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Pectin content of selected local fruit by-products

(Kandungan pektin dalam hasilan sampingan buah-buahan tempatan terpilih)

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Key words: pectin, fruit by-products

Abstrak

Kandungan pektin dalam hasilan sampingan buah-buahan tempatan seperti koko *(Theobroma cacao)*, limau kasturi *(Citrus microcarpa)*, jambu batu *(Psidium guajava)*, pisang Mas (*Musa* sp. cv. Mas), belimbing *(Averrhoa carambola)*, nanas *(Ananas comosus)*, betik *(Carica papaya)*, limau bali *(Citrus grandis)*, pisang tanduk (*Musa* sp. cv. Tanduk), rambutan *(Nephelium lappaceum)*, markisa *(Passiflora incarnata)* dan cempedak *(Artocarpus integer)* telah ditentukan dengan kaedah oleh McCready (1970). Kulit limau bali mengandungi kandungan pektin yang tertinggi (6.87–5.12%) manakala kulit nanas yang terendah (0.01–0.05%). Kandungan pektin berkurangan mengikut susunan berikut: kulit limau kasturi, hampas jambu batu, hampas belimbing, kulit betik, kulit markisa, kulit cempedak, kulit rambutan, kulit pisang tanduk, kulit koko and kulit pisang Mas. Walaupun pektin yang dihasilkan boleh dikategori sebagai pektin metoksil tinggi, tetapi kelarutan dan keupayaan membentuk jel adalah rendah.

Abstract

Pectin content in local fruits such as cocoa *(Theobroma cacao),* calamansi lime *(Citrus microcarpa)*, guava *(Psidium guajava)*, *Mas* banana (*Musa* sp. cv. Mas), carambola *(Averrhoa carambola),* pineapple *(Ananas comosus)*, papaya *(Carica papaya)*, pomelo *(Citrus grandis)*, plantain (*Musa* sp. cv. Tanduk), rambutan *(Nephelium lappaceum)*, passionfruit *(Passiflora incarnata)* and *cempedak (Artocarpus integer)* by-products were determined using the method by McCready (1970). Pomelo peels had the highest pectin content (6.87–5.12%) while pineapple skins had the lowest (0.01–0.05%). Pectin content decreased in the following order: calamansi skins, guava press cake, carambola press cake, papaya skins, passionfruit pods, *cempedak* skins, rambutan skins, plantain skins, cocoa pods and *Mas* banana skins. Even though the pectin extracted can be categorised as high methoxyl pectins (HMP), its solubility and gel forming ability were poor.

Introduction

Pectic substances are high molecular weight polysaccharides widely spread in the plant kingdom. They can be found as an integral part of the primary cell wall and middle lamella of higher plants. Pectic substances are complex polymers composed of a backbone of partially methylated 1–4 linked a-D-galactopyranosyluronic acid residues with some 1–2 linked a-L-rhamnopyranose

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residues (Pilnik and Voragen 1970). Levels of pectic substances range from about 0.1% to 4% by weight of the plant's whole fruit (Ehrlich 1977).

The more commonly used term 'pectin' designates those pectic substances soluble in water and capable of forming gels under suitable conditions. Pectins vary considerably in composition and structure. Molecular weight can also vary with both botanical origin and maturity of the source materials (May 1989). Pectin is used as a jellying and thickening agent in the preparation of jams, jellies and marmalades, as a fat replacer in various food formulations and in the pharmaceutical industry for the treatment of diarrhoea. It has also been used as a haemostat agent. It is estimated that more than 50% of the world's pectin production is in the making of jellies, jams, marmalades and confectionery products, where the ability of pectin to form gels is the most important property (Ikkala 1986).

The two main sources of commercial pectin are apple pomace and citrus rinds. Other potential sources are from papaya (Sarode et al. 1964), lime and lemon peels (Rouse and Crandall 1978), guava (El Tinay et al. 1979), mango (Srirangarajan and Shrikhande 1979), sugar beet pulp (Phatak et al. 1988), sunflower head residue (Chang et al. 1994) and tropical agrowastes (Suhaila and Zaharah 1995).

