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# Physicochemical properties of pectin extracted from jackfruit and chempedak fruit rinds using various acids

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**Abstract**

This study investigated the effect of citric, nitric and sulfuric acid on the yield and physicochemical properties of pectin extracted from jackfruit and chempedak fruit rinds. Yield and physicochemical properties such as uronic acid content, degree of esterification, degree of acetylation and colour of pectin solution were determined and compared. Yield of pectin from jackfruit and chempedak fruit rinds with nitric acid as extractant were 14.81 ± 1.02% and 17.62 ± 0.69%, respectively, which were the lowest. The uronic acid content of all extracted pectin was more than 65%. All jackfruit and chempedak fruit rind pectins in this study were high methoxyl pectin with degree of esterification ranging from 72-75% for jackfruit rind pectin and 66-69% for chempedak rind pectin. The degree of acetylation of all extracted pectin was lower than 1%. For both jackfruit and chempedak fruit rind pectins, the citric acid-extracted pectin produced darker, more reddish and yellowish solution and thus is least preferable. Among the acids studied, sulfuric acid was the best extractant due to the high yield of pectin and the solution of this pectin was brighter, less reddish and yellowish.

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## Introduction

Jackfruit *(Artocarpus heterophyllus)* and chempedak *(Artocarpus integer)* are fruits that are available in Malaysia. They can be consumed as table fruits or processed into other types of delicacies such as dried chips and battered-fried snacks. Jackfruit is one of the largest tree-borne fruit in the world and it is basically round-cylindrical in shape with the dimension of about 30 – 100 cm in length and 25 – 50 cm in diameter (Corner, 1938). Chempedak is smaller and slightly more oblong in shape if compared to jackfruit. Both fruits are covered with pyramidal spines that are either pointed or blunt. About 40-60% of each matured jackfruit and chempedak is composed of its fruit rind (unpublished data from preliminary study; Chadha, 1985). According to the statistic from Ministry of Agriculture and Agro-based Industry Malaysia, the productions of jackfruit and chempedak in Malaysia at 2012 were 19,712 tonnes and 35,412 tonnes, respectively. This means that at least 7884 tonnes and 14,164 tonnes of jackfruit and chempedak fruit rinds were produced, respectively. Some of the rinds were used as feed for cows and goats while the rest were disposed as waste. Disposing these rinds will be a burden for the environment. One way to overcome this problem is to utilize these wastes and produce useful material such as pectin.

Pectin can be found in the middle lamella of most plant cell wall and generally divided into 2 major groups, high methoxyl (HM) pectins and low methoxyl (LM) pectins, which are differentiated on the basis of their degree of methylation (DM). The DM value of commercial HM pectin is >50% whereas for LM pectin is <50% (Sriamornsak, 2003). Pectin is widely used as ingredient in food, pharmaceutical and cosmetic industries owing to its gelling properties. The most common commercial pectin obtained from fruit wastes are from apple pomace and citrus peel (May, 1990).

Most of the studies on pectin extraction investigated extraction parameters such as time, temperature, pH, extractant concentration and sample:solvent ratio during extraction, for example; pectin extraction on orange peel (El-Nawawi and Shehata, 1987), banana peels (Emaga *et al*., 2008), soy hull (Kalapathy and Proctor, 2001), passion fruit peel (Pinheiro *et al*., 2008) and others. The yield and characteristics of pectin from different fruits were different if extraction was performed using different

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acids (Virk and Sogi, 2004; Canteri-Schemin *et al*., 2005; Kliemann *et al*., 2008; Yapo, 2009a; Kumar and Chauhan, 2010).

However, information on pectin from jackfruit and chempedak fruit rind is scarce. Therefore, the objective of this study was to investigate the effect of different extractants on yield and characteristics of pectin from jackfruit and chempedak rinds.

