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Determination of SPF (Sun Protection Factor) Value of Lotion Preparation of Ethanol Extract of Akway Bark (Drymis Piperita Hook F.) of West Papua

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ABSTRACT

Akway bark (Drymis piperita Hook f.) has antioxidant activity. It contains secondary metabolite compounds, one of which is flovonid so that it can be utilized as protection against UV rays and developed into pharmaceutical preparations, namely lotions. The aim of the study was to test the physical stability and determine the SPF value of lotion preparations of ethanol extract of akway bark analyzed in vitro. The test includes the determination of SPF value by UV-vis spectrophotometry method. The absorbance was measured at the wavelength of UV B light (290-320 nm) with an interval of 5 nm; the Mansur formula calculated the results obtained. The test results showed that The results of the stability test showed that the lotion preparation of akway bark extract was stable in organoleptic testing, elution type test, homogeneity test, the stickiness test, spreadability test, pH test, protective power test and the SPF value of lotions with concentrations of 0.5%, 1.0%, 1.5% obtained an average SPF value of 5.20 ± 0.00 in the medium protection category that protected the skin for 52 minutes; 11.52 ± 0.00 maximum protection that protected the skin for 115 minutes; and 5.20 ± 0.00 in the maximum protection category that protected the skin for 115 minutes. 16.00 ± 0.03 ultra protection will protect the skin for 160 minutes from UV B exposure. Thus, it can be concluded that the variation in concentration of akