

Interaction of Temperature and Extraction Time on the Rendement and Some Properties of Lime Pectin (*Citrus Aurantifolia* S)

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Received: 2023-02-13

Accepted: 2023-04-13

Publication: 2023-05-04

Abstract

This study aims to determine the right temperature and extraction time to get the best yield and quality of pectin from lime peel. It is hoped that the results of this study can become a reference for studying and utilizing lime peels as a pectin producer. This research was carried out at the Food Technology Laboratory, Faculty of Agriculture, University of Sam Ratulangi. This study used a completely randomized design (RAL) with 2 factors, namely extraction temperature (60, 70, and 900C) and extraction time (40, 60, 80, and 100 minutes) with 3 replications. The results showed that the yield of orange peel pectin lime ranged from 3.1826% to 5.2211% wet weight. The highest yield of pectin was produced and the extraction temperature was treated at 900C for 100 minutes. The methoxy content ranges from 7.1706% to 8.3534% so that it is classified as a high methoxyl content pectin, the pectin water content ranges from 9.734% to 13.7703% while the ash content ranges from 1.8009% to 2.5339%. From these results it was concluded that the best conditions for extracting lime peel pectin were at 900C with an extraction time of 100 minutes and lime peels could be used for commercial processing of pectin.

Keywords— Lime, Pectin Extraction Time

Introduction

Citrus fruit is a fruit that is very popular and very familiar to our society because it is often used in everyday life, both as a table fruit that can be eaten directly and as an ingredient to add aroma and taste to food or as a fresh drink. One type of citrus that cannot be eaten directly is lime (*Citrus aurantifolia* Swingle). In use, the part that is taken is only the flesh of the fruit while the skin is generally discarded. Orange peel biagan 30% of the total weight of the fruit and this part can still be used as a pectin-producing material.

Pectin is widely used in food products as an emulsifier, texture stabilizer, as well as a food additive that is permitted for use in Indonesia. The use of pectin is often found in the jam, jelly, and so on industry. Apart from the food industry, pectin is also used in the pharmaceutical and cosmetic industries. In early research it was found that the most suitable pH for pectin extraction from lime peel was pH 2, while the temperature and extraction time were unknown. The higher temperature will be accelerating the protopectin hydrolysis process from orange peel tissue in water so that more pectin is obtained (Yoakhim, 2021). Using the right temperature and extraction time can maximize pectin production and produce high quality pectin products. Basically, a high extraction temperature with a longer extraction time can increase the recovery of pectin. High water content is caused by low temperatures being unable to evaporate water in pectin, on the other hand the higher the temperature, the more evaporation of the amount of water during the extraction process, thus facilitating the drying process (Ahmad, 2015). However, if the temperature and extraction time used are excessive, it will cause a decrease in the quality of the

resulting dry pectin product, namely a decrease in the ability to form gel from pectin. Based on the facts above, a study was conducted to obtain the most effective temperature and extraction time in producing pectin from lime peel with good quality.

Literature Review

This study aims to determine the right temperature and extraction time so as to obtain the best yield and quality of pectin from lime peel. To study and utilize lime peel as a pectin producer. It is suspected that differences in temperature and extraction time can affect the yield, methoxyl content, moisture content and ash content of lime peel pectin. This research was conducted at the Food Technology Laboratory, Faculty of Agriculture, University of Sam Ratulangi. The tools used in this study were scales, stainless steel knives, blenders, containers, measuring cups, beakers, measuring pipettes, dropping pipettes, petri dishes, mortar, ovens, pH meters, temperature gauges, timers, volumetric flasks, baths, cloth filters, desiccators and tools for analysis.

The materials used are lime peels of harvest age (ripe green) collected from consumers in Manado City and chemical substances such as distilled water, absolute HCl, absolute ethanol, silver nitrate solution and substances used to analyze the pectin produced.

Research Method

This research was conducted using the factorial method in a completely randomized design (CRD) consisting of two factors with three replications. Factor A was the extraction temperature treatment with 4 levels:

A1 : temperature 60°C

A2 : temperature 70°C

A3 : temperature 80°C

A4 : temperature 90°C

Factor B is the long extraction treatment with 4 levels:

B1 : 40 minutes

Long B2 60 Minutes

Long B3 80 Minutes

Long B4 100 minutes long

Observed Things

The things observed in this study were the yield of pectin and the properties of the pectin itself including methoxyl content, moisture content and ash content.

