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FORMULATION OF RED BETEL LEAF EXTRACT LOTION (*Piper crocatum* Ruiz & Pav) AS SUNSCREEN

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Abstract

Betel leaf is a natural ingredient that people use as a traditional medicine. One of the ingredients found in red betel leaves is a phenolic compound in the form of flavonoids, a photoprotective substance that can be used as protection from UV radiation. Sunscreen is a cosmetic preparation that can protect the skin from UV radiation and reduce the amount of radiation harmful to the skin. This study aimed to formulate a lotion preparation from red betel leaf extract that has activity as a sunscreen. Red betel leaf (*Piper crocatum* Ruiz & Pav) was extracted using the maceration method. Preparation formulations were made with variations of red betel leaf ethanol extract in each formula, namely with concentrations of 0.04% (F1), 0.05% (F2), 0.06% (F3), and base control (F0). The activity of sunscreen lotion extract is based on the Sun Protection Factor (SPF) value using UV-VIS spectrophotometry at wavelengths of 290–320 nm using the Mansur equation. The results of the SPF value of the lotion are F1 of 13.48, F2 of 16.71, and F3 of 18.7. The evaluation of the lotion results obtained a light green lotion with M/A emulsion type that met the requirements, including homogeneity, pH, spreadability, viscosity, centrifugation test, and freeze and thaw test. Research results show that betel leaf extract lotion is physically stable and has a high SPF value, so it has the potential to be a sunscreen.

Keywords: betel leaf, lotion, piper crocatum ruiz & pav, sunscreen

Introduction

Sun exposure is very beneficial for the body, including vitamin D synthesis. Exposure to sunlight causes 7-dehydrocholesterol in our skin to uptake UV B radiation and transform into pre-vitamin D3, which is then isomerized into vitamin D3. Still, overexposure to these rays can cause skin redness or burning.[1] Frequent long-term exposure to ultraviolet light can cause several types of abnormalities in the body, especially the skin, including erythema, pigmentation, and DNA abnormalities that can lead to cancer. Although, these effects can be minimized with the use of sunscreen.[2]

Sunscreens are cosmetic products that reflect and block UV radiation to decrease the radiation that can be dangerous to the body.[3] Sunscreens can be divided into two types, namely synthetic sunscreens and natural sunscreens. The public widely uses natural sunscreens because they are considered safer and affordable.[4] One of

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the ingredients for using sunscreen is the content of photoprotective ingredients such as flavonoids. Flavonoids are potential sunscreens because they feature chromophore groups that generally contribute colour to plants. Chromophore groups are conjugated aromatics that can take up intense light in the UV wavelength range.[5] Compounds with high flavonoid content have the best potential to be used as sunscreen.[6]

One of the ingredients for using sunscreen is the content of photoprotective ingredients such as flavonoids. One of the natural sunscreens is red betel leaf (*Piper crocatum* Ruiz & Pav). Red betel leaves contain active compounds that can play a role and provide activity as a sunscreen, such as flavonoids, tannins, alkaloids and saponins.[7] Using natural ingredients as active substances allows minimizing irritation to the skin compared to synthetic ingredients.[6] Previous research shows that betel leaf fractions have sunscreen activity with an SPF value of more than 15 and have potent antioxidant activity with an IC50 value of 53.91.[8]

Lotion dosage formulations are more often used because they are more effective as sunscreens. The lotion is a suspension, emulsion, or solution with or without drugs for topical use whose liquidity allows even application to dry quickly on the skin after application. It leaves a thin layer of the drug component on the skin's surface.[9]

The research was carried out further by the formulation and effectiveness test of sunscreen lotion preparations of ethanol extract of red betel leaves with concentrations of 0.04%, 0.05%, and 0.06%. The benefits of this study are that it provides information or knowledge about the formulation, evaluation, and effectiveness of red betel leaf extract lotion (Piper crocatum Ruiz & Pav), which can be used as a sunscreen product.