## Materials and Methods

*Raw materials*

Jackfruit rinds of the cultivar J6 were supplied by a jackfruit seller in Serdang, Selangor. Chempedak of a local cultivar, 'paya jeras', were purchased from Universiti Putra Malaysia's Agriculture Park (TPU), Selangor. The selected fruits were all at commercial maturity level. All fruit rinds were washed and cleaned under running tap water after collection. The parts of the fruit rind used were according to Koh *et al.* (2014). The rinds were diced immediately into cube sizes of about 1cm * 1cm * 1cm. The cubed rinds were then used to prepare alcohol insoluble solids (AIS) and pectin was extracted from the AIS. Yield and physicochemical properties of the extracted pectin were determined using various chemical and spectrometry methods. Distilled water was used throughout the research and all chemicals purchased were of analytical grade.

*Alcohol insoluble solid preparation*

Alcohol insoluble solid (AIS) was prepared according to the method of Yapo and Koffi (2006) with slight modifications. The cleaned fruit rind cubes were boiled in 95% ethanol (1:2, w/v) at 80°C under reflux for 15 minutes to inactivate any possible enzyme and dried at 55°C until constant weight was achieved. The dried fruit rinds were then grounded into powder. The powdered fruit rinds were boiled in 80% ethanol (1:4, w/v) at 80°C under reflux for 45 minutes. Then the rind powder was filtered and washed three times with 60% ethanol, once with 95% ethanol and then dried at 35°C until constant weight was achieved. The dried residue was labeled as AIS and stored in capped Schott bottles at room temperature (25 ± 2°C).

*Pectin extraction*

Pectin was extracted from AIS according to the method of Yapo *et al.* (2009b) with slight modifications. The three acids selected as extractants in this study were citric acid, nitric acid and sulfuric acid. The AIS was suspended in one of the selected extractant at solid-liquid ratio of 1:25 (w/v). The pH of the suspension was adjusted to pH 2.0 and extraction was done at 90°C for 60 minutes in a shaking water bath at 120 rpm. After that, the AIS were filtered and the slurry obtained was concentrated to 2/5 of the initial volume under vacuum evaporation. The filtered AIS were then extracted one more time under the same condition (pH 2.0, 90°C for 60 minutes) and the slurry obtained was collected and concentrated. Both concentrated slurries were combined and pectin was precipitated by the addition of 95% ethanol at a ratio of slurry:ethanol (1:2, v/v). The mixture was stirred at low speed for 30 minutes at room temperature (25 ± 2°C). Then it was put at 5°C for 90 minutes to allow pectin flotation. The precipitated pectin was filtered and washed twice with 70% ethanol and then with 95% ethanol until the washing filtrate was colourless. Then the pectin was dried at 35°C until constant weight was achieved, grounded into powder and kept in a vacuum desiccator until further analysis. Pectin extraction using each extractant was carried out in triplicates.

*Yield*

The yield of pectin from each extraction process was calculated on a dry weight basis using Equation 1:

$$\text{Yield (\%)} = \frac{\text{(dry weight of pectin extracted, g)}}{\text{(dry weight of AIS used for extraction, g)}} \times 100 \quad (1)$$

*Uronic acid content*

Firstly, pectin molecules were hydrolyzed according to the method of Ahmed and Labavitch (1977). Then, uronic acid content was estimated using colorimetric method of Blumenkrantz and Asboe-Hansen (1973) with slight modifications according to Filisetti-Cozzi and Carpita (1991) and Ibarz *et al*. (2006). A sample control was also prepared for each hydrolysate. Aliquots of 100 µL from each hydrolysate and reagent control were transferred to their respective test tubes. Then, 300 µL of water were added to each test tube. A 40 µL of 4M potassium sulfamate solution were added and shake vigorously for 10 sec. After that, 2.4 mL of 75mM sodium tetraborate in 96% sulfuric acid solution was added and shake vigorously for 20 sec. The test tubes were capped and placed in boiling water for 20 min and cooled in ice bath for 10 min. Then, 80 µL of m-hydroxydiphenyl solution was added to both blank and sample, while 80 µL of 0.5 % NaOH was added to sample control (all solution was added just before absorbance value was read). The highest absorbance at 525 nm was read with D-galacturonic acid as standard with concentrations ranging from 25 - 225

µg/mL.

