

Effectiveness of Sunscreen Cream with Ethanol Extract of Sungkai Leaves (*Peronema canescens* Jack)

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ABSTRACT: Sungkai (*Peronema canescens* Jack) (PCL) contains flavonoid compounds that can absorb UV light therefore having the potential to be developed as a raw material for cosmetics that functions as a sunscreen. The research aims to formulate PCL extract into a sunscreen cream. Sungkai leaves are macerated using 96% ethanol solvent. Specific and non-specific parameter tests were carried out on the extract as well as antioxidant tests using the DPPH method and SPF tests using the Mansur method. The results of this research showed that 96% ethanol extract of PCL had a blackish-green color, pH 5.25; the ethanol and water-soluble essence levels were 6.25% and 5.17%. The water content obtained is 27.4%; drying shrinkage is 0.46%; total ash content is 0.12% and acid insoluble ash content is 0.1%. The antioxidant value obtained was 35.06 ppm. Three cream formulations were prepared (FI of 0.1753%, FII of 0.3506%, and FIII of 0.5259%) and evaluated for organoleptic, homogeneity, cream type, viscosity, pH, particle size, and spreadability tests for one month at 25°C and 40°C. The organoleptic test results of the cream have a soft texture, with varying colors; light green (FI), green (FII), and dark green (FIII) color. Other evaluation test results show that the cream has a pH of 5.16 - 6.73, a spreadability of 6.47 - 8.07 cm, a viscosity of 15466.67 - 38933.33 cPs, a particle size of 9.88 - 16.17 μm and an SPF of 17.68 (FI), 27.46 (FII), and 33.64 (FIII) with ultra protection on all three. PCL extract after being formulated into creams shows high SPF activity with F3 as the best formula. PCL extract can be formulated into a cream form that meets physical and chemical quality parameters.

KEYWORDS: antioxidant; formulation; sungkai; sunscreen.

1. INTRODUCTION

Indonesia is a country located on the equator, making it a tropical country, of course, we feel the effects of the sun throughout the year. However, sunlight contains ultraviolet rays, and excessive exposure to sunlight without additional protection can cause various complications on our skin, such as burning, the appearance of black spots, wrinkles, and even skin cancer [1].

Indonesia is rich in herbal plants that have various benefits, including as raw materials for cosmetics. One of the natural plants that has the potential to be developed as a cosmetic preparation is Sungkai (*Peronema canescens* Jack). *Peronema canescens* (PCL) is a medicinal plant from the Verbenaceae family. In Indonesia, Sungkai is known as the name Sungkai or Sabrang Teak. PCL has also been reported to have a wide range of secondary metabolites such as phenols, tannins, alkaloids, steroids, flavonoids, saponins, and triterpenoids, which display a variety of biological effects such as antioxidant, immune-boosting, and anti-inflammatory, and immunity. Empirically, PCL leaves are known to be used for a few treatments such as bruises, colds, and fevers, and even as a mouthwash to prevent dental diseases [1], [2].

Antioxidants are compounds that can be used to prevent damage to cells in the body caused by free radicals by neutralizing the free radicals by replenishing electron deficiency. Using the DPPH method, an IC_{50} value is obtained. This determines the value of the sample that can scavenge 50% of DPPH free radicals in the DPPH free radical scavenging method. The lower the IC_{50} value, the higher the antioxidant, the stronger the sunscreen activity to protect the skin. Research conducted previously regarding IC_{50} values of PCL extract by Fadlilturrahmah *et.al*, 2021 obtained a value of 42,21 ppm and research by Okfrianti *et.al*, 2022 obtained a value of 45,7 ppm which puts the two research with strong antioxidant results [3], [4], [5].

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Ultraviolet rays triggers the production of melanin pigments, causing the skin cells to produce a thicker epidermis, making a person tanner than they were before. These rays are also the cause of sunburns and other skin conditions such as loss of skin elasticity and wrinkling. An attempt to protect the skin from harsh ultraviolet rays is by using sunscreen. Sunscreens are divided into two categories based on their action mechanism; chemical or mineral. Chemical sunscreens work by absorbing UV light and converting it into heat energy that is then released from the skin while mineral sunscreens or also known as sunblocks work by reflecting and scattering UV lights; hence protecting the skin [6].