Method

Tool

Instruments are analytical balance, pH meter, Brookfield viscometer, UV-Vis spectrophotometer, mixing rod, ultra turrax (IKA®), bottle, plate glass, centrifugation, drops pipette, 10 mL volumetric flask (pyrex®), 100 mL volumetric flask (pyrex®), 10 mL measuring cup (pyrex®), 100 mL measuring cup (pyrex®), 100 mL beaker (pyrex®), 250 mL beaker (pyrex®), ultrasonication instrument and lotion storage unit.

Ingredient

The ingredients used are stearic acid (PT. DPH), cetyl alcohol (PT. DPH), lanolin (PT. DPH), triethanolamine (PT. DPH), propylene glycol (PT. DPH), span 80 (PT. DPH), tween 80 (PT. DPH), methylparaben (PT. DPH), propylparaben (PT. DPH), distilled water, ethanol 96% (Daya Chemical), ethanol Pa % (Daya Chemical) and the active ingredient ethanol extract of red betel (red betel leaves collected in Kubang Hamlet, Sukajaya Village, Sumedang Sub-district, Sumedang Regency, West Java).

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Procedure

Basic Formulation of Red Betel Leaf Ethanol Extract Lotion Preparation

Table 1. Red Betel Leaf Ethanol Extract Lotion Formula

Material	F0 (%)	F1 (%)	F2 (%)	F3 (%)
Red betel leaf extract	0	0,04	0,05	0,06
Stearic acid	5	5	5	5
Cetyl alcohol	5	5	5	5
Lanolin	2	2	2	2
Propylene glycol	5	5	5	5
Span 80	2,12	2,12	2,12	2,12
Tween 80	7,4	7,4	7,4	7,4
Triethanolamine	1	1	1	1
Metyl paraben	0,25	0,25	0,25	0,25
Propyl paraben	0,5	0,5	0,5	0,5
Aquades	Ad 100	Ad 100	Ad 100	Ad 100

The sunscreen lotion formulation process begins with making a lotion base to obtain the best lotion formula base. Lotions were produced by melting the oil phase, namely stearic acid, cetyl alcohol, propylparaben, and lanolin, span 80 and tween 80 in a porcelain cup heated in a water bath to approximately 75°C. The water phase was methylparaben, triethanolamine, and propylene glycol. Phase oil was added to the water phase slowly at 75°C, distilled water was added, and then ethanol extract of red betel leaves with three different concentrations was stirred until homogeneous. Furthermore, the lotion preparation that has been formed is put into a closed package and evaluated for 28 days. Physical evaluation of ethanol extract of red betel leaf lotion includes organoleptic test, emulsion type, homogeneity, pH, spreadability, viscosity, centrifugation test, and freeze and thaw test.[10]

Physical Stability Testing of Red Betel Leaf Ethanol Extract Lotion Preparation

Organoleptic Observation

This test is carried out for organoleptic examination, including shape, colour, and odour, which are observed visually.[10]

Determination of Dosage Type

The type of preparation is tested using methylene blue solution. The lotion is dripped on the preparation glass and then dripped with methylene blue and the spread of methine blue. If the methylene blue spreads, then the lotion is type O/W. [10]

Homogeneity

The preparation should display a homogeneous structure when applied on a glass piece or other suitable transparent material. Without granules or particles, the preparation is declared homogeneous.[10]

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pH Measurement

The pH of the preparation was then evaluated using a pH meter. The preparation is placed in a container, and then the electrode is dipped. The number displayed by the pH meter is the pH value of the lotion.[10]

Viscosity Test

The viscosity test was carried out for 28 days in weeks 1, 2, 3 and 4, using a Brookfield Viscometer with spindle no. 6 and a speed of 100 rpm. The preparation was placed on a plate, the cone lowered, and the viscometer was run. The device's scale determines the viscosity value.[10]

Spreadability Test

Viscosity tests were carried out for 28 days in weeks 1, 2, 3 and 4, using lotion preparations taken as much as 0.5 grams. Then they put on a transparent glass and covered it with clear glass. Glass was given a load weighing 150 grams and allowed to stand for 1 minute, and then the diameter was measured.[10]

