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Pectin Extraction from Kepok Banana Peel (*Musa acuminata* × *balbisiana*); Case Study on Procedure of Pre-Treatment Process

Ekstraksi Pektin dari Kulit Pisang Kepok (Musa acuminata x balbisiana): Studi Kasus pada Prosedur Proses Pra-Perlakuan

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ABSTRAK

Ekstraksi pektin dari kulit pisang merupakan salah satu cara meningkatkan nilai guna dari limbah kulit pisang. Metode ekstraksi yang diaplikasikan adalah ektraksi padat-cair menggunakan pelarut asam. Tujuan penelitian ini yakni untuk mengetahui pengaruh praperlakuan yakni jenis dan konsentrasi pelarut serta waktu dan konsentrasi substrat yang optimum terhadap rendemen yang dihasilkan. Penelitian ini dilakukan secara eksperimental dengan metode OFAT (*One Factor at The Time*). Hasil penelitian menunjukkan pelarut terbaik adalah asam sulfat 0,5N dengan konsentrasi substrat 10% selama 80 menit menghasilkan rendemen sebesar 10,02%.

Kata Kunci:

ekstraksi;

limbah kulit pisang;

pektin

ABSTRACT

Keywords: extraction; banana peel waste; pectin The extraction of pectin from banana peels is one way to increase the use value of banana peel waste. The extraction method applied is solid-liquid extraction using an acid solvent. The purpose of this study was to determine the effect of pre-treatment, the type and concentration of solvent as well as the optimum time and substrate concentration, on the resulting yield. This research was conducted experimentally with the OFAT (One Factor at a Time) method. The results showed that the best solvent was 0.5N sulfuric acid with a substrate concentration of 10% for 80 minutes, resulting yield of 10.02%.

INTRODUCTION

In meeting national pectin needs, Indonesia still imports 455,035 kg of pectin and spends US\$6,635,609 in 2021(Badan Pusat Statistik, 2021). Meanwhile, the need for pectin in Indonesia is increasing. This is evidenced by the increasing quantity of pectin imports. Pectin demand increases by 10-15% every year (Astuti et al., 2022). Imports have a negative and significant effect on economic growth (Hodijah & Angelina, 2021) because it will reduce the country's foreign exchange.

Pectin extraction is one way to produce pectin by separating pectin from natural sources such as fruit peels, seeds or agricultural waste (Husni et al., 2021). Pectin has the ability to form gels when exposed to acidic environments and is widely used in food, pharmaceutical, cosmetic and other industries as a thickener, stabilizer and emulsifier (Sri et al., 2016). Pectin is a polysaccharide found in plant tissues, especially cell walls. Among the producers of pectin is Cavendish banana weevil waste with a yield of 3.11-4.57% (Andini et al., 2022) and kepok banana peel with a yield ranged from 5.79% to 22.57% (Pagarra et al., 2019).

In general, the utilization of banana peel waste is quite low, because most banana peelers use their waste for animal feed or dispose of it directly as biowaste. According to data from the Central Statistics Agency, Indonesia produces around 2,048,948 tons of bananas per year 2021 and 1,231,218 quintals come from Lumajang district. In line with this fact, the probability of banana peel waste will also be higher, because the number of banana peels is quite a lot, which is about 1/3 of whole bananas that have not been peeled (Lukankubo, 2007: Palupi et al., 2020).

The process of extracting pectin from banana peel waste involves several stages including selecting the appropriate pectin source, shrinking the size of the raw material, extracting pectin with solvents such as acids, filtration to separate solids and liquids, precipitation and drying of pectin and finally crushed into powder (Kamal et al., 2023). Some of the factors that affect pectin extraction are the type of acid (Koubala et al., 2008) and solvent concentration (Astuti et al.. 2022): substrate concentration (Siddiqui et al., 2021); Extraction time (Kamal et al., 2023). Therefore, in this study, pectin extraction from banana peels will be carried out with various factors to determine various approaches to increase pectin capacity.