The manufacture of pectin is an expensive and complicated process involving the preparation of raw materials including deactivation of enzymes, removal of bitter glycosides and crude sugars, conversion of protopectin into soluble pectin, filtration of the extracted pectin, precipitation of the pectin, purification and drying of the pectin. There will be minor variations in the process as different fruit varieties vary in their pectin content. It varies also at different stages of ripeness and due to different growing conditions.

Even though the current commercial sources of pectin are by-products of the fruit juice industry especially apple and citrus, there is abundance of local fruits that are not consumed as well as waste materials from agricultural practice and other fruit processing industries which can be used to produce pectin. This work deals with the extraction and determination of pectin in selected local fruits, with the ultimate aim of sourcing for new potential utilisation for such fruits.

Materials and methods

Acquisition of samples

All samples analysed were acquired from nearby markets except for cocoa pods (from MARDI Station, Hilir Perak, Perak), pomelo (from markets in Perak) and passionfruit pods (from MARDI Station, Bukit Ridan, Pahang). The materials investigated were cocoa pods *(Theobroma cacao)*, calamansi lime *(Citrus microcarpa)*, guava press cake *(Psidium guajava)*, *Mas* banana peels (*Musa* sp. cv. Mas), carambola press cake *(Averrhoa carambola)*, pineapple skins *(Ananas comosus)*, papaya skins *(Carica papaya)*, pomelo skins *(Citrus grandis)*, plantain skins (*Musa* sp. cv. Tanduk), rambutan skins *(Nephelium lappaceum)*, passionfruit pods *(Passiflora incarnata)* and *cempedak* skins *(Artocarpus integer)*. Three lots of each sample were obtained over the year in consideration for seasonal variations. The analyses were carried out in triplicates.

Extraction and determination of pectin

Five methods of extraction and determination of pectin were evaluated for accuracy, ease of extraction and optimum yield using orange peels as raw material. Analysis of variance using one-way ANOVA with multiple range tests was carried out and the significance level was established at $p \leq 0.05$.

- **Method I** (Simpson et al. 1984)
  Sample materials (50 g) were boiled in 95% ethanol for 15 minutes, washed with distilled water and dried at 40 ± 5 °C. The dried sample was extracted in

200 mL distilled water with initial pH adjusted with 0.1N hydrochloric acid (HCl) to values of 1 to 3 and were left for 8–12 h. The extracts were filtered through nylon gauze and the residues washed with 200 mL distilled water. The filtrate was concentrated over boiling water to one fifth of the initial volume and was added to acidified ethanol (pH between 0.7 and 1.0) in the ratio of 1:3. The mixture was stirred for 30 min and left to stand for 1 h. The mixture was filtered and washed with more acidified ethanol. The residue was dried at 60 °C to a constant weight.

- **Method II** (McCready 1970)
  Samples (50 g) were placed in 2.5 L boiling water and 8 mL HCl was added to give a pH of 2.2 ± 0.1, followed by addition of 20 g paper pulp filter aid. The mixture was heated at 95–100 °C for 30 min with constant stirring, after which the mixture was filtered and the residue was washed once with 500 mL boiling water. The filtrate was cooled before adding to 1.5 volumes 95% ethanol containing 2 mL/L HCl. The mixture was slowly stirred and left to stand for 30 min. The residue was collected and dried at 60 °C overnight.

- **Method III** (Sarode et al. 1964)
  Sample materials (50 g) were minced and soaked in water containing 0.03% sodium metabisulphite for about 1 h. The rinds were then washed with water and pressed to remove the water soluble non-pectic substances. The pressed sample was added to 100 mL 0.2% HCl solution and boiled at 100 °C for 30 min. It was strained through muslin cloth and the filtrate was quickly cooled. For second, third and fourth extractions, the residue was boiled in 50 mL 0.2% HCl solution for 30 min, strained and cooled before combining all the filtrates together. The filtrate was then concentrated to one quarter of its original volume. Pectin was extracted with 3 volumes of 95% ethanol. The mixture was allowed to stand for 4 h before filtration.