Centrifuge Test

The ethanol extract of red betel leaf lotion underwent mechanical test method stability testing. A centrifuge tube is filled with up to 1.5 mL of lotion. After that, the centrifuged tube was placed in a centrifugator and spun for 2.5 hours at 3800 rpm. Additionally, separating the emulsion phase was a defining feature of the physical alterations.[11]

Freeze and Thaw

This method is carried out by storing the preparation at four °C for 48 hours, then transferring it to 24°C for 48 hours. The treatment is counted as one cycle. The freeze and thaw method was carried out for five cycles. Organoleptic, physical changes in lotion were observed.[12]

Sunscreen Activity Test on Red Betel Leaf Extract Lotion

The lotion preparation containing red betel leaf extract was dissolved in 1 g in 10 mL of ethanol p.a. and then homogenized using ultrasonication for 10 minutes. The solution was then measured at UV-Vis spectrophotometry every 5 nm in the wavelength range between 290-320 nm. The Mansur method can determine the lotion preparation's SPF or Sun Protection Factor value.[3]

Result

Physical Evaluation of Red Betel Leaf Ethanol Extract Lotion Preparation

Physical stability testing of the ethanol extract of red betel leaf ethanol conducting organoleptic examinations, determining the type of preparation, homogeneity examination, pH measurement, viscosity test, spreadability test, centrifuge test and freeze and thaw test. The following are the results of observations for each test that has been carried out:

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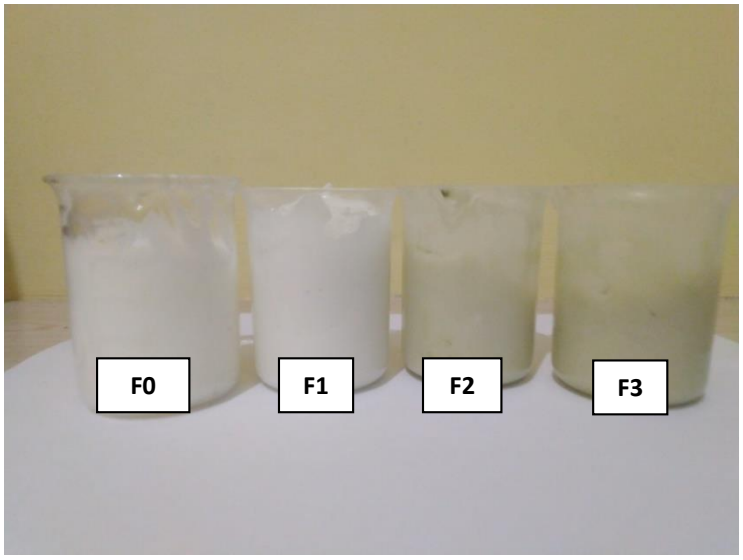

Figure 1. Formulation of the ethanol extract of red betel leaf lotion preparation
Description :
F0 = lotion preparation with a concentration of red betel leaf ethanol extract 0%
F1 = lotion preparation with a concentration of red betel leaf ethanol extract 0.04%
F2 = lotion preparation with a concentration of red betel leaf ethanol extract 0.05%
F3 = lotion preparation with a concentration of red betel leaf ethanol extract 0.06%

Table 2. Physical Appearance Results of Red Betel Leaf Ethanol Extract Lotion Formula

The following are the results of the physical appearance of the ethanol extract of the red betel leaf lotion formula :

Observation		Formula			
		F0	F1	F2	F3
Organoleptic	Odour	-	-	-	-
	Colour	White	Yellowish	Light Green	Green
	Texture	Soft			
Homogeneity		Good			
Emulsion Type	Dye with Methylene blue	O/W			

Table 3. Results of pH Measurement of Red Betel Leaf Ethanol Extract Lotion

Preparations	pH Observation				
	Day-0	Day-7	Day-14	Day-21	Day-28
F0	6,04	6,04	6,01	5,98	5,95
F1	6,35	6,37	5,95	5,80	5,71

