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Microwave Assisted Extraction of Pectin from Balinese Orange Peel using Different Power Levels and Times

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***Abstract*—The extraction of pectin from Balinese orange peel was conducted using Microwave Assisted Extraction (MAE). The extraction was conducted with various microwave powers (180, 300, 450, and 600 W) and extraction times (10, 15, 20, 25, and 30 min). The extraction with various powers was conducted to 10 g of the substance in 300 mL of hydrochloric acid with the 20 min extraction time. Meanwhile the extraction with various times was conducted to 10 g of substances in 300 mL of solvent with the 300 W of power. The obtained pectin was then analyzed the composition using Fourier Transform Infrared and the physico-chemical properties using manual procedures. The experimental result showed that the extraction yield of pectin from orange peel using MAE was bigger than conventional method. The various powers influenced extraction yield of pectin, the optimal power in extraction of pectin from Balinese orange peel using MAE was 300 W, it resulted in 40.5% w/w. The extraction time also influenced the extraction yield of pectin. The most optimal time was about 20 min. The extracted pectin was able to fulfill the standard quality of pectin based on the International Pectin Producers Association. The produced pectin can be used as edible film and the quality is promising as food commodity.**

Keywords— Balinese orange peel, edible film, microwave assisted extraction, pectin

I. INTRODUCTION

Pectin was one of the basic ingredients for edible film [1-4]. Pectin is located in the middle of lamella on the cell wall [3-5]. Basically, all plants which can photosynthesize contain pectin but on different amounts depending on the kinds of plants and ripeness [6]. Orange, as one species of plants, contain many nutrients. Most of the orange components are located on its peel, some of them are alkaloid, flavonoid, lycopene, vitamin C, and the most dominant ones are pectin and tannin [7]. The content of pectin was located mostly on the layers between albedo cell walls, the content was between 20–35% [8].

Several researchers have conducted the study on the extraction of pectin [2-4,8-13]. The studies reveal that in order to obtain the highest yield of pectin, hydrochloric acid is used on the extraction temperature of 80 °C and the extraction time of 2 h. It can be concluded from the previous study that the higher the degree of the solvent acidity, the temperature and the time then the higher the extraction yield of pectin [14]. Conventionally, pectin extraction is conducted in a hot solvent solution. The high temperature and the long time effect the pectin degradation, so this methods are ineffective to extrac the pectin [8]. Zhongdong and co-workers reported that 10 min of microwave irradiation time disintegrate the orange peel than 18 h of traditional heating time [15]. Recently, application of microwave heating as power source in orange peel extraction can improve extraction yield and quality of pectin [15-18]. In addition, the extraction of pectin from orange peel with oxalate using microwave radiation on various times has also been conducted [19], but the extraction yield was still low due to the big power which cause pectin to be degraded. Meanwhile the extraction time was too short so that the content of pectin has not dissolved into the solvent. Therefore, it is necessary to conduct a study about pectin extraction from orange peel using MAE method with various powers of microwave oven and times of extraction in order to increase the extraction yield of pectin.

II. METHODS

A. Preparation of Balinese Orange Peels

The Balinese orange was chosen due its abundance. Before the substance was used in the study, some preparation needed to be conducted. The preparation process began with drying the Balinese orange peel using the electrical oven on the temperature of 55 °C until it reaches its constant weight. After the peel was dried, then it was milled using blender then it was screened using 34 mesh sieve so that it resulted in ready powder to extraction.

B. Balinese Orange Peel Extraction using Conventional Method

The extraction was conducted with the following conditions: 5 g of Balinese orange peel in the 150 mL of 0.2 N hydrochloric acid, the temperature of 80 °C, and the duration of 2 h. After finishing the extraction process, the solution was then dehydrated in order to separate pectin from its solvent by adding 96% v/v ethanol. The addition of ethanol could dehydrate pectin so that it disturbed the stability of its colloidal solution, then it resulted in coagulated pectin [5,20]. The dehydration result was transparently white pectin gel. The pectin gel was then dried and purified using ethanol. The purified pectin was dried in the oven on the temperature of 50 °C then it was milled until it became pectin powder.

