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DEVELOPMENT OF SUNSCREEN PRODUCTS CONTAINING GREEN TEA LEAF EXTRACT

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ABSTRACT

Objective: The purpose of this research was to determine antioxidant activity of green tea leaf extract, the value of sun protection factor (SPF), physical properties, and stability of green tea cream.

Methods: Green tea leaf extract was obtained by maceration using 96% ethanol. Cream was prepared in three formulas with various concentration of the green tea leaves extract. The physical evaluations included organoleptic, pH, viscosity, adhesion, spreadability, and stability tests were done. The determination of the SPF value is calculated using the Mansur equation. Extract of green tea leaf has strong antioxidant activity (IC_{50} 2.19 µg/ml).

Results: The green tea leaf extract showed high antioxidant activity (2.19 µg/m). All formulas are organoleptically creamy brownish-green to brown, with a distinctive green tea odor and homogeneous. All formulas met the requirements of physical properties of cream. The creams showed significant change while they were stored at 4°C and at 40±2°C, but showed no difference when they were stored ad 26°C. SPF values of cream are 0.54; 2.03, and 2.41, respectively.

Conclusions: It is clearly indicated that the sunscreen cream of green tea leaf extract is potential to be further developed as cosmetic preparations.

Keywords: *Camellia sinensis* L., Cream, Sunscreen, Antioxidant.

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INTRODUCTION

Sunscreen serves to protect skin from ultraviolet (UV) radiation by absorbing or reflecting radiation so as to reduce the effects of skin damage due to sun exposure. At present, attention to natural active ingredients is increasing [1,2]. Green tea leaves are plants that are popular throughout the world. Indonesia is the 7th tea producer in the world in 2015 [3]. Usually, tea leaf is brewed to drink. In addition, the leaves of this plant are also used for cosmetics.

Green tea leaf extract in cosmetic preparations can protect the skin from UV damage and aging of the skin [4]. Catechin compounds contained in green tea are polyphenol substance [5]. Polyphenols are secondary metabolites of plants and are generally involved in defense against UV radiation or aggression by pathogens [6]. The catechin compounds found in green tea are 2-epicatechin (EC), EGC, (2)-EC-3-gallate, and epigallocatechin (EGCG) [7]. EGCG is the main polyphenol contained in green tea which has an anti-inflammatory and antioxidant function [8]. Green tea leaves are a potential antioxidant with IC_{50} of 3.17µg/ml [9]. These antioxidant compounds provide absorption at the wavelength of the UV B area (290–320 nm) so that they can be used as active ingredients for sunscreen. Its UV protection efficacy and potent antioxidant activity are resulting synergistic effect in photoaging protection.

Sunscreen preparations can contain both physical photo protective ingredients, and chemistry. Physical photoprotective materials such as titanium dioxide (TiO_2) and zinc oxide works by reflecting or scattering UV light while photoprotective chemical substances such as p-amino benzoic acid (PABA), PABA esters, cinnamic, salicylate, antranylate, oxybenzone, benzophenone, and phenolic compounds work by absorbing light UV so it does not get into the skin [10]. Green tea extract with a concentration of 18.1 mg% had an SPF value of 5.87 [11] so that it could be used as a sunscreen. A cream preparation is an effort to increase the usage of green tea. Cream is very suitable for skin care because it is an easy to use, soothing, moisturizing, and easy to penetrate the skin so as to provide the desired effect in healing. Cream of green tea leaf extract with the addition of 1% Vitamin C has a higher antioxidant activity compared to green tea leaf extract cream with the addition of 1% Vitamin E [12]. Another research proved that preparations of green tea with 1–4% green tea extract and 5% TiO_2 have relatively good physical stability [13].