- **Method IV** (Suhaila and Zaharah 1995)
  Ground and dried materials (100 g) were weighed into tared 2 L beaker containing 500 mL distilled water. Sodium hexametaphosphate (12 g) was added and the initial pH was adjusted with 3N HCl to 2.2 ± 0.1, heated with constant stirring at 80 ± 5 °C for 1 h. The extract was filtered through muslin cloth and the residue washed with 200 mL warm water. The washings were added to the filtrate, which was concentrated by evaporation on a hot plate to approximately one fifth of its initial volume. The concentrated pectin solution was cooled to 50 °C and poured into a volume of ethanol in a ratio of 1:3, the ethanol containing 0.5M HCl. The mixture was stirred for 0.5 h and allowed to stand for 1 h. The precipitate was centrifuged at 3500 G for 15 min, washed with more ethanol-HCl solution and centrifuged at the same speed for 15 min. Finally, the precipitate was washed with acetone and the precipitate was dried at 60 °C to constant weight.

- **Method V** (Anon. 1986)
  Samples (50 g) were heated for 5–10 min and then washed with water. The samples were treated with HCl at a ratio of 1:2 at 60 °C for about 30 to 60 min. The pH of the mixture was 1.2–1.3. The mixture was immediately cooled and strained to remove the residue from the liquid. The filtrate was then precipitated in ethanol (1:2), stirred and left to stand overnight to allow colloidal particles to separate. Filtration was done to recover the precipitate and then it was air-dried for several hours before grinding to pass a 60 mesh screen.

Analysis

The moisture and ash contents of the extracted pectin were determined according to Egan et al. (1981). The methoxyl content was determined as described by Ranganna (1977). The colour and solubility were determined by visual observation. The ability to form gel was evaluated by using a standard gel formulation as given by Srirangarajan and Shrikhande (1979).

Results and discussion

Five methods of pectin extraction were evaluated to determine optimum extraction parameters and conditions as well as ease of extraction using orange peels as raw material. From the results given in *Table 1*, it was observed that different methods of extraction gave varying amount of pectin. In method I, extraction was carried out under ambient temperature using acidic conditions for 8–12 h. Methods II, III, IV and V utilised heat for the extraction process with temperatures ranging from 80 °C to 100 °C. All methods of extraction produced dry pectin powder except method III where liquid pectin was produced. Method II produced the highest amount of pectin (3.65 ± 0.53) followed by methods III (3.64 ± 0.55), V (3.51 ± 0.56), I (3.33 ± 0.28) and IV (3.32 ± 0.36). Method II was chosen as the method for the extraction of pectin from different materials based on its ease of extraction as well as significantly high recovery compared to the other methods ($p \leq 0.05$).

Analysis results showing the range and mean values for various parameters recorded for the pectin extracted from by-products of different local fruits are given in *Table 2* to *Table 5*.

The highest pectin was from pomelo peel ranging from 5.12% to 6.87%. Relatively high yields were obtained from guava press cake (3.11–3.89%), calamansi skin (2.99–4.08%) and carambola press cake (2.09–3.56%). Intermediate pectin content was obtained from *cempedak* skin (0.98–2.53%), papaya skin (1.36–2.10%) and passionfruit pods (1.63–1.97%). Low amount of pectin was extracted from rambutan skins (1.05–1.89%), plantain skin (0.55–1.08%), cocoa pods (0.54–0.91%) and *Mas* banana skin (0.25–0.67%). Pineapple skin gave very low to almost negligible level of pectin (0.05–0.10%).

Table 1. Pectin content (%) of orange peels extracted using different methods

Method	Range	*Mean ± SD
I	3.10–3.71	3.33 ± 0.28a
II	2.99–4.26	3.65 ± 0.53b
III	2.67–4.01	3.64 ± 0.55a
IV	2.95–3.33	3.32 ± 0.36a
V	2.89–4.19	3.51 ± 0.56a

*The values were expressed as mean ± standard deviation

Means with the same letter are not significantly different at $p \leq 0.05$

Comparison of data from this study with available data is shown in *Table 2*. It is noted that the pectin content of the fruit by-products differed from those obtained by Suhaila and Zaharah (1995) and Simpson et al. (1984). This may be due to differences in variety and maturity of the starting materials. It is also possible that the method chosen could be inefficient due to the differences in the raw materials used.

