Characterization of pectin from tongka langit banana peels with various extraction temperature

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Abstract. Banana peel waste can be used in the food industry as a source of pectin. Pectin can be obtained from the extraction process tongka langit banana peel. This research's objective was to characterize pectin's properties extracted from banana tongka langit peel with different extraction temperatures. A completely randomized experimental design with 3 level treatment extraction temperature of banana tongka langit peel, i.e., 80°C, 85°C, and 90°C, with three replicates was applied in this research. The properties evaluated, including equivalent weight, methoxyl content, galacturonate acid, esterification degree, yield, moisture content, and ash content. Result showed that equivalent weight (3104.3 mg, 1084.0 mg, and 699.8 mg), methoxyl content (3.25%, 3.87%, and 5.44%), galacturonate acid (24.56%, 38.06%, and 56.08%), esterification degree (76.02%, 57.45%, and 55.05%), moisture content (11.39%, 10.79%, and 9.85%), ash (4.42%, 5.11%, and 6.47%), and yield (8.37%, 8.80%, and 9.20%).

1. Introduction

Banana tongka langit (Musa troglodytarum L.) is one of the local foods in the Maluku region containing high carbohydrate, fiber, vitamins and carotenoid compounds to be beneficial for health. With this potential, efforts were made to develop tongka langit bananas into several processed products including instant porridge [1], crackers [2], brownies [3], starch [4], and several other products.

Processing the tongka langit banana to produce a product is in the stage of peeling the fruit peel to be taken from the fruit flesh so that the fruit skin will only be disposed of as organic waste or used as animal feed. Waste can cause environmental damage problems, waste elimination, and banana peels can be used as a product with a sale value, one of which is pectin. Banana peels could be used as a food source with high value, such as pectin [5].

Pectin is a polysaccharide compound with a high molecular weight with a functional food value due to its suitable emulsion and stability to be used as a gelling agent and stabilizer, for example, in jelly manufacture jam, marmalade, edible film, and edible coating.

Several research results use banana peels to be used as pectin, including pectin from cavendish banana peels [6], saba banana peels [7], and raw banana peels [8]. Based on the study results, the pectin content in the banana peel tongka langit is 10.41% [9], so that the tongka langit banana is made to produce pectin compounds. Pectin can be obtained from the source through the extraction process. Pectin extraction can be relied on by several factors, including pH, type of solvent, extraction, and extraction temperature. So these factors need to be considered to get the best quality pectin.
This research carried out pectin extraction from tongka langit banana peels with various extraction temperatures based on the description above.

2. Methods
This research's raw material was the tongka langit banana obtained from Soya State, Ambon City, Maluku. The chemicals used include hydrochloric acid (Merck, Germany) and 96% ethanol (Merck, Germany). The equipment consists of a cabinet dryer, analytical scales (Ohaus, USA), and hot plates.

2.1. Pectin extraction
Pectin extraction refers to the method of Ramdja[10] with modifications. 20 g of cleaned tongka langit banana peel is dried using a cabinet dryer. After drying, the banana peel is crushed with a crusher. The powder of the tongka langit banana peel was added with 500 mL of HCl (0.1 N) and extracted based on temperature treatment (80 °C, 85 °C, and 90 °C) for 2 hours. Furthermore, filtering is carried out to separate the precipitate from the filtrate. Pectin phytate was precipitated with 96% ethanol (1: 1). The precipitated pectin is separated from the filtrate using filter paper. The residue was washed with 96% ethanol. The results of the filtration were dried using an oven at 40 °C for 24 hours. Then pectin analysis was carried out for the equivalent weight[11], methoxyl content [11], galacturonic acid [11], degree of esterification [11], moisture content[12], ash content [12], and yield.