*Degree of esterification*

The degree of esterification of extracted pectin was determined according to the potentiometric titration method of Bochek *et al*. (2001).

*Degree of acetylation*

The degree of acetylation of extracted pectin was determined according to the method of Pippen *et al*. (1950).

*Colour of pectin solution*

Pectin solution was made by dissolving 0.5 g of pectin in 50 mL of distilled water and allowed the pectin to be hydrated. The dispersion was stirred at 50°C until dissolved. The colour of the solution was determined using Hunter LAB colorimeter (Model UltraScan PRO) coupled with EasyMatch QC software for data collection and display. The data reported were L (lightness; 0 = black, 100 = white), a (-a = greenness, +a = redness) and b (-b = blueness, +b = yellowness).

*Statistical analysis*

All analysis was done in triplicates and the data obtained were analyzed and interpreted by one-way analysis of variance (ANOVA) using MINITAB v.14 Statistical Package (Minitab Inc., State College, Pennsylvania). Values expressed were mean ± standard deviation and significance level was set at $p \leq 0.05$.

## Results and Discussions

*Yield*

Figure 1 shows the yield of pectin extracted from jackfruit and chempedak fruit rind using citric acid, nitric acid and sulfuric acid. The yields of pectin from jackfruit rind ranged from 14.8-18.6% (w/w) while pectin from chempedak rind was from 17.6-20.5% (w/w). For both jackfruit and chempedak fruit rind, the yield of pectin obtained using nitric acid were the lowest ($p \leq 0.05$) compared to citric acid and sulfuric acid, and there are no differences ($p > 0.05$) between citric acid and sulfuric acid. Effect of types of acid on pectin yield might be different on different samples or even samples from different sources. Yapo (2009a) extracted pectin from yellow passion fruit rind and suggested that the factor that affecting pectin yield might be due to the acids strength instead of the acids protic nature. Yapo (2009a) found that pectin yield obtained using strong acid was higher than weak acid. However Kliemann *et al.* (2008) also extracted

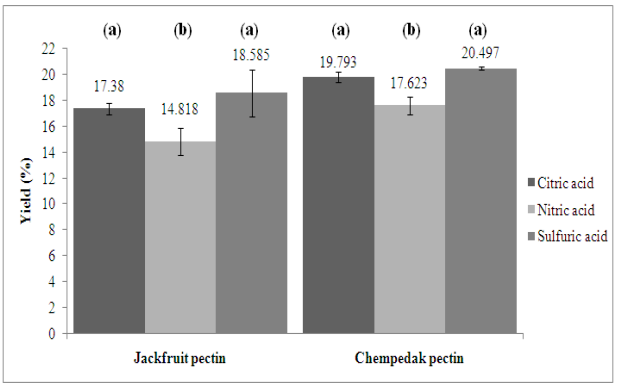


Figure 1. Yield of pectin extracted from jackfruit and chempedak fruit rind using various acids. Values having different alphabet within parenthesis are significant different ($p \leq 0.05$)

pectin from passion fruit rind but found that weak acid (citric acid) were able to extract significantly higher pectin yield than strong acid (nitric and hydrochloric acid), which was contrary to Yapo (2009a). It were also shown that pectin yield from apple pomace were higher if extraction was done using weak acid (citric acid) compared to strong acids (Virk and Sogi, 2004; Canteri-Schemin *et al*., 2005). Possible explanation for this is that the type, source and even variety of sample will affect the solubility of pectic materials in different type of acids.