METHODS

Reagents

Dried green tea leaves are obtained from PT. Rumpun Sari Kemuning), ethanol 96%, cetyl alcohol, paraffin oil, methylparaben, propylparaben, stearic acid, cera alba, glycerine, tween 80, and span 80, all are pharmaceutical grade from PT. Brataco Chemica; potassium dihydrogen phosphate (Merck), 1,1-dipehnyl-2picrylhidrazyl (DPPH) (Sigma Aldrich), absolute EtOH (Merck), and sodium hydroxide (Merck).

Green tea leaf extracts preparation

Extraction was done by maceration using 96% ethanol. A total of 500 g of green tea powder were extracted using 2 L 96% ethanol. It was stirred continuously for 3 h, and then allowed to stand for 18 h. The macerate was filtered using a Buchner funnel and then evaporated using a vacuum rotary evaporator (IKA RV 10) and evaporated on a waterbath (Memmert) at 60°C until thick extracts formed.

Antioxidant activity of green tea leaf extract

The measurement of antioxidant activity with DPPH radical scavenging method is based on the ability of a sample to react with radical DPPH on wavelength 517 nm. A total of 1.0 ml of DPPH solution (0.5 Mm) was put into a test tube, then added with 50 µl of various

concentration of extract and completed until 5.0 ml with ethanol. The mixture was stirred using vortex until evenly mixed and allowed to stand for 30 min. The extract concentration is made in such a way to result an IC_{50}.

Formulation of green tea leaf extracts cream

The oil phase consisting of paraffin oil, cera alba, glycerine, cetyl alcohol, span 80, stearic acid, and propylparaben and water phase consisted of green tea extract, methylparaben, tween 80. Each phase was mixed separated. The oil phase and water phase are melted in a ±75°C waterbath (Memmert). The oil phase and fused water phase are mixed in a warm mortar, constantly stirred to form a good cream preparation. The concentration of extracts used in creams is 1%, 2%, and 4%. The cream formula of green tea extract is listed in Table 1.

Physical evaluation and stability test of cream

In brief, the cream is tested for its physical properties which include organoleptic, pH (pH meter Ohaus), viscosity (ViscotesterRion VT-06), stickiness, and spreadability.

The stability parameters of each cream formula measured are odor, color, and pH for 4 weeks with observations every 1 week. The cycling test was carried out for six cycles by the way the sample was stored at 4°C for 24 h and then transferred to an oven (Memmert) at 40±2°C for 24 h, the storage time of the two temperatures was considered as one cycle. Cream preparations were observed for phase separation and inversion.

***In vitro* photoprotective efficacy assessment**

The sun protection factor (SPF) was assessed by dissolving 1.0 g of cream in ethanol to 100.0 mL volumetric flask. The solution was ultrasonicated for 5 min then it filtered with filter paper. Removed a 10 mL of the first filtrate. A 5.0 mL aliquots was transferred into a 50 mL volumetric flask and diluted with ethanol. Then 5.0 mL aliquots were diluted again into a 25 mL volumetric flask with ethanol. The solution was read on a UV-Vis spectrophotometer (Genesis 10S) to determine the spectrum of sample absorption at wavelengths of 290–320 nm with ethanol as blank. Absorption values are recorded at 5 nm intervals. SPF values are calculated using the Mansur equation [14].

$$SPF = CF\, x \sum_{290}^{320} EE(\lambda)\, x\, I(\lambda)\, x\, Abs\ (\lambda)$$

Where: EE-erythemal effect spectrum; I-solar intensity spectrum; Abs-absorbance of sunscreen; CF-correction factor (=10).

Statistical analysis

The parameters were compared using ANOVA test with significance level of p<0.05 using SPSS program version 21.0.