2.2. Equivalent weight
Equivalent weight was defined using weighing 0.5 g of pectin in 100 mL conical flask and moistened with 5 mL of ethanol. 1 g of sodium chloride would be added for the more incisive endpoint. The mixture was then stirred until it reached homogeneity. Titration was then slowly performed (to avoid the possibility of de-esterification) using standard 0.1 N of NaOH until the red indicator turned pink (pH 7.5) and lasted 30 seconds at most. Neutralizing solvent was then used to determine methoxyl content. The equation below was used to calculate the equivalent weight:

\[(\text{mg}) \text{Equivalent weight} = \frac{\text{weight of sample} \times 1000}{\text{mL titrated NaOH} \times N \text{NaOH}}\]

2.3. Methoxyl content
In determining the methoxyl content, 25 mL of 0.25 N NaOH was added to titration solvent, shaken, and left for 30 minutes at room temperature in a stoppered flask. And 25 mL of 0.25 N HCl was then added and titrated with 0.1 N NaOH solution until the same previous endpoint.

\[\% \text{ Methoxyl content} = \frac{\text{mL titrated NaOH} \times N \text{NaOH} \times 31}{\text{pectin weight}}\]

2.4. Galacturonic acid
Galacturonic acid would then be determined using the following:

\[\% \text{ Galacturonic acid} = \frac{\text{eq (equivalent content)-methoxyl content} \times 176 \times 100}{\text{pectin weight}}\]

2.5. Degree of esterification
Degree of esterification in pectin was defined as follows:

\[\% \text{Degree of esterification} = \frac{\text{Methoxyl content} \times 176 \times 100}{\text{galacturonic acid} \times 31}\]

2.6. Moisture content
Weight about 5 g of sample to the dish. Place the dish with the sample in the oven. Dry for 3 hours at
105 °C. After drying, transfer the dish with a partially covered lid to the desiccator to cool. Reweight the dish and its dried sample.

\[
\% \text{ Moisture content} = \frac{\text{weight of sample before drying} - \text{weight of sample after drying}}{\text{weight of sample before drying}} \times 100
\]

2.7. Ash content
Weight about 1.5 g sample into the crucible. Heat over low Bunsen flame with the lid half covered. When fumes are no longer produced, place the crucible and lid in the furnace. Heat at 550 °C in the electric furnace until ashes are complete. Transfer the dish with a partially covered lid to the desiccator to cool and then weighed.

\[
\% \text{ Ash content} = \frac{\text{weight of ash}}{\text{weight of sample}} \times 100
\]

2.8. Pectin yield
Pectin yield was calculated as follows:

\[
\% \text{ Yield} = \frac{\text{weight of dried pectin}}{\text{weight dried banana peel taken for extraction}} \times 100\%
\]

2.9. Statistical analysis
The research design was carried out using a completely randomized design with three treatment levels of pectin extraction temperature. Each treatment level was repeated three times. Analysis of variance was used to analyze the data obtained and the Tukey test for the average treatment difference test using Minitab 17 software.

3. Results and discussion
3.1. Weight equivalent
The average value of the equivalent pectin weight of tongka langit banana peel with temperature treatment can be seen in Table 1. The results of the analysis of variance show that the extraction temperature has a significant effect on the weight equivalent of the pectin tongka langit banana peel.

The pectin extraction of tongka langit banana peels at 80 °C produces an equivalent weight greater than 85 °C and 90 °C. The results showed that the higher the extraction temperature, the smaller the equivalent weight value. This is due to the de-esterification process, namely the change in pectin to pectic acid, which can increase the number of free acid groups so that the amount of pectin equivalent weight will decrease. By the research of Aziz [13], the higher the extraction temperature, the lower the equal weight; this is due to many esterified compounds.

