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Development of Skin Lotion Containing Antioxidant Extract from Coffee Pulp and Study on Its Stability

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Abstract. Coffee pulp, a major coffee waste in the coffee industry, has been found to contain bioactive compounds that can provide high antioxidant activity and hence, can be utilized as a natural antioxidant source. One of the beneficial utilizations of coffee pulp extract is its application in skin cosmetics, such as skin lotion. The application of antioxidant on the skin is one way to avoid skin damages and diseases from happening, which can be caused by free radicals generated through ultraviolet (UV) radiation from sunlight. The aim of this study is to apply the coffee pulp extract powder into skin lotion formulation, and to study the phenolic content, antioxidant activity and the stability of the formulated lotions. First, the coffee pulp (CP) was extracted at sample to solvent ratio of 1:10 (w/v) using ethanol/water mixtures at various extraction temperatures and times to obtain the optimum extraction condition. The resulting coffee pulp extract at optimum condition (50°C, 120 minutes) was pulverized into coffee pulp extract powder. The yield of the pulverization was 0.217146 g powder/g CP. The coffee pulp extract powder was then added into skin lotion formulation with four different concentrations (0.125 %, 0.25 %, 0.5 % and 1 % by mass). All coffee pulp skin lotions were found to show good physical stability and considerably higher antioxidant activity (IC₅₀ : 5,805 ppm – 55,776 ppm) compared to commercial lotions (IC₅₀ : 505,018 ppm – 557,218 ppm).

1. Introduction

Indonesia as a tropical country located at both sides of the equator has relatively long daytime duration per year, that causes the people to be often exposed to sunlight and essentially endangered by the amount of ultraviolet (UV) radiation reaches their skin. Exposure to chronic ultraviolet radiation can cause skin damage or serious diseases such as skin cancer [1], since free radicals can be generated through various interactions of UV with the surroundings. The application of antioxidant on the skin is one way to fight free radicals generated from ultraviolet radiation, which can avoid further skin damages from happening. Although human body already retains natural antioxidant coming from different source of external intake, applying antioxidant topically is important to protect the skin from UV radiation, dust, pollution etc. Hence, applying cosmetics products such as skin lotions and skin gels are the possible ways to provide antioxidant to the skin directly.

Nowadays, many cosmetics products use natural plant extract as the natural antioxidant source. Such extracts can be obtained from many plants, among other is coffee. Coffee is one of the most popular beverages consumed by Indonesian people. The coffee plants themselves are spread throughout several provinces of Indonesia, and it has been reported that Indonesia is a top coffee producer after Brazil, Vietnam and Colombia during the crop season in 2016-2017. Moreover, the amount of coffee produced in Indonesia in 2017-2018 is 636,000 tons of coffee cherries and it has



been estimated to increase by 30,000 tons in 2018-2019 according to Global Agricultural Information Network. Due to a large coffee cherries production, it also results in a large amount of coffee waste in the coffee industry. A high amount of coffee waste during the coffee processing, known as coffee pulp, has become a major problem in the coffee industry, since it represents 40 % of the whole coffee cherries by weight [2]. In the effort to find solution to this waste problem, several studies have found that coffee pulp still contains hydroxycinnamic acids, which have been identified as a natural antioxidant, such as caffeic acid, chlorogenic acid and ferulic acid. Coffee pulp has been found to be able to provide high antioxidant activity [3], hence it can be considered as natural antioxidant source.

Researches on the utilization of coffee wastes extract, coming from coffee silver skin and coffee pulp, as antioxidant source in cosmetic products have been thoroughly conducted. In previous studies, it was confirmed that the extract of coffee pulp and coffee silver skin show high antioxidant activity, hence is still very beneficial to be utilized [4] [5]. Efforts to apply coffee pulp extract on skin cosmetics have also been made. Coffee pulp extract has been applied to skin gel [6], while coffee silver skin extract was applied to skin lotion by Kusumocahyo [4]. However, the skin lotion was found to be unstable during the storage time, shown by a phase separation after certain period and after going through a mechanical test.

A comparison was also made between coffee silver skin and coffee pulp extract, which led to a result that the antioxidant activity of coffee pulp extract is higher than coffee silver skin [6]. Realizing this fact, this research was aimed at developing a skin lotion with coffee pulp extract as antioxidant source. The stability of the skin lotion is an important parameter to be studied, since it can be affected by the physicochemical properties of the skin lotion. Therefore, a stability study of the different formulations of skin lotion was also performed.

