

Extraction of pectin from pomelo (*Citrus maxima*) peels with the assistance of microwave and tartaric acid

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Abstract

Pectin is a component of the plant cell and can be found in the primary cell wall and the middle lamella of plant tissues. Currently, pectin is a very important raw material, especially in the food and the pharmaceutical industries. There are several ways to extract pectin from different materials and solvents. One way to synthesize pectin is from the extraction of pomelo peel and this research specifically studies the extraction using tartaric acid as the solvent and under microwave assistance. Some properties of the synthesized pectin were tested such as DE and viscosity. The pH value, the rate of pomelo peels/solvent (w/v), the level of irradiation power and the irradiation time affected the productivity of pectin. The results showed that at pH value of 1.5, rate of pomelo peel/solvent was 1/40 and the irradiation time during 9 minutes at 660W of microwave power. The yield of pectin obtained 23.83%, pure pectin was 80.88% and it was rated as a high methoxyl pectin (HMP) (DE = 92.75%) with a low viscosity. The study proved that the use of tartaric acid solvent and microwave increased the quality and yield of synthesized pectin; moreover, this method was also time and energy saving.

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Keywords

Citrus

DE value

Grape fruit

Viscosity

Yield

Introduction

Among other polysaccharides that are extracted from plant materials, pectin is an extremely complex polysaccharide and is widely used as a functional ingredient in food and pharmaceutical industries. In food technology, it is used as a thickening and gelling agent (May, 1990). Its medical uses are anti-diarrhea, detoxification and blood glucose reducer (Voragen *et al.*, 1995). The worldwide annual consumption of pectin is approximately 45 000 tones every year, occupying the global market value of at least 400 million Euros (Savary *et al.*, 2003). Pectin consists of D-galacturonic acid units and is classified into two groups: high methoxyl pectin (HMP) and low methoxyl pectin (LMP) that depend on the degree of esterification (DE). There are many sources of pectin in nature such as pomelo peel, apple pomace, citrus peel, sugar beets, dragon fruit peel, etc... Apart from the apple pomace and the citrus peel which are the most common commercial sources for producing pectin, other novel sources, including sugar beets and sunflower heads, have also been investigated (Joye and Luzio, 2000).

Pomelos (*Citrus Maxima*) belongs to *Citrus* group and *Rutaceace*. Pomelo is widely planted in Vietnam with different varieties (Quoc *et al.*,

2014). The spongy white peel can account up to 30% of the total fruit weight and is a good source for pectin extraction (Methacanon *et al.*, 2013). The extraction of pectin usually uses two kinds of solvents: inorganic acid solvent such as hydrochloric, sulfuric or nitric acid and organic acid solvent such as oxalic, tartaric or acetic acid. All of them utilize traditional heating. However, it is proved that the microwave extraction shortens the pectin extraction time, also reduces the solvent consumption and higher yields than the conventional method did (Seixas *et al.*, 2014). Utilizing the microwave to aid the heating process and tartaric acid as solvent to extract pectin from orange peel (Liang *et al.*, 2011) and passion fruit (Seixas *et al.*, 2014). The extracted pectin from both studies were proved to have high yield, also time and energy saving. Recognizing the potential of microwave assistance and tartaric acid solvent in pectin extraction, this research focuses on pectin extraction using pomelo peels as raw material due to its abundant growth in Vietnam region. The following properties would be reported to rate the effectiveness of this synthesis: the pH value, the ratio of pomelo peel/solvent, the level of irradiation power and the extracted time.

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Material and Method

Materials

The ripe “Nam Roi” pomelo was chosen as the raw material in these experiments. It was harvested from Vinh Long province (Vietnam). The spongy white peels were removed using a paring knife and cut into small pieces, then they were blanched in steam at 90°C for 5 minutes (Guo *et al.*, 2011). After blanching, the peels were dried at 50°C for about 10 hours (Moisture of dried peels were approximate 9%) and grounded into powder (Particles size was under 0.6 mm).

Isolation of pectin

Five gram of pomelo peels were acidified with 0.25% (v/v) tartaric acid solution with ratio of peels/acid 1/70, 1/60, 1/50, 1/40 and 1/30 (Joye and Luzio, 2000) and acid pH 1, 1.5, 2, 2.5 and 3 (Zhou *et al.*, 2010). It was then placed in the microwave for 3, 6, 9 and 12 minutes (Seixas *et al.*, 2014) at the power 195, 379 and 660W (Quoc *et al.*, 2014). After microwave heating, the mixture was cooled down to room temperature and filtered using filter fabric. Then 96% (v/v) ethanol solution was used to precipitate pectin (the pectin solution/alcohol ratio was 1/3) for 60 minutes at pH 3.5. The precipitated pectin was washed with 96% ethanol to remove the mono and disaccharides. At the end, the mixture was dried at 70°C for 4 hours and then stored in bags.

