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Effect of S/L Ratio and Particle's Diameter on The Percentage of Extracted Polyphenol In The Extraction Polyphenol from Malang Apple Skin

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Abstract

The objective of this research was to extract polyphenol from Malang apple skin in a batch stirred tank by use methanol-HCl 1% as solvent. Beside it, this research also evaluated the impact of ratio of Solid-Liquid (S/L) and particle's diameter to percentage of extracted polyphenol.
The study was conducted in three stages, namely preparation of raw materials, extraction process, and results analysis. In the preparation of material, apple skins is withered for 24 hours, crushed (size reducted) and then screen to obtain the desired particle's size. The extraction process takes place in a batch stirred tank, which placed inside a water bath. In the extraction experiment, variation of ratio of Solid-Liquid are 7.5:100; 10:100; 15:100; 10:150; 10:200 (w/v) and average diameter of particles are 8, 12, 16, 20 and 24 mesh. The process of extraction polyphenol from apple skin for each ratio S/L and diameter of particles is run at temperature 70^{o}C during 4 hours. As much as 1 mL samples were taken every 30 minute interval. The polyphenol concentration measured using spectrophotometer.
The results showed that the polyphenol can be extracted methanol-HCl 1% from Malang apple skin well. The percentage of extracted polyphenol increased by decreasing diameter of particles at constants temperature, stirring speed and ratio of Solid-Liquid (S/L). The highest percentage is 12.93% for particle's diameter is 20 mesh. Increasing S/L also decreased the percentage of polyphenol. The highest percentage obtained at S/L 10:200(w/v) is 21.58%.

Keywords: batch stirred tank, extraction, Malang apple skin, particle's diameter, polyphenol, S/L

Introduction

Air which is polluted by motor vehicle fumes, cigarette smoke, polluted water, radiation of ultraviolet rays from sun and foods that containing unsaturated fats is a source of free radicals that exist around us. Free radicals can cause premature aging and source of various diseases, such as hardening of blood vessels, coronary heart, stroke and cancer (Anonim, 2010).

According to the World Health Organization (WHO), every 11 minutes there is one of the world population died because of cancer and every 3 minutes there is a new cancer patients. The data of Department of Health in 2009 declare that around 6% or 13.2 million people Indonesia suffering from cancer and cancer is the 5th leading cause of death in Indonesia.

Free radicals are atoms or molecules that are highly unstable and highly reactive, and damage the tissues. This free radical compounds can be formed due to the chemical processes that occur in the body, such as oxidation, cell metabolism, excessive exercise and

inflammation (Anonim, 2010). One way used to counteract free radicals by consuming foods that contain lots of antioxidants such as vegetables and fruits. Apples are fruits that contain lots of antioxidants.

Based on the research, apples can reduce the risk of colon cancer, prostate cancer, and lung cancer. Compared with other fruits and vegetables, apples contain vitamin C which is not much, but rich in other antioxidant compounds. Many antioxidant compounds of polyphenol contained in apples is 0.68% (Alberto, 2006).

Polyphenol is one type of bioflavonoid antioxidant products which very strong and powerful in counteracting free radicals. Various studies and research has shown that free radicals are the major cause of cancer, cholesterol, diabetes, heart disease and stroke. Thus, polyphenols are necessary to prevent or overcome these diseases (Nurhasim, 2011).

According to Halliwell and Gutteridge (1999), there are a variety of herbs, vegetables and fruits that contain polyphenolic compounds, such as: red wine, green tea, duwet fruit and apple skin (Widowati et al, 2010). In this study selected Malang apple skin as a raw material for the extraction of polyphenol because had greater levels of polyphenol than Washington apple peel. The apple skin is a waste that is not widely used, so that availability is more secure than red wine, green tea and duwet fruit (Alberto et al, 2006).

Apple (Malus sylvestris Mill)

Apple is a plant that grows in subtropical región. In autumn season, the plants will shed their leaves and during the winter they will rest. In the spring season the plants grow together with flower buds (Rahardi, 2004)

The nutrient contents in 100 grams of apples are 58 kcal of energy, 4 g fat, 3 g protein, 14.9 carbohydrates, 900 IU vitamin A, 7 mg of thiamine, 1 mg riboflavin, 2 mg of niacin, 5 mg of vitamin C, 0, 04 mg of vitamin B, 6 mg calcium, 3 mg iron, 10 mg of phosphorus and 130 mg of potassium (Zia, 2009).