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Table 3. (Extension)

Preparations	pH Observation				
	Day-0	Day-7	Day-14	Day-21	Day-28
F2	6,20	6,01	5,83	5,77	5,54
F3	6,13	6,15	5,78	5,56	5,40

Table 4. Measurement Result of Spreadability of Red Betel Leaf Ethanol Extract Lotion

Preparations	Observation Measurement of Spreadability g (cm/s)				
	Day-0	Day-7	Day-14	Day-21	Day-28
F0	5	5	5	5	5
F1	5,5	5,5	5,5	5	5
F2	5,5	5,5	5,5	5	5
F3	5,5	5,5	5,5	5	5

Table 5. Viscosity Measurement Result of Red Betel Leaf Ethanol Extract Lotion

Preparations	Viscosity Observation (Cps)				
	Day-0	Day-7	Day-14	Day-21	Day-28
F0	2800	9367	12000	12200	13000
F1	4600	4967	8033	8100	10000
F2	4583	6367	9000	9000	10133
F3	4417	6900	9317	9000	10200

Sunscreen Activity Test on Red Betel Leaf Extract Lotion

Sunscreen activity test of the ethanol extract of red betel leaf sedan lotion on formula I (0.04%), formula II (0.05%) and formula III (0.06%). The following are the results of observations of sunscreen activity tests of lotion preparations:

Table 6. SPF Value of Red Betel Leaf Ethanol Extract Lotion Preparations

Group	SPF	Significance
Base	2,163±0,003	
Formula 0,04%	13,431±0,088	0,308
Formula 0,05%	16,710±0,002*	0,042
Formula 0,06%	18,745±0,004*	0,002

Notes:
*) significantly different compared to the base group ($p<0.05$), n=three repetitions

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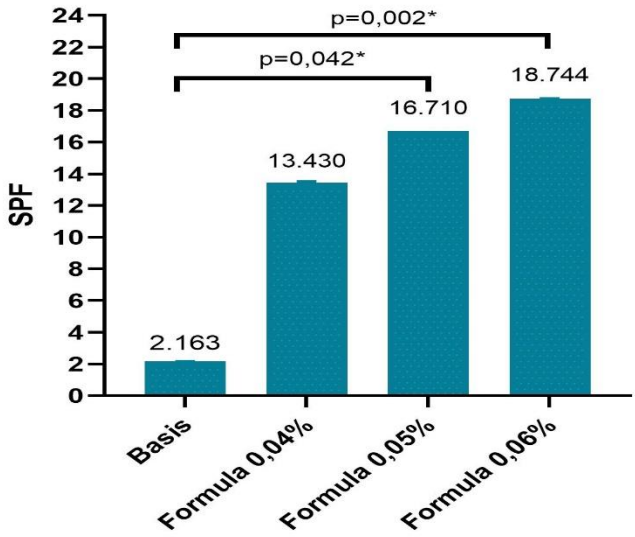

Figure 2. SPF value of the ethanol extract of red betel leaf lotion preparation

Discussion

The formulation is done by determining the best base formula, then formulated with the addition of a thick extract of red betel leaf. Additional ingredients in the formulation have their respective functions and roles. Stearic acid and cetyl alcohol are emulsifiers and function as thickening agents that maintain the stability of the preparation, and lanolin functions as a softener. It improves the lotion base, propylene glycol as a humectant, tween 80 and span 80 function as emulsifying agents, triethanolamine as an emulsifier and alkalizing, propylparaben and methylparaben function as anti-bacterial and antifungal.

The next step was to evaluate the physical characteristics and stability of the preparation during storage. The evaluation was carried out for 28 days. Organoleptic tests are carried out to see the physical appearance of the preparation by observing the colour, odour and texture of the preparation that has been made. Evaluation of lotion base and evaluation of sunscreen lotion formula added with red betel leaf extract. The results showed that the four formulas were stable in storage for 28 days.