The tongka langit banana peel's pectin equivalent weight at 80 °C is 3104.3 mg, 85 °C is 1084.0 mg, and 90 °C is 699.8 mg. For pectin extracted with a temperature of 90 °C, which meets the quality standards of IPPA (International Pectin Producers Association) [14] because it is in the range between 600-800 mg.

| Pectin characteristics | Treatment extraction temperature (°C) | Quality standard IPPA (International Pectin Producers Association) |
|------------------------|----------------------------------------|---------------------------------------------------------------|
|                        | 80          | 85      | 90      |                                             |
| Equivalen Weight (mg)  | 3104.3 a    | 1084.0b | 699.8c  | 600-800 mg                                   |
| Methoxyl content (%)   | 3.25 a      | 3.87b   | 5.44c   | < 7% (low methoxyl)                         |
| Galacturonic Acid (%)  | 24.56 a     | 38.06b  | 56.08c  | Min 35%                                     |
### 3.2. Methoxyl content

The average methoxyl pectin content of tongka langit banana peels with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the methoxyl pectin content of the tongka langit banana peel. The pectin extraction of Tongka Langit banana peels at 90 °C resulted in a methoxyl content greater than 80 °C and 85 °C. The results showed that the higher the extraction temperature, the greater the methoxyl content. The increase in methoxyl levels is due to higher temperatures, which will cause a faster protopectin hydrolysis reaction and result in breaking the bonds of the pectin galacturonic component with other compounds, so that there will be an increase in carboxyl groups which will react with alcohol and will produce esters. According to the research results of Triandini\[15\], the increasing extraction temperature will cause higher methoxyl pectin levels due to the rising esterified free carboxyl groups.

The methoxyl pectin content of tongka langit banana peels at 80 °C was 3.25%, 85 °C was 3.87%, and 90 °C was 5.44%. Based on the quality standards of IPPA [14], the banana peel pectin of tongka langit is less than 7%, classified as low methoxyl pectin. According to Goycoolea and Adriana\[16\], pectin is high methoxyl if the methoxyl content is equal to or more than 7%, and low methoxyl if the methoxyl content is less than 7%.

### 3.3. Galacturonic acid

The mean of galacturonic acid pectin levels of tongka langit banana peels with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the galacturonic acid levels of the pectin peels of Tongka Langit banana peels.

The pectin extraction of tongka langit banana peels at 90 °C resulted in a higher galacturonic acid level than 80 °C and 85 °C. The results showed that the higher the extraction temperature, the higher the galacturonic acid levels. High temperature will cause protopectin's hydrolysis reaction to pectin, which is the basic component of D-galacturonic acid. Galacturonic levels can affect the structure and texture of the gel that is formed. According to Desmawarni & Hamzah [17], the higher the temperature causes the hydrogen ions produced to substitute calcium and magnesium from the more protopectin, the hydrolyzed protopectin produces more pectin.

Galacturonic acid levels of pectin of tongka langit banana peel at 80 °C were 24.56%, 85 °C temperatures were 38.06%, and 90 °C was 56.08%. For the extracted pectin with temperatures of 85 °C and 90°C which meet the quality standards of IPPA [14] because it has a galacturonic acid content of more than 35%.

### 3.4. Degree of esterification

The average degree of esterification of the tongka langit banana peel pectin with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the degree of esterification of the tongka langit banana peel pectin.

The pectin extraction of Tongka Langit banana peels at a temperature of 80 °C resulted in a greater degree of esterification than 85 °C and 90 °C. The higher the extraction temperature, the higher the degree of esterification. This is due to the presence of a glycosidic bond of the methyl ester group of pectin, which tends to be hydrolyzed to produce galacturonic acid. According to Budiyanto & Yulianingsih\[18\], the degradation of the methyl ester group in pectin becomes carboxyl acid in the

| Esterifikasi | Degree |
|-------------|--------|
| (%)         |        |
| Moisture content (%)  | 11.39 a | 10.79b | 9.85c | Max 12% |
| Ash content (%)        | 4.42 a  | 5.11b  | 6.47c | Max 10% |
| Yield (%)             | 8.37 a  | 8.80b  | 9.20c |

Numbers followed by different letters at the same column are significantly different at 5% of Tukey.
presence of acid. The glycosidic bonds of the methyl ester group of pectin tend to be hydrolyzed to produce galacturonic acid.