Total antioxidant activity of the apple skin is around 83 moles of vitamin C, which means that the antioxidant activity of 100 grams of apples is proportional to 1500 mg of vitamin C (Mey, 2009). Antioxidant activity is affected by the physical properties of the apple itself; opaque-skinned apple have polyphenols two times more than which light-skinned apples (Lola, 2008). Other factors that influence the content of polyphenols in apple skin include (Mey, 2009): the variety, the time and method of harvesting, storage, and the withering process.

Polyphenol

Polyphenol is chemical compounds contained in the plant and be a powerful antioxidant. This substance has a distinctive mark that has a lot of the phenol group in the molecule (Diastuti, 2009). Polyphenol is a group of chemical compounds derived from the bark, roots, leaves and fruit of the plant, the structure of polyphenol (Kumala, 2010):

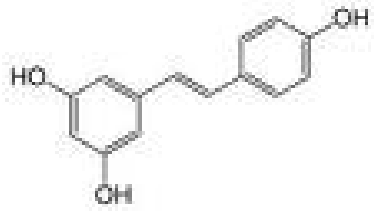

Fig.1 Polyphenol Compound Structure

Polyphenol is a group antioxidants that naturally occurring in vegetables (broccoli, cabbage, celery), fruit (apples, pomegranates, melons, cherries, pears, and strawberries), nuts (walnuts, soybeans, peanuts), olives oil, and beverages (such as tea, coffee, chocolate and red wine) (Diastuti, 2009). Higher concentration of polyphenols can be found on the skin of the fruit, such as grapes, apples, and oranges (Nuryanti, 2010).

Extraction

Extraction is a process in wich a material dissolved in one liquid phase is transferred to a second liquid phase by using solvent. The basic of separation is the difference of component solubility in the solvent. The extraction that involved solids is often called Solvent Extraction, Washing or Leaching. This extraction process is influenced by several factors, namely temperature, contact time, and the ratio of feed to solvent (Brown et al, 1950).

Solid-liquid extraction, also known as leaching is a process of taking soluble component (solute) in a solid using a solvent (Treybal, 1980). The solvent used must be able to extract the desired substance without dissolving other material. The interaction between the components of dissolved solids is very influential in the extraction process. In the extraction process, the dissolved components trapped inside solids, moving through the pores of the solid. Solute diffuses out of the surface of the solid particles and moving film layer around solids, further to the solution (Coulson and Rhichardson, 2002).

Rate of extraction also depends on several factors, namely (Coulson and Richardson, 2002):

a. Temperature

   High temperatures tend to increase the rate of extraction, but the too high temperature causing the desired components evaporate.

b. Particle's surface area

   Increasingly surface area of the particles, the extraction process can run more easily because of the greater possibility of particles to collide with each other.

c. Ratio of Solid and Liquid (S/L)

   The smaller ratio of solute and solvent used in extraction process, rate of extraction increases because of increasing the movement of particles in a solvent

d. Speed and duration of stirring

   High stirring speed will increase turbulence so that extraction can take place more quickly.

In the extraction process of solute (polyphenol) from the solid phase (apple skin) contained several stages, stage of solute diffusion in solids to the surface of a solid, the next stage of phase equilibria and phase of mass transfer from the solid surface to the solvent (Sudarmi and Siswanti, 2011).

Methodology

Research Materials

The materials used in this study are aquabidest, HCl, apple skin, methanol, Na_2CO_3 and Folin Cioteu reagent.

Research Tools

Extraction of polyphenols from Malang apple skins run in a batch stirred extractor (1000 mL glass beaker), equipped with a water bath to keep the operating temperature. In addition, also installed thermometer to ensure that the operating temperature in accordance with the design.