2. Methodology

The coffee pulp underwent a sequence of pre-treatment, consisted of washing with water, drying, milling to reduce the size and washing with n-Hexane by stirring for 1 hour. The extraction process of coffee pulp was divided into 2 phases. The first phase was extraction of coffee pulp at sample to solvent ratio of 1 : 40 and 1 : 10 (w/v) at 60°C for 60 minutes, by using a mixture of ethanol : water (50:50) (v/v) as solvent. In the second phase, coffee pulp was extracted at sample to solvent ratio of 1 : 10 (w/v) at various temperatures (Room temp., 40°C, 50°C and 60°C) and extraction times (40, 60, 90, 120, 180 and 240 minutes). The extract obtained from the second phase was further prepared to undergo spray drying process. The extract solution was filtered using vacuum filtration, then it was dried using spray dryer with inlet temperature of 175°C and outlet temperature of 125°C, at a feed flow rate of 17 mL/min to produce powder extract.

In the next stage of the research, the pulverized CP extract was used for the further process, which was skin lotion formulation. The coffee pulp extract was applied into the skin lotion formulation in the form of powder at various extract concentrations, namely 0.125 %, 0.25 %, 0.50 %, and 1.00 % CP powder extract by mass. During the storage time, the antioxidant activity and the physicochemical properties of skin lotion were analysed, while stability testing according to CTFA [7] was conducted. The physicochemical properties observed in this research include organoleptic, spreadability, pH measurement, homogeneity and viscosity.

3. Results and Discussion

A different sample to solvent ratio could affect the phytochemical characteristic of a sample. In this study, the phytochemical observed was the total phenolic content. Dewi [5] has discovered the optimum extraction condition for CP Extract solution with sample to solvent ratio of 1:40 (w/v). However, according to Tan, et al. [8], a sample to solvent ratio of 1:10 (w/v) could increase the total soluble solid (TSS) of the yielded extract, which would be more suitable for a spray drying process subsequently.

In this experiment, both sample to solvent ration were applied in the extraction of coffee pulp using a mixture of ethanol and water as the solvent. The result has shown that the TPC of CP extract

obtained through extraction using sample to solvent ratio of 1:40 (w/v) and 1:10 (w/v) was 53.378 ± 0.974 mg GAE/ g CP and 45.945 ± 0.795 mg GAE/ g CP respectively and the difference was statistically significant ($p < 0.05$). Even though the ratio of 1:10 (w/v) was found to be resulting in a lower TPC of the extract, the TSS obtained was much higher and has reached 36.106 %, which is sufficient for spray drying process subsequently without the need for additional filler. This ratio is found to be more beneficial to be used since it requires less solvent and less energy to evaporate the solvent completely prior to spray drying.

Table 1. Total Phenolic Content of CP Extract Solution at Various Condition

Sample to solvent ratio 1:10 (w/v)				
No.	Solvent	Temp. (°C)	Time (min)	TPC (mg GAE / g CP)
1		Room		48.872 ± 3.359
2		40	60	52.655 ± 0.795
3		50		55.704 ± 1.110
4		60		45.945 ± 0.79
5	Ethanol/Water Mixtures (50:50) v/v			40
6			60	33.463 ± 1.153
7		50	90	38.655 ± 0.760
8			120	54.427 ± 9.521
9			180	32.631 ± 1.269
10			240	33.993 ± 0.675

Since the ratio of 1:10 (w/v) was chosen, the selection of best extraction temperature and time was conducted. It was concluded from the results that the optimum extraction condition is 50°C and 120 minutes, when using ethanol/water mixtures (50:50) as the solvent. After the CP extract solution was prepared, the sample was spray-dried to change the form of the extract from liquid into powder form. The aim of the spray drying process was to make sure that due to the hot steam, the ethanol from the extract solution can be totally evaporated to make sure that the CP extract powder does not contain any ethanol anymore as it will be applied to the skin lotion formulation. The yield of the pulverization was 0.2171416 g powder/g CP.