Moisture values *(Table 3)* differed in each of the pectin extracted with carambola press cake pectin giving the highest range (30.00–32.12%). Other pectins in the high moisture group are those from cocoa pods, guava press cake, rambutan skin and *cempedak* skin with moisture content ranging from 10.87% to 23.56% while the remaining pectins have their moisture content of less than 10%. Suhaila and Zaharah (1995) reported that calamansi lime, cocoa pod husks, pineapple skin, immature starfruit rejects, rambutan skin, *duku* skin, melon skin, jackfruit skin, durian skin and banana skin produced pectin having moisture ranging from 9% to 14% which was considered a normal moisture content, commercially.

Table 2. Yield (% fresh weight basis) of pectin extracted from by-products of different fruits compared with other studies

	S1		S2	S3
	Range	Mean ± SD		
Carambola press cake	2.09–3.56	2.24 ± 0.18	1.0 ± 0.4	
Cempedak skin	0.98–2.53	1.32 ± 0.62	4.7 ± 0.1 (jackfruit skin)	
Cocoa pods	0.54–0.91	0.75 ± 0.21	1.7 ± 0.3	0.06 ± 0.04
Guava press cake	3.11–3.89	3.49 ± 0.38		2.74 ± 0.36
Calamansi skin	2.99–4.08	3.50 ± 1.69	3.05 ± 0.05	
Mas banana skin	0.25–0.67	0.50 ± 0.14	1.6 ± 0.1 (banana skin)	0.41 ± 0.08
Papaya skin	1.36–2.01	1.82 ± 0.38		
Passionfruit pods	1.63–1.97	1.75 ± 0.07	3.02 ± 0.22	
Pineapple skin	0.05–0.10	0.07 ± 0.02	0.65 ± 0.15	0.15 ± 0.07
Plaintain skin	0.55–1.08	0.95 ± 0.21		
Pomelo peel	5.12–6.87	5.39 ± 0.81		
Rambutan skin	1.05–1.89	1.25 ± 0.21	1.9 ± 0.6	

S1 = this study
S2 = Suhaila and Zaharah (1995)
S3 = Simpson et al. (1984)

Table 3. Moisture content and total ash content of pectin extracted from by-products of different fruits

	Moisture content (%)		Total ash content (%)	
	Range	Mean + SD	Range	Mean ± SD
Carambola press cake	30.00–32.12	30.15 ± 0.30	8.22-9.23	8.81 ± 0.31
Cempedak skin	12.06–13.85	12.66 ± 0.95	6.95–8.88	7.54 ± 0.78
Cocoa pods	20.35–23.56	21.33 ± 2.23	7.96–9.21	7.77 ± 1.24
Guava press cake	13.97–15.77	14.05 ± 0.33	2.17–3.55	2.57 ± 0.21
Calamansi skin	7.12–8.56	7.70 ± 0.12	2.05–2.87	2.40 ± 0.21
Mas banana skin	4.63–5.55	4.95 ± 0.42	9.88–12.55	10.63 ± 2.01
Papaya skin	7.21–8.96	7.32 ± 1.32	4.88–7.86	6.36 ± 1.62
Passionfruit pods	5.10–7.05	5.21 ± 1.43	4.95–6.60	5.29 ± 1.31
Pineapple skin	7.65–9.98	8.70 ± 1.18	18.65–20.36	19.31 ± 1.05
Plaintain skin	4.98–6.25	5.29 ± 1.36	3.25–4.55	3.40 ± 1.20
Pomelo peel	7.02–8.65	7.76 ± 0.69	8.63–10.55	8.97 ± 0.83
Rambutan skin	10.87–14.58	12.43 ± 2.52	7.40–9.87	8.73 ± 0.56

Total ash content is an indication of the inorganic matter and it was observed that pineapple skin pectin has the highest ash content (18.65–20.36%) followed by *Mas* banana skin pectin (9.88–12.55%), pomelo peel pectin (8.63–10.55%), carambola press cake pectin (8.22–9.23%) and rambutan skin pectin (7.40–9.87%) *(Table 3)*. Other pectins contained less than 8% total ash content.

