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Physicochemical Properties of Pectin Extracted from Selected Local Fruits By-Product

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Abstract- Pectin was extracted from selected local fruits by-product, which was a banana, mango, and papaya peel by using the citric acid solution as a solvent. The primary objectives of this study were to extract pectin from selected local fruits by-product as well as, to determine and compare the physicochemical properties of extracted pectin to that of commercial pectin. Pectin extracted from mango peel gave the highest yield significantly (15.4%) as compared to banana peel pectin (14.47%) and papaya peel pectin (12.77%). The physicochemical properties of pectin obtained were compared with commercial pectin. These include color, degree of esterification (DE), ash, moisture, solubility, and viscosity. The color measurement of pectin showed it has a significant difference between other types of pectin. The color parameter L (lightness) showed commercial pectin had the highest value of 89.17 significantly, yet significantly the lowest of a* (redness) value of 1.72. Hence, the color parameter b* (yellowness) showed that the papaya peel pectin was significantly the highest (17.65) as compared to other types of pectin. All pectins in this study were of high methoxyl pectin as the DE was found to be more than 50%. No significant difference was noted for DE between commercial pectin and both banana and mango peel pectin. As for the ash content, banana peel pectin has significantly the highest ash content of about 4.60% compared to other types of pectin. Moisture content also showed a significant difference between all kinds of pectin. Solubility analysis also indicated significant difference between all types of pectin in this study with commercial pectin to possess the highest solubility (98.37%) as compared to other types of pectin. No significant difference was noted for viscosity between commercial pectin and mango peel pectin. Based on this study, it can be concluded that pectin extracted from mango peel possess high chances to be exploited and commercialized as most of the physicochemical analysis carried out showed no significant difference to that of commercial pectin.

Keywords-pectin; by-products; degree of esterification (DE); local fruit.

I. INTRODUCTION

Pectin is a high-value functional ingredient widely used in food as a gelling agent in jams and jellies, as a thickening agent in sauce and ketchup and also as an emulsifier in an acidic milk drink. Pectin is a complex mixture of polysaccharides that consist of repeating units of Dgalacturonic acid which are joined by α -1-4-linkage. It is located in the cell wall and the middle lamellae of plants [1]. Pectin appears in a different amount in fruit cell walls and has gel forming capabilities, especially for jams, marmalade, and concentrate [2]. The gelling abilities of pectin depend on the degree of methyl esterification (DE) that can be divided into two categorized, low *methoxyl* pectin and high *methoxyl* pectin. This categorized pectin is differentiated by the degree of esterification (DE). The DE value of commercial HM pectin is more than 50%, while for LM pectin is less than 50% [3].

Pectin is one type of substances that can be recovered from fruits waste. The local fruit waste that has sources of pectin are lime and lemon peel, papaya, mango, sunflower head residues, sugar beet pulp, as well as tropical agricultural wastes [4]. Malaysia has a large variety of fruits, whether fruits are seasonal or perennial. The examples of seasonal fruits are durian, rambutan, duku, langsat, rambai, mangosteen, and so on. Hence, the example of perennial or off-season fruits like banana, guava, mango, papaya, pineapple, and many more. The rise in production of products fruit-based in the processing industry, especially using off-season fruits due to availability throughout the year that results in generating massive fruit by-products. However, these by-products have the ability for conversion into valuable products such as pectin [5].

Food wastes are major problems in food processing industry due to higher cost and lack of landfill to dispose of it. The increase in the food processing industry such as canned foods, fruit juice, and puree processing can cause an increasing in by-products. It can lead to a large volume of wastage to the environment. Waste disposal represents a growing problem because it is prone to microbial spoilage

and resulting in serious environmental problems [6]. This study can prove there is no significant difference between pectin extracted from fruits by-product to the physicochemical properties of commercial pectin.