Sunscreens are often formulated into cream form, a topical preparation that's applied on the skin. The definition of creams itself is "viscous liquid or a semi-solid emulsions of either an oil-in-water or water-in-oil type". This type of formulation are used for localized effects in which the drugs are delivered into the underlying layer of the skin. Creams contained one or more active ingredients that are dissolved or dispersed into a suitable base, oil-in-water creams or water-in-oil creams [6], [7].

Cream types are determined by the basis of phases. Therefore, the formulation of sunscreen products affects the Sun Protection Factor (SPF). The ability to withstand ultraviolet rays from sunscreens is assessed in terms of the (SPF), which is the ratio of the ultraviolet energy required to produce minimum erythema on the skin. SPF values can be determined using in vivo methods but in vitro approaches using spectrophotometric tools are also allowed. If the SPF value of a sunscreen product is higher, then the effectiveness of the sunscreen will also be higher [2], [6].

2. MATERIALS AND METHODS

2.1. Materials and Equipments

Sungkai leaves (*Peronema canescens* Jack) (PCL) were obtained from Palembang. Chemicals such as 96% ethanol, ethanol pro analysis (Merck, Germany), stearic acid (Wilmar International, Singapore), triethanolamine (Merck, Germany), cetyl alcohol (Akoma International, United Kingdom), glyceryl monostearate (Fagron, Netherlands), isopropyl myristate (Wilmar International, Singapore), phenoxyethanol (Chem On, Vietnam), propylene glycol (Dow Chemical Pacific, Singapore), and purified water.

The equipment used is UV-Vis Spectrophotometer (Shimadzu 1900), rotary vacuum evaporator (Heidolph), analytical scales (Kern), stirrer (Ika Eurostar), viscometer (Brookfield RV type), water bath (Mettler, W 600), pH meter (Hanna Instruments), glassware (Pyrex, Iwaki), spreadability test kit, microscope (Optika), object glass, evaporating basin, spatula, and filter paper.

2.2. Procedure

2.2.1. Determination of PCL

Determination was conducted at the Biology Department, Faculty of Mathematics and Natural Science, Sriwijaya University, Palembang (No. 302/UN9.1.7/4/EP/2021).

2.2.2. Preparation and Extraction of PCL

The leaves are first dried using an oven at 40°C. The dried leaves are then blended down into a powder using a blender. Sieves with the values of 4 and 18 are used to determine the degree of fineness. Place 100 g of powder upon sieve number 4 and shake [8]. Weigh the amount of simplicia remaining on the sieve and the receiving pan and repeat the instructions with the number 18 sieve. Sungkai simplicia was macerated with 96% ethanol (1:10) for 24 hours. After 24 hours, it was filtered using a filtration paper. The filtrate obtained was then concentrated using a rotary vacuum evaporator [9], [10]. The yield and DER-Native of the extract are then calculated using the formula below [8]:

$$\text{Yield} = \frac{\text{Weight of extract}}{\text{Weight of simplicia}} \times 100\%$$

$$\text{DER - Native} = \frac{\text{Weight of simplicia}}{\text{Weight of extract}}$$

2.2.3. Quality Test of Extract

The extract then undergoes a series of quality tests, such as phytochemical screenings and specific and non-specific parameter tests. To create a homogenous cream, the solubility of the extract needs to be known by referring to the 6th edition of the Farmakope Indonesia.

2.2.4. Antioxidant Test with DPPH Method

A solution of ethanol extract from PCL was made with a concentration of 1000 ppm, then dilution was carried out with varying concentrations, namely 20; 40; 60; 80, and 100 ppm [11]. Then 1 mL of each solution was taken, then a 0,4 mM DPPH solution was added to 5,0 mL, then the mixture was incubated for 30 minutes and the absorbance was measured at the maximum wavelength using a UV-Vis spectrophotometer. The antioxidant activity is then calculated using the formula below [12]:

$$\% \text{ Inhibition} = \frac{\text{Blanko Absorbance} - \text{Sample Absorbance}}{\text{Blanko Absorbance}} \times 100\%$$

The IC₅₀ value was determined by calculating the curve concentration (x) and % inhibition (y) using a regression line with the equation of $y = a + bx$ where IC₅₀ is stated as x.

