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Utilization of Coffee Silverskin By-Product from Coffee Roasting Industry through Extraction Process for the Development of Antioxidant Skin Gel

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Synopsis

Coffee roasting industries generate a by-product called coffee silverskin that is usually disposed of as waste. The valorization of this abundant waste is necessary because of the antioxidant compounds in coffee silverskin. In this study, coffee silverskin was extracted in different extraction conditions to obtain an extract with high antioxidant activity and to use it as an additive for antioxidant skin gel. The extracts were characterized for the total phenolic content by using the Folin–Ciocalteu method. The antioxidant activity was determined by using the 2,2-diphenyl-1-picrylhydrazyl free radical scavenging assay. It was found that the extraction time and temperature strongly affected the total phenolic content and the antioxidant activity of the extracts. The extraction at 40°C and 60 min resulted in an extract with a high total phenolic content of 31.15 ± 2.77 mg Gallic Acid Equivalent/g coffee silverskin and a high antioxidant activity of 68.44 ± 0.76%. The extract solution was spray-dried to produce extract powder, which was then added to a basic skin gel with different extract concentrations. It was observed that the antioxidant activity of the gel increased with increasing extract concentration in the gel. This result showed that coffee silverskin has great potential as a source of antioxidants for various skin care products.

INTRODUCTION

Antioxidants are very important for the human body because they protect the human body against reactive oxygen species, known as free radicals, which can cause oxidative reactions in human cells. Although the human body has an internal defense system toward free radicals (1), it still requires the intake of antioxidants from outside that usually can be obtained from synthetic or natural products, such as fruits and vegetables, containing antioxidants (2). Recently, the interest in natural products for healthy foods, beverages, supplements, and health care products has been rising along with the increasing awareness of a healthy life style (3–5).

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One of the most popular and valuable natural products is coffee. Because of the high consumption of coffee as a beverage, the coffee industry is categorized as one of the largest industries in the world (6). Besides its good taste, coffee is beneficial for human health because many studies have shown that coffee contains phenolic compounds such as chlorogenic acids, which show antioxidant activity (7,8). The coffee industries also produce a large number of by-products, such as coffee spent grounds and coffee silverskin (9). Coffee silverskin is produced as a by-product during the roasting process of coffee beans. It is a thin layer surrounding the coffee bean, and it is usually still attached to the coffee bean even after the depulping and dehulling process of the coffee cherries. When the coffee beans are heated at high temperature during the roasting process, the coffee beans will crack because of loss of moisture content, resulting in the detachment of the coffee silverskin from the beans. Because of its lightness, the coffee silverskin escapes from the roasting container through the roaster exhaust or cyclone, and is usually disposed of as waste. However, recent studies on the revalorization of coffee silverskin reported that coffee silverskin contains phenolic compounds and shows high antioxidant activity (10–12). Bresciani et al. (13) reported that chlorogenic acids are the sole phenolic compounds in coffee silverskin. The studies on the extraction of coffee silverskin mostly used a mixture of Robusta and Arabica coffee silverskin because the roasting industry usually blends these two types of coffee. However, our recent study showed that Robusta coffee silverskin showed a higher phenolic content and, thus, a higher antioxidant activity than Arabica coffee silverskin (14). This result is in accordance with the study conducted by Farah et al. (8) who reported that Robusta coffee beans contain a higher amount of chlorogenic acid than Arabica coffee beans.

Rodrigues et al. (15) reported that coffee silverskin was an effective ingredient in the improvement of skin hydration and firmness. De Hond et al. (16) studied the use of coffee silverskin extract to protect accelerated skin aging. In spite of the high antioxidant activity of coffee silverskin and its great potential for industrial application, to the best of our knowledge, there is no study on the development of skin care products containing coffee silverskin extract as an antioxidant. Moreover, a proper extraction method and the optimization of the extraction condition to obtain an extract of coffee silverskin with a high phenolic content and a high antioxidant activity are not studied yet. Furthermore, the extraction of coffee silverskin was usually conducted by maceration process, which means the raw material is immersed in a solvent without any mechanical agitation. This conventional method takes a long time to extract the phenolic compounds, and results in a low phenolic content.

In this work, coffee silverskin of Robusta type was extracted using a hydroalcoholic solvent with the aid of mechanical agitation at various extraction times and temperatures with the purpose to maximize the phenolic content and the antioxidant activity of the coffee silverskin extract. The extract solution was then dried using a spray dryer to produce coffee silverskin extract in powder form. The study on the effect of drying of the extract is important because extracts are usually produced in powder form. Furthermore, to develop an antioxidant skin care product, a basic skin gel was prepared and the coffee silverskin extract powder was added as an active ingredient in the skin gel. The total phenolic content and the antioxidant activity of the skin gel were measured to study the effect of the addition of the coffee silverskin extract to the skin gel.