Determination of content of pectin

According to Mui (2001), crude pectin (0.15 g) was added in 250 ml flask, then adding 100 ml of 0.1 N NaOH. Crude pectin was soaked in NaOH solution for 7 hours, then added 50 ml of 1 N CH₃COOH and 50 ml CaCl₂ after 5 minutes and kept it in 1 hour. The solution was boiled for 5 minutes, filtered by filter paper and dried for 1 hour. Calcium pectate was washed with hot water until not having Cl⁻ ion in the solution. After washing, it was dried for about 2 hours at 105°C. The pure level of pectin was calculated according to the following formula below:

$$P = \frac{m \times 0.92 \times 100}{M}$$

P (%): the pure level of pectin

m (g): weight of calcium pectate

M (g): weight of crude pectin

0.92: pectins have 92% in volume of calcium pectate

The yield of pectin was determined by formula:

$$Y = \frac{a_0}{a} \times 100$$

Y (%): the yield of pectin

a₀ (g): the weight of pectin (a₀ = P×M)

a (g): the weight of dried pomelo peel

Determination of degree of esterification (DE)

This method was slightly modified from titrimetric method of Owens *et al.* (1952) and Pinheiro *et al.* (2008). Pectin (0.5 g) was added in 250 ml flask and dissolved in 5 ml ethanol, 1 g NaCl and some drops of phenolphthalein. Adding 100 ml of warm deionized water dissolved pectin. The solutions were titrated with 0.1 N NaOH and the result was recorded as V₁. Then 25 ml of 0.25 N NaOH was added in this solution which were stirred at room temperature for 30 minutes. After that, 25 ml of 0.25 N HCl was added and the solutions were shaken until the pink color disappeared. The solution was titrated again with 0.1 N NaOH and the final result was recorded as V₂. The DE value was calculated according to the following formula below:

$$\%DE = \frac{V_2 \times 100}{V_1 + V_2}$$

Determination of viscosity of pectin

Determination of viscosity by Rheometer (Brookfield DV-III Ultra-USA), spindle “61” and speed 100 rpm.

Color evaluation

Color parameters were measured on pectin samples (powder form) and color was determined using by a Chroma Minota CR-410. Values were recorded as lightness (L*, ranging from 0 to 100, corresponding to black to white, respectively), chroma (C*, representing color intensity or saturation), and hue angle (h*, representing by degrees of the angle) (Cook, 2000).

Results and Discussion

Effect of pH on the extraction yield of pectin and DE value

The extraction of pectin is influenced by several variables such as temperature, extraction time, pH, solid/solvent ratio (May, 1990), irradiation time and irradiation power (Wang *et al.*, 2007; Seixas *et al.*, 2014). Figure1 shows that there are significant differences at p_{value} = 0.05 from the pH values. As the pH increases, the pectin yield decreases. The highest yield was 32.43% at pH = 1.5 but DE had the minimum value (76.39%). The lowest yield was 6.23% at pH = 3. The lower the pH values were the presence of H⁺ ions increased, hence it would

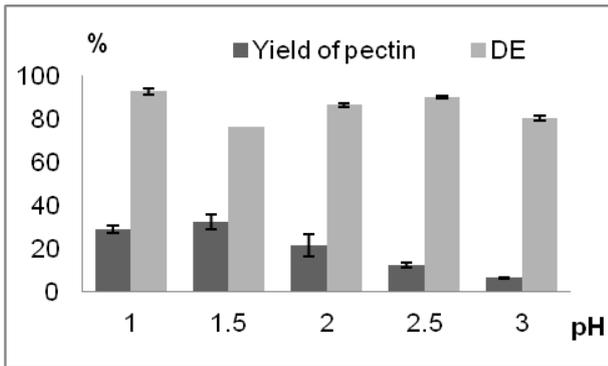


Figure 1. Effect of pH values on the extraction yield of pectin and DE value

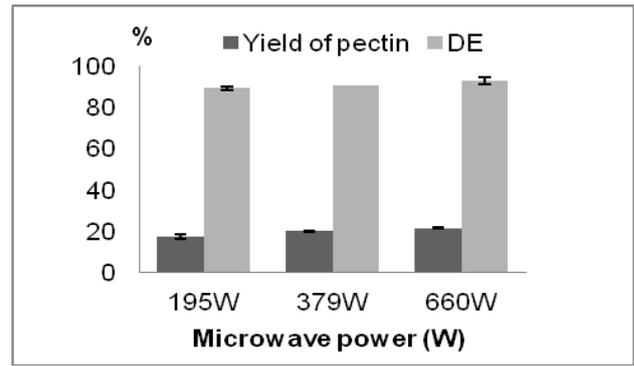


Figure 3. Effect of microwave power on the extraction yield of pectin and DE value