Table 1: Green tea leaf extract cream formula

Phase	Ingredients	Formula (%)		
		F1	F2	F3
Water	Green tea leaf extract	1	2	4
	Methylparaben	0.2	0.2	0.2
	Tween 80	6.9	6.9	6.9
Oil	Paraffin oil	6	6	6
	Cera alba	5	5	5
	Glycerin	10	10	10
	Cethyl alcohol	4	4	4
	Span 80	1.9	1.9	1.9
	Stearic Acid	3	3	3
	Propylparaben	0.3	0.3	0.3
	Perfume	1	1	1
	Phosphate Buffer pH 7.4 until	100	100	100

RESULTS AND DISCUSSION

All formulas have a distinctive smell of green tea and homogeneous. The color of the cream is determined from the concentration of green tea extract. The higher the concentration of green tea extract the browner the cream color. pH test showed that the cream was in the range pH5.45–5.81 (Table 2). A good cream should have a pH range that matches the normal skin pH range of 4.5–6.5 [15]. The results indicated that the cream is acceptable and does not irritate the skin because they are still in the normal pH range of the skin. If a cream is at a pH that is too alkaline it will cause scaly skin, if the pH is too acidic it will cause irritation to the skin [16]. Statistical test results showed the difference between F1: F2 and F1: F3 with a p=0.00154 (<0.05), so it could be interpreted that the increase in levels from 2% to 4% does not change the pH. However, increasing levels from 1% to 2% or 1% to 4% could change the pH of the cream.

Viscosity test results obtained in the cream preparation were in the range 65–95 dPas (Table 2). A good cream has a viscosity range of 2000–4000 cps, equivalent to 20–40 dPas [15]. However, if the cream has a higher viscosity than those range, it does not become a problem as long as it is easily removed from its container, easy to spread, and able to attach well to the skin. A cream that has low viscosity will affect the length of time to adhere when used [17]. The statistical tests indicated that an increase in extract levels from 1% to 4% is not cause significant changes in viscosity.

The results of stickiness test showed creams were in the range 0.61–0.87 s (Table 2). To be able to protect the skin from UV radiation in a relatively long-time cream preparations are expected to have stickiness to the skin for a long time. The results of statistical tests showed that the increase in levels of green tea extract from 1% to 2% or from 1% to 4% caused changes in stickiness in the cream.

The results of the spreadability test of cream were in the range of 15.55–20.00 cm^2 (Table 2). All formulas have a large spread. If a formula has a large spreadability, then no large pressure is needed so that the spread of the active ingredient in the skin is more evenly distributed and the effect is more optimal while if the preparation has a small spreadability then a large pressure is needed. The statistical test results obtained showed that the increase in levels of green tea extract from 1% to 4% is not cause a change in the spreadability of cream.

During storage of all formulas did not show the presence of oil phase separation and water phase. The creams that stored at 40±2°C tend to turn brownish, while creams stored at 4°C tend to be brownish green and the color of the cream stored at 26°C was not change. The higher the concentration of green tea extracts the browner after being stored. This is because high temperatures cause the polyphenols in the extract more easily oxidized [13].

All formulas of the cream showed change in pH while they were stored for 4 weeks at 4°C, 26°C, and 40±2°C (F2). The pH change tends to be more acidic. This is because green tea extract contains weak acidic polyphenols. In addition, hydrolysis reactions between polyphenols and glycosides occur more quickly so that the polyphenols are released from glycosides and are in a more acidic free form [13].

Table 2: Physical properties of green tea leaf extract cream

Physical properties	F1	F2	F3
Color	Brownish green	Light brown	Brown
Odor	Green tea	Green tea	Green tea
Homogeneity	Homogeneous	Homogeneous	Homogeneous
pH	5.81±0.01	5.45±0.01	5.45±0.01
Viskosity (dPas)	65±0.07	75±0.07	95±0.07
Stickiness (s)	0.61±1.41	0.81±2.12	0.87±1.41
Spreadability (cm^2)	20.00±0.57	15.90±1.98	15.55±2.47