MATERIALS AND METHODS

MATERIALS

Robusta coffee silverskin was obtained from a coffee roasting company located in Bandar Lampung, Indonesia. Technical grade ethanol (96% v/v) was purchased from PT Sumber Abadi (Tangerang, Indonesia). Sodium carbonate (BDH, London, England), Folin–Ciocalteu reagent (Merck, Darmstadt, Germany), gallic acid (GA) powder (Aktin Chemical, Chengdu, China), aluminum chloride (Merck), potassium acetate (BDH), 2,2-diphenyl-1-picrylhydrazyl (DPPH, Sigma-Aldrich, St. Louis, MO), and analytical grade ethanol (Smart Lab, Bogor, Indonesia) were used. Chemicals to prepare the basic skin gel were carbomer 940, propylene glycol, methylparaben, and propyl paraben, all purchased from PT Intralab Ekatama (Bogor, Indonesia), whereas triethanolamine (TEA) and ethylene diamine tetra acetic acid (EDTA) were purchased from PT Sumber Abadi. Distilled water was used.

EQUIPMENT

Standard laboratory equipment was used in this work, namely, hot plate (Cimarec, Waltham, MA), beaker glasses, Erlenmeyer flasks, magnetic stirrer, thermometer, volumetric glass (Pyrex, Corning, NY), water bath shaker (Memmert, Schwabach, Germany), micropipette (Eppendorf, Hamburg, Germany), micropipette tips, cuvette (Brand GMBH, Wertheim, Germany), rotary vacuum evaporator (IKA HB 10, Shanghai, China), spray dryer (BUCHI mini spray dryer B-290, Flawil, Switzerland), analytical balance (Ohaus PA214, Parsippany, NJ), UV-Vis spectrophotometer (Genesys 10-S, Waltham, MA), vortex (Vortex-Genie 2, St. Louis, MO), filter instrument, Whatmann filter paper 1001 125 (GE, Little Chalfont, Buckinghamshire, UK), desiccator, moisture content analyzer (Sartorius MA35, Goettingen, Germany), mixer (IKA Labortechnik, Staufen, Germany), pH meter (Lutron pH-208, Taipei, Taiwan), and viscometer (Brookfield DV-E, Lorch, Germany).

EXTRACTION PROCEDURE

First, the coffee silverskin sample was rinsed with water and dried in an oven at 40°C. The dried sample was milled using a mixer grinder to reduce the size. Then, 100 mL of water–ethanol mixture with a weight ratio of 50:50 was poured into a glass beaker and heated using a hot plate. The temperature was varied at 30, 40, 50, and 60°C. When the desired temperature of the solvent was reached, 2 g of coffee silverskin were poured into the solvent, and the extraction process was carried out by stirring using a magnetic stirrer at a speed of 350 rpm. Thus, the weight ratio of the coffee silverskin and water–ethanol solvent was 1:50. The beaker was covered with aluminum foil to prevent loss of solvent because of evaporation during heating. The extraction time was varied at 5, 10, 20, 30, 40, and 60 min. The extract solution was then filtered using a filter paper and stored in a refrigerator before analysis.

ANALYSIS OF TOTAL PHENOLIC CONTENT AND ANTIOXIDANT ACTIVITY

The total phenolic content was determined by using the Folin–Ciocalteu method. First, the Folin–Ciocalteu reagent solution was made by diluting concentrated Folin–Ciocalteu

reagent in distilled water at a ratio of 1:10. Sodium carbonate solution (7.5% w/v) was made by diluting 7.5 g of solid sodium carbonate with 100 mL of distilled water. For the standard curve of GA, a solution of GA was prepared by dissolving 0.1 g of solid GA in 100 mL of distilled water to obtain 1,000 mg/L of GA stock solution. Then the standard solutions of GA were made by diluting the GA solution into concentrations of 10, 20, 50, 70, 100, 250, and 500 mg/L.

Furthermore, the coffee silverskin extract solution was diluted with a weight ratio of 1:10, and 500 µL of the diluted extract solution was mixed with 2.5 mL of the Folin–Ciocalteu reagent solution and 2 mL of the sodium carbonate solution. The mixture was then mixed using a vortex mixer and incubated in the dark at room temperature for 1 h. After the incubation, the mixture was poured into a cuvette and directly checked by using a UV-Vis spectrophotometer at 765 nm to measure the absorbance. The total phenol content (TPC) was then calculated by using the following equation:

$$\text{Total Phenolic Content (mg GAE/L)} = \frac{\text{Abs} \times \text{DF}}{m}, \tag{1}$$

where Abs is the absorbance, m is the gradient of the GA standard curve, and DF is the dilution factor. The total phenolic content was expressed in mg of GA equivalent per liter extract solution [mg Gallic Acid Equivalent (GAE)/L] and then converted to mg GAE/g dry coffee silverskin (mg GAE/g CS).

To determine the antioxidant activity of the extract, the extract samples were tested using the DPPH free radical scavenging assay. A stock solution of DPPH (250 µM) was prepared by diluting 11 mg of DPPH powder in 20 mL ethanol (96% v/v). The stock solution was covered with aluminum foil and stored at a temperature of 4°C. Next, 100 µL DPPH, 50 µL sample, and 850 µL ethanol were mixed in a test tube. For the control, 100 µL DPPH, 50 µL distilled water, and 850 µL ethanol were mixed in a test tube. Furthermore, both test tubes were wrapped with aluminum foil and stored in a dark room for 30 min. The absorbance reading was performed using a UV-Vis spectrophotometer at a wavelength of 515 nm. The antioxidant activity was expressed as an inhibition percentage and calculated by using the following equation:

$$\text{Antioxidant Activity}(\%) = \frac{(A_c - A_s)}{A_c} \times 100\%, \tag{2}$$

where A_c is the absorbance of the control and A_s is the absorbance of the sample. The antioxidant activity (the free radical scavenging activity) obtained by this method was expressed as IC_{50} (in ppm), which means the concentration of the sample needed to inhibit 50% of the DPPH as the free radical.

