin yield was decreased when microwave power increased to 800 W (17.21%). A similar trend of pectin yield due to application of different power levels was reported by Bagherian *et al.* (2011) on MAE of pectin from grapefruit. The decrease in yield due to further increase of microwave power could be caused by pectin degradation where pectin disaggregates into its smaller component parts (Bagherian *et al.*, 2011; Fishmann *et al.*, 2006). Zhongdong *et al.* (2006) studied the structure of pectin extracted from microwave heating using scanning electron microscopy. According to Zhongdong *et al.* (2006), microwave has swelling effect that results in cell splitting. The swelling effect breaks down the cluster form of pectin into smaller particle similar to crystal. In other words, higher microwave power results in more breakage of pectin and thus lower pectin yield.

The moisture content of pectin extracted using conventional extraction was significantly higher ($p \leq 0.05$) than pectin extracted from MAE. This result showed that pectin from MAE had lower water holding capacity relative to that extracted by conventional extraction. In term of safety, pectin powder is favourable to have lower moisture content for safe storage and prevent deterioration of pectin quality due to production of pectinase as a result of microorganism growth (Ismail *et al.*, 2012). In short, pectin powder extracted by conventional extraction is more susceptible to quality deterioration by action

of pectinase due to higher moisture content. On the other hand, the moisture contents among pectin extracted using MAE with different power levels were not significant different ($p > 0.05$).

Based on Table 2, ash content of pectin extracted using conventional extraction was significantly higher ($p \leq 0.05$) than that by MAE. All ash contents of pectin extracted using MAE with three power levels were not significant different ($p > 0.05$). Sönmez and Giray (2011) suggested that short extraction time of MAE for pectin extraction is a factor contributing to the low ash content. Ash contents of pectin from jackfruit rinds found in this research were lower than that reported by Mohamed and Hasan (1995) which was 9.05%. According to Ismail *et al.* (2012), low ash content is desired for good gel formation of pectin. A gel is considered as good quality when ash content is lower than 10% (Ismail *et al.*, 2012). As shown in Table 2, all pectin extracted from jackfruit rinds with various extraction treatment conditions contained ash content lower than 10%. This recommends that jackfruit rinds are a good source for producing good quality pectin.

The results in Table 2 showed that crude protein contents were not significantly different ($p > 0.05$) for pectin extracted from various treatments. A similar protein content of commercial citrus pectin (1.84%) was observed (Jiang *et al.*, 2012). The crude protein content of pectin extracted by MAE (1.67-1.74%) was similar to that extracted by conventional extraction (1.53%). The crude protein content measured was due to dissolution of nitrogen from jackfruit rind in the water as a result of high temperature generated by both conventional extraction and MAE (Liu *et al.*, 2006).

Galacturonic acid is one of the criteria for characterising pectin other than neutral sugar content, degree of esterification and molecular weight distribution (Canteri *et al.*, 2012). Higher galacturonic acid and lower ash content define the purity of the pectin (Liang *et al.*, 2012). According to Food and Agricultural Organisation (n.d.), pectin is known to be pure only if the galacturonic acid content is not less than 65%. Pectin from both conventional extraction and MAE did not have significant difference ($p > 0.05$) in term of galacturonic acid content. As shown in Table 2, all pectin extracted from jackfruit rinds contained more than 65% of galacturonic acid content regardless of treatment conditions. This shows that pectin extracted from jackfruit rinds is considered to be pure. In addition, the galacturonic acid content of pectin from jackfruit rinds (69.47-75.34%) was comparable to the galacturonic acid content of pectin from apple (73.5%) and citrus (72.1%) which are

sources of commercial pectin (Savary and Nuñez, 2003). Similar galacturonic acid contents of pectin extracted from grapefruit (74.86%), navel orange peel (~70%) and fresh orange peel (66.4-70.1%) relative to the galacturonic acid contents of pectin from jackfruit rinds (69.47-75.34%) were reported by Bagherian *et al.* (2011), Guo *et al.* (2012) and Kratchanova *et al.* (2004), respectively. This indicates that jackfruit rinds are source that potentially producing pure pectin. Although MAE involved shorter extraction time than conventional extraction, galacturonic acid contents of jackfruit pectin for MAE was similar to that of conventional extraction because penetration of solvent into tissues structure was facilitated due to improvement of capillary-porous characteristics by microwave heating (Kratchanova *et al.*, 2004). In other words, microwave heating is a desired alternative extraction method in maintaining quality of pectin but further increase in microwave power could decrease the pectin quality.

