



Microwave Assisted Extraction of Watermelon Rind Pectin

Indah Hartati¹ and Endah Subekti²

¹Department of Chemical Engineering, Faculty of Engineering, Wahid Hasyim University, Indonesia

²Department of Agribusiness, Faculty of Agriculture, Wahid Hasyim University, Indonesia

Abstract: Rind of watermelon is reported to contain approximately 13% of pectin. Hence, watermelon rind is considered as a potential raw material for pectin production. Pectin separation can be conducted by applying microwave assisted extraction. In the present study, pectin was extracted by using microwave extractor conducted at different condition. Sulfuric acid solution concentration was varied of 0.25-1.5 M; extraction time of 5-25 min; solid liquid ratio of 1:6-1:14; and microwave power of 39.9, 119.7W and 199.5W. Microwave assisted extraction of watermelon rind pectin was proved to give a high yield of extraction. The highest extraction yield of 11.25% was achieved at 15 min of extraction time, 0.5 M of sulfuric acid solution, and solid liquid ratio of 1:08. Microwave assisted extraction of watermelon rind pectin conducted at lower power system gave better extraction yield.

Keywords: watermelon rind, pectin, microwave assisted extraction.

Introduction

Citrullus lanatus, a tropical fruit commonly known as watermelon is native to Africa. It has been cultivated for thousands of years in many Middle East and South East Asia countries¹. Watermelon scientific name is derived from both Greek and Latin roots. A Greek word "citrus" which refer to the fruit is the root for the *Citrullus* part. Meanwhile "lanatus" which has the meaning of being wooly is come from Latin root².

Watermelon is widely consumed around the world and enjoys worldwide popularity for its aesthetic tastes and nutritional compositions. Nowadays, China, Turkey, Iran, Brazil and Unites stated are reports as the big five watermelon producers in the world. The world wide watermelon production reaches 109.2 million tons at 2013³.

Flesh, seed and rind are the three main components of watermelon biomass⁴. The flesh constitutes approximately 68% of the total weight, the rind approximately 30%, and the seeds approximately 2%. Watermelon rind is the area of white-colored flesh between the colored flesh and the outer skin of watermelon. The rind of watermelon is reported to contain approximately 20% of cellulose, 23% of hemicelluloses, 10% of lignin, 13% of pectin, 7 mg/g silica, and 12% silica free minerals⁵.

Various attempts and studies already conducted in order to separate and utilize all of the watermelon biomass. Watermelon rind was used as the raw material for sugar extraction⁴. Watermelon rind was also used for candy, cheese and pickle¹. Moreover, watermelon rind pectin extraction was investigated by several researchers^{5,6}.

Pectin is present within the primary cell wall of almost all higher plants. Pectin acts as glue that holds cellulose together. Chemically, pectin is a heterogeneous polymer. The dominant component is a linear chain of 1→4 linked galacturonic acid. The proportions of the carboxyl acid groups of pectin are present as methyl esters and, in amidated pectin, are replaced with amide groups. Further, regions of neutral sugars exist in the molecules of pectin are including arabinose, galactose, and rhamnose⁷.

Pectin has important nutritional and technological properties, mainly because of its ability to form gels. Pectin is having properties such as gelation and emulsion stabilization which make it useful in the manufacture of food, cosmetics, and medicine^{6,8}.

In food industry, pectin has been widely applied as thickening, gelling, and emulsifying agent for jams, soft drinks, fish and meat products, fruit juice, desserts and dairy products⁶. Pectin is also applied in medicine, since it helps in lowering serum cholesterol level, removing heavy metal ions from the body, stabilizing blood pressure and restoring intestinal functions and weight reduction⁹. Pectin is also has been used potentially as a carrier for drug delivery to the gastrointestinal tract, such as matrix tablets, gel beads, film-coated dose form.

At present, commercial pectin is produced by extraction of edible plant material. The dominant raw material is the rind of citrus fruit. Apple pomace and sugar beet pulp are also used¹⁰. As stated above, watermelon rind is a potential alternative sources for pectin production.

The most commonly method for pectin extraction is direct boiling in hot, acidified water followed by isolation of the pectin from the ensuing solution. It takes approximately two hours to obtain good yield of pectin. Due to relatively long period of direct heating, pectin undergoes thermal degradation¹¹.

Microwave assisted extraction (MAE) is a potential method that can be applied in the pectin production. MAE is generally more effective in term of pectin yield and give better quality product. Usually MAE takes shorter period than the direct boiling¹².

The purpose of the present study was to investigate the influence of process parameter of the microwave assisted extraction of pectin from watermelon rind.