STATISTICAL ANALYSIS

Statistical analysis of the data obtained in this work was performed by using analysis of variance and Tukey test. The difference among data of different samples was considered as significant if the probability was less than 0.05 ($p < 0.05$).

DRYING OF COFFEE SILVERSKIN EXTRACT

To produce coffee silverskin extract in powder form, the coffee silverskin extract solution was dried using a spray dryer with an inlet temperature of 175°C and an outlet temperature of 125°C with a feed flow of 16.7 mL/min. The coffee silverskin extract powder was then analyzed for its total phenolic content and antioxidant activity using the method as previously described.

PREPARATION OF COFFEE SILVERSKIN SKIN GEL

A basic gel was prepared by using the cold mechanical method as described by Schmolka (17) with some modifications. The composition of the ingredients for the basic skin gel is shown in Table I (18).

First, Carbomer 940 was dissolved in distilled water in a glass beaker and stirred using a mixer to obtain a gel. The mixing process was carried out at room temperature at 600 rpm for 20 min. To reduce the bubble, the gel was kept for 24 h at room temperature. TEA was then added to the gel and mixed. Methylparaben and propylparaben were dissolved in propylene glycol, whereas EDTA was dissolved in distilled water by stirring. Then, these solutions were added into the gel and stirred until a homogeneous mixture was attained. Finally, the coffee silverskin extract powder which was already dissolved in distilled water, was added into the gel, and stirred at room temperature for 20 min. The concentration of the coffee silverskin extract powder in the skin gel varied at 0.125%, 0.25%, 0.5%, and 1%. The total phenolic content of the skin gel was analyzed using the method described previously. The antioxidant activity of the skin gel was also analyzed and expressed as IC_{50} value using the method described previously. The pH of the gel was measured using a pH meter, whereas the viscosity was measured using a viscometer using spindle number 6 at a speed of 3 rpm. All measurements were carried out at room temperature.

RESULTS AND DISCUSSION

TOTAL PHENOLIC CONTENT AND ANTIOXIDANT ACTIVITY OF COFFEE SILVERSKIN EXTRACT SOLUTION

The coffee silverskin sample was extracted using water–ethanol (50:50 w/w) as solvent at different extraction times and temperatures. Water–ethanol mixture with a weight ratio

Table I
Composition for the Basic Skin Gel

Ingredient	Composition (g/100 g water)
Carbomer 940	1
TEA	1
Propylene glycol	15
Methylparaben	0.15
Propylparaben	0.05
EDTA	0.05
Ethanol (96% v/v)	5
Perfume	0.4
Distilled water	100

of 50:50 was chosen as the solvent because of the intermediate polarity of the phenolic compounds Ballesteros et al. (12). The extract solutions were then analyzed for their total phenolic content and antioxidant activity. Figures 1 and 2 show the effect of extraction time on the total phenolic content and the antioxidant activity of the coffee silverskin extract solution. The extraction was carried out at a constant extraction temperature of 40°C. As shown in Figure 1, a longer extraction time resulted in a higher amount of total phenolic content of the coffee silverskin extract solution. Because phenolic compounds are known to have antioxidant property, the antioxidant activity increased with increasing extraction time, as shown in Figure 2. The extraction time of 60 min showed the highest total phenolic content and antioxidant activity compared with the other extraction times.

To study the effect of extraction temperature, the extraction experiment was carried out at different extraction temperatures at a constant extraction time of 60 min. Figures 3 and 4 show the effect of extraction temperature on the total phenolic content and the antioxidant activity of the coffee silverskin extract solution. When the extraction temperature was elevated from 30°C to 40°C, an increase in the total phenolic content was observed. However, a further increase in temperature above 40°C did not result in a significant increase in the total phenolic content as shown in Figure 3 ($p > 0.05$). A similar phenomenon was also observed for the antioxidant activity as shown in Figure 4. An increase