2.2.5. Cream Formulation and Preparation

The formula used is adopted from Prima with a few adjustments [13]. This study aims to formulate a cream with a high sunscreen activity, therefore the extract used in each formula is slightly more.

Table 1. Formulation of Cream with PCL Extract.

Material	Formula (%)		
	F1	F2	F3
PCL Extract	50x IC ₅₀	100x IC ₅₀	150x IC ₅₀
Stearic Acid	3	3	3
Cetyl Alcohol	3	3	3
Triethanolamine	0.4	0.4	0.4
Glyceryl Monostearate	2.1	2.1	2.1
Isopropyl Myristate	3	3	3
Phenoxyethanol	0.5	0.5	0.5
Propylene Glycol	15	15	15
Purified Water	ad 100	ad 100	ad 100

Each material is weighed and melted down in the water bath at a temperature of 70°C using evaporating basins. The water phases (propylene glycol, triethanolamine, phenoxyethanol, and pure water) are poured inside a beaker glass. The beaker is then placed under a homogenizer with the spindle set to 400 rpm. The oil phases (stearic acid, cetyl alcohol, isopropyl myristate, and glyceryl monostearate) and PCL extract are then poured inside the beaker glass slowly until a cream consistency is formed [14].

2.2.6. Evaluation of Sungkai Leaves Extract Cream

The evaluation of cream includes organoleptic; homogeneity test, cream type, pH, viscosity, particle size, and spreadability test. Additionally, a SPF test was conducted on each formula. The evaluation of cream was also observed every 2 weeks for 4 weeks stability test [15].

2.2.7. SPF Test using the Mansur Method

Ten grams of cream of each formula is weighed and dissolved using pro-analysis ethanol inside a 25 mL volumetric flask. The absorbance of each sample is measured using a wavelength of 290-320 nm with intervals of 5 nm. The SPF value is then calculated using the formula below [10] :

$$SPF = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times absorbance(\lambda)$$

- EE : Erythematous effect spectrum
- I : Solar intensity spectrum
- Abs : Absorbance of sunscreen product
- CF : Correction factor (10)
- λ : Wavelength (nm)

After the wavelengths obtained are calculated, the SPF value can be determined based on the SPF category. The SPF category ranges from minimal to ultra as listed down below [16]:

Table 2. SPF Category.

Protection Type	SPF Value
Minimal	1-4
Medium	5-6
Extra	7-8
Maximal	8-15
Ultra	>15

2.2.8. Stability Testing

The stability testing was conducted for 4 weeks where the cream was stored at temperatures of 25°C and 40°C. The parameters tested are organoleptic, homogeneity, viscosity, pH, particle size, cream type, and spreadability.

3. RESULTS AND DISCUSSIONS

3.1. Preparation of Sungkai Leaves Extract

Before the research is conducted, the Sungkai leaves must undergo the determination process. The determination was conducted at the Mathematics and Natural Science Faculty of Sriwijaya University, Palembang. Based on the results, the leaves determined are PCL of the Verbenaceae family as seen on the report of No. 302/UN9.1.7/4/EP/2021. Determining the degree of fineness of simplicia is an important step because if a powder is too fine, it can cause the filtering process to be hindered, taking longer to obtain a filtrate. On the contrary, if a powder is too big, it can cause quite a less optimal diffusion of the analytes process as the surface area of the simplicia is too small. A fineness degree of 4/18 must be obtained which indicates that 100% of powder must go through sieve number 4 but no more than 40% should go through sieve number 18. The results obtained are 100/9,5%. The yield of extract obtained is 7,06% with a DER-Native of 14,16. The bigger the number of yields of extract obtained indicates that the extract produced is bigger as well [8].

3.2. Phytochemical Screening

The results shown in Table 2 follow the previous study done by Fitriyani *et.al* (2023).

Table 3. Results of Phytochemical Screening of PCL Extract.