Experimental

Materials

Watermelon was bought from local market of Gunungpati Semarang. Ethanol (Merck) was purchased from CV Damai Sejahtera Prima. In the laboratory, the rind of the watermelon was removed from the flesh. The rinds were first washed with water to remove some of the sugars and ground in an electric grater. It dried, initially at room temperature and then at 50°C, to a constant weight with air circulation. This dry watermelon pool was then crushed and mixed. The product was called watermelon flour and it was used as the raw material for all the pectin microwave assisted extraction assays.

Apparatus

Microwave assisted extraction of watermelon rind pectin was conducted in a modified domestic microwave. The microwave was modified and equipped with extraction flask and a spiral condenser as shown on Figure 1.



Figure 1. Modified microwave extractor

Extraction

Twenty grams of watermelon rind pectin dried powder was subjected in a certain volume of sulfuric acid solution. The mixture placed in 500 ml round bottom flask and extracted in a modified microwave extractor for certain extraction duration. The extraction process parameters were including the sulfuric acid solution concentration was varied of 0.25-1.5 M; the extraction time of 5-25 minutes; and the microwave power of 39.9 and 119.7W. The mixture then was allowed to stand for 1 hour and then filtered. After the microwave heating process completed, the mixture was then filtered and supernatant was cooled at 8-10°C. The pectin was precipitated with two volumes of alcohol (ethanol) for one volume of supernatant. The obtained precipitate was washed with 6.6 % alcohol and centrifuged (10000 rpm during 20 min). The precipitated was then dried and weighed.

SEM analysis

In order to understand the effect of microwave power toward the extraction mechanism, powder of watermelon rind and residue of watermelon rind after the MAE process conducted at different power level were subjected to SEM. Samples were dried at 70°C in 2 hours for scanning by SEM. Samples were tested under high vacuum conditions at a voltage of 20 kV (50 µm, 300 and 500×magnification).

Results and Discussion

Effect of extraction time

Period of heating in the microwave assisted extraction is one of important factor to be considered. Compared to conventional techniques, extraction times in microwave assisted extraction are very short. Conventional extraction of pectin by direct boiling in hot, acidified water is time-consuming which commonly takes more than one hour to obtain a certain amount of pectin. While microwave assisted extraction of pectin usually vary from a few minutes to a half-hour¹³⁻¹⁴.

Twenty grams of watermelon rind pectin dried powder was subjected in a 160mL of 0.5 M sulfuric acid solution. The mixture placed in 500 ml round bottom flask and extracted in a modified microwave extractor that is set 39.9 W of power level. The extraction duration was varied 5-25 minutes. Yield of the microwave assisted extraction of watermelon rind pectin conducted at sulfuric acid solution of 0.5 M; solid liquid ratio of 1:8; and microwave power of 39.9W was shown in Figure 2.

The microwave assisted extraction of watermelon rind pectin showed that there was a positive linear correlation between extraction yield and irradiation time. The longer exposures caused higher values of extraction yield, whereas further increase in irradiation time not only resulted in no improvement in the extraction performance, but led to a fall in the extraction yield. It was observed that the extraction yield was increase with time up to an exposure of 15 min and then the extraction yield started to decrease.

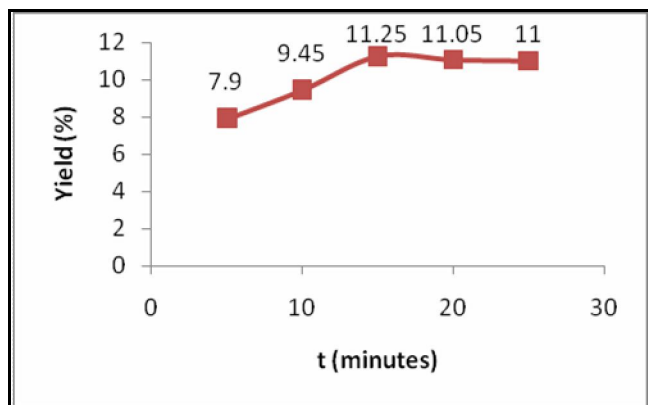


Figure 2. Yield of the microwave assisted extraction of watermelon rind pectin conducted at sulfuric acid solution of 0.5 M; solid liquid ratio of 1:8; and microwave power of 39.9W

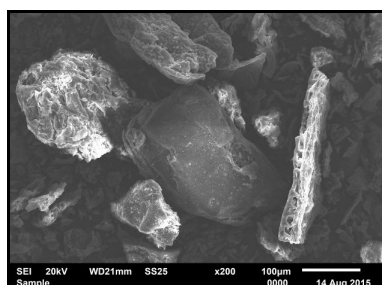
In the microwave assisted extraction berry pectin with water as the solvent, it was found that microwave assisted extraction reached its maximum at 30 min¹⁵. In the case of pectin extraction from apple pomace, there was an increase in yield with time up to an exposure of 20 min and then the extraction yield started to decrease¹⁶.