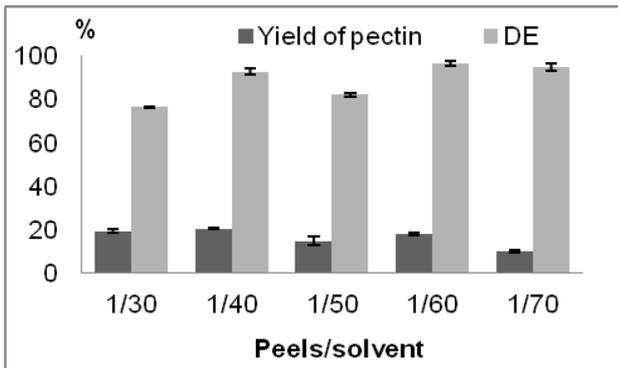


Figure 2. Effect of peels/solvent ratio on the extraction yield of pectin and DE value

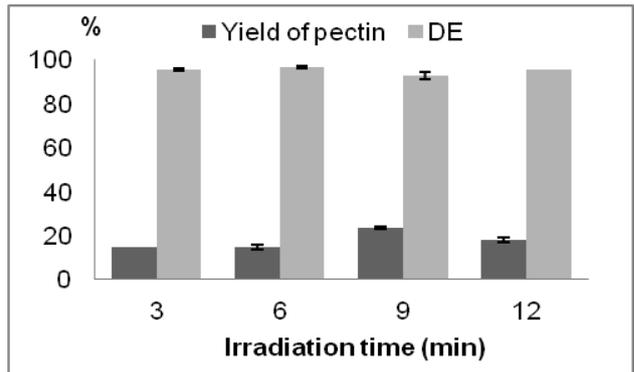


Figure 4. Effect of irradiation time on the extraction yield of pectin and DE value

increase the hydrolysis protopectin (Kertész, 1951). Besides, reducing pH value could promote the release of the pectin molecules from raw peel because the linkage of pectin with hemicellulose was separated (Rombouts and Thibault., 1996). Using pomelo, the content of pectin was higher than using from lemon, grapefruit, orange whose the content of pectin were respectively 28.7%, 22.9% and 19.3% with HCl solvent ratio of 1/60 at 80°C, for 1 hour and pH of 1.6 (Huang, 1973). The range of DE was greater than 50% (from 76.39 to 92.85%), so the type of pectin was HMP. Pomelo extracted pectin also had higher DE than lemon extracted one (DE = 64.15 ± 1.65%) with citric acid solvent (pH = 2), microwave operated at 628W at pH 2 and irradiation time of 9 minutes (Seixas et al., 2014). Thus, the optimum pH value at 1.5 was chosen for the next survey.

Effect of peels/solvent ratio on the extraction yield of pectin and DE value

There are significant differences at $p_{value} = 0.05$ from peels/solvent ratios (w/v). At the peels/solvent ratio of 1/40, the extraction yield obtained its highest value (20.44%) and the DE value was 92.75% (Figure 2). The use of large amounts of water would reduce the viscosity of the solution, so extraction process was more efficient. In addition, the high concentration gradient of solvent/peels can impulse the extraction

speed (Coulson and Richardson, 1978). However, this ratio was quite low, the concentration of pectin in the extraction solution was also low. The water in solution increased, thus the alcohol volume for precipitating pectin also increase dramatically and this would not be effective economy (El-Nawawi and Shehata, 1987). The obtained results were higher compared the study conducted by Zhou *et al.* (2010). In their research, pectin was extracted from pomelo using tartaric acid as a solvent and conventional heating. The content pectin was 18.73% at pH of 1.5, 90°C, 60 minutes and the material/solvent ratio of 1/15.

Effect of microwave power on the extraction yield of pectin and DE value

Theoretically, the microwave irradiation disintegrates wall of cells and cut off the soft tissues in the cell. Thus, the cells decays quickly due to electromagnetic energy (Bagherian *et al.*, 2011). As the microwave capacity increases, the solution temperature also increases. At the moment, the dielectric constant of water decreased and it related to the pectin soluble in water (Hoshino *et al.*, 2009). Rising temperatures promoted the extraction because the diffusion coefficient would increase (Coulson *et al.*, 1978; Treybal, 1980).