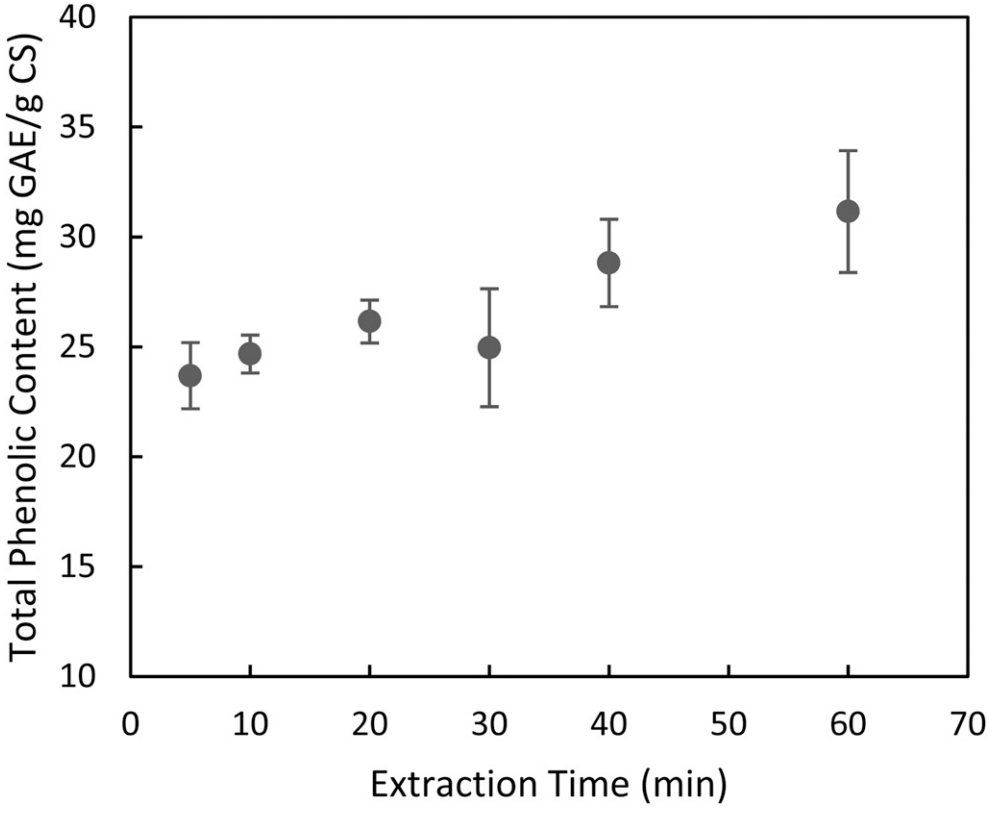

Figure 1. Effect of extraction time on the total phenolic content of the coffee silverskin extract solution (extraction temperature: 40°C).

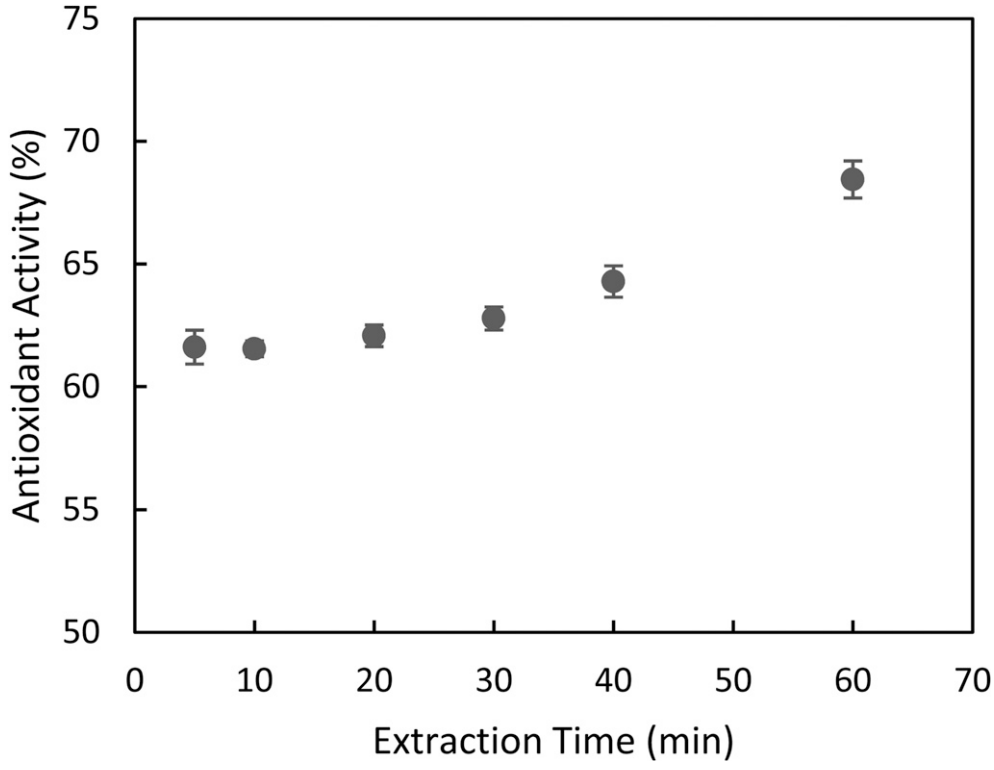

Figure 2. Effect of extraction time on the antioxidant activity of the coffee silverskin extract solution (extraction temperature: 40°C).

in the extraction temperature above 40°C did not result in a significant increase in the antioxidant activity of the extract ($p > 0.05$). This result indicated that all phenolic compounds were completely extracted from the coffee silverskin at an extraction temperature of 40°C and an extraction time of 60 min. This is very interesting because an extraction temperature of 40°C is appropriate for the industrial scale extraction process because of the lower energy consumption for heating.

