Microwave Assisted Extraction of Watermelon Rind Pectin with Different Kind of Acid Solution

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Abstract — **Watermelon biomass can be categorized as three main components which are the flesh, seed, and rind. Watermelon rind is the area of white-colored flesh between the colored flesh and the outer skin of watermelon. It is constituted approximately 13% of pectin, and made it as a potential raw material for pectin production. Therefore it is necessary to separate pectin of watermelon rind. Pectin can be separated from watermelon rind matrix by microwave assisted extraction (MAE). Commonly, pectin separation is conducted by subjected in an acid solution. The aim of this work was to investigated the utilization of different kind of acid solution in the microwave assisted extraction of watermelon rind pectin. The acid solution were hydrocloric acid, citric acid, acetic acid and sulphuric acid. The molarity of the acid solution was varied of 1 and 2 N. 20 grams of watermelon rind powder was subjected into 200 ml od acid solution. The mixture was then irradiated in microwave extractor for 15 minutes at 39,9W. The research showed that 1 N of sulphuric acid gave the highest yield of extraction, namely 18%.**

Key words - pectin, watermelon rind, MAE, sulphuric acid.

I. INTRODUCTION

The energy of the conventional heating process is transferred to the material by convection, conduction, and radiation phenomena through a thermal gradients. In contrast, the energy of the microwave heating is delivered directly to materials through molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy. Energy transfer of the microwave heating occurs by two mechanisms: dipole rotation and ionic conduction [1, 2].

The unique properties of microwave have promote the application of microwave in various area. Microwave heating has been applied in various chemical synthesis, reactions, analysis and processes such as drying, sintering, and extraction [3].

In microwave assisted solvent extraction, the mass and the heat transfers occur from the inside of the extracted material to the bulk solvent In conventional heating, heat transfer occurs from the outside to the inside whilst mass transfer occurs from the inside to the outside [4].

Compared to conventional extraction, microwave assisted extraction (MAE) offers many advantages, including shorter extraction time, less solvent consumption, higher extraction rate and better product with lower cost because the microwaves heat the solvent or the mixture directly [5].

Due to its advantages, microwave assisted extraction has been applied in the separation of many phytoconstituents {4,5,6]. Pectin is one of them.

Pectins are high molecular weight heteropolymers containing a majority of galacturonic acid units. The acid group may be free, a simple salt or naturally esterified with methanol. Pectins are also contain a range of neutral sugars, including rhamnose, galactose, arabinose and lesser amounts of other sugars. Pectin is present within the primary cell wall that hold cellulose together of almost all higher plants [7].

Because of its ability to form gels, pectin has important nutritional and technological properties. Pectin is having properties such as gelation and emulsion stabilization which make it useful in the manufacture of food, cosmetics, and medicine [8].

In food industry, pectin has been widely applied as thickening, gelling, stabilizer and emulsifying agent [8]. Pectin is used as a gelling agent in a wide range of fruit-based products. Pectin can be used to improve the mouth-feel and the pulp stability in juice based drinks and as a stabiliser in acidic protein beverages**.** Pectin also increases the gel strength of low calorie jams. Pectin is also used in confectionery jellies to give a good gel structure [9]. In medicine, pectin helps in lowering serum cholesterol level, removing heavy metal ions from the body, stabilizing blood pressure and restoring intestinal functions and weight reduction [10]. 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. Moreover, cosmetic product used pectin as stabilizer.

Nowadays the major sources of pectin production are citrus peel and apple pomace. Alternative sources of pectin are continously investigated and developed, which one of them is watermelon rind.

Watermelon biomass can be categorized as three main components which are the flesh, seed, and rind. 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% cellulose, 23% hemicellulose, 10% lignin, 13% pectin, 7 mg/g silica, and 12% silica free minerals [11].

Due to its high rind pectin content and the advantages of microwave assisted extraction, hence it is urge to conduct and investigate the microwave assisted extraction of watermelon rind pectin.

Pectin extraction could be conducted by using acid solution. Commonly, the acid used in the pectin extraction were hydrochloric acid, nitric acid, acetic acid and sulfuric acid.

The purpose of the present study was to investigate the utilization of different kind of acid, i.e hydrochloric acid, acetic acid and sulfuric acid in the microwave assisted extraction of watermelon rind pectin.

II. METHODS

A. Materials

Water melon (Citrullus lanatus, L.) was bought from local market of Gunungpati Semarang. Ethanol (Merck) was purchased from CV Damai Sejahtera Prima.