C. Balinese Orange Peel Extraction using MAE Method

The MAE method has been conducted in a microwave oven (Samsung, ME731K, maximum output power of 800 W with 2450 MHz of microwave irradiation frequency). The reactor for extraction was a 1 L short-necked horizontal cylinder Pyrex glass equipped with a condenser. The extraction of pectin using MAE method was conducted with various powers and times. The extraction of pectin with various powers was conducted on 10 g of substance in 300 mL of 0.2 N hydrochloric acid with 20 min extraction time. Meanwhile, the extraction with various times was conducted on 10 g of substance with the 300 mL of solvent on the 300 W of power. The analysis of pectin composition was conducted using Fourier Transform Infrared (FTIR). In addition, the analysis of physico-chemical properties was conducted using manual procedures.

D. The Making of Edible Film of Balinese Orange Peel Pectin

The produced pectin was used a raw material for edible film by adding plasticizer. The making of edible film was conducted by dissolving 0.8 g Carboxy Methyl Cellulose (CMC) into 200 mL of aqua with the temperature of 70 °C. After that, 4 g of pectin was added in the CMC solution then agitated until it dissolved, 6 mL of sorbitol, 0.22 g of potassium sorbate, and 0.14 g of stearate acid were added consecutively. Then, the solution was poured into the glass cast and heated in the oven on the temperature of 50 °C until the film dried.

III. Results and Discussion

Before the extraction on Balinese orange peel was carried out, preparation had to be organized. The drying of the raw material was conducted with the purpose of removing water content in the raw material which could cover its surface so that the process of solvent diffusion in to the raw material in to the raw material was more optimal [4,8,11]. The drying process was carried out on the temperature of 55 °C because if the drying was carried out on higher temperature, then the pectin in the orange was degraded. After the drying process, the orange peel was milled in order to multiply the surface contact between the material and the solvent, so that the extraction yield of pectin increased [4,8,12,13].

The result of pectin extraction with various powers of microwave oven can be seen in Table 1. It shows that the most optimal condition for orange peel extraction was 300 W. From the experiment, 300 W of power produced pectin with extraction yield of 40.5% w/w. This yield value was the highest amongst all extraction with other power variation. On the power of 180 W, the yield was smaller because when the power was smaller, the heating process took longer time so that the content of pectin on the Balinese orange peel had not dissolved properly in the solvent. Meanwhile, on the power of 450 and 600 W, the yield decreased due to the pectin content was degraded. The bigger the power, the higher the microwave which lead to excessive heat then degraded the pectin. The research on the structure of pectin using MAE method with electron microscope has been conducted by [15]. Based on the research, microwave caused swelling effect which lead to the burst of pectin cell. This swollen part caused degradation on the pectin so that the particle was smaller and looked like crystal.

TABLE I. The Influence of Power Level to Pectin Yield of Extraction

Power (w)	Yield (%)
180	12.1
300	40.5
450	32.9
600	30.6

The time variation during extraction on Balinese Orange peel had been investigated (see Table 2). However, the yield was still low, so that this current study was necessary to be conducted. The time variation in this study was conducted for 10, 15, 20, 25, and 30 min. The longer the extraction, the more the yield would be. Even though, the additional extraction time was not linear with the result of the yield. Therefore, the optimal extraction time was important to be investigated. The extraction was conducted as long as the solvent was not saturated. The saturated solvent could not extract anymore or the ability of extracting became lower because the driving force decreased. It resulted in the longer extraction time but it did not guarantee to obtain more yield significantly [21]. In addition to the saturated solvent, the decreasing yield of pectin was also caused by the longer period of contact between solvent and the pectin which lead to hydrolyzed pectin then turned pectin into peptic acid. Based on Table 2, it can be seen that the 20 min extraction time gave the biggest yield which was 40.5% w/w.

TABLE II. The Influence of Time to Pectin Yield of Extraction

Time (min)	Yield (%)
10	8.8
15	20.2
20	40.5
25	36.6
30	22.9

The pectin obtained from the extraction result was then analyzed using FTIR in order to identify the main chemical substances in the pectin. The main functional groups of pectin are on the area of 1.00 and 2.000 cm^{-1} from the FTIR spectrum which can be seen in Figure 1 [22]. Table 3 shows that the composition of pectin from the current study. The existence of carbonyl compound on 1637.03 cm^{-1} shows that the sample contained a cluster of ester. The absorption bond on 1100.67 cm^{-1} which comes from ether and absorption area on 1734.81 cm^{-1} shows carboxylate.