METHODOLOGY

Materials

The materials used in this study include banana peel waste from CV. Strudel Lumajang, citric acid (C₆H₈O₇), acetic acid (CH₃COOH), and oxalic acid (C₂H₂O₄) ex china, 96% ethanol (technical), aquades, sulfuric acid (H₂SO₄) by smartlab, hydrochloric acid (HCl), NaOH and Phenolphthalein indicators by Merck.

Sample Preparation

Banana peels are thoroughly washed to remove adhering dirt. After draining, the banana peels were dried in the oven until they reach the moisture content $\pm 10\%$ in Wet basis (Wb). In order to reduce the size of dried banana peels, grind them using a blender. Banana peel powder will be used as a substrate to be extracted.

Design of Experiments

This study used the One Factor At the Time (OFAT) method. This optimization design is used to find out the best value of the variable that is varied where other variables that are considered influential are assumed to be fixed. In this study there are four variables that will be varied alternately. The optimum result of each variable will be set as a fixed value for the next running. The design used in this study can be seen in Table 1.

Run count	Solvent	Solvent (N)	Time (m)	Substrate (%)
1	H_2SO_4	0,2	70	5
2	HC1	0,2	70	5
3	$C_6H_8O_7$	0,2	70	5
4	$C_2H_2O_4$	0,2	70	5
5	CH ₃ COOH	0,2	70	5
6	X1	0,1	70	5
7	X1	0,3	70	5
8	X1	0,5	70	5
9	X1	0,7	70	5
10	X1	X2	60	5
11	X1	X2	80	5
12	X1	X2	100	5
13	X1	X2	X3	7,5
14	X1	X2	X3	10
15	X1	X 2	X3	12.5

Table 1. Design One Factor at The Time

X1 : Solvent with optimum response

X2 : Molarity of solvent with optimum response

X3 : extraction time with optimum response

Extraction of Pectin

The extraction method applied was solid-liquid extraction using acid solvents. The substrate was extracted according to the DoE that has had been designed, in a waterbath with a temperature of 85°C. Filtration was done using filter paper and vacuum pump. The filtration result consists of two phases. The filtrate was further processed and the residue was removed. 96% ethanol was added to the filtrate with a ratio of 1:1 then precipitated for 18 hours. Filter with filter paper. The filtrate was removed and the residue was further processed. The residue was dried in an oven at 60°C for 4 hours. Dry residue was grounded using a mortar to produce pectin powder._The entire research flowchart can be seen in the Figure 1.



Figure 1. Overall flow chart of pectin extraction

Qualitative test

The pectin solution was prepared (0.05 grams in 5 ml of water) into a test tube contain 5 ml of 95% alcohol solution and vortex for 2 minutes. Will be after 30 minutes the precipitate forms. The precipitate was added by 5 mL of 10% sugar solution to the test tube. Vortex for 2 minutes and leave for 30 minutes until a gel forms. If a gel is formed, the test sample contains pectin (Laemmli, 1970).

Yield Analysis

The yield value was analyzed by weighing the dried pectin produced and



Figure 2. Effect of acid type on pectin yield



Figure 4. Effect of time extraction on pectin yield

The solvents used are acids of different types. Sulfuric acid (H₂SO₄) and hydrochloric acid (HCl) are strong acids while citric acid (C₆H₈O₇), acetic acid (CH₃COOH), oxalic acid (C₂H₂O₄) are weak acids. The main difference between the bond strengths in a strong acid and a weak acid lies in the degree to which the

then dividing by raw material weight (DB). The yield calculation is as follows:

yield =
$$\frac{\text{Weight of pectin}}{\text{Weight substrat of banana peel powder}} x100\%$$

RESULTS AND DISCUSSION

Pectin is obtained from banana peel waste tissue by extraction process using a variety of types of acid solvents. The effect of variations in the research variables on the yield is described in the graph in figure 2-5.



Figure 3. Effect of Solvent Concentration on pectin yield



Figure 5. Effect of substrate concentration on pectin yield

acid can release hydrogen ions (H^+) in solution. Strong acids have strong hydrogen bonds and are relatively easy to release hydrogen ions (H^+) and counter ions in solution compared to weak acids. This difference results in different acidity properties between strong acids and weak acids. Strong acids tend to have higher acidity and stronger reactivity due to their ability to completely release hydrogen ions. Meanwhile, weak acids have lower acidity and lower reactivity because only part of the acid molecules dissociates in solution (Brown, 2012).