In addition, this study will provide information on pectin also can be obtained from local fruits by-product, even though the commercial pectin is mainly from citrus by-products and apple *pomace*. In addition, the uses of fruits waste also can reduce the environmental problems and the cost of waste disposal due to a large number of wastes generated in the food processing industry. The objectives of this research are to extract pectin from selected local fruits by-product such as banana peel, mango peel, and papaya peel by using acidic extraction method as well as, to determine and compare the physicochemical properties of extracted pectin to that of commercial pectin.

II. MATERIALS AND METHODS

A. Dried sample preparation

The selected local fruits by-product, papaya peel, banana peel, and mango peel were obtained from *Pasar Moden*, *Section 6*, and *Shah Alam*. The papaya peel was cut into smaller size and slightly washed with tap water to remove any impurities on peels. The peel was blanched for 5 minutes in boiling water [7] to inactivate any microorganisms that appear on peel. Then, the blanched peel was allowed to drain out of water. The blanched peel was loaded on perforated stainless steel tray for kept in cabinet dryer with $60 \pm 1^{\circ}$ C for 1 day [8]. After that, the dried peel was ground into fine particles through 30 meshes of sieves and store in air-tight container [9]. This method was repeated using mango peel and banana peel.

B. Extraction of Pectin

The pectin extraction method was followed instructions as in [9] with minor modifications. A total of 10 g of papaya peel powder was weighed on an analytical balance. Then, the powder was blended with 250 ml distilled water and acidified with different volume of 0.1 N citric acid until the pH was shown of 2.0 on pH meter. The mixture was stirred by using stirrer until the powder homogenizes wet with acidified water. This procedure was continued by treated the acidified samples at 70°C for 75 minutes in shaking water bath. After that, the mixture was kept at room temperature for 24 hours. Then, the sample was filtered, and a double volume of 95% ethanol (1:2 v/v) was added for pectin precipitation. The sample was stored in dark condition at room temperature of 25°C in 24 hours for pectin floatation. After that, the pectin was separated by filtration process and wash with 70% ethanol twice. Acetone was added in drop wise for the unwanted removed color of pectin. Then, the pectin was dried in a drying oven at 60°C overnight or until a constant weight was reached. This method was repeated with mango peel and banana peel to getting pectin from different samples. The yield of pectin from papaya peel, mango peel, and banana peel was calculated as follows:

Pectin yield (%) =
$$\frac{\text{Weight of extracted pectin}}{\text{Weight of dried sample (10 g)}} \times 100$$
 (1)

C. Viscosity Measurement

The viscosity measurement was determined by using viscometer (Brookfield Engineering Inc., Model RV-DV+1) by following the method as in [10] with modifications. The pectin was measured in 2.0% w/v with viscometer by using spindle at room temperature. The spindle number 1 was used that operated at 10 rpm. The viscosity reading in centipoises (cP) after 30 seconds of rotation was recorded.

D. Color Measurement

The color of pectin was determined by using chromameter (CR-400 Konica Minolta, Japan) by following the method as in [11]. The pectin was kept in airtight plastic to measure the color of pectin. The color parameters were L (lightness; 0 = black, 100 = white), a (-a = greenness, +a = redness) and b (-b = blueness, +b = yellowness). The colorimeter was calibrated with reference white tile before each measurement of samples. The measurement was repeated three times to get an average.

E. Solubility analysis

A total of 0.2 g of pectin was dissolved in 20 ml distilled water. The solution was heated by using hotplate to dissolved pectin. The additional water was added if the solution was running out of water. The solubility of pectin was tested by using the method from [12] with slight modifications. The ashes filter was weighed on an analytical balance, and the weight of ashless filter paper was recorded. After pectin was dissolved, the pectin solution was cooled and filtrated by using pre-weighed ashless filter paper to measure the solubility of pectin. After filtration, the ashless filter paper was transferred into the aluminum dish and dried in the oven at 60°C overnight. The degree of solubility of pectin was calculated based on the difference in weight of ashless filter paper before and after drying. The calculation can be simplified as follows:

Solubility=100 -
$$\left(\frac{W_{after\ drying} - W_{before\ drying}}{W_{before\ drying}} \times 100\right)$$
 (2)

F. Determination of Degree of Esterification

The Degree of Esterification (DE0 was followed by the instruction as in [13] with slight modifications. The DE is calculated using the following formula.

$$DE = \frac{V2}{(V1+V2)} \times 100 \tag{3}$$

A total of 0.2 g dried pectin was moistened with ethanol and dissolved in 20 ml of deionized water. The solution was heated on a hotplate until the pectin was dissolved. After pectin was dissolved, a total of 5 drops of phenolphthalein was added to the solution. Then, the solution was titrated with 0.5 M NaO, and the volume of NaOH solution was recorded when the color of the solution was changed from colorless to a faint pink, and as an initial titer V1. Hence, a total of 10 ml of 0.5 M NaOH was added into the solution. The solution was shaken strongly and rest for 15 minutes. Then, a total of 10 ml 0.5 M HCl was added into the solution and shaken strongly until the pink color was disappeared. Then, the solution was titrated with 0.5 M NaO, and the colorless of the solution is back to faint pink again. The volume of NaOH at this point as final titer, V2 was recorded. The degree of esterification (DE) was calculated

G. Determination of ash

The method for ash determination was taken from [14] with some modifications. The bottom of the porcelain dish was labeled by using liquid paper clearly and dried in an oven at 105°C for 3 hours. Then, the porcelain dish was cooled in desiccator and weigh soon after it has attained room temperature. A total of 1 g of the pectin was weighed into porcelain dish. The dried pectin was burned gently over a Bunsen burner until no smoke was evolved. The dish was placed in a muffle furnace at 550°C for 3 hours until whitish or greyish ash was obtained. The dish was removed, and then, it was cooled in a desiccator and weigh soon after attaining room temperature. The ash content was calculated as formula follows:

Ash content (%) =
$$\frac{\text{weight of ash}}{\text{weight of the sample (pectin)}}$$
 x 100 (4)

H. Determination of moisture

The moisture determination method was adapted from [14] with some modifications Aluminum with cover was dried for 3 hours in an oven at 105°C. The dish was transferred into the desiccator to let it cool and weigh after it has attained room temperature as W1. The dried aluminum dish with covers was labeled and weighed accurately. Then, a total of 1 g of pectin was weighed into an aluminum dish. The aluminum dish with sample (pectin) was weighed as W2. The aluminum dish with sample uncovered was placed in an oven at 105°C overnight. After that, the cover of the aluminum dish was replaced before remove it from the oven. The dish with the sample was cooled in a desiccator and weigh soon after attaining room temperature. The weight obtained was recorded as W3. The percentage of moisture content was calculated as described below:

Moisture content (%) =
$$\frac{W2-W3}{W2-W1} \times 100$$
 (5)

I. Statistical analysis

All analysis was done in triplicate, and the data obtained were analyzed and interpreted by IBM SPSS Statistic version 20.0. One-way analysis of variance (ANOVA) was applied to determine significant difference at p < 0.05.

III. RESULTS AND DISCUSSION

A. Pectin extraction yield

Three different samples, which was banana, mango and papaya peels were used in this study. The pectin was extracted for 75 minutes at 70°C and maintained at a pH of 2.0 to enhance the maximal yield of pectin. The organic acid, which was citric acid used in this study due to environmental considerations to keep the environment safe and clean. Also, hydrolyzing capacity of organic acid was lower due to lower of the dissociation constant that likely pectin to cause proton catalyzed depolymerization [15]. The yield of pectin from different peels was presented in Fig 1. Figure 1 below shows that values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP:

Papaya Peel Pectin. There were significantly different in pectin extraction yield from three types of selected local fruits by-product. The mango peel pectin showed significantly the highest pectin yield, which was 15.4%, followed by banana peel pectin and papaya peel pectin with 14.47% and 12.77% respectively. The amount of pectin in fruits also depends on on the cultivar, ripeness, and extraction condition.



