Effects of microwave power and irradiation time on pectin extraction from watermelon rinds (*Citrullus lanatus*) with acetic acid using microwave assisted extraction method

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**Abstract.** The aims of this research are to study the effect of microwave power (119.7 W, 199.5 W and 279.3 W) and irradiation time (6, 9 and 12 min) on pectin extraction by using Microwave Assisted Extraction (MAE) with acetic acid and to do a preliminary characterization of pectin from watermelon rinds. A randomized factorial design with two factors was used to determine the effect of microwave power and processing time on the yield, equivalent weight, degree of methoxylation (DM), galacturonic acid content (GA) and the degree of esterification (DE) of extracted pectin. The results showed that extracted pectin from watermelon rinds using MAE method have yield ranged from 3.925% to 5.766%, with equivalent weight ranged from 1249.702 to 2007.756. Extracted pectin have a DM value ranged from 3.89% to 10.81%. Galacturonic acid content that meets with IPPA standard resulted from extraction condition of 279.3-watt microwave power for 9 min and 12 min. The degree of esterification (DE) value ranged from 56.86% to 85.76%, and this value exhibited a relatively high methoxyl pectin (>50%). The best pectin properties was obtained at a microwave power of 279.3 watts for 12 min.

**Keywords:** microwave assisted extraction, pectin, watermelon

1. **Introduction**

Watermelon (*Citrullus lanatus*) is a fruit usually consumed as a fresh fruit by many people in Indonesia. In 2013 Indonesia produced approximately 460,628 tons and the number was increased in 2014 to 653,995 tons [1]. The consumption of watermelon left waste rind which makes environmental problems due to watermelon rind compose approximately 30% of watermelon biomass [2]. According to the production of watermelon in 2014 at least 196,198 tons of watermelon rind was generated. Riped watermelon rinds contain 20% of cellulose, 23% of hemicellulose, 10% of lignin, and 13% of pectin [3]. Due to the pectin content, it was potentially used as raw material for pectin extraction.

Pectin is polysaccharide that composes 30% of all plant cell-wall midle lamella of the plant tissues. It is consisting of (1-4) α-D- galacturonic acid residues and some neutral sugar namely L-arabinose, D-galactose and L-rhamnose that partially esterified at carboxylic acid with methanol or acetic acid. Every source of pectin has different composition and structure that depend on the source
of pectin, the degree of ripening and the condition during extraction [4,5,10]. In food industries, pectin usually used as food thickener, food stabilizer, water binder, emulsifier and gelling agent. The application of pectin in food product is depend on the degree of esterification (DE) which is represent the percentage of esterified carboxyl group with metil alcohol. Pectin with DE higher than 50% is classified as high methoxyl pectin (HMP) and pectin which has DE lower than 50% is classified as low methoxyl pectin (LMP) [10].

Various source of pectin is extracted commercially by acid sovent or diluted mineral acid at high temperature. Commercial pectin usually extracted from the waste of juice industries such as citrus peel and apple pomace. Many studies have been reported pectin extraction from other fruit waste such as pectin from orange peels [14], apple pomace [4,11], banana peels [7], watermelon rinds [2,5], and pomelo peels [13].

There are many methods use to pectin extraction that affects the yield and pectin quality. A dilute mineral acidic solvent in high temperature was used in a conventional method. Pectin extraction by using conventional methods was considered not appropriate to obtain high quantity and quality of pectin due to the length of time processing. Therefore, some novel extraction techniques have been developed to increase the process efficiency (yield per time) such as enzyme-assisted extraction (EAE), subcritical water extraction (SWE), microwave-assisted extraction (MAE), ultrasounds-assisted extraction (UAE), supercritical fluid extraction (SFE), and pressurized solvent extraction (PSE) [2,12]. The comparison between conventional and novel method has been reported by [6]. They were compared conventional method, MAE, and UAE to extract grapefruit pectin.

Some previous study has been reported extraction pectin from watermelon rinds by using various method. [5] evaluated the properties of pectin from watermelon rind by using conventional extraction including rheological, emulsifying and foaming properties. Pectin was extracted with nitric acid solvent using conventional method. Extraction of pectin from watermelon rinds with destilled water using MAE exhibited maximum yield (25,79%) that obtained with microwave power of 477 W for 128 s [2]. Extraction pectin from watermelon rind with a weak solvent has not been reported. The weak acids such as citric acid are considered more interesting than strong acid from a safety application point of view. Although strong acids are more effective but weak acids solvent can minimize the environmental problems [7]. The objectives of this research were to investigate the effect of microwave power and irradiation time on pectin extraction by using Microwave Assisted Extraction (MAE) with acetic acid and to characterize the properties of pectin from watermelon rinds.

2. Experimental

2.1. Raw material and reagent
Watermelon rinds were provided by Lor In Hotel located in Karanganyar, Central Java, Indonesia. Before extraction, the fresh materials were dried and converted be a rind powder. Chemical reagent for analysis and all solvents used for extraction were purchased from Sigma-Aldrich, Singapore and Merck.