Compared to the conventional method of watermelon rind pectin, MAE of water melon rind pectin was much faster. In the conventional extraction of watermelon rind pectin with citric acid as the solvent, it was reported that the highest yield achieved at 110 minutes of extraction⁶.

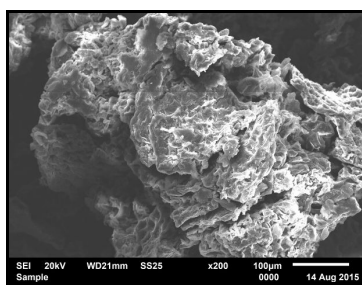
MAE gave better extraction yield compared to the conventional methods because theoretically, the application of microwave towards the cell wall matrix could cause severing of parenchyma cells which results in opening of skin tissues by microwave¹⁷. Hence, the solvents were able to penetrate into the skin tissues leading to increased pectin yield due to the the interaction between solvent and tissues

The severing of the cell was shown on the Scanning Electron Microscopy (SEM) analysis of the watermelon rind flour before and after used in the MAE process (Figure 3).

On the microwave assisted extraction, irradiation time is also influenced by the dielectric properties of the solvent and the power level of the microwave¹⁸. Microwave power is directly related to the quantity of sample and the extraction time required. However, the power provides localized heating in the sample, which acts as a driving force for MAE to destroy the plant matrix so that the solute can diffuse out and dissolve in the solvent. Therefore, increasing the power will generally improve the extraction yield and result in shorter extraction time. This behavior match with research conducted by Maran *et al.*(2014). They found that the optimum MAE conditions for the highest pectin yield from waste *C. Lanatus* fruit rinds (25.79%) were obtained with microwave power of 477 W, and irradiation time of 128 s, solid–liquid ratio of 1:20.3 g/ml respectively¹⁹.



(a)



(b)

Figure 3. SEM analysis of the water melon rind pectin (a) before and (b) after it used as the raw material for microwave assisted extraction of pectin

Effect of acid solution concentration

The investigation of the acid solution concentration effect toward the extraction yield was conducted in a series. Twenty grams of watermelon rind pectin dried powder was subjected in a 160mL of 0.25-1.5M sulfuric acid solution. The mixture placed in 500 ml round bottom flask and extracted in a modified microwave extractor that is set 39.9W of power level. The extraction duration was set of 15 min.

The microwave assisted extraction of watermelon rind pectin showed that there was a positive linear correlation between extraction yield and acid solution concentration. The higher the acid concentration caused higher values of extraction yield, whereas further increase in acid concentration gave no improvement in the extraction performance, but led to a fall in the extraction yield. It was observed that the extraction yield was increase with acid concentration up to 0.5M and then the extraction yield started to decrease (Figure 4).

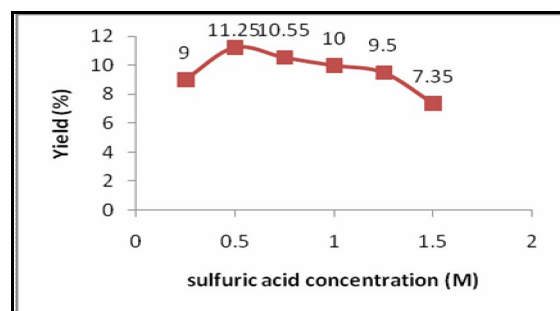


Figure 4. Yield of the microwave assisted extraction of watermelon rind pectin conducted at extraction time of 15 min; solid liquid ratio of 1:8; and microwave power of 39.9W

High concentration of strong acid solution are reported could cause a partial hydrolysis of the pectin^{9,20}. Thus it would produce smaller pectin particles which then lead to the increasing of its solubility to the point that no precipitate was formed by the addition of alcohol. Hence, this could be the reason why the use of a high concentration of strong acid resulted in a lower pectin yield.

Effect of solid liquid ratio

The solid liquid ratio was varied in range of 1:6-1: 14 in order to investigate the effect of solid liquid ratio to the extraction yield of watermelon rind pectin. The 20 grams of watermelon rind pectin dried powder was subjected in a varied volume of 0.5M sulfuric acid solution. The mixture placed in 500 ml round bottom flask and extracted in a modified microwave extractor that is set 39.9 W of power level. The yield of watermelon rind pectin by raising the liquid solid ratio from 6-14 was shown on Figure 5.

It was observed that the extraction yield was increase with solid liquid ratio up to 1:8 and then the extraction yield started to decrease. The increasing ratio of liquid-solid indicates that bigger liquid-solid ratio firstly led to higher yield of pectin. Bigger solid liquid ratio implicated to the extraction contact area because the volume of the liquid is bigger. The higher the extraction contact area, led to a higher of the extraction efficiency.