Cure Procedure

The work procedure used in this study is a modification of the work procedure. Orange peel washed with water then weighed as much as 50 gr. added with 250 ml of distilled water and then crushed with a blender. The mixture is then acidified with hydrochloric acid until it reaches a pH of 2. Extraction was carried out at 60°C, 70°C, 80°C and 90°C with extraction times of 40, 60, 80 and 100 minutes.

The extraction results are filtered through a filter cloth, and the residue is squeezed out until as much liquid extract as possible is obtained. The extract liquid is then filtered again through

another filter cloth. Before settling, the liquid extract is introduced by heating it over an electric heater to 1/2 its original volume. Precipitation is carried out with 95% ethanol containing 2 ml of concentrated hydrochloric acid per liter.

Ethanol was added little by little to the liquid extract until it used up 1.5 times the volume of the liquid extract. Then precipitate for 12 hours without being disturbed. Furthermore, the pectin precipitate is separated using a filter cloth and washed with 95% ethanol to neutralize the hydrochloric acid that is still left behind. there is still silver chloride, then washing still needs to be done.

Results and Discussion

Yield

In this study, a combination of temperature and extraction time was sought (temperature 60°C, 70°C, 80°C, 90°C and 40, 60, 80, and 100 minutes at pH 2) to obtain the highest yield. From the pectin precipitate is then dried in an oven at 37°C -40°C for 16 minutes. The dried pectin is then finely ground. The pectin product is then put in a small bottle.

a. Methoxyl content

Methoxyl content was measured to determine the type of pectin (HMP or LMP type).

b. Water content

Measurement of water content was carried out to evaluate the pectin drying process

c. Ash content

Measurement of ash content was carried out to calculate the purity of the pectin produced and to evaluate the pectin washing process.

The research results obtained that the highest average yield was 5.22% (temperature 90°C and 100 minutes long) and the lowest was 3.18% (70°C temperature and 40 minutes long). Figure 8 shows the yield curve of dry pectin lime peel from different temperatures and extraction times.

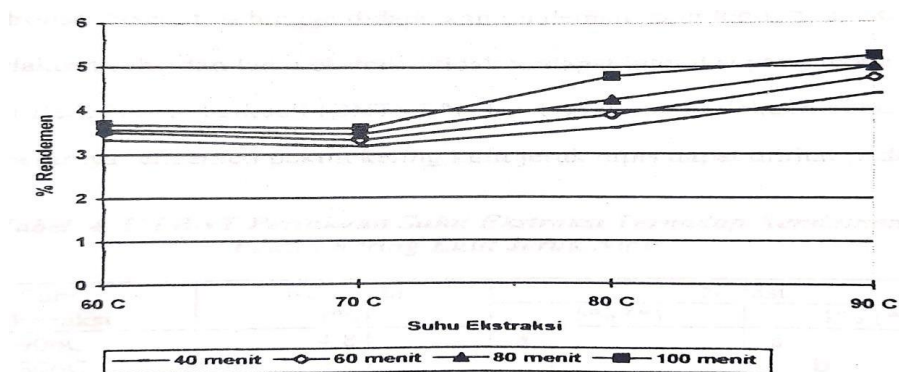


Figure 8. Yield of Lime Peel Pectin from Different Temperatures and Extraction Time

The results of the analysis of the yield variance of dry pectin lime peel showed that the temperature of the extraction had a very significant effect. Increasing the extraction temperature from 60°C to 90°C was followed by a trend of increasing pectin yields. Besides that, the extraction time also had a very significant effect on the dry pectin yield of lime peel. Increased extraction time from 40 minutes to 100 minutes also followed by an increase in the yield of pectin produced. The Least Significant Difference (LSD) test of 5% and 1% for the extraction temperature treatment for the yield of dry lime peel pectin can be seen in table 4.