Research Procedures

10 grams of withered apple skin for 24 hours first, then blended and sieved using a screen with a certain particle size (8, 12, 16, 20 and 24 mesh). After that, the skin of apples mixed with methanol-1% HCl (7:3) as much as 100 mL. Then a mixture of apple skin and solvent poured into the beaker and extracted with stirring speed of 500 rpm. Extraction of polyphenols from apple skins for each rate of stirring is done at 70 ° C for 4 hours. As much

as 1 mL samples were taken every 30 minutes interval. Repeating the experiment for the variation of the S / L are 7.5:100; 10:100; 15:100; 10:150; 10:200 (w / v), with a particle diameter of 20 mesh.

Product Analysis

1 mL of samples diluted with the same solvent is methanol-HCl 1% (7: 3) until 50 mL. Then each 1 mL sample was taken and added to 2 mL of Folin Cioteu reagent 10% polyphenols allowed to stand 8 minutes, then added 2.5 mL of 7.5% Na_2CO_3 solution and allowed to stand for 30 minutes, and then absorbance was measured with a spectrophotometer at a wavelength of 740 nm. Spectrophotometer used was a GENESYS 2 spectrophotometer with a wavelength range of 325-1100 nm, the stability of ≤ 3 mA / hour drift, wavelength accuracy of ± 0.003 A and 3.4 mL volume of cuvette.

Variables Research

Fixed Variable

The study was conducted at 70 ° C, with a mass of sample is 10 grams, stirring speed of 500 rpm within 4 hours. Used methanol-HCl 1% (7: 3) as a solvent.

Free Variables

a. Particle sizes of 8, 12, 16, 20 and 24 mesh.
b. Ratio of solid-liquid (S/L), among others 7.5:100; 10:100; 15:100; 10:150; 10:200 (w / v).

Result and Discussion

Effect of Solid-Liquid Ratio (S/L) to The Percentage of Polyphenols in Solvents

Effect of solid-liquid ratio (S/L) to percentage of extracted polyphenol is shown in Table 1 and Figure 2. From Table 1 and Figure 2 can be seen that the greater number of solid (apple skin), the concentration of polyphenols decreased. It can be explained that if the number of solvent is constant and the number of solid decreased, means the polyphenols dissolved in solvent will decrease because the polyphenols in equilibrium (solid-liquid). so polyphenols from the solids can not be transferred into the solvent again. Highest percentage of polyphenols obtained when 7.5:100 ratio (w / v) is equal to 15.48%. However, in that ratio, concentration of extracted polyphenols smaller compared to the ratio of 10:100 and 15: 100.

While, the percentage of extracted polyphenol on S/L ratio variation can be seen in Table 1 and Figure 3. Highest percentage of polyphenol obtained when the ratio of 10:200 (w / v) is equal to 21.58%. In fact, this ratio has the smallest concentration of extracted polyphenol

when compared with the ratio of 10:100 and 10:150 (w / v). The concentration of extracted polyphenol is inversely proportional to the percentage of the extracted polyphenols due to the amount of solvent used is different (solid constant). This shows that the more solvent used in the extraction, the levels of polyphenol extracted in the solvent will be greater. This is according to research by Inayah (2007) who study the effect of solvents on the yield of oleoresin turmeric oil (1:4; 1:5; 1:6 (w / v)), and generating optimum conditions at a ratio of 1: 6.

Table 1. The Percentage of extracted Polyphenol on Various Solid-Liquid Ratio

No	Extraction Time (minutes)	Percentage of Extraction Polyphenol (%)				
		7.5:100	10:100	15:100	10:150	10:200
1.	30	9.86	10.62	6.47	8.72	11.63
2.	60	12.83	11.01	7.667	9.68	12.90
3.	90	15.33	11.18	7.99	11.01	14.68
4.	120	14.23	11.29	8.13	11.54	15.39
5.	150	14.95	11.30	8.46	13.39	17.85
6.	180	14.86	12.35	8.47	14.99	19.98
7.	210	14.91	12.56	8.51	16.31	21.75
8.	240	15.48	12.93	8.51	16.18	21.58