Besides the type of acids, it was also shown that the concentration of acids used have effect on yield of jackfruit and chempedak rind pectin. The reason that nitric acid extracted significantly ($p \leq 0.05$) lower yield than citric acid and sulfuric acid was probably due to the concentration of nitric acid used had reached the point in which it will degrade pectin molecule and thus reducing the recovery of pectin. It has also been reported before that acid concentration has effect on soy hull pectin yield (Kalapathy and Proctor, 2001). Soy hull pectin was extracted using hydrochloric acid at different concentrations (0.05, 0.1, 0.2, 0.3 N) and at a lower concentration, higher yield of pectin was extracted than using higher concentration of acid.

*Uronic acid content*

Table 1 and 2 shows the uronic acid content (UA), degree of acetylation (DA) and degree of esterification (DE) of pectin extracted using 3 different acids from jackfruit and chempedak fruit rinds, respectively. According to the Food Chemical Codex (FCC), Food and Agriculture Organization (FAO) and European Union (EU), pectin must consist of at least 65% of galacturonic acid (Willats *et al*., 2006). The UA content of pectin extracted from jackfruit and chempedak fruit rinds were all more than 65%, regardless of the type of acid being

Table 1. Physicochemical properties of pectin extracted from jackfruit rind using various acids

| Pectin source | Extractant | Uronic acid (%) | Degree of acetylation (%) | Degree of esterification (%) |
|---|---|---|---|---|
| Jackfruit | Citric acid | 79.07 ± 5.13[a] | 0.478 ± 0.119[a] | 75.82 ± 3.62[a] |
| | Nitric acid | 81.34 ± 4.45[a] | 0.418 ± 0.142[a] | 74.82 ± 2.76[a] |
| | Sulfuric acid | 79.95 ± 4.47[a] | 0.567 ± 0.090[a] | 72.82 ± 2.99[a] |

Mean values having different superscript low case letter within the column are significant different ($p \leq 0.05$)

Table 2. Physicochemical properties of pectin extracted from chempedak rind using various acids

| Pectin source | Extractant | Uronic acid (%) | Degree of acetylation (%) | Degree of esterification (%) |
|---|---|---|---|---|
| Chempedak | Citric acid | 76.54 ± 4.68[a] | 0.060 ± 0.119[a] | 69.01 ± 1.66[a] |
| | Nitric acid | 83.65 ± 6.10[b] | 0.328 ± 0.179[b] | 69.10 ± 1.37[a] |
| | Sulfuric acid | 75.44 ± 3.17[a] | 0.328 ± 0.179[b] | 66.34 ± 1.43[b] |

Mean values having different superscript low case letter within the column are significant different ($p \leq 0.05$)

used as an extractant. This indicates that jackfruit and chempedak fruit rind pectin was high in purity and all 3 acids studied were effective extractants of extracting high purity pectin.

*Degree of esterification*

Pectin composes of mainly galacturonic acid as the structure backbone. These galacturonic acid subunits are partially esterified, mostly with methyl group and some with acetyl group. Degree of esterification is the sum of degree of methylation (DM) and degree of acetylation (DA).

In this study, DA of all jackfruit and chempedak fruit rind pectin extracted was lower than 0.6% and 0.4%, respectively. These were similar to pectin with low DA such as pectin from passion fruit rind, which is between 0.3 - 0.5% (Yapo and Koffi, 2006; Kliemann *et al*., 2008) while apple peel pectin, source of commercial pectin, have DA of 0.7% (Virk and Sogi, 2004). The DA of pectin was very low and therefore the DE of jackfruit and chempedak fruit rind pectin was assumed to be corresponded to DM because the DA was considered as negligible. Yapo (2009a) also made similar assumption in their study.