This is compatible with the research results contained in Figure 2. It is known that the solvent with the highest yield is H₂SO₄ compared to other solvents, which is 6.48%. Sulfuric acid has more hydrogen which function to hydrolyze ions protopectin into soluble pectin so that the vield of pectin produced will be higher. (Kesuma et al., 2018). According to (Jong 2023) in acidic conditions, et al.. protopectin tends to hydrolyze into soluble pectinic acid or pectin. The process of dissolving pectin into pectinic acid can occur due to the substitution of protopectin divalent ions into hydrogen ions or due to breaking the bond between pectinic acid and cellulose. So that H₂SO₄ is designated as X_1 (Solvent with optimum response) which will be used for the next run.

The concentration of the solvent used varies from 0.1N; 0.3N; 0.5N; and 0.7N. The higher the solvent concentration, the more pectin content is produced. High solvent concentration will increase the release of protopectin from banana peels so that the yield obtained is even greater (Fakhrizal et al., 2015). In addition, from the figure 3 can also be seen that pectin is produced the most at a solvent concentration of 0.5 N (7,4% yield) which is due to more release of protopectin into pectin than solvents in other normality variations. The acid in the solvent reacts with the ester bonds in the protopectin, depolymerization resulting in and deesterification of the pectin. The acid breaks the methoxyl ester bonds of pectin to produce polygalacturonic acid which dissolves in water (Rodríguez Robledo & Isabel Castro Vázquez, 2020). However, if the concentration of solvent used is too high, pectin will continue to degrade into pectic acid so that the yield obtained will also decrease (Tuhuloula et al., 2013). A solvent concentration of 0.5N is designated as X_2 (solvent concentration with maximum response) and will be used in all subsequent treatments.

The time used varies from 60 minutes, 80 minutes and 100 minutes. The effect of solution concentration on the yield is shown in Figure 3. From Figure 4 it can be seen that the effect of time and substrate concentration is directly proportional to the yield produced. This is because the opportunities for contact with the solvent will be greater so that the yield of pectin produced will be greater, but if the reaction has reached a maximum, the reaction will run out of the substrate so that the yield produced will decrease (Winata & Yunianta, 2015). Extraction for 80 minutes produces optimum yield (7,5%) so that it is used as the value of X_3 (extraction time with optimum response) which will be used to find the value of substrate concentration in the optimum extraction process.

Substrate concentration varied from 5%; 7.5%; 10%; and 12.5% of the total solvent volume. The effect of substrate concentration on the yield response can be seen in Figure 5. The optimum yield was obtained with a substrate concentration of 10% with a yield value of 10.02%. It can be seen that the effect of substrate concentration is directly proportional to the yield produced. The higher the substrate concentration, the higher the yield due to the principles of diffusion and saturation. In the diffusion process, the solvent will penetrate into the substrate. The pectin contained in the banana peel will dissolve into the solution due to differences in concentration (Prayudo et al., 2015). When the concentration of substrate containing pectin is higher, there will be more pectin available to diffuse until saturation point is reached. When the saturation point has been reached, no more reactions occur.

Based on the results of qualitative tests, the product produced in this study is pectin. This can be proven by the formation of precipitates with the addition of 96% ethanol (Daniarsari & Hidajati, 2005). Pectin will precipitate if alcohol is added. This is in accordance with the nature of pectin which is insoluble in organic solvents such as alcohol. Alcohol acts as a dehydrating agent which can disrupt the stability of the colloid causing clots to form. In addition, alcohol can break down pectin bonds with unwanted compounds (Fauzan et al., 2022).

CONCLUSION

Based on the results of the study, it can be concluded that pectin extraction from kepok banana peel (Musa acuminata \times balbisiana) for the best results uses 0.5N sulfuric acid solvent with process conditions of 10% substrate concentration and 80 minutes time, resulting in an average yield of 10,02%.

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