Table 2. DPPH Inhibition and TPC of CP Extract Solution and CP Extract Powder

	Extract Solution	Extract Powder	% Reduction
DPPH Inhibition	926.003 mg DPPH/ g solid	734.960 mg DPPH/ g powder	20.63 %
TPC	170.595 mg GAE/ g solid	135.313 mg GAE/ g powder	20.68 %

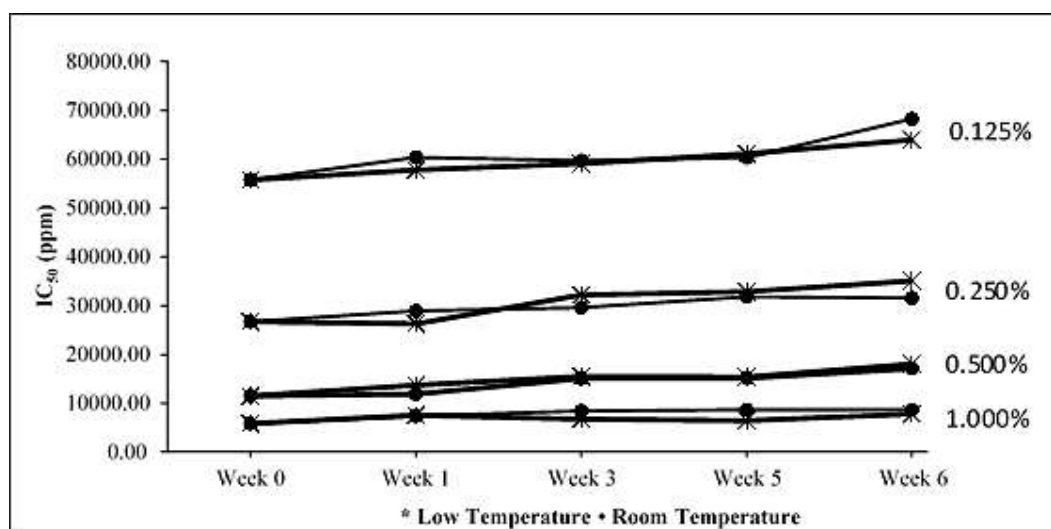
After CP extract solution was spray-dried, there was a degradation of the TPC in the CP extract powder when compared to its liquid form, due to the high temperature during spray drying process. Table 2 shows the data of DPPH inhibition and total phenolic content before and after spray drying. The reduction of the antioxidant activity and total phenolic content was due to the hot temperature by spray drying, which caused a theviscosityrml degradation in which a compound who was sensitive to heat such as ferulic acids, can be damaged. The IC_{50} value for extract solution and extract powder are 148.328 ppm and 308.853 ppm respectively.

Table 3. DPPH Inhibition of Coffee Pulp Skin lotion before and after CP extract powder was added

	DPPH Inhibition (%)
Basic lotion (without CP extract powder)	3.678
F2 (with 0.125 % CP Extract powder)	30.036
F3 (with 0.25 % CP extract powder)	69.602

In the formulation stage, four different concentrations of CP extract powder (0.125 %, 0.25 %, 0.50 %, and 1.00 % by mass) was added into the skin lotion formulation. Table 3 shows the %-inhibition of the skin lotion with and without coffee pulp extract powder. The DPPH inhibition was observed before and after adding the antioxidant compound into the skin lotion formulation. Before CP extract powder was added, the DPPH Inhibition was only 3.678 %, however, after adding 0.125 % and 0.25 % of CP extract powder into the formulation, the CP skin lotion could inhibit as much as 30.036 % and 69.602 % DPPH respectively.

The CP skin lotion was stored at room temperature and low temperature for 6 weeks. The antioxidant activity and physicochemical properties were analysed during storage. In both storage temperatures, the IC_{50} of the CP skin lotion was considered stable as it only showed slight increase from time to time, as there is no sudden change on the IC_{50} value. While the IC_{50} between week 0 and week 6 from each concentration increased insignificantly ($p > 0.05$), the CP extract powder concentration significantly affect the IC_{50} ($p < 0.05$). The higher the IC_{50} value indicates the smaller amount of CP extract powder in the lotion formulation.

**Figure 1.** IC_{50} of CP Skin Lotion stored at room temperature and low temperature for 6 weeks

Physicochemical Properties of CP Skin Lotion

The physicochemical properties, including organoleptic, pH, viscosity, spreadability and homogeneity of the CP skin lotion were analysed for 6 weeks to study their stability. Organoleptic is one of the physicochemical properties for semisolid preparations, to describe colour, consistency, and homogeneity. Organoleptic properties of a skin lotion could affect the aesthetics and comfort when applied, and are also related with the acceptability of the skin lotion. Through the observation on the lotions within a study period of 6 week, the colour did not visually show significant change in real condition.