Table 4. BNT Test of Extraction Temperature Treatment on Dried Pectin Yield of Lime Peel

Suhu Ekstraksi	Rata-rata (%)	Notasi	
		5% (*)	1% (**)
90oC	4,84	a	a
80oC	4,13	b	b
60oC	3,52	c	c
70oC	3,39	d	c

(*) 0.200730547 (**) 0.270327623

From the results of the BNT test for the extraction temperature treatment of the dry pectin yield of lime peel, it showed that the 90°C extraction temperature treatment was greater and significantly different from the dry pectin yield obtained from the 60°C, 70°C and 80°C extraction temperatures. Likewise, there is a very significant difference between the yield of pectin and the temperature treatment 80°C with the yield of pectin from 60°C and 70°C, while between 60°C and 70°C there was no significant difference. This is because at an extraction temperature of 90°C the effectiveness of HCl in hydrolyzing protopectin which is not soluble in water to pectin which is soluble in water reaches the highest effectiveness, namely by decreasing the viscosity of the media as the extraction temperature increases so as to facilitate the diffusion of HCl solvent in the lime peel tissue so that the hydrolysis process runs faster and can produce more pectin yields. Likewise, the 800C temperature treatment is still more effective than the 60°C and 70°C temperature treatments for the same reason. Meanwhile, the yield of pectin from the 600C and 700C temperatures did not show a very significant difference, because it only met the 5% BNT test. Both of them produce the smallest yield of pectin.

The results of this study indicate that the extraction time greatly affects the yield of dry pectin from lime peel. The BNT test of the long extraction treatment on the dry pectin yield of lime peel is presented in table 5.

Table 5. BNT Test of Old Extraction Treatment on Dried Pectin Yields of Lime Peel

Lama Ekstraksi	Rata-rata (%)	Notasi	
		5% (*)	1% (**)
100 menit	4,32	a	a
80 menit	4,07	b	b
60 menit	3,87	c	c
40 menit	3,62	d	d

(*) 0.200730547 (**) 0.270327623

From the results of the BNT test, the extraction time for the dry pectin yield of lime peel showed that the 100-minute extraction time was very significantly different from the 80-minute, 60-minute, and 40-minute treatment, as well as the 80-minute extraction time, which was very significantly different from the 60-minute treatment minutes and 40 minutes, and also the 60 minutes treatment was very significantly different from the 40 minutes extraction treatment.

From this study, the yield of lime peel pectin ranged from 3.18 (at 70°C and 40 minutes) to 5.23 % wet weight (at 90°C and 100 minutes). These results indicate that the pectin content of siem oranges, arrowroot oranges, sweet oranges and grapefruits ranged from 3.10 - 4.78%. However, the results of this study cannot be compared because the type of orange peel used is not the same.

Methoxyl Levels

Methoxyl levels have an important role in the ability to form a pectin jelly. Pectin with Low methoxyl content has less gel forming ability, while pectin with high methoxyl content has high gel forming power. From the results of this study the dry methoxyl pectin content of orange peels ranged from 7.17% - 8.35%.

Figure 9 shows the curve of dry methoxyl pectin content of lime peel from different temperatures and extraction times.

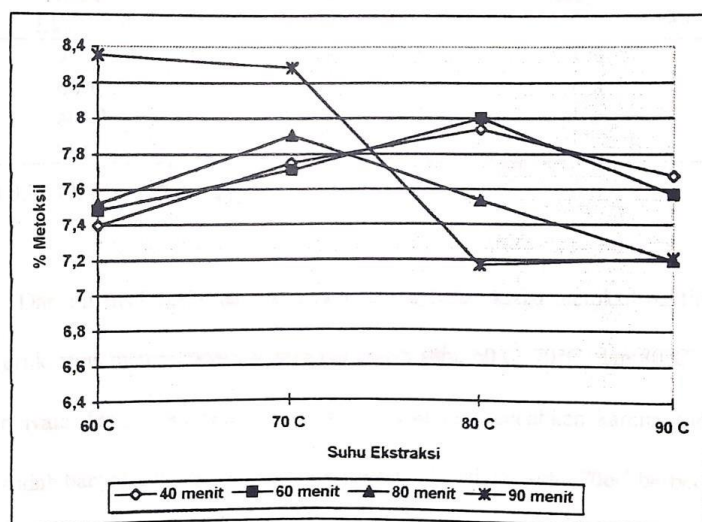


Figure 9. Methoxyl Pectin Levels of Dried Lime Peel from Different Temperatures and Extraction Time.

The results of the analysis of variance for the dry methoxyl pectin content of lime peels showed that the extraction temperature treatment had a very significant effect on the methoxyl pectin content of the peels. BNT test for temperature treatment of extraction of methoxyl pectin content lime, besides that there is a significant interaction between temperature and extraction time on lime peel methoxyl pectin content, while the extraction time does not have a significant effect.