The degree of esterification of tongka langit banana peel pectin at $80^\circ C$ was 76.02%, $85^\circ C$ was 57.45%, and $90^\circ C$ was 55.07%. Based on the IPPA [14] quality standard, the banana peel pectin produced in this study belongs to the high ester pectin class because it has an esterification degree of more than 50%.

3.5. Moisture content

The average moisture content of the tongka langit banana peel pectin with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the moisture content of the Tongka Langit banana peel pectin.

The pectin extraction of tongka langit banana peels at a temperature of $80^\circ C$ resulted in a greater moisture content than temperatures of $85^\circ C$ and $90^\circ C$. The results showed that the higher the temperature, the lower the moisture content. This decrease in moisture content is due to the high temperature, facilitating water evaporation to speed up drying. The results of Roikah [19] showed that the water content of the extracted pectin at a temperature of $60^\circ C$ was higher than the extraction temperature of $100^\circ C$. High extraction temperatures can hydrolyze the pectin polymer so that the shorter molecular chains will dry out more quickly.

The tongka langit banana peel pectin's water content at an extraction temperature of $80^\circ C$ was 11.39%, a temperature of $85^\circ C$ was 10.79%, and a temperature of $90^\circ C$ was 9.85%. Based on the IPPA [14] quality standard, pectin’s maximum moisture content is 10%, so the moisture content of the banana peel pectin from this study meets the quality standard.

3.6. Ash content

The mean ash content of the tongka langit banana peel pectin with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the ash content of the tongka langit banana peel pectin.

The pectin extraction of tongka langit banana peels at $90^\circ C$ resulted in a more significant ash content than $80^\circ C$ and $95^\circ C$. The higher the extraction temperature, the higher the ash content. The increase in ash content is due to a heating process, which can result in a protopectin hydrolysis reaction, which will cause an increase in Mg and Ca components in the extract solution so that more magnesium and calcium minerals will increase the ash content [20].

The ash content of the tongka langit banana peel pectin at an extraction temperature of $80^\circ C$ was 4.42%, a temperature of $85^\circ C$ was 5.11%, and a temperature of $90^\circ C$ was 6.47%. Based on the IPPA [14] quality standard, the maximum pectin ash content is 12%, so that the ash content of the banana peel pectin from this study meets the quality standard.

3.7. Yield

The average yield of tongka langit banana peel pectin with temperature treatment can be seen in Table 1. The results of the analysis of variance showed that the extraction temperature had a significant effect on the yield of tongka langit banana peel pectin.

The pectin extraction of tongka langit banana peels at $90^\circ C$ resulted in a higher yield than $80^\circ C$ and $85^\circ C$. The higher the extraction temperature, the yield will increase. This is because the higher the extraction temperature, the hydrolysis of protopectin to pectin will increase. Protopectin that still binds to cellulose is released and converted into water-soluble pectin by hydrolysis. According to Aziz [13], the higher the temperature caused, the higher the yield because the hydrogen ions produced will substitute more calcium and magnesium from protopectin. The hydrolyzed protopectin produces more pectin.

4. Conclusion

Based on the results of the study, it was concluded that the pectin characteristics of the tongka langit banana peel based on extraction temperatures of $80^\circ C$, $85^\circ C$, and $90^\circ C$ resulted in equivalent weight
(3104.3, 1084.0, and 699.8 mg), methoxyl content (3.25, 3.87, and 5.44%), galacturonic acid (24.56, 38.06 and 56.08%), degree of esterification (76.02, 57.45 and 55.05%), moisture content (11.39, 10.79 and 9.85%), ash content (4.42, 5.11 and 6.47%) and yield (8.37, 8.80 and 9.20%).

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