TABLE III. The Composition of Pectin from Balinese Orange Peel

Absorption Bond (cm^{-1})	Compound
1637.03	Carbonyl
1734.81	Carboxylate
1100.67	Ether
1227.04	Alcohol, ester

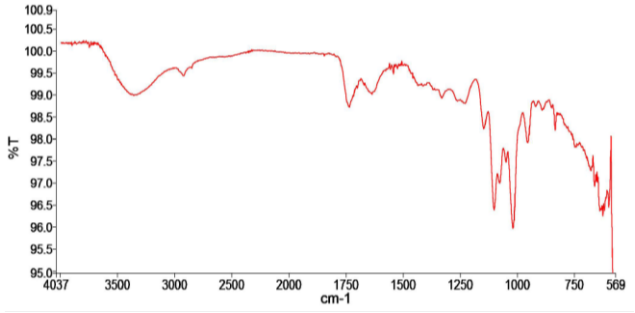

Figure 1. The spectrum of pectin from the balinese orange peel extraction with hydrochloric acid using MAE method

In addition to composition analysis, the analysis of methoxyl, the level of galacturonate, the degree of esterification, water content and equivalent weight were also conducted. The level of methoxyl in pectin has important role in determining the functional properties of pectin and the structure and texture of pectin gel. Polygalacturonic acid is a basic structure of pectin compound which describes the purity of pectin. The degree of esterification is a percentage of the amount of D-galacturonate residue whose carboxyl cluster is esterified with ethanol [14]. The higher the degree of esterification then the better the gel formation process would be. The water content means the amount of water in the pectin and it affects the storage period. The high water content leads to susceptibility to microbial activity. The ash content shows the existence of inorganic components which were left behind in the pectin after combustion. The inorganic components are calcium and magnesium which was hydrolyzed along with protopectin. The ash content influences the purity of pectin. The equivalent weight is a unit of content of free-galacturonic acid cluster (unesterified) in the pectin chain molecules [14]. The lighter the equivalent weight resulted in the greater the content of methoxyl pectin. The results of all analysis can be seen in the Table 4.

TABLE IV. THE COMPARISON OF PECTIN FROM BALINESE ORANGE PEEL EXTRACTION WITH INTERNATIONAL PECTIN PRODUCERS ASSOSIATIONBLE

Quality Factor	Standard	Experiment
Methoxyl, %	>7.12	8.5
Galacturonic Acid, %	≥35	66.5
Esterification Level, %	≥50	72.5
Water, %	≤12	17.6
Ash, %	≤10	4
Equivalent Weight	600 – 800	961.538

The pectin from the extraction had been turned into powder and then used as the basic ingredient of edible film. Sorbitol as the plasticizer was added during the process of making edible film. The sorbitol has the highest molecular weight which is 182.17 g/mole amongst other kinds of plasticizer. The molecular weight of plasticizer depends on the tensile strength of the edible film. The bigger molecular weight of the plasticizer increased the tensile strength of the edible film. The dough of edible film was then molded and dried in the oven on the temperature of 50 °C. The brownish color of edible film was caused by the brown pectin. In addition, there were spots on the surface of the edible film. However, the spots could be solved by filtering them using vacuum filter. Then, water vapor permeability observation was conducted on the edible film, it resulted in the high value, it could be seen when the edible film was taken out of oven and placed it on the room with low humidity. The edible film had high tensile strength and elasticity so that it shows promise as commodity in food industry.

IV. CONCLUSION

Based on this study, the yield of pectin from the Balinese orange peel extraction with hydrochloric acid using the microwave assisted extraction method was 40.5% w/w. The yield was higher than the one using conventional method. The variation of power in the pectin extraction using MAE method influenced the yield of pectin, the most optimal power was 300 W. The extraction time using MAE method also influenced the yield of pectin, the optimal time to extract Balinese orange peel was 20 min. The yield of pectin is able to fulfill the quality standard of International Pectin Producers Assosiation, except for the water content and the equivalent weight. The pectin can be used as edible film and the result shows promise of becoming food commodity.

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