The DE of all extracted pectin was more than 50%, which means that they were all high methoxyl (HM) pectin. Jackfruit rind pectin obtained in this study was not affected ($p > 0.05$) by the type of acids used as extractants, with the DE ranging from 72 – 75%. This is in accordance to the findings of Seggiani *et al.* (2009) who found that the type of acid had no effect on DE of lemon peel pectin. In contrast, Yapo (2009a) found that the DE of passion fruit rind pectin extracted with nitric acid and sulfuric acid were lower than that of citric acid. When extraction pH was lowered from pH 2.5 to 1.8, DE of citric acid-extracted pectin dropped drastically and the deesterifying action of citric acid becomes similar to nitric acid and sulfuric acid. The extraction pH in this study was low (pH 2.0) and maybe it was low enough to increase the deesterifying power of citric acid to the same level as nitric acid and sulfuric acid. This might be the reason that the DE of all extracted pectin in this study showed no significant differences ($p > 0.05$), except sulfuric acid-extracted chempedak rind pectin. Sulfuric acid shows greater deesterifying effect on chempedak pectin compared to nitric acid and citric acid. Hence, the DE of sulfuric acid-extracted chempedak rind pectin was lower ($p \leq 0.05$) than citric acid- and nitric acid-extracted pectin.

*Colour of pectin solution*

Figures 2 and 3 show the colour parameters of solutions produced using jackfruit and chempedak fruit rind pectin extracted using citric acid, nitric acid and sulfuric acid, respectively. The colour parameters were L (lightness; 0 = black, 100 = white), a (-a = greenness, +a = redness) and b (-b = blueness, +b

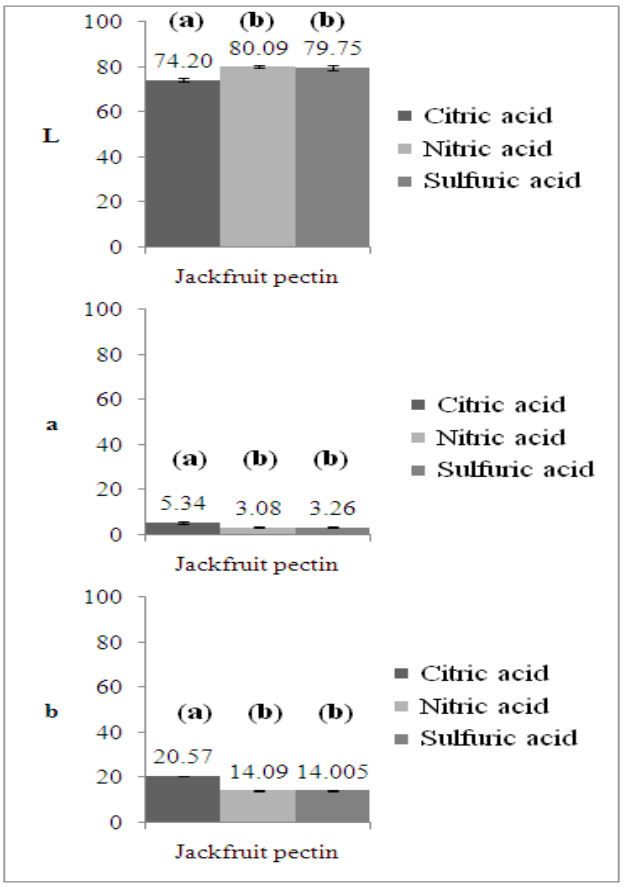


Figure 2. Colour parameters (L, a, and b) of solutions made from jackfruit rind pectin extracted using citric acid, nitric acid and sulfuric acid. Values having different alphabet within parenthesis are significant different ($p \leq 0.05$)