COFFEE SILVERSKIN EXTRACT POWDER

Based on the aforementioned results, coffee silverskin extract in powder form was prepared by drying the coffee silverskin extract solution using a spray dryer. First, the extract solution was prepared by extracting the coffee silverskin at 40°C for 60 min because this extraction condition resulted in a high total phenolic content and a high antioxidant activity. Then, the filtered extract solution was dried using a spray dryer at an inlet temperature of 170°C and an outlet temperature of 125°C. Figure 5 shows the picture of the extract solution before drying and the extract powder after drying.

The measurement of the moisture content showed that the dried coffee silverskin extract powder had a moisture content of 5.6%. The analysis of the total phenolic content using the Folin–Ciocalteu method with GA as a standard showed that the coffee silverskin

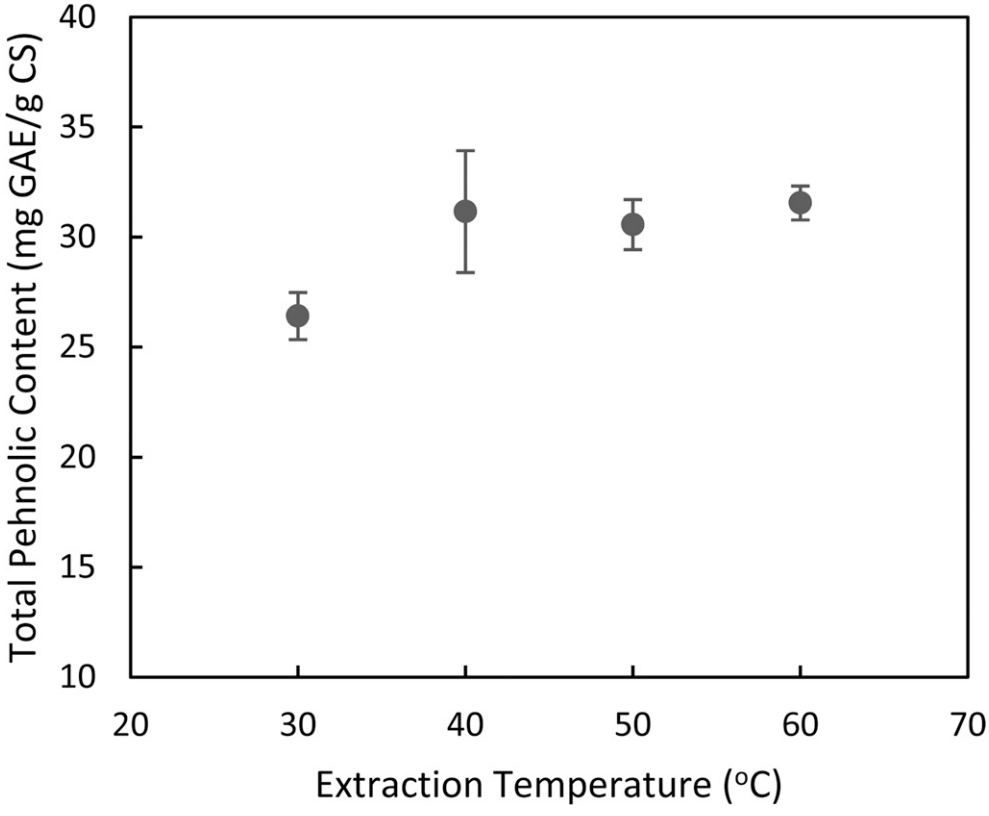

Figure 3. Effect of extraction temperature on the total phenolic content of the coffee silverskin extract solution (extraction time: 60 min).

extract powder had a total phenolic content of 188.83 mg GAE/g solid extract, which is slightly lower than that of the extract solution before drying of 210.27 mg GAE/g solid extract, as shown in Table II. The decrease in the total phenolic content after drying occurred because the extract was treated at a high temperature of 170°C during the spray drying process. The heating of the extract degraded some phenolic compounds, resulting in a lower phenolic content of the dried extract. A similar phenomenon was also reported by Orphanides et al. (19) who studied the effect of drying on the phenolic content and antioxidant capacity of spearmint. The effect of drying on the antioxidant activity can be seen in Table II. The antioxidant activity of the extract is expressed as IC_{50} value, which means the concentration of the sample needed to inhibit 50% of DPPH as the free radical. Thus, a low value of IC_{50} corresponds to a high antioxidant activity. As can be seen, the IC_{50} value of the extract powder was 433.64 ppm, higher than that of the extract solution of 358.15 ppm. This result is in accordance with the decrease in the total phenolic content after the drying process as described earlier.