Fig 1 The extraction yield of pectin from different samples by using the citric acid extraction method

Reference [16] found the fruit maturity or cultivar have influenced in quality and amount of pectin. They also proven about blanching and pressing the fresh mango peel can enhance both quality and amount of pectin. Also, the amount of pectin yield is increased at the beginning of fruit peel become tenderer [17]. There are scarce researches on pectin extraction from papaya peel, but a study from [18] said that pectin and protease could be recovered from papaya peel with an adequate yield of pectin.

B. Viscosity measurement

The viscosity was examined by 2.0% (weight per volume) of concentration by using viscometer under room temperature. The result of viscosity measurement for each sample were presented in Fig 2. Fig 2 showed the commercial pectin had high in viscosity, which was 28.67 cP and followed by mango peel pectin, banana peel pectin and papaya peel pectin with viscosity measurement of 28.33, 23.00 and 22.33 cP, respectively.

The commercial pectin and mango peel pectin showed no significant differences among each other. It showed the degree of gelling properties of commercial pectin and mango peel pectin was the same. Also, the result also showed there was no significant difference between banana peel pectin and papaya peel pectin.

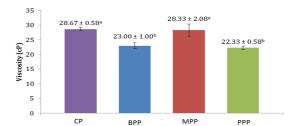


Fig 2 The viscosity of pectin solution of four different samples

Figure 2 indicates that values are expressed as mean ± standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin

C. Color measurement

Color analysis will influence the likeness of extracted pectin as it might affect the appearance of the final products. The least or transparent color of pectin solution is more preferable because it shows pectin will have the least effect in color of final products such as in jams and jellies [3]. Fig 3, 4, and 5 show the color parameters of pectin produced using banana, mango, and papaya peels extracted by using citric acid.

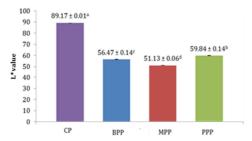


Fig 3 Color parameter L (lightness) of pectin from different samples

Figure 3 describes that values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin. As shown in Fig 3, there was a significant difference (p<0.05) between four samples. It showed that the commercial pectin had the highest lightness (more to white) value of 89.17 significantly. Then, it followed by papaya peel pectin, banana peel pectin and mango peel pectin with 59.84, 56.47 and 51.13, respectively. The mango peel pectin was significantly the lowest in lightness, indicated the mango peel pectin was closely to darker appearance. It was contrary to color parameter a*, where it showed that the commercial pectin had the least value (1.72) significantly. Fig 4 showed the papaya peel pectin had significantly the highest value of color parameter a* with 6.25, which was close to redness. Then, it was followed by banana peel pectin and mango peel pectin with the value of 5.08 and 1.85, respectively.

For the yellowness (color parameter b*) as presented in Fig 5, it showed that the papaya peel pectin had the highest b* value of 17.65 significantly. Then, followed by commercial pectin, banana peel pectin and mango peel pectin with the value of b* were 10.45, 8.89 and 6.17, respectively. All color parameters of pectin showed there was a significant difference (p<0.05). This means the banana peel pectin, mango peel pectin, and papaya peel pectin were more likely to affect the color of final products due to the significant difference in color parameters as compared to commercial pectin. In the future, the new methods might be applied to remove the unwanted color of pectin solution to keep the better color of final products as commercial pectin.



Fig 4 Color parameter a* (redness) of pectin from different samples

Figure 4 above highlights that values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at the level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin

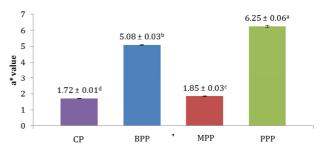


Fig 5 Color parameter b* (yellowness) of pectin from different samples

Figure 4 above shows that values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin

D. Solubility analysis

The solubility of pectin was important to ensure the pectin was solubilized to produce good quality of end products. Insoluble pectin can affect final products quality in terms of the appearance of the product itself. Fig 6 showed that the solubility analysis was conducted on four different types of pectin.