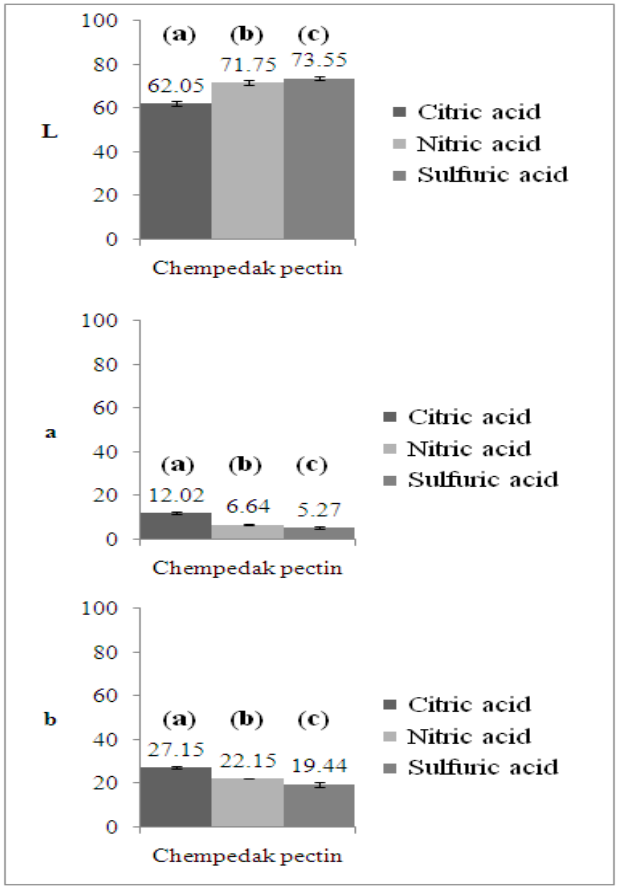


Figure 3. Colour parameters (L, a, and b) of solutions made from chempedak rind pectin extracted using citric acid, nitric acid and sulfuric acid. Values having different alphabet within parenthesis are significant different ($p \leq 0.05$)

= yellowness). Pectin is used as an ingredient in both food and non-food systems. Colour analysis is important because it would provide information on the likeliness of the extracted pectin to affect the appearance of the final product. The least coloured solution will be more preferable because it shows that the respective pectin will have least effect on the colour of the final product.

For jackfruit rind, citric acid-extracted pectin solution has lowest ($p \leq 0.05$) brightness and highest ($p \leq 0.05$) redness (colour parameter a) and yellowness (colour parameter b) compared to solution made from nitric acid- and sulfuric acid-extracted pectin (Figure 2). All colour parameters between nitric acid- and sulfuric acid-extracted pectin solutions showed no significant differences ($p > 0.05$). This means that citric acid-extracted pectin will produce product with darker appearance and more likely to affect the original colour of the end product as compared to pectin obtained using nitric acid and sulfuric acid, and thus will be least preferable. As for chempedak rind, the brightness between pectin solutions studied has significant differences ($p \leq 0.05$) with the sequence: citric acid < nitric acid < sulfuric acid; while the redness and yellowness were: citric acid > nitric acid > sulfuric acid ($p \leq 0.05$).

The reason that citric acid pectin solutions were darker, more reddish and yellowish was undetermined, but it was suspected that the concentration of acid used plays a major role in this observation. Much higher concentration of citric acid (about 0.1 M) was needed to obtain the extraction pH of 2.0 as compared to nitric acid (about 0.01 M) and sulfuric acid (about 0.005 M). This might have solubilised more colour and/or other compounds from fruit rind during the extraction. Therefore, in terms of colour, nitric acid and sulfuric acid were better extractants for jackfruit rind pectin whereas sulfuric acid was the better extractant for chempedak rind pectin.

## Conclusion

The potential of jackfruit and chempedak fruit rind as the source of pectin has been shown. The yield of jackfruit rind pectin was 14.8% - 18.6% and chempedak rind pectin was 17.6% – 20.5%, in which nitric acid extracted the lowest yield of pectin. The type of acids does not affect the UA and

DE of jackfruit rind pectin. Chempedak rind pectin extracted using nitric acid has significantly higher UA, while pectin extracted using sulfuric acid has lowest DE. It was shown that colour of the pectin solution made using citric acid-extracted pectin was the least bright, most reddish and yellowish. Overall, sulfuric acid was the best extractant among the acids studied because sulfuric acid was able to extract high amount of pectin in which the pectin solution has highest brightness and less reddish and yellowish.

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