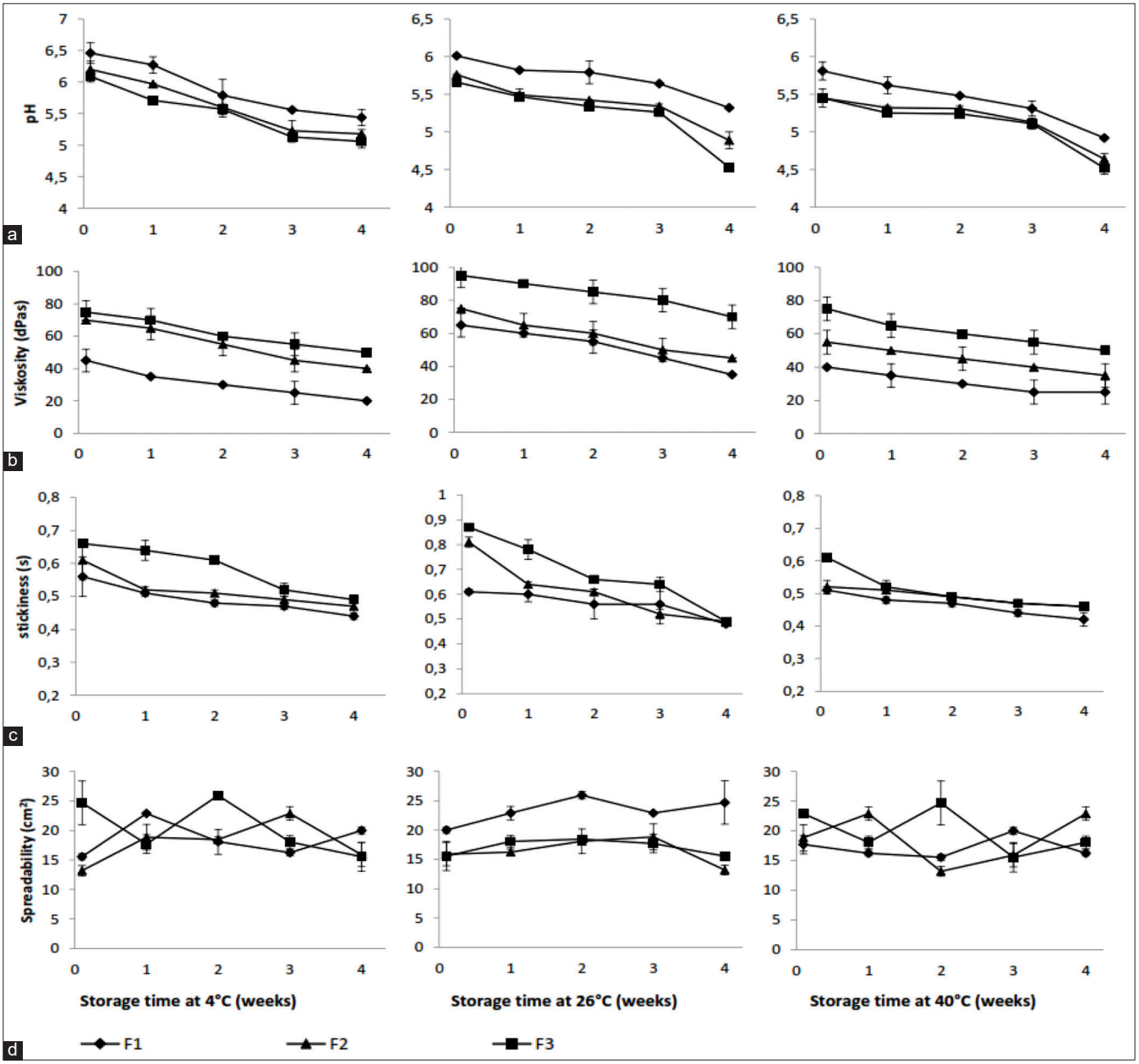

Fig. 1: (a-d) Stability of the green tea leaf extract cream

The viscosity of all creams was decreased when stored at 4°C, 26°C, and at 40±2°C for 4 weeks, except for formula F1 and F2 which are stored at 40±2°C (Fig. 1b). After 4 weeks the viscosity of F1 decreased almost a half than before.