There was no specific trend seen in gel strength of pectin from jackfruit rinds by applying conventional extraction and MAE with different power levels. Refer to Table 2, changing extraction method and microwave power did not affect gel strength of pectin from jackfruit rinds significantly ($p > 0.05$). Similar results were reported by Kratchanova *et al.* (2004) where the gel strength of pectin extracted from fresh orange peels that had been pretreated with microwave heating at power of 450 W, 630 W and 900W was in the range of 150-182°TB (5.29-6.42 g). Kratchanova *et al.* (1996) found that the gel strength of pectin extracted from microwave-pretreated fresh Navel orange peels was 165-190°TB (5.82-6.70 g). This indicates that the gel strength of pectin from jackfruit rinds is comparable with that of orange peels which are commercial source of pectin. Observation of the insignificant gel strength variation was because of the combination of effects from ash content, crude protein content and galacturonic acid content of pectin. Ash content is the indication of inorganic impurities in pectin (Mohamed and Hasan, 1995). According to Masmoudi *et al.* (2012), lower protein content indicates higher purity level of pectin. Yap (2009) claimed that low ash and protein content coupled with high galacturonic acid content are vital for good gelling properties of pectin. This indicates that high purity of pectin is essential in contributing to the good gel strength of pectin.

Colour of pectin is an important factor affecting appearance of gel formed. Lighter colour of pectin is preferred in order to have minimal effect on the final appearance of end product. In general, colour of pectin is varied according to source (Mohamed

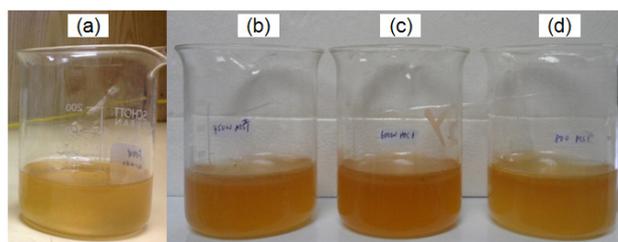


Figure 2. Gel of pectin extracted from jackfruit rinds: (a) Conventional, microwave-assisted extraction (b) 450 W, (c) 600 W and (d) 800 W

and Hasan, 1995). Table 2 shows that gel colour of pectin from jackfruit rinds was not significantly ($p > 0.05$) affected by extraction condition. The lightness of gel from pectin extracted using MAE did not vary in specific trend with increase in microwave power level. In visual observation, the gel colour of pectin from jackfruit rinds was brownish orange. Figure 2 shows the colour of pectin gel. Colour appeared on pectin is possibly due to entrapment of polyphenols or water soluble pigments in pectin during precipitation. Improvement of pectin colour could be achieved by performing filtration using filter aid, activated carbon, diatomaceous earth and others (Mohamed and Hasan, 1995).

Conclusions

Microwave-assisted extraction was found to be more efficient than conventional extraction method in extracting pectin from jackfruit rinds. Shorter time was needed for MAE than conventional extraction in extracting comparable amount and quality of pectin from jackfruit rinds. Moreover, significant lower moisture and ash content of pectin extracted using MAE gave additional advantages to the application of MAE by providing better quality of pectin. Increase in microwave power level for MAE did not significantly affect yield and quality of pectin extracted from jackfruit rinds. Economically, MAE at 450 W was the most effective extraction condition among the different power levels for extracting pectin from jackfruit rinds due to its efficiency to extract pectin with similar yield and quality relative to a conventional extraction.

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