The result showed that the commercial pectin had great solubility characteristic, which was 98.37%. This value was close to 100% that make the commercial pectin as good quality of pectin for gelling properties for jams, marmalade, and jellies. Commercial pectin had significant differences in terms of solubility between banana peel pectin, mango peel pectin, and papaya peel pectin.

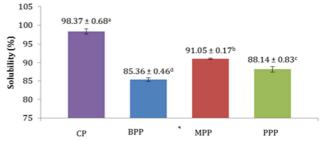


Fig 6 the solubility analysis for different types of pectin

Figure 6 above shows that values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin. Mango peel pectin also well solubilized in water, which was 91.05%, followed by papaya peel pectin and banana peel pectin with the percentage of solubility 88.14% and 85.36%, respectively. There were showed a significant difference between these four different samples. The banana peel pectin had significantly the lowest soluble pectin, and it might contain certain amount of insoluble compounds in pectin's such as impurities and other minerals. Mango peel pectin also showed the good solubility of pectin in water compared to banana peel pectin and papaya peel pectin.

E. Degree of esterification determination

The gelling properties of pectin are important to produce high quality of pectin. It can be divided into two categories, which were pectin with a degree of esterification more than 50% which are known as high *methoxyl* pectin (HM) and low *methoxyl* pectin (LM) which has less than 50% of DE. The degree of esterification of different types of pectin was presented in Fig 7.

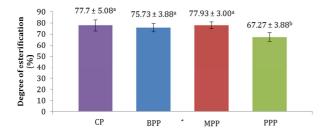


Fig 7 Degree of esterification for four different types of pectin

The values in Figure 7 are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin. In this stud, the commercial pectin and extracted pectin from peels were categorized as high methoxyl pectin as the DE was more than 50%. It indicated that the pectin structure more esterified and ability to form better gels in food products. The commercial pectin, banana peel pectin, and mango peel pectin showed no significant difference (p<0.05). It means the banana peel pectin and mango peel pectin has the same power as commercial pectin. However, the papaya peel pectin was significantly differenced to another pectin (CP, BPP, and MPP). Even though the DE of these four types of pectin were high methoxyl content (HM), but only papaya peel pectin showed it was a significant difference (p<0.05) among others.

F. Ash determination

Ash content determination was important as ash represent the total mineral content in pectin. Fig 8 shows the ash content in four different types of pectin. Banana peel pectin has significantly the highest in ash content, which was 4.60% compared to commercial pectin, papaya peel pectin and mango peel pectin with ash content of 2.40%, 1.20%,

and 0.12%, respectively. There were no significant differences noted between commercial pectin and papaya peel pectin. Also, the mango peel pectin and papaya peel pectin showed no significant differences among each other. Figure 8 indicates the values are expressed as mean ± standard deviation. Values with different alphabet were significantly different between samples at a level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin. Mango peel pectin had the lowest inorganic residue after ignition process was conducted. However, commercial pectin had high in ash content compared to mango peel pectin and papaya peel pectin. This result was contrary to study as in [17]. They found the commercial pectin had the lowest ash content, which was 1.76%.

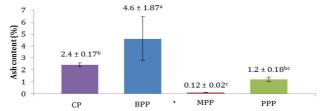


Fig 8 The ash content in four different types of pectin

In contrast, this study showed the ash content was higher (2.40%) compared to study as in [17]. Reference [19] was reported the ash content of banana peel pectin and mango peel pectin were 0.47% and 0.915%, respectively. However, this study showed the ash content of banana peel and mango peel pectin were 4.60% and 0.12%, respectively. This result showed a big difference percentage of ash content of these two types of pectin. These results can be improved through a better pectin extraction method, types of extractors, and addition of chelating agent.