Tests	Reagents	Results
Alkaloids	Dragendorff	+
Flavonoids	Mg + HCl	+
Tannins	FeCl ₃	+

Saponnins	H ₂ O + HCl	+
Steroids	CH ₃ COOH anhidrate + H ₂ SO ₄	+
Triterpenoids	CH ₃ COOH anhidrate + H ₂ SO ₄	-

Note: (+) = Positive, (-) = Negative

Phytochemical screening was conducted to determine the content of secondary metabolite compounds. Based on the tests conducted, the PCL extract had tested positive for every secondary metabolite except triterpenoids. Flavonoids have the potential to prevent exposure to UV radiation on the skin due to the presence of a chromophore group that can absorb UV A and UV B rays. This test shows different results than the ones conducted by Fitriyani *et.al* (2023) which showed a negative result on the alkaloid test. Flavonoids contain a free form (aglycone) or are similar to glycosides, such as polyhydroxy aglycones, which are semi-polar, and polymethoxy aglycones, which are non-polar [17], [18].

3.3. Specific and Non-Specific Parameter Tests

Table 4. Results of Specific and Non-Specific Parameters Test.

Tests	Results
Organoleptic	Blackish-green sticky, thick extract with a distinct scent
pH	5.25
Water Content	27.4
Water-Soluble	6.25
Ethanol-Soluble	5.17
Drying Shrinkage	0.46
Total Ash Content	0.12
Acid Insoluble Ash Content	0.1

An organoleptic test showed that the extract produced was a blackish-green, sticky thick extract with a distinct scent and a pH of 5,25 which can be the effects of acid metabolites [19]. The aim of determining water content is to find out whether the amount of water content in the extract exceeds the limit range or not. High water levels have the potential to increase the growth of fungi, microbes, or microorganisms which can affect the quality of the extract. The water content obtained was 27,4% which meets the criteria [20].

Determination of water-soluble essence content aims to determine the number of compounds that can be extracted with water from an extract while ethanol-soluble tests are conducted to find out how much of the compound content is dissolved in the ethanol solvent [21]. Based on the research done, the water-soluble obtained is 6,25% and the ethanol soluble obtained is 5,17%. Drying shrinkage testing aims to determine the maximum limit (range) regarding the number of compounds that disappear during the drying process, the results obtained are 0,46% [22].

A total ash content test is carried out so that the total internal and external mineral composition present from the initial stage until it becomes an extract can be determined [23]. The results obtained are 0,12%. The remaining ash from the previous test is then tested for another test called acid insoluble ash content. The acid insoluble ash content test aims to determine the results of pollution originating from external factors such as dirt that sticks to the drying process [24]. The results obtained are 0,1%.

3.4. Antioxidant Test with DPPH Method

Antioxidant activity is determined by calculating the IC₅₀ value, namely the concentration needed to reduce 50% of DPPH free radicals. If the IC₅₀ value is smaller, the antioxidant activity is greater. Based on the antioxidant activity test done, the IC₅₀ value of the PCL extract obtained is 35,73 ppm. Based on the level of antioxidant power, PCL extract is in the "very strong" category.

3.5. Evaluation of Sungkai Leaves Extract Cream

Tables 4 and 5 show the stability results of cream formulations (F1, F2, and F3) for one month. The formulations are generally stable during storage although there are some changes although not quite significant.

Table 5. Characteristics of Cream in 25°C for One Month.

Parameters	Room Temperature (25°C)								
	F1			F2			F3		
	Week 0	Week 2	Week 4	Week 0	Week 2	Week 4	Week 0	Week 2	Week 4
Organoleptic	Pale green color	Pale green color	Pale green color	Green color	Green color	Green color	Dark Green Color	Dark Green Color	Dark Green Color
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous
Cream Type	O/W	O/W	O/W	O/W	O/W	O/W	O/W	O/W	O/W
pH	6.71	6.68	6.73	6.38	6.47	6.45	6.13	6.17	6.17
Viscosity (cPs)	31333.33	30533.33	30133.33	26133.33	25600	24800	23600	20266.67	19466.67
Particle Size (µm)	9.88	10.23	11.89	11.40	12.57	12.88	13.67	14.84	15.26
Spreadability (cm)	6.92	6.96	7.22	7.09	7.32	7.42	7.16	7.27	7.47