Methoxyl content is an important factor in evaluating the setting time of pectin, its sensitivity to polyvalent cations and usefulness in low solids gels and films (McCready 1970). From the results in *Table 4*, all sources produced pectins with quite high methoxyl content of more than 7% categorising them into high methoxyl pectin (HMP) (May 1989). These values, however, differed from those reported by Suhaila and Zaharah (1995), Simpson et al. (1984) and El Tinay et al. (1979) *(Table 4)*. Differences in the methoxyl contents may be

Table 4. Methoxyl content (%) of pectin extracted from by-products of different fruits compared with other studies

Source	SI				
	Range	Mean ± SD	S2	S3	S4
Carambola press cake	12.05–15.77	13.99 ± 2.89	10.1 ± 0.3		
Cempedak skin	11.58–12.99	12.35± 1.18	12.8 ± 0.8 (Jackfruit skin)		
Cocoa pods	10.07–12.41	10.54 ± 1.33	10.0 ± 0.6		
Guava press cake	11.00–12.55	11.38 ± 0.42		7.93 ± 0.25	3.03–3.23
Calamansi skin	11.74–13.25	12.04 ± 0.98	10.3 ± 0.1		
Mas banana skin	8.15–10.35	8.21 ± 1.04	7.9 ± 0.1 (Banana skin)		
Papaya skin	9.36–10.85	10.01 ± 0.55			
Passionfruit pods	10.33–11.17	10.65 ± 1.36		7.56 ± 0.62	
Pineapple skin	10.17–10.95	10.33 ± 0.69	10.2 ± 0.3		
Plaintain skin	7.28–9.36	7.93 ± 2.51			
Pomelo peel	10.66–12.58	11.26 ± 2.91			
Rambutan skin	10.13–12.07	10.99 ± 1.03	11.7 ± 0.0		

S1 = this study
S2 = Suhaila and Zaharah (1995)
S3 = Simpson et al. (1984)
S4 = El Tinay et al.(1979)

Table 5. Physical properties of pectin extracted from by-products of different fruits

Source	Colour	Solubility	Gel
Carambola press cake	Whitish grey	Low	Very soft
Cempedak skin	Brown	Low	No gel
Cocoa pods	Brown	Low	No gel
Guava press cake	Whitish grey	Low	Very soft
Calamansi skin	Blackish green	Medium	Soft
Mas banana skin	Brown	Low	No gel
Papaya skin	Brown	Low	No gel
Passionfruit pods	Dark brown	Low	No gel
Pineapple skin	Yellowish brown	Low	No gel
Plaintain skin	Dark brown	Low	Very soft
Pomelo peel	Whitish grey	Medium	Soft
Rambutan skin	Black	Low	No gel

attributed to the different cultivars used, different stages of maturity of the source materials as well as the extraction methods employed.

Physical properties of the pectin extracted include colour, solubility and gel forming ability. Colour of pectin is important as it affects the appearance of the gel produced. The colours of pectin vary with the source materials *(Table 5)*. The colours of pectin from carambola press cake, guava press cake and pomelo peel were the lightest followed by pineapple skin, *cempedak* skin, cocoa pods, *Mas* banana skin, papaya skin, passionfruit pods, plantain skin, calamansi skin and rambutan skin. All pectins except for calamansi and pomelo pectins have low solubilities. It was also observed that pectins from calamansi and pomelo produced soft gel while those from carambola press cake, guava press cake and

plantain skins produced very soft gel. Other pectins did not form gel at all.

Conclusion

Pomelo (*C. grandis)* peel had the highest pectin content followed by calamansi *(C. microcarpa)* skin, guava *(P. guajava)* press cake, carambola *(A. carambola)* press cake, papaya *(C. papaya)* skin, passionfruit *(P. incarnata)* pods, *cempedak (A. integer)* skin, rambutan *(N. lappaceum)* skin, plantain (*Musa* sp. cv. Tanduk) skin, cocoa *(T. cacao)* pods, *Mas* banana (*Musa* sp. cv. Mas) skin and pineapple *(A. comosus)* skin. Variation in the pectin content may be due to differences in variety and maturity of the starting materials. It is also possible that the method chosen could be inefficient due to the differences in the raw materials used. Further work in this area of extraction may need to be carried out to optimize the process.

Even though the pectin extracted can be categorised into high methoxyl pectins (HMP), solubility and gel forming ability were poor as such their use in food products would be limited.

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