SKIN GEL CONTAINING COFFEE SILVERSKIN EXTRACT

The basic skin gel was prepared using the formulation and the method described previously. The coffee silverskin extract powder was then added to the basic skin gel and mixed

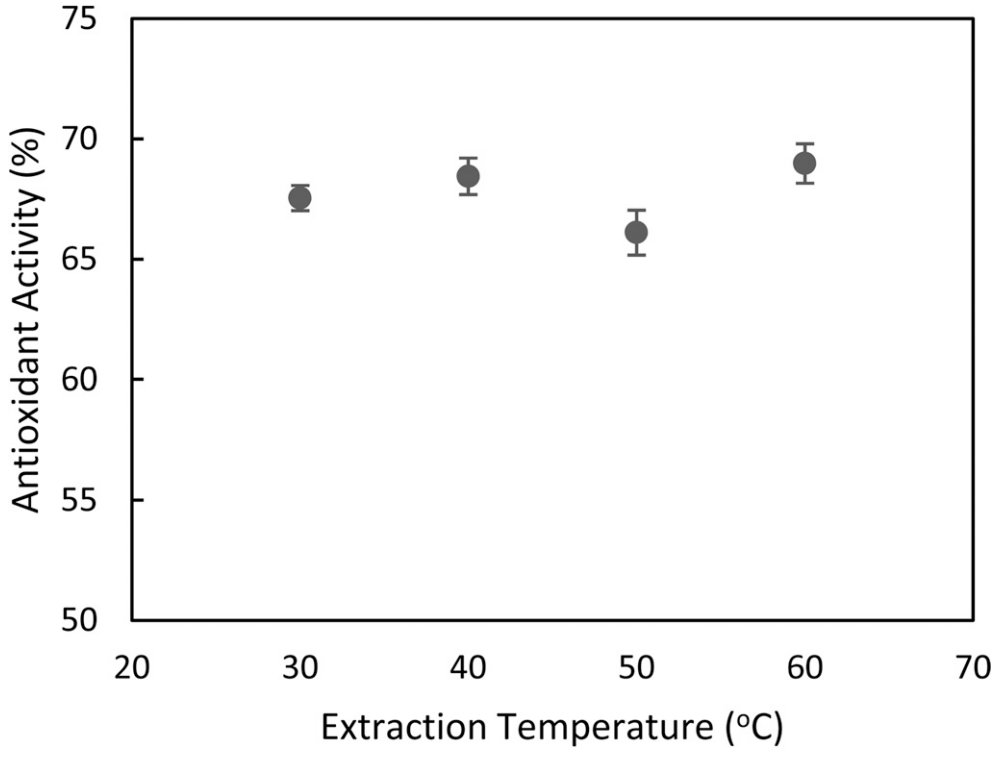

Figure 4. Effect of extraction temperature on the antioxidant activity of the coffee silverskin extract solution (extraction time: 60 min).

together at room temperature to obtain a homogeneous gel. The concentration of the coffee silverskin extract powder in the skin gel varied at 0.125%, 0.25%, 0.5%, and 1%. Figure 6 shows the physical appearance of the skin gels with different extract concentrations.

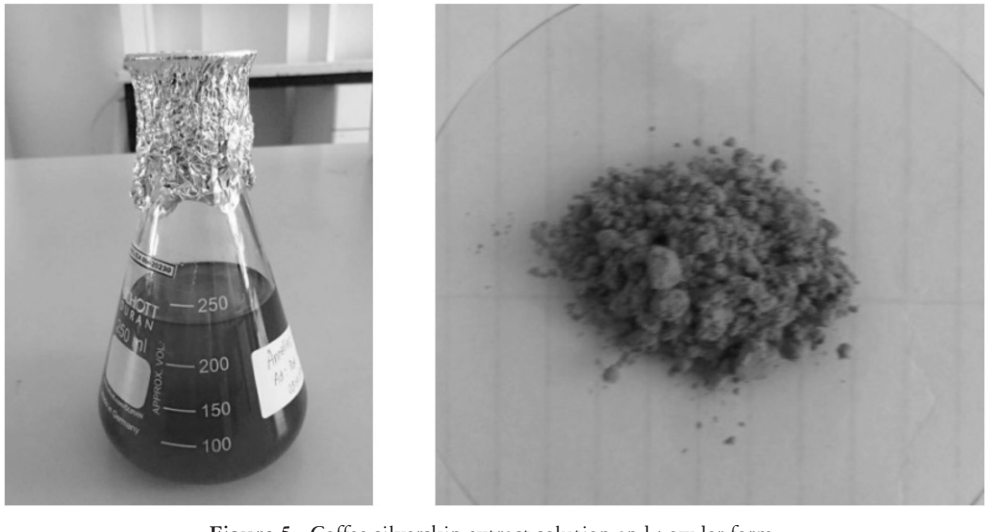

Figure 5. Coffee silverskin extract solution and powder form.

Table II
Total Phenolic Content and Antioxidant Activity of Coffee Silverskin Extract before and after Drying

	Coffee silverskin extract solution (before drying)	Coffee silverskin extract powder (after drying)
Total phenolic content (mg GAE/g solid extract)	210.87	188.83
IC_{50} (ppm)	358.15	433.64

The color of the skin gel became darker when more extract powder was added to the gel. The gel viscosity ranges between 98,300 and 109,500 cps, with pH ranges between 6.5 and 7.0.

Figure 7 shows the total phenolic content of the skin gels with various extract concentrations. As can be seen, the total phenolic content in the skin gel increased by adding more coffee silverskin extract to the skin gel. The addition of the extract of more than 1% is still possible to obtain a skin gel with higher total phenolic content, but because the color of the skin gel will become darker, the color acceptance by the users should be considered.