There are significant differences at $p_{value} = 0.05$

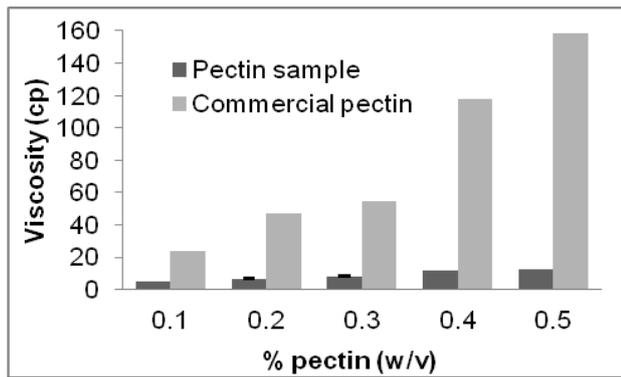


Figure 5. Comparing viscosity between pectin sample and commercial pectin

from microwave powers, the highest pectin content was 21.66% at 660W and the lowest pectin content was 17.44% at 195W. DE values were approximate 89.23 - 92.75% (HMP) (Figure 3). Thus, the irradiation power at 660 W was the optimum value in this survey.

The yield achieved the highest value at the highest microwave power. It was consistent with the study by Bagherian *et al.* (2011), this results was higher than one from Liang *et al.* (2011) (18.73%), which combined tartaric acid and microwave to extracting pectin from orange peel at pH 2.0, 280W, irradiation time of 5 minutes and the material/solvent ratio of 1/10.

Effect of irradiation time on the extraction yield of pectin and DE value

There are significant differences at $p_{\text{value}} = 0.05$ from irradiation time, the highest pectin content was 23.83% at 9 minutes and the lowest pectin content was 14.72% at 6 minutes. DE values were approximate 92.75 - 96.7% (HMP) (Figure 4). Thus, the irradiation time at 9 minutes was the optimum value.

Extraction time was very important to extract pectin; if the extraction process was extended for long time, the pectin content can be destroyed by high temperatures (Vo and Luong, 2010). Besides, increasing the extraction time resulted pectic acid by product which was hydrolyzed by soluble pectin. Thus it reduced extraction yield of pectin. When extraction time was short, the links between pectin with other components such as cellulose, hemicellulose could not be severed, protopectin was also hydrolyzed to soluble form. It could influence the extraction efficiency (Kertest, 1951; Albersheim *et al.*, 1960).

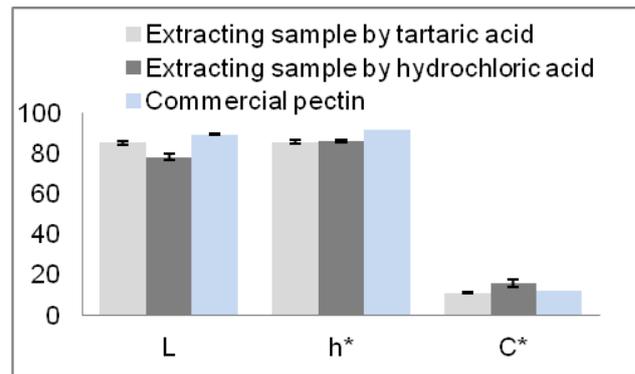


Figure 6. Comparing some parameters of color between pectin samples

Properties of pectin sample

Viscosity

Pectin solution is a non Newtonian fluid but it can become the Newtonian fluid at the low concentration ($< 0.5\%$, w/v) (Voragen *et al.*, 1995). The pectin concentration and the viscosity value are directly proportional (Figure 5). The viscosity of sample was much lower than commercial pectin and this viscosity was similar with result of Quoc *et al.* (2014) ($< 20\text{cp}$), extracting pectin from pomelo peel used oxalic acid as a solvent and combined with microwave.

Color of pectin samples

There are significantly differences ($p_{\text{value}} < 0.05$) from L, h^* and C^* between the three kinds pectin (extracted pectin using tartaric acid, extracted pectin using hydrochloric acid, and commercial pectin). The L^* value of received pectin (85.19) was higher than pectin extraction by HCl (78.13) (control sample) and lower than commercial pectin (89.37). Conversely, the C^* value of received pectin was the lowest value (10.96) (Figure 6). Pectin product in study was bright white color similar the commercial pectin.

Conclusions

In this study, acid tartaric and microwave heating affected strongly to the pectin extraction and its properties. The optimum condition to extract pectin were at pH (1.5), rate of material/solvent (1/40), irradiation time (9 minutes), irradiation power (660W), the pure pectin peaked 23.83%. The obtained pectin was high methoxyl pectin (DE value was over 90%) and low viscosity. It can use in food industry, especially beverage and jam processing.

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