Table 6. Characteristics of Cream in 40°C for One Month

Parameters	Accelerated Temperature (40°C)								
	F1			F2			F3		
	Week 0	Week 2	Week 4	Week 0	Week 2	Week 4	Week 0	Week 2	Week 4
Organoleptic	Pale green color	Pale green color	Pale green color	Green color	Green color	Green color	Dark Green Color	Dark Green Color	Dark Green Color
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous
Cream Type	O/W	O/W	O/W	O/W	O/W	O/W	O/W	O/W	O/W
pH	6.67	6.69	6.70	6.46	6.52	6.38	6.15	6.12	6.12
Viscosity (cPs)	22666.66	20933.33	19466.66	21466.66	19200	18933.33	19333.33	17333.33	15466.67
Particle Size (µm)	9.88	10.40	11.24	11.40	12.37	13.04	13.67	14.34	15.20
Spreadability (cm)	6.65	7.11	7.45	7.13	7.23	7.3	7.3	7.37	7.48

The organoleptic test is carried out by using the human senses to observe the color, smell, and form of the PCL cream extract. Each formula has a distinct smell with a thick, creamy texture. Formula 1 has a pale green color to it, formula 2 has a green color while Formula 3 has a dark, leaf green color.

The homogeneity test is carried out to determine whether every particle in each product merges well with one another. A homogeneous product shows that it has good quality because it shows that all the materials in the formulation are evenly dispersed. Based on the test results, PCL extract cream is homogeneous.

The cream type test is carried out to determine the type of cream formulated. If the outside phase of the cream is blue, it indicates that the cream type is oil in water (O/W) but if the outside phase is red then it indicates that the cream type is water in oil (W/O).

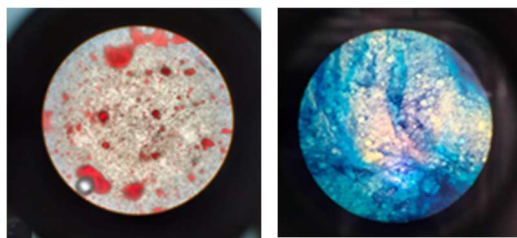


Figure 1. Cream type test results under microscope

Based on the test carried out, the cream type of each formula is oil in water (O/W) as it shows the outside phase of the cream-colored blue and the inside phase colored red.

The viscosity test is carried out to determine the level of viscosity in a sample. Based on SNI 16-4399-1996, the standard viscosity of cream is 2.000 - 50.000 cPS. [25]. The spindle size used is number 5 with the rpm speed of 10. The test results show a viscosity range between 18933,33 - 31333,33 cPS indicating that the PCL extract cream meets the requirements.

The pH test is carried out to determine the level of acidity or alkalinity in a sample. The pH standard for skin is 4.5-6.5 therefore the cream that is formulated should have a pH level nearing one of the skin [26]. By measuring using a pH meter, it is obtained a pH level of formula 1, formula 2, and formula 3 at 6,71; 6,38; and 6,13 respectively which indicates that the cream formulated meets the requirements.

The ability to spread on the skin surface when the cream formula is applied can be determined by a spreadability test. A good topical preparation according to SNI is around 5-7 cm for spreadability [26]. The spreadability obtained from the three formulas are 6,67; 7,31; and 7,73 cm respectively which indicates that formulas 2 and 3 do not meet the requirements as each spreadability exceeds that of 7 cm.

Based on the results of the organoleptic tests on each formula, there were no changes physically from week 0 to week 4 indicating that storage duration and temperature do not have a significant effect on the three products. In the chemical stability test, the pH is tested on each formula. The results were no significant changes as well.

3.6. SPF Test of Cream PCL Extract

The SPF test was carried out on the three formulas. This test determines the SPF category which ranges from minimal to ultra. The test was conducted using UV-Vis spectrophotometer with a wavelength of 280-320 nm. The absorbance obtained are then calculated using the Mansur method and the SPF obtained from the three formulas range from 17 to 33 which puts them in the ultra category as they exceed the value of 15.

Table 7. SPF Value Results of Sungkai Leaf Ethanol Extract Cream.

Formula	SPF Value
F1	17.68
F2	27.46
F3	33.72

Results shows that the higher the concentration of extract in the formula will result in a higher SPF value as well, indicating that PCL extract has high photoprotection activity and is therefore effective as a main ingredient in a sunscreen.

4. CONCLUSION

PCL extract after being formulated into a cream showed high SPF activity based on the values obtained, namely 17.68; 27.46; and 33.72 with F3 as the best formula. PCL extract can be formulated into a cream that meets physical and chemical quality parameters.

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