Homogeneity will affect the effectiveness of the skin lotion. If an emulsion is homogeneous, the active ingredients will spread evenly. By applying a CP skin lotion to a certain area, there is an opportunity for the active ingredients to occupy the area applied. All skin lotion formulations from F1 to F5 from this study were examined, and after 6 weeks all of the formulations were still found to be homogenous and no coarse particle was felt when rubbed on skin, nor a phase separation could be observed during this time period.

The CP skin lotion acidity is one of the important physical characteristics in the formulation, because it could affect drug solubility, activity, absorption, stability, and patient comfort [9]. The pH measurement was conducted on CP skin lotion, as it is important to ensure that the lotion is in a tolerable range of pH to be applied topically on skin, to avoid any irritation. According to SNI 16-4399-1996 a good and safe skin lotion should have pH around 4.5 – 8. If it was below 4.5, it would be too acidic and could cause skin irritation, and if it was above 8, it would be too alkali which could cause the skin to become scaly. During the stability testing conducted in 6 weeks period, all skin lotion formulations with different amounts of CP extract added, have shown a stable pH condition in the range of 6.6 – 7.34 when stored at room temperature, and in the range of 5.62 – 7.1 when stored in refrigerator.

Viscosity is one of the parameters of physical properties in semisolid preparations. The viscosity determination could determine the tendency of a semisolid preparation to breaks into oil phase and water phase. According to Stoke Law, the time to reach phase separation of an emulsion is inversely proportional to the viscosity. The higher the viscosity of an emulsion, the stable the emulsion is as the time to reach phase separation is longer. Furthermore, viscosity of an emulsion related to the therapeutic effects and comfort in use. CP skin lotions formulated in this research have a viscosity that is not too thick nor too damp, and is within the range of viscosity regulated by Standar Nasional Indonesia (SNI), which is 2,000-50,000 cP. After six weeks of stability testing, the viscosity of all skin lotion formulation was found to remain within the regulated range, which was 3,733 – 9,933 cP for storage at room temperature, and between 4,333 cP and 8,800 cP for storage in refrigerator.

Table 4. Comparison of skin lotion properties

	SNI	CP Skin Lotion	Commercial Lotion
pH	4.5-8	5.62 – 7.34	7.38 – 8.50
Viscosity (cP)	2,000-50,000	3,733-9,933	11,200-19,800
TPC (mg GAE/g lotion)	-	4.368 – 9.261	1.706 – 1.742
IC₅₀ (ppm)	-	5,805-55,776	505,018-557,218
Mechanical Test	Stable	Stable	Stable
Cycling Test	No Phase separation	No Phase separation	-

After conducting all of the stability testing and physicochemical analysis, the CP skin lotion can be concluded to show a good behaviour during 6 weeks storage period. This was confirmed through the observation on increasing viscosity with time, which is indication of a stable lotion; the pH was maintained stable; and through the positive results of mechanical test and cycling test. Table 4 shows the comparison between CP skin lotion, the standardized requirement by SNI and several commercial lotions. The CP skin lotion characteristic was positioned between the SNI standard and the commercial lotions that are widely marketed in Indonesia. In term of pH and viscosity, CP skin lotion are still in the range of SNI's requirement. On the other hand, in term of TPC and IC₅₀, CP skin lotion has much higher TPC value and much lower IC₅₀ value than the commercial lotions. This shows an antioxidant activity that is 10 to 100 times higher than the commercial lotion.

4. Conclusion

A development of skin lotion containing coffee pulp extract as antioxidant source was conducted in this research. The research began with the search for the most optimum condition of coffee pulp extraction, which can provide highest yield of coffee pulp powder extract at maintained antioxidant activity level. The optimum extraction method was determined to be applying sample to solvent ratio of 1:10 (w/v) using ethanol/water mixtures (50:50) as solvent, and conducted at 50°C for 120 minutes. After spray drying process, the yield of CP powder obtained was 0.2171416 g powder/g CP.

The application of the CP extract into skin lotion with different formulations (0.125 % - 1.0 % by mass) was successfully performed. All formulations showed good physicochemical stability and comply with SNI requirement as can be seen in Table 4. When compared to commercial lotions in Indonesia, the developed CP lotions have shown antioxidant activity that is 10 to 100 times higher.

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