Dry lime peel can be seen in the table

Suhu Ekstraksi	Rata-rata (%)	Notasi	
		5 % (*)	1 % (**)
70°C	7,91	a	a
60°C	7,69	a	a
80°C	7,66	b	a
90°C	7,41	c	b

(*) 0,22393219 (**) 0,301573713

From the BNT test, the extraction temperature treatment on the dry methoxyl pectin content of lime peel showed that the 60°C, 70°C, and 80°C temperature treatments were very significantly different from the 90°C temperature treatments. This is due to the temperatura 900C has a lot of deesterification processes going on. The 70°C temperature treatment was significantly different from the 80 0C treatment, while the 60°C and 700C temperatures were not significantly different. This causes a decrease in the content of methyl ester or methoxyl groups at 90°C faster than at 60°C, 70°C and 80°C. Thus the methoxyl content at temperatura 90°C it becomes lower than other temperature treatments. The BNT test for the interaction between temperature and extraction time on dry lime methoxyl pectin content can be seen in table 9.

Table 9. Test of BNT interaction, temperature and extraction time on methoxyl pectin content of

dried lime

Suhu Ekstraksi	Lama Ekstraksi	Rata-rata (%)	Notasi	
			5% (*)	1% (**)
60°C	100 menit	8,35	a	a
70°C	100 menit	8,28	a	a
80°C	60 menit	8,00	a	a
80°C	40 menit	7,92	a	a
70°C	80 menit	7,90	b	a
70°C	40 menit	7,75	c	b
70°C	60 menit	7,71	c	b
90°C	40 menit	7,67	c	c
90°C	60 menit	7,57	c	c
80°C	80 menit	7,54	d	c
60°C	80 menit	7,52	d	c
60°C	60 menit	7,49	d	c
60°C	40 menit	7,40	e	c
90°C	100 menit	7,21	f	d
90°C	80 menit	7,20	f	d
80°C	100 menit	7,17	f	d

(*) 0,447864379 (**) 0,603147426

The results of the LSD test on the interaction of temperature treatment and extraction time on dry methoxyl pectin content of lime peel showed that there was no significant difference between the 600C and 700C temperature treatments with an extraction time of 100 minutes, as well as there was no significant difference between the 700C, 80°C temperatures. and 90°C at extraction times of 40 minutes and 60 minutes. Treatments at 80°C and 90°C with extraction times of 80 and 100 minutes also showed no significant difference.

The temperature treatment of 80°C at an extraction time of 100 minutes produced lowest methoxyl content. This can happen because at high temperatures, the hydrolysis process of protopectin into pectin runs fast so that with a not too long extraction time the protopectin will be completely hydrolyzed. Thus the remaining time will be used to hydrolyze the methyl ester group so that the resulting methoxyl pectin content decreases.

Water content

The highest average yield of dry pectin water content of lime peel was 13.7703% (temperature 800C and 40 minutes long) and the lowest was 9.7345% (temperatura 600C and 100 minutes long). The moisture content of dry lime peel pectin in this study met the requirements for dry pectin moisture content, which ranged from 7 - 14%. In storage, the moisture content of the pectin obtained must always be maintained to avoid excessive moisture due to the hygroscopic nature of pectin.

Ash Content

In a food product in the form of flour or powder, the ash content becomes more important, especially to determine the purity of the product.

The highest average yield of dry pectin ash content of lime peel was 2.5339% (temperature 700C and 80 minutes long) and the lowest ash content was 1.8009% (temperature 900C and 40 minutes long). The ash content produced in this study has met the quality requirements for pectin ash content, which is below 10%.

Conclusion

The yield of dry pectin obtained from lime peels ranges from 3.18-5.22%. Pectin obtained from lime peel has the following characteristics:

- Methoxyl content : 7.17- 8.35%

- b. Moisture content : 9.73-13.77%
- c. Ash content : 1.80-2.53%

The properties of pectin, namely methoxyl content of more than 7%, moisture content between 7-14% and ash content below 10% meet the requirements. The most suitable temperature for pectin extraction from lime peel is 90°C and the most suitable extraction time is 100 minutes. Lime peel can be used as a source in the dry pectin manufacturing industry.

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