To study the effect of the addition of the coffee silverskin extract on the antioxidant activity of the skin gel, an analysis using DPPH free radical scavenging assay was conducted. It was observed that the basic skin gel that contains no coffee silverskin extract exhibited a DPPH inhibition percentage of only 3% because of the oxidation of the bulk skin gel by the DPPH. Interestingly, the skin gel containing 0.125% coffee silverskin extract exhibited a DPPH inhibition percentage of 26.3%, indicating that the coffee silverskin extract was effective as a source of antioxidants in the skin gel. Further, Figure 8 shows the effect of the addition of the coffee silverskin extract on the antioxidant activity

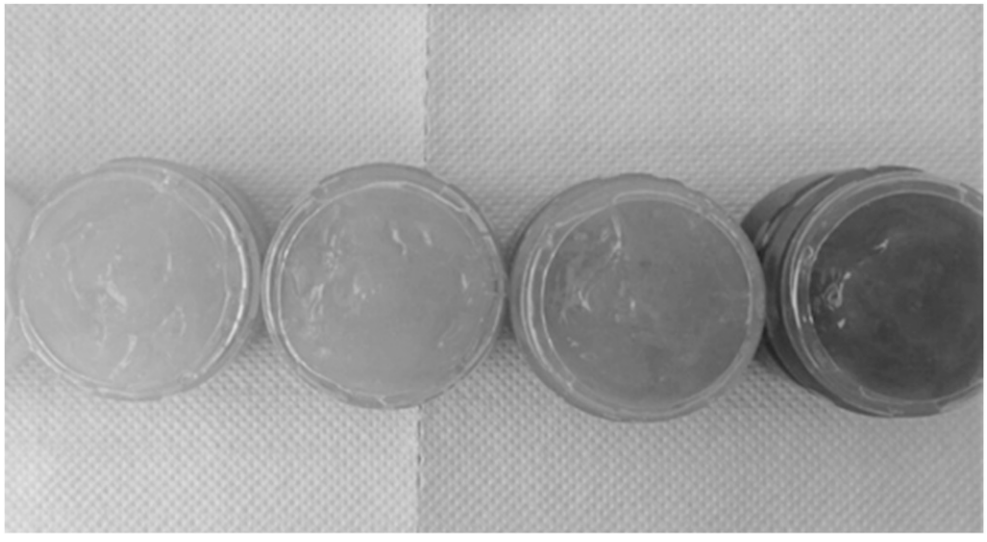

Figure 6. Physical appearance of skin gels containing coffee silverskin extract at different concentrations (left to right: F1: 0.125%, F2: 0.25%, F3: 0.5%, F4: 1%).

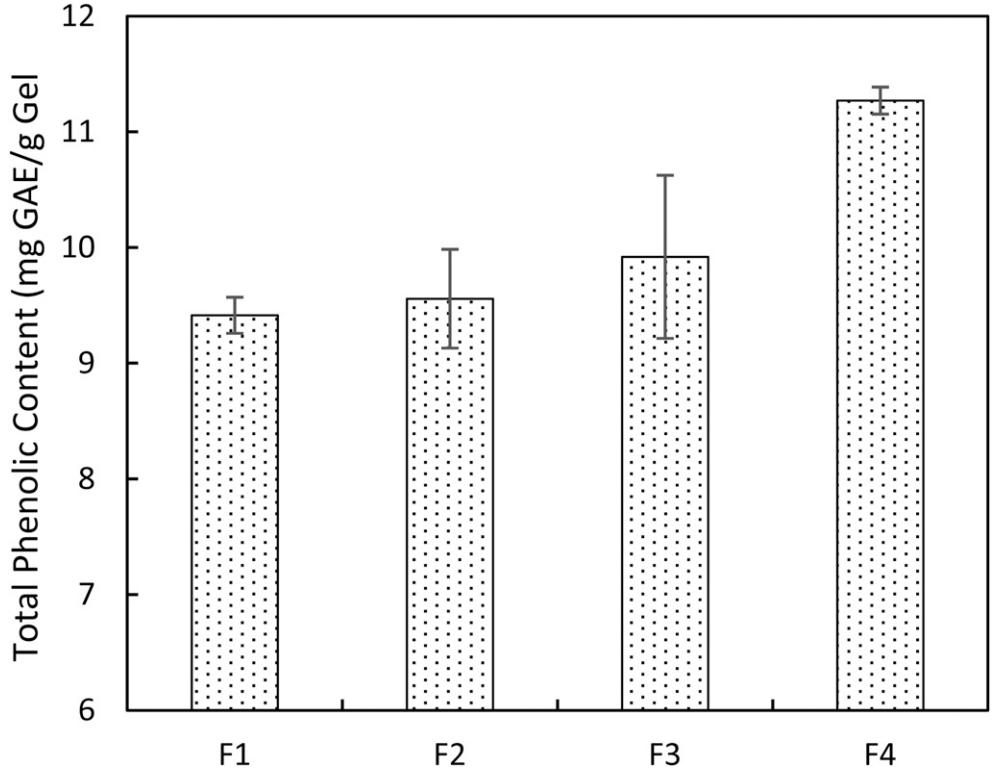

Figure 7. Total phenolic content of skin gels with various concentrations of coffee silverskin extract (F1: 0.125%, F2: 0.25%, F3: 0.5%, F4: 1%).