Fig 1. Watermelon rind

Fig 2. Watermelon rind flour

The rind of the watermelon was removed from the flesh (Fig 1). It was done with a knife. All the rinds were first washed with water to remove some of the sugars and ground in an electric grater.

The watermelon rind then was dried, initially at room temperature and then at 50ºC, to a constant weight with air circulation. This dry watermelon rind was then crushed and mixed. The product was called watermelon rind flour (Fig 2) and it was used as the raw material for all the pectin microwave assisted extraction assays.

B. 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 condensor (Fig 3).

Fig 3. Modified microwave extractor

Extraction

20 grams of watermelon rind pectin dried powder was subjected in a 200 ml of acid solution. The acid concentration was varied of 1 and 2 N, whilst the acid used were hydrochloric acid, acetic acid and sulfuric acid. The mixture placed in 500 ml round bottom flask and extracted in a modified microwave extractor for 15 minutes of extraction duration. the The mixture then was allowed to stand for 1 hour and then filtered. After the microwave heating process completed, the mixture then was filtered and supernatant was cooled at 8-10^{0}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 presipitated was then dried and weighed.

III. RESULT AND DISCUSSION

The separation of pectin basically involved the aqueous extraction, isolation, purification and the drying process of pectin. The pectin extraction process should use a suitable method to obtain the maximum yield and quality of pectin. The extraction conditions, such as temperature, extraction time, pH, type of extraction solvents, and the drying method of pectin were the parameter process that influence the yield of the pectin separation [12].

Pectin extraction could utilized several acids as the solvent. The acids used for pectin extraction were the tartaric, malic, citric, lactic, acetic, ammonium oxalate/oxalic acid and phosphoric acids [9]. Citric acid was used in the extraction of Kafir lime pectin. Ammonium oxalate/oxalic acid was used in the extraction of dragon fruit pectin [12].

Nowadays there was a tendency to use the cheaper mineral acids, such as sulfuric, hydrochloric and nitric acids. Hydrochloric acid was used in the apple pomace pectin extraction process [9] and nitric acid was utilized in the citrus peel and lemon pomace pectin extraction [13].

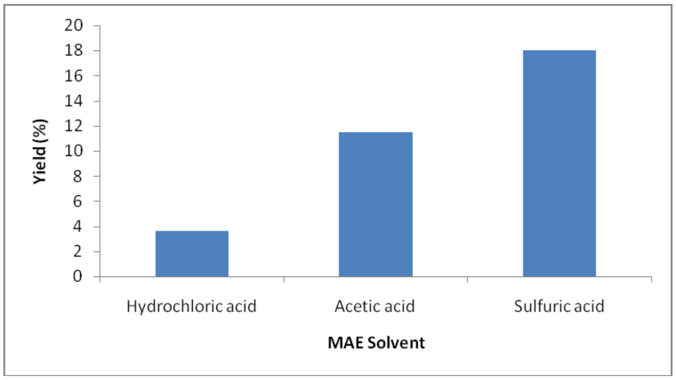


Fig. 4. Yield of watermelon rind microwave assisted extraction conducted at power level of 39,9W, 15 minuted and acid concentration of 1N

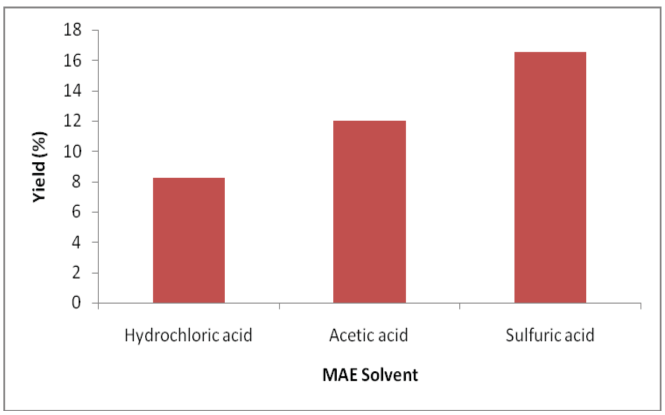
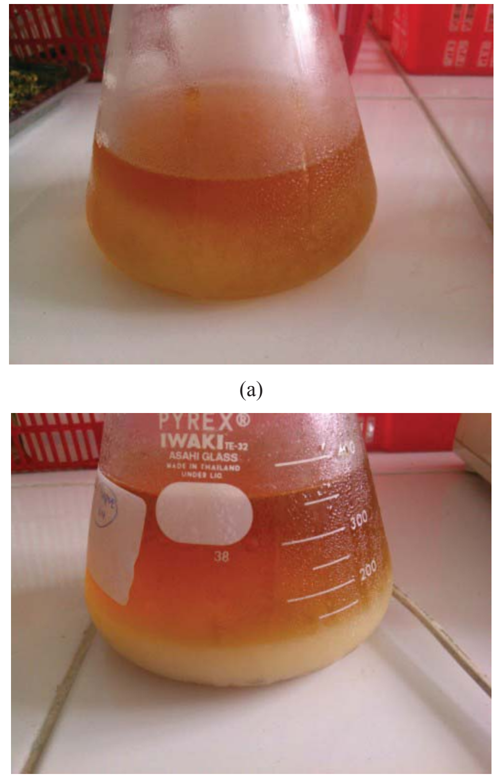


