

# Microwave Power and pH Regulating Solution Effect on Characteristics of Pectin from Sukun Peel (*Artocarpus altilis*) using Microwave Assisted Extraction (MAE)

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## ABSTRACT

Pectin, a biopolymer which is a natural additive, is widely used in various countries. In the food industries, it is used as a basis for functional food, including fat replacer. In Indonesia, however, pectin is still imported. Previous studies reported that pectin can be obtained through the extraction of sukun peel (*Artocarpus altilis*) using reflux for about 180 minutes. In this study, pectin extraction was carried out using Microwave Assisted Extraction (MAE) because it has a shorter time advantage. This study aims to determine the effect of various power from the microwave on the yield of pectin from sukun peel and the effect of the type of pH regulating solution on the extraction process on the characteristics of extracted pectin. The characterization carried out was the determination of water content, equivalent weight, methoxyl content, galacturonic content and degree of esterification. The results of the pectin extraction using MAE at pH 1.5 for 3 minutes showed that the use of 450 Watt microwave power and sulfuric acid pH regulating solution were the best method because it produced the highest yield of pectin of 4.30% and 1.91% respectively. The characterization results showed that pectin had a moisture content of 0.014%, equivalent weight using sulfuric acid and citric acid were 4471.224 and 1595.793, respectively. Methoxyl content using sulfuric acid and citric acid was 12.60% and 8.78%, respectively. Galacturonic content using sulfuric acid and citric acid was 76.08% and 60.89%, respectively. The degree of esterification using sulfuric acid and citric acid was 94.82% and 81.87, respectively. Molecular weight using sulfuric acid and citric acid were 4471.242 and 1595.793, respectively. Based on FT-IR analysis, both extracted pectins obtained from the results of this study have a functional group in the same wavenumber range as the functional group in the standard pectin. Based on the above, pectin from sukun peel has met the specifications based on International Pectin Producers Association, thus can be used both in the food and pharmaceutical industries.

**Keywords:** pectin, sukun peel, MAE, Characterization, pH regulating solution

## 1. INTRODUCTION

Pectin, a biopolymer, is a natural additive. In food industries, it is used as a basis for functional food. Some researchers report that sukun peel contains about 32-49% pectin [1].

Pectin is a polygalacturonic acid with  $\alpha$ - (1,4) glycosidic linkage which forms most of methyl esters. In water the pectin would dissolve almost completely, forming a viscous liquid, acidic and practically insoluble in organic solvents [2][3]. In general, pectin is classified based on the amount of galacturonic acid, methoxyl content, and the degree of esterification. These three parameters will affect the role of pectin in the industries both in food and pharmaceutical. Pectins

can be classified according to their Methoxyl content. The most important use of pectin is based on its ability to form gels. High Methoxyl- pectin (HM-P) forms gels with sugar and acid, whereas for Low Methoxyl – pectin (LM-P) they would form if there are calcium ions and a small amount of sugar [4].

Previous studies reported that pectin can be obtained through the extraction process of sukun peel using thereflux method for about 180 minutes [1]. In this study, pectin extraction was carried out using the MAE because it has several advantages, namely the extraction time used is relatively shorter so that the solvent and energy used are relatively low [5].

This study aims to determine the effect of various power (Watt) from the microwave on the yield of pectin from sukun peel and the effect of the type of pH regulating solution on the extraction process on the characteristics of pectin from sukun peel.

## **2. BACKGROUND**

The utilization of functional food has become a lifestyle for modern society. In addition to obtaining nutritional food, it is also low in calories and low in fat content. Indonesian local food has the potential to be developed into functional food, one of which is sukun (*Artocarpus altilis*). Sukun, one of the natural vegetable resources in Indonesia is used in the fields of food, pharmacy, and cosmetics, both for domestic and industrial needs.

The Ministry of Agriculture stated that sukun production in Indonesia continues to increase. West Java is the largest sukun production center, which is around 14,262 tons per year. However, there will also be an increase in its production of waste, namely the sukun peel which is the residual [6]. On one hand, the results of research from [1] reported that the sukun peel contains about 32-49% pectin.

Pectin, a complex carbohydrate which is a natural additive, is widely used in various countries. In food industries, it is used as a thickener, texture giver, emulsifier, stabilizer and fat replacer [7]. In the cosmetic and pharmaceutical industry, it is used as a natural texturizer, thickener and stabilizer, lotion and tonic [3].

In Indonesia, pectin needs are met with imports. The Central Bureau of Statistics stated that the amount of pectin imports in Indonesia from 2008 to 2012 reached 240.8 - 291.9 tons with a value of 2,977,479 US Dollars [4].

The results of the study [1] reported that pectin can be obtained by extracting the sukun peel by reflux in about 180 minutes.

In the last few years, researchers have begun to develop an extraction method using Microwave Assisted Extraction (MAE). The MAE has the advantage of 2-20 minutes shorter extraction time with higher yield [3][7][8].

## **3. METHODOLOGY**

### **3.1. Material and chemicals**

Sukun peel used comes from the building yard of the Politeknik Negeri Bandung, Chemical Engineering Department. The solvent used is distilled water. Variations of sulfuric acid and citric acid were used to regulate the pH of the solvent.