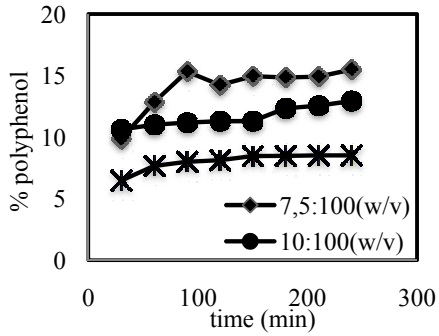

Fig. 2 Percentage of Polyphenol on Various S/L Ratios

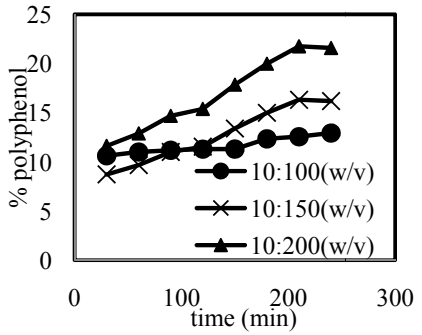

Fig. 3 Percentage of Polyphenol on S/L Ratio Variation

Effect of Particle's Diameter to The Percentage of Extracted Polyphenol

In this research, variation of particle size was performed 8, 11, 16, 20, and 24 mesh. Laboratory results showed the influence of the diameter of solids to the percentage extracted polyphenols can be seen in Table 2 and Figure 4. From Figure 4 can be seen that the percentage of extractable polyphenols increased with increasing extraction time and the small size of the particles. It can be explained that the longer extraction time, means the contact time between the solid particles and solvent are also getting longer, furthermore the percentage of extracted polyphenols also increase. Then, smaller solid particle size can

increase contact's surface area between solid particles and solvent.

The results showed that the percentage of polyphenol obtained are still quite small when compared to the study by Alberto et al (2006) which can extract polyphenol from apple skin by 68%. This significant difference is due to differences in pretreatment and treatment samples of the extraction process. In the study conducted Alberto et al (2006), apple skin before extracted freeze dried beforehand, whereas samples extracted concentrated in rotary evaporator at 35°C, then stored at -18°C to avoid hydrolysis reactions, redox , the polymerization reaction and direct contact with oxygen. Whereas in this study, as well as a sample of the raw materials are not being subjected to extraction as did Alberto et al (2006), so the probability of happening the reactions that can change the composition of the sample cannot be avoided.

Table 2. Percentage of Polyphenol on Various Particle's Diameters

No	Extraction Time (minutes)	Percentage of Extracted Polyphenol (%)				
		8 mesh	12 mesh	16 mesh	20 mesh	24 mesh
1.	30	1.97	3.01	3.31	10.62	6.55
2.	60	3.64	4.65	5.05	11.00	8.03
3.	90	4.41	4.68	5.41	11.18	9.73
4.	120	5.87	5.56	6.59	11.29	9.87
5.	150	6.01	6.30	8.07	12.18	10.01
6.	180	7.04	7.48	8.95	12.35	10.07
7.	210	8.11	8.54	9.55	12.56	10.38
8.	240	9.37	8.87	10.08	12.93	10.53

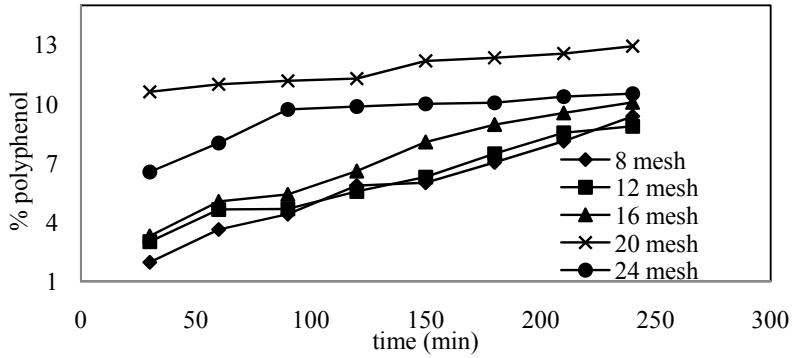

Fig. 4 Percentage of Polyphenol in Various Time and Particle's Diameter

Conclusions

1. Extraction of polyphenol from apple skin using methanol-HCl as a solvent at 70°C for 4 hours obtained the highest percentage of extracted polyphenol in S/L ratio of 10:200 (w / v), which is 21.58%.

2. The percentage of extracted polyphenol increased by decreasing diameter of particles at constants temperature, stirring speed and ratio of Solid-Liquid (S/L). The highest percentage is 12.93% for particle's diameter of 20 mesh.

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