expressed by the IC_{50} value of the skin gel. The IC_{50} value indicates the concentration of the antioxidant, which is necessary to inhibit 50% of the DPPH as the free radical. The IC_{50} is given in ppm, which means milligram skin gel per liter solution. As can be seen in Figure 8, the addition of more coffee silverskin extract reduced the IC_{50} value, which means it resulted in a skin gel with a higher antioxidant activity. The increase in the antioxidant activity of the skin gel is correlated with the increase in the total phenolic content of the skin gel due to the addition of the coffee silverskin extract. The statistical analysis showed that there is a significant difference of IC_{50} with the variation of the extract concentration ($p < 0.05$). This result indicates that the coffee silverskin extract has great potential to be used as an additive for skin gels to obtain antioxidant-rich skin gel products.

The pH value of a topical preparation should be within the skin pH range between 4.0 and 7.0 (20). The value of the pH should not be too acidic as it causes skin irritation and should not be too alkaline as it may cause scaly skin. The skin gels containing coffee silverskin extract showed pH values of 7.0, 6.63, 6.50, and 6.50, for the extract concentrations of 0.125%, 0.25%, 0.5%, and 1%, respectively. The addition of more coffee silverskin extract into the skin gel resulted in a slight decrease in the pH value. This is reasonable because the phenolic compounds of the coffee silverskin extract are mostly chlorogenic acids, which lead to an acidic condition of the gels. However, the pH values of the skin gels are all still within the pH range for topical preparations.

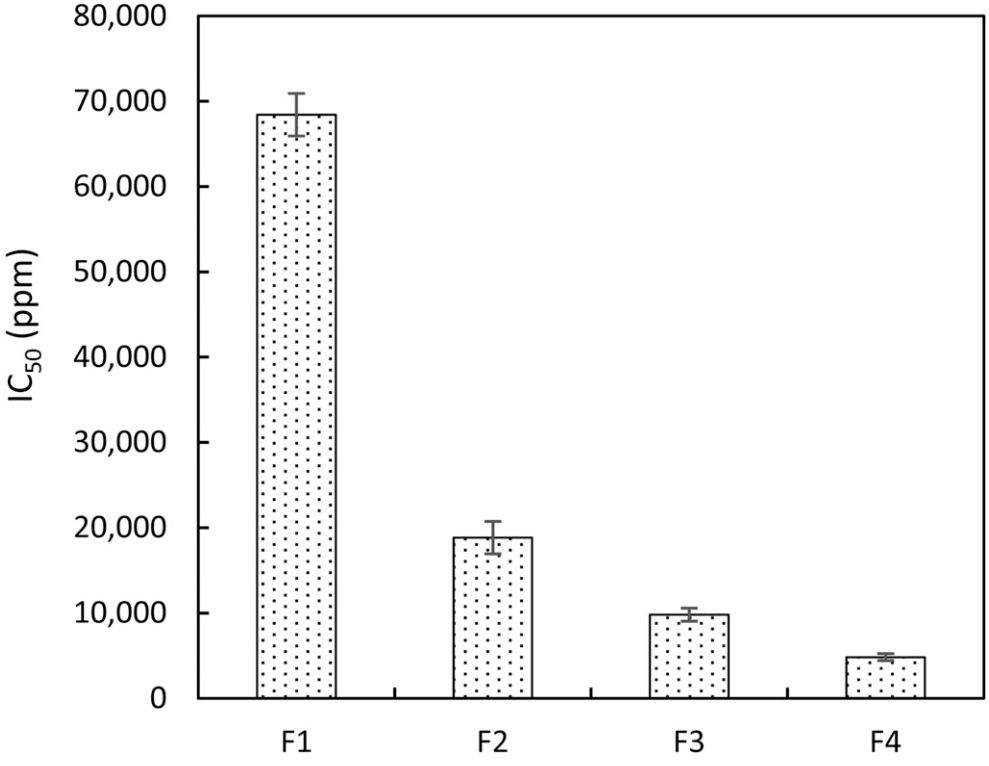

Figure 8. IC_{50} of skin gels with various concentrations of coffee silverskin extract (F1: 0.125%, F2: 0.25%, F3: 0.5%, F4: 1%).

CONCLUSION

This study demonstrated the utilization of coffee silverskin through an optimized extraction process to produce an extract with high total phenolic content and high antioxidant activity. It was observed that the extraction time and temperature strongly affected the total phenolic content and the antioxidant activity of the coffee silverskin extract. The extraction at 40°C and 60 min resulted in an extract with a high total phenolic content of 31.15 ± 2.77 mg GAE/g coffee silverskin and a high antioxidant activity of 68.44 ± 0.76%. It was found that the addition of the coffee silverskin extract powder to a basic gel resulted in a gel having antioxidant property. The antioxidant activity of the gel increased with increasing coffee silverskin extract concentration in the gel. The result of this study showed that coffee silverskin has great potential to be used as a source of antioxidant and the extract can be applied as an additive for various skin care products.

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