Fig. 5. Yield of watermelon rind microwave assisted extraction conducted at power level of 39,9W, 15 minuted and acid concentration of 2N

Microwave assisted extraction of watermelon rind pectin showed that in the extractions that were conducted by using sulhuric acid gave the highest yield (Fig 4 and 5). Yield of watermelon rind microwave assisted extraction conducted at power level of 39.9W, 15 minuted and sulfuric acid concentration of 1 and 2N were 18 and 16.5% respectively. Compared to the yield of the watermelon rind pectin microwave assisted extraction by using sulfuric acid, the yield of the watermelon rind pectin microwave assisted extraction using acetic acid were lower, followed by hydrochloric acid as the extraction solvent. Yield of watermelon rind microwave assisted extraction conducted at power level of 39.9W, 15 minuted and acetic acid concentration of 1 and 2N, were 11.5 and 12% respectively. Pectin obtained from microwave assisted extraction with sulfuric acid and hydrochloric acid as solvent were shown on Fig 6.



(a)

(b)

Fig 6. Pectin obtained from microwave assisted extraction with acid solution of: (a) hydrochloric acid, (b) sulfuric acid

The high yield of watermelon rind pectin microwave assisted extraction has proved the advantages of microwave heating. A conventional solvent extraction of watermelon rind by using nitric acid at extraction conditions of 45 min, pH 1.65, and solid to liquid ratio of 0.258 g/mL using a waterbath has gained extraction yield of 12% [11]. Moreover, pectin yield for enzymetic extraction of watermelon rind pectin by utilized Fibrilase and Multifect XL at solid to liquid ratios of 0.18 and 0.25 g/mL using enzyme loadings of 4.6 and 3.0 FPU/g, respectively, at 50CC for 15 hours using 50 mM citrate buffer could reach 20% [11]. Though enzymatic extraction of pectin gave a higher yield, the drawbacks of enzymatic process were the high cost of the enzyme and the long duration of the process.

The high yield of microwave assisted extraction conducted with sulfuric acid is probably due to its high dielectric constant and its low difference of Hildebrand solubility parameter of sulfuric acid and pectin (Table 1).

The microwave absorbing properties of the solvent is represented by its dielectric constant. The microwave absorbing properties dictating the selection of solvent for microwave assisted extraction. The higher the dielectric constant values of a solvent, the faster the heating rate of the solvent under the influence of microwave irradiation [5,14] . Hence it is logical that sulfuric acid microwave assisted extraction gave the highest yield of pectin. Table 1 showed that the dielectric cosntant of sulfuric acid is 21.9. The lowest one is hydrochloric acid, namely 5.

TABLE 1. DIELECTRIC CONSTANT AND HILDEBRAND SOLUBILITY PARAMETER OF VARIOUS SOLVENTS

Solvent	δ_t (MPa$^{1/2}$)	$\Delta\delta$	Dielectric constant ε'
Acetic acid	21.4	36,91	6.2
Hydrochloric acid	23.25	35,06	5
Sulfuric acid	35.89	22,42	21.9

Hildebrand solubility parameter was also an important concept that can be used, among other applications, for the rational selection of solvents. Both the total solubility parameter and its constituent partial solubility parameters (Hansen solubility parameters) are widely used for the selection of appropriate solvents for givens solutes [15]. The Hildebrand solubility parameter of pectin is 58,31 MPa$^{1/2}$[16]. The closeness of the Hildebrand solubility parameter of pectin and solvent could give us assistance in the solvent selection of microwave assisted extraction of watermelon rind.

IV. Conclusions

Based on the experimental data, sulfuric acid gave the highest yield in the microwave assisted extraction of watermelon rind. The experimental data was validated with the dielectric constant and the Hildebrand solubility parameter.

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