A homogeneity test is performed to see the mixing of lotion preparations. The results show that all formulations are homogeneous. The emulsion type test using Methylene blue solution shows spread, so the lotion is A/M type. This type provides advantages: it can be rinsed with water and is not sticky. The pH measurement is carried out to see that the preparation is safe when used so that it will not irritate the skin.10 The results obtained pH 5-6 follow the provisions of SNI 16-4349-1996, which is 4.5-8. Spreadability testing aims to see a picture of the ability to spread lotion on the skin, and the results show that the lotion has a spreadability of 5 to 5.5 cm. These results follow the theory of lotion preparations with a spreadability of 5-7cm.10 Viscosity test is done to see the viscosity of a preparation. The results obtained follow the provisions of SNI 16-4349-1996, which ranges from 2,000-50,000 cps.

A freeze and thaw stability test was conducted to determine the lotion's stability at two different temperatures, namely four °C and 24°C, or freeze-thaw cycles, to see the effect of temperature on the phase separation of the preparation. Observations showed that all lotion formulations were stable, with no phase separation until the 5th cycle. The freeze-thaw process can be successful, or no phase separation occurs, depending on the ability of the preparation to recover from the pressure of crystal water immediately. While freezing, crystalline water is formed that has a more structured and

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tight structure that prevents the cream from flowing. With a temperature of 4°C, the aqueous phase freezes and tends to shrink, reducing the aqueous phase space and causing the oil globules to stick together or merge to form tighter inter-particle bonds, increasing the product's viscosity. When the recovery speed of the cream is slow, instability may occur.[12]

The physical stability of the lotion was further evaluated by centrifugation at 3800 rpm for 150 minutes. The evaluation showed that the formula remained stable,[12] with no phase separation. Based on this evaluation, the cream was stable against gravity for one year of storage.

Sunscreen activity test of the ethanol extract of red betel leaf sedan lotion on formula I (0.04%), formula II (0.05%) and formula III (0.06%). Results of the sunscreen activity test showed that the lotion of red betel leaf ethanol extract produced an average value of SPF lotion F0, F1, F2, and F3, respectively, of 2.163 ± 0.003 which shows minimal protection; 13.431 ± 0.088, which shows maximum protection; 16.710 ± 0.002 which shows ultra protection and 18.745 ± 0.004 which shows ultra protection. The study showed that the lotion has sunscreen activity, which means that the lotion with ethanol extract of red betel leaves can protect the skin from exposed sunlight directly.[13]

Chemicals of red betel leaf's ethanol extract include flavonoids, alkaloids and tannins. Flavonoids, tannins, interquinones, cinnamates, and others have been reported to be able to protect against UV rays. The main compounds that play a role in sunscreen activity are flavonoids and tannins, which have chromoform groups that can block UV A and UV B rays.[14] Flavonoids inhibit the enzyme xanthine oxidase and impair superoxide activity.[15] However, alkaloid compounds have a mechanism of action as antioxidants by donating H atoms towards free radicals, confirming that alkaloids work as primary antioxidants.[16]

SPF test data were analyzed using non-parametric analysis using Kruskal-Wallis with Post Hoc Dunn Test. The resulting statistical analysis showed no significant effect between small concentrations of 0.04% against the control (base), and there was a considerable effect when the concentration was increased, namely at a concentration of 0.05% and 0.06% with a p-value <0.05. This study concludes that the ethanol extract of red betel leaf ethanol lotion has the best sunscreen activity in F2 and F3.

Conclusion

Overall, this study reports that betel leaf extract lotion is physically stable and has activity as a sunscreen. The results of the lotion evaluation showed that the lotion was light green with an O/W emulsion type and met the requirements, including homogeneity, pH, spreadability, viscosity, centrifugation test and freeze and thaw. The SPF values at each concentration were 0.04%, 0.05% and 0.06%. The SPF values were 13.48, 16.71, and 18.7.

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