2.2. Preparation of watermelon rind powder
The watermelon rinds obtain from Lor In Hotel were cut ± 1cm in length, wide and thick after the red flesh was removed. After that, the rinds were washed and dried at 55 °C. The dried watermelon rinds were pulverized and sieved through a 60-mesh sieve. The rind powders were stored in plastic bags for pectin extraction.

2.3. Microwave assisted extraction of pectin
In this research, pectin extraction was carried out according to the method developed by Wang et al. [4], with a some modifications. Extraction was conducted by microwave equipment (SHARP) with different microwave power (119.7-watt, 199.5-watt and 279.3-watt) and various time processing (6
12 min). A 10 g of rind powder were loaded in 1000 ml Pyrex beaker with 100 ml distilled water. An acetic acid solvent 1 N was added to adjust pH until pH of mixture was 2. After that, the mixture was placed in a microwave and heated with different microwave power and irradiation (processing) time. After the extraction process has been done, the mixture was cooled down and filtered using Whatman paper no 1. The filtrate was precipitated by adding ethanol 96% with the same volume with the filtrate (v/v) for overnight (12 h). The coagulated pectin mass was washed with 70% ethanol to remove acidity. Then, the wet pectin was dried at 45 °C in cabinet dryer until constant weight was obtained. The driedpectin was grinded in order to obtain pectin powder.

2.4. Method of analysis

The powdered pectin was analyzed for yield of pectin, equivalent weight, degree of methoxylation (DM), galacturonic acid content (GA) and the degree of esterification (DE). The yield of pectin was calculated as a ratio between the weight of dried pectin and the weight of dried watermelon rind, according to this equation:

\[ PY = \frac{m_0}{m} \times 100 \]

where \( m_0 \) is the weight of dried pectin and \( m \) is the weight of watermelon rind powder. Equivalent weight, DM, and GA content were determined by titration with a method described by Ranggana [8]. The DE of pectin was calculated based on DM and GA content [9]. The result of pectin yield, DM value, DE, equivalent weight and GA content were analyzed statistically with two way ANOVA using SPSS.

3. Result

3.1. Effect of process variable on pectin extracted yield and equivalent weight

3.1.1 Yield of extracted pectin. The extraction were carried out at different process condition (microwave power and duration of irradiation time) in order to study the interaction between two factor to the yield of extracted pectin. The data showed that the yield of extracted pectin was increased linearly to the enhancement of microwave power and duration of irradiation time (Fig 1a). The yield of extracted pectin from this study was ranged between 3.925%–5.766%. The interaction of microwave power and irradiation time was influenced the yield of pectin.

Some previous studies were reported the optimum condition to obtain the highest pectin using response surface methodology (RSM). [4] have been reported their study of pectin extraction using MAE from apple pomace. Their experiment data showed that the microwave power have significantly effect on the yield of extracted pectin. According to the prediction by SAS, the longer extraction time the higher yield was. The extracted pectin from watermelon rind with distilled water as a solvent that exhibited the same trend with the result in this study [2]. The optimum conditions to obtain maximum yield (25.79%) of pectin were determined to be microwave power of 477-watt, duration of time irradiation of 128 s with pH of 1.52. The enhancement of microwave power will increase the penetration of solvent into the material and lead to rapid energy transfer to the solvent and the material, generating dissolution of extracted pectin [2].
3.1.2. Equivalent weight. The result of the effects of condition process to equivalent weight extracted pectin has been showed in Fig 1b. Increasing of microwave power energy lead to decrease of equivalent weight. In other hand, increasing the duration of irradiation time significantly decreased the equivalent weight. The greatest equivalent weight of pectin was obtained at 279.3 watt of microwave power and 6 min of irradiation time. According to ANOVA, interaction between microwave power and irradiation time have influenced the equivalent weight of pectin.

3.2. Effect of of process variable on qualitative characterisitics of pectin extracted
The data exhibited a significant influence of microwave power and irradiation time on the degree of esterification of the dried pectin. The average of DE value was increased significantly with an increase in microwave power. In different irradiation time, the average of the DE value showed significantly difference.

The average DM value resulted in this study showed significant enhancement with an increasing of heating power and duration irradiation time. The highest DM value was 10.812% that obtained at 279.3-watt of microwave power and 12 min of irradiation time.

The data showed an interaction of microwave power and duration time process could effect the GA content of extracted pectin. Based on ANOVA anlysis, the average of GA content was increased with an increase in microwave power and the lenght of irradiation time. The highest GA content was exhibited in condition 279.3-watt of heating power during 12 min irradiation.

4. Conclusion
Based on the data resulted from this presen study, pectin was extracted succesfully from watermelon rind with acetic acid solvent by using MAE method. There was an influence of the microwave power and irradiation time on the yield, equivalent weight, DM value, DE value and GA content of extracted pectin from watermelon rind. The best characteristic of extracted pectin and the highest yield was obtain in extraction condition 279.3-watt of microwave power and 12 min of irradiation time.
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