Stickiness of all formulas was changed when stored at 4°C, 26°C, and 40±2°C for 4 weeks except in formula F1 which was stored at 4°C and 26°C not there are changes. The stickiness of the cream decreases, due to its alteration of viscosity. While the viscosity of the cream decreases, the adhesive strength of the cream also decreases (Fig. 1c).

All formulas changed its spreadability when stored at 4°C and 40±2°C. However, when it stored at 26°C all formulas did not show change in spreadability. The change in spreadability could be affected by its viscosity. The results of this study depicted that the viscosity undergoes a change at a temperature of 26°C but has no effect on its dispersion (Fig. 1d).

From the results of cycling tests conducted for six cycles between 4°C and 40±2°C, all formulas did not show a separation between the oil phase and the water phase.

Evaluating of antioxidant activity is one of the general procedures to establish the safety and quality of the nature product used in cosmetics. The same natural source (plants) but from different area of cultivating may result a different antioxidant activity [19]. Compared to previous research, antioxidant activity of green tea leaf extracts has value that almost same (<5 µg/ml) [9].

SPF as an indicator for efficacy of sunscreen products could be assessed using *in vivo* or *in vitro* method. *In vitro* method was selected due to its efficient, cheaper, and more ethical [20]. Furthermore, *in vitro* method also more applicable in industrial practice.

Protection of skin from dangerous UV rays is important for preventing of skin aging and photoaging. A product could be claimed has sunscreen protection if it has SPF value from 2 to 100 values [21]. Sunscreen products that containing antioxidant are highly recommended for protection from skin damage [22].

The results of the SPF study showed that the cream containing of 1% green tea extract had limited protection. The creams containing green

Table 3: Antioxidant activities of the green tea leaf extract

Sample	Antioxidant activity (IC_{50}) (µg/ml)	
	Present study	Previous study
Green tea leaf extract)	2.19	3.16 [9]
Vitamin E	3.11	3.11 [18]

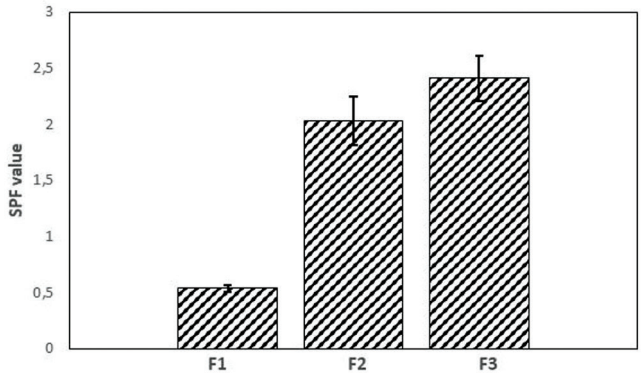

Fig. 2: Sun protection factor value of green tea leaf extract cream

tea extract 2% and 4% provide protection (2.03 and 2.41). The higher the concentration of green tea extract the higher the SPF value (Fig. 2). The results of the statistical test of the SPF value obtained that the increase in levels from 1% to 2% or 1% to 4% can cause changes in the SPF value of the cream. The structure of green tea polyphenols (i.e., 2-EC, EGC, (2)-EC-3-gallate, and EGCG) contains a chromophore system and an auxochrome group which is bound to the chromophore system. The existence of this system causes green tea polyphenols has ability to absorb UV radiation.

CONCLUSIONS

All formulas meet the requirements of physical test for cream preparation. All formulas show significant changes while they were stored at 4°C and at 40±2°C, but showed no difference when they were stored ad 26°C. The cream that containing 2–4% green tea leaf extracts provides protection from UV-B.

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CONFLICTS OF INTEREST

There are no conflicts of interest.

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