G. Moisture determination

Moisture content analysis of different types of pectin was presented in Fig 9. The result showed moisture content in commercial pectin was significantly the highest compared to banana peel pectin, mango peel pectin, and papaya peel pectin. Moisture content in commercial pectin was 8.32%, followed by banana peel pectin (5.68%), mango peel pectin (3.67%) and papaya peel pectin (0.96%). There were significant differences between all types of pectin.

Reference [17] found the moisture content of commercial pectin was 12.03%. However, the moisture content in this study was 8.32%, which indicated it significantly the highest moisture content among other pectin. A study from [19] found the moisture content of banana peel pectin and mango peel pectin were 8.94% and 8.82%, respectively.

On the other hand, the moisture content of these two types of pectin was different in this study. This study showed the moisture content of banana peel pectin and mango peel pectin was 3.67% and 0.96% respectively. It showed this pectin are safe for storage purposes at room temperature. The improved method of processing might be introduced to ensure the moisture content of pectin was less than 10%. A good quality of pectin powder, it must be below 10% for storage stability [17].

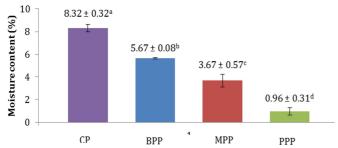


Fig 9 The moisture content in four different types of pectin

Figure 9 indicates the values are expressed as mean \pm standard deviation. Values with different alphabet were significantly different between samples at level of p<0.05. CP: Commercial Pectin; BPP: Banana Peel Pectin; MPP: Mango Peel Pectin; PPP: Papaya Peel Pectin

H. Recommendations

This study was important to recover waste product into a valuable product. In a future study, it was recommended to compare more local fruits by-products in Malaysia such as pineapple, durian, mangosteen, rambutan, and many more. The good quality of pectin can be determined by conducting further analysis such as methoxyl content, sugar composition, galacturonic acid, and emulsifying properties. In addition, the response surface methodology also can be used to identify the right temperature of pectin extraction to produce good quality and adequate amount of pectin. The extraction of pectin by using different extraction solvents also can give a better comparison in a future study. The pectin obtained also can be applied to food product to determine the suitability as a gelling agent. The investigation on pectin also can be extended to the sensory evaluation in order to determine any loss of color or flavor in the food products.

IV. CONCLUSIONS

From this study, it can be concluded that pectin can be extracted from selected local fruits by-product such as banana peel, mango peel, and papaya peel. The pectin yield from mango peel was significantly the highest, which was 15.40% whereas banana peel pectin and papaya peel pectin were 14.47% and 12.77%, respectively. Hence, the physicochemical properties of extracted pectin to that of commercial pectin can be determined and compared. The commercial pectin has high in viscosity, which was 28.67 cP and followed by mango peel pectin, banana peel pectin and papaya peel pectin with viscosity measurement was 28.33, 23.00 and 22.33 cP, respectively. The commercial pectin and mango peel pectin has a significant difference between banana peel pectin and papaya peel pectin. The color of pectin can be determined by using chromameter. The three color parameter L, a* and b* were identified.

For solubility analysis, the commercial pectin showed it was significantly the highest soluble pectin compared to another pectin. The commercial pectin was 98.37% soluble, followed by mango peel pectin, papaya peel pectin, and banana peel pectin with a solubility of 91.05%, 88.14%, and 85.36%, respectively. All pectin showed high *methoxyl* pectin as it had a degree of esterification was more than 50%

— no significant difference between commercial pectin, banana peel pectin, and mango peel pectin. Mango peel pectin showed it was the high degree of esterification (DE), which was 77.93%, and its value that is closely related to commercial pectin (77.67%). Banana peel pectin has significantly the highest of ash content (4.60%) compared to commercial pectin, papaya peel pectin and mango peel pectin with ash content was 2.40%, 1.20, and 0.12%, respectively. On the other hand, commercial pectin had significantly the highest of moisture content which was 8.32 and then followed by banana peel pectin (5.68%), mango peel pectin (3.67%) and papaya peel pectin (0.96%).

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