Moreover, further expanding the liquid-solid ratio, has led to a decreasing of the extraction yield. It could be due to the effect of the dielectric properties of acid solution (sulfuric acid) and water. Sulfuric acid and water has a high dielectric constant i.e and 80 respectively. Solvents with high dielectric constant are the best absorber of microwave radiation may heat up tremendously on longer exposure²¹, thus risking the future of thermo labile constituents such as pectin and may led to a decreasing of the extraction yield.

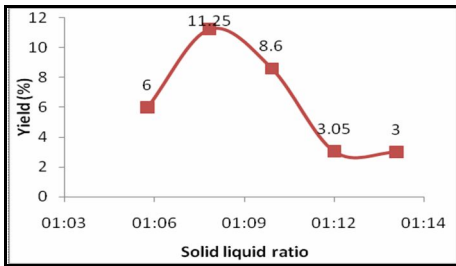


Figure 5. Yield of the microwave assisted extraction of watermelon rind pectin conducted at sulphuric acid solution of 0.5 M; extraction time of 15 min; and microwave power of 39.9W

Effect of microwave power level

In order to investigate the effect of microwave power level toward the yield of watermelon rind pectin, the extraction parameters were set of 0.5 M sulfuric acid solution, extraction duration of 15 minutes, solid liquid ratio of 1:10 and the microwave power were varied of 39.9W, 119.7W and 199.5W. The experiments result showed that MAE at lower power gave better extraction yield (Figure 6).

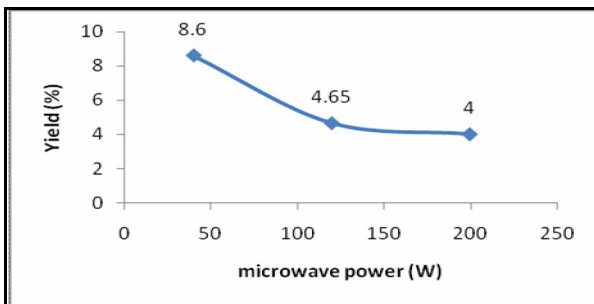
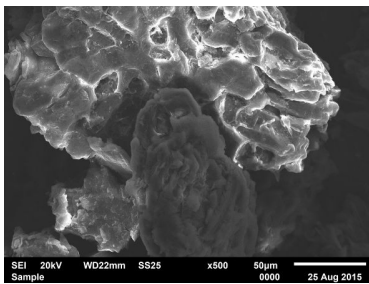
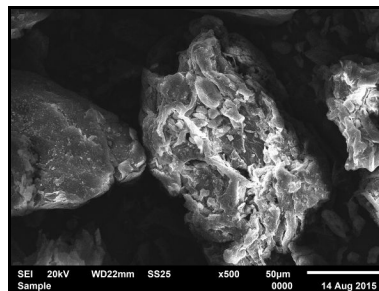


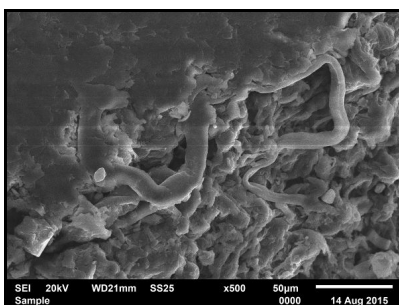
Figure 6. Yield of the microwave assisted extraction of watermelon rind pectin conducted at sulfuric acid solution of 0.5 M; extraction time of 15 minutes; and solid liquid ratio of 1:10



(a)



(b)



(c)

Figure 7. SEM analysis of the water melon rind pectin residue from MAE conducted at power level of (a) 39.9W, (b) 119.7W and (c) 199.5W

The Scanning Electron Microscopy analysis toward the residue of the microwave assisted extraction of watermelon rind conducted at different power level can be seen at Figure 7.

Figure 7 showed that that higher power level of the microwave caused more severed cell of the watermelon rind flour. It was supposed to give us a better yield of the extraction conducted at higher power level of the microwave. But, research result showed that the extraction yield at higher power was lower than the extraction yield at lower power.

The lowering of the extraction yield at higher power could be due to at higher power, pectin undergo degraded. The pectin degradation on high power level of microwave was also reported¹⁷. They stated that the decreasing of pectin yield from grapefruit extracted by MAE at a higher power could be caused by pectin degradation where pectin disaggregates into its smaller component parts¹⁷. Higher microwave power also reported as the cause in more breakage of pectin and thus lower pectin yield.

Conclusions

Microwave assisted extraction of watermelon rind pectin was proved to give a high yield of extraction. The highest extraction yield of 11.25% was achieved at 15 min of extraction time, 0.5 M of sulfuric acid solution, and solid liquid ratio of 1:08. The experiments showed that microwave assisted extraction of watermelon rind pectin conducted at lower power system gave better extraction yield.

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