### **3.2. Microwave Assisted Extraction (MAE)**

A domestic microwave oven (SHARP type R-728(W)-IN/R-728(K)-IN/R-728(S)-IN maximum delivered the power of 800 W) was used to employ MAE with a wave frequency of 2450 MHz.

Five grams of dried sukun peel powder and 100 mL of aquadest were placed in the reaction flask and heated by microwave irradiation with 360, 450, and 540 W for 3 minutes and solution with pH of 1.5. From the microwave power variations above, pectin extract would be obtained and the results would be calculated in % yield. Microwave power with the highest extract % yield would be used for the next process to vary pH regulating solution, so that the best pH regulating solution could be obtained.

### **3.3. Characterization of Extracted Pectin**

This stage aims to classify the extracted pectin, a characterization which consisted of:

#### **3.3.1. Determination of Water Content Using Gravimetric Method with Oven Drying**

This method involves weighing a moist extracted pectin oven and drying it at 105°C for 24-48 h, reweighing, and calculating the mass of the lost water in the percentage of the mass of the dried extracted pectin.

#### **3.3.2. Determination of Equivalent Weight**

The equivalent weight of the pectin was determined by titrating a known weight of pectin against a standardized 0.1N NaOH solution to a faint pink endpoint. This titer is known as an initial titer (IR) or free acid titer.

#### **3.3.3. Determination of Methoxyl Content**

The methoxyl content was determined by saponification of extracted pectin and titrating the liberated carboxyl group against standardized 0.1N NaOH solution using phenol red as an indicator to a faint pink endpoint.

#### **3.3.4. Determination of Galacturonic Acid Content**

The Galacturonic acid content was determined from the milliequivalent of NaOH obtained from the determination of equivalent weight and methoxyl content.

**3.3.5. Determination of Esterification Degree**

The esterification degree was determined from the content of methoxyl and galacturonic acid content produced.

**3.4. Identification of Functional Group Using FT-IR**

This stage aims to identify the presence of a functional group in the extracted pectin based on its wave

number (cm<sup>-1</sup>), then compare it with the functional groups in standard pectin.

**4. RESULTS AND DISCUSSIONS**

The results of the pectin extraction from sukun peel using MAE at pH 1.5 for 3 minutes showed that the use of 450 Watt microwave power and sulfuric acid pH regulating solution were the best method because it produced the highest yield of pectin of 4.30% and 1.91% respectively as shown in Table 1 and 2 below.

**Table 1** The Effect of Various Power (Watt) on The Yield of Extracted Pectin

Sukun peel weight (g)	Power (W)	Solvent volume (ml)	Filtrate Volume (mL)	Pectin weight (g)	Yield (%)
5.0001	360	100	67.5	0.184	3.68
4.9998	450	100	64	0.215	4.30
5.0002	540	100	56	0.165	3.31

**Table 2** The Effect of Type of pH Regulating Solution on The Yield of Extracted Pectin

Solvent	Powder weight (g)	Power (W)	Solvent volume (mL)	Filtrate volume (mL)	Pectin weight (g)	Yield (%)
Sulfuric Acid	10.0001	450	200	152	0.1915	1.91
Citric acid	10.0007	450	200	150	0.1508	1.507

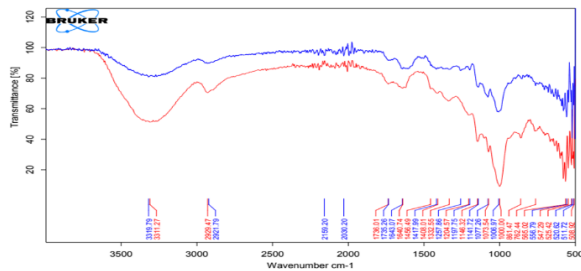
At 540 Watt, the yield of pectin decreased significantly. This happens because the microwave radiation is too high and will damage the active compounds present in the sukun peel. The higher the power, the heat generated will be also higher and it can cause hydrolyzed pectin polymers into shorter chains. The shorter the polymer chain the smaller the yield will be.

Sulfuric acid has a higher equilibrium constant (K) than citric acid. The greater value of K causes the increasing number of dissociating acids and the stronger acid to attract divalent ions that will be replaced with hydrogen ions. This replacement of hydrogen ion aims to hydrolyze protopectin into soluble pectin to produce a greater yield of pectin [9].

The characterization result of the extracted pectin from sukun peel using MAE at pH 1.5, 450 Watt for 3 minutes can be shown in the following Table 3.

**Table 3** The Characterization Result of The Extracted Pectin

Spesification	Extracted Pectin		Pectin Standard	
	Sulfuric Acid	Citric Acid	IPPA (International Pectin Producers Association)	Farmakope V Indonesia
Water Content (%)	0.14	0.67	Max. 12	≤ 10
Equivalent Weight	4471.242	1595.793	600-800 mg	-
Methoxyl Content (%)	12.60	8.78	High = > 7.12 Low = 2.5-7.12	≤ 6.7
Galacturonic Acid Content (%)	76.08	60.89	Min. 65	≤ 74.0
Esterification Degree (DE) (%)	94.82	81.87	High = Min. 50 Low = Max. 50	-
Molecular Weight (g/mol)	4471.242	1595.793	-	-



**Figure 1** FTIR spectra of pectin from sukun peel extraction using either citric acid or citric acid as a pH regulating solution

Based on the results of the characterization above, pectin from sukun peel has met the specifications based on IPPA.

Pectin from the sukun peel is included in the high methoxyl pectin (HM -pectin) because the methoxyl

content is  $\geq 7\%$ . In general, HM-pectin is thermally reversible, so it is widely

used both in the food and pharmaceutical industries as thickener, emulsifier, and stabilizer [3].

Based on the water content, the extracted pectin will have high stability in its storage because of the minimum number of insects and microorganisms [10][11].

The results of the identification of functional groups using the FT-IR showed that, pectin from sukun peel extraction

using either sulfuric acid or citric acid as a pH regulating solution produced the same pectin characteristics, as shown in Figure 1.

**Table 4** The Comparison of Functional Groups in Standard Pectin and in Extracted Pectin using Two Types of pH Regulating Solution

Annotation	Hydroxyl group (O-H stretching) (cm <sup>-1</sup> )	Methoxyl (O-CH <sub>3</sub> stretching) (cm <sup>-1</sup> )	Carboxylate ion stretching (COO <sup>-</sup> ) (cm <sup>-1</sup> )	Ester carbonyl (COO-R) (cm <sup>-1</sup> )	Cyclic (C-C) (cm <sup>-1</sup> )
Extracted Pectin using Citric Acid	3319.79	2921.79	1643.07	1735.26	1197.50
Extracted Pectin using Sulfuric Acid	3311.27	2929.47	1640.74	1736.01	1204.57
Standard Pectin	3446.17	2934.16	1698.02	1746.10	1200

The FT-IR spectra of the two extracted pectin did not differ significantly. The spectra of the two extracted pectin and standard pectin showed absorption of functional groups for methoxyl, ester carbonyl and carboxylate ion stretching. The three functional groups are peculiar to the functional groups in pectin as shown in Table 4.

Based on Figure 1, the examination of the spectral regions revealed the existence of two bands occurring at 1736.01–1735.26 cm<sup>-1</sup>, and at 1643.07–1640.74 cm<sup>-1</sup>, which were assigned to the ester carbonyl groups (COO- R) and the carboxylate ion stretching band (COO<sup>-</sup>), respectively.

The correlation between the intensity of ester carbonyl and carboxylate groups established the basis for the quantitative analysis of the degree of esterification (DE) of pectins by FTIR. In this study, the spectrum of extracted pectin using H<sub>2</sub>SO<sub>4</sub> has a higher absorption intensity than extracted pectin using citric acid, this corresponds to its DE value as shown in Table 3.

The broad area of absorption between 3500 and 2500 cm<sup>-1</sup> refers to O-H stretching absorption due to inter and intramolecular hydrogen bonding of the galacturonic acid backbone. The O-H stretching

vibrations occur within a broad range of frequencies and indicate several features of a compound in the extracted pectin [12]. Bands around 3000-2900 cm<sup>-1</sup> refer to C-H absorption. These include CH, CH<sub>2</sub> and CH<sub>3</sub> stretching and bending vibrations. In the case of esterified pectins, an O-CH<sub>3</sub> stretching band is

showed between 2929.47-2921.79 cm<sup>-1</sup> due to methyl esters of galacturonic acid. This is in accordance with Table 3 which showed that pectin from the sukun peel is included in the high methoxyl pectin (HM -pectin). The bands detected between 1100 and 1200 cm<sup>-1</sup> represent R-O-R ether bonds and C-C cyclic bonds present in the ring structure of extracted pectins. An increase in the intensity of these peaks was observed in two extracted pectins which could indicate the presence of more cyclic chains and the reduction of pectin ramifications [12][13].

Table 4 showed a slight difference in structure and molecular composition between standard pectin and extracted pectin that can cause a significant shift in the wavenumber of the absorption peaks in FT-IR. However, pectin obtained from the results of this study has a functional group in the same wavenumber range as the functional group in the standard pectin [14].

#### 4. CONCLUSION

The results of the pectin extraction from sukun peel using MAE at pH 1.5 for 3 minutes showed that the use of 450 Watt microwave power and sulfuric acid pH regulating solution were the best method because it produced the highest yield of pectin of 4.30% and 1.91% respectively.

Based on the results of the characterization, extracted pectin from sukun peel using MAE has met the specifications based on International Pectin Producers Association.

Pectin from the sukun peel is included in the high methoxyl pectin (HM –pectin) because the methoxyl content is  $\geq 7\%$ .

Pectin from the sukun peel has a functional group in the same wavenumber range as the functional group in the standard pectin.

#### ACKNOWLEDGMENT

This research is funded by Politeknik Negeri Bandung through its Center for Research and Community Services. Contract number : B/186.53/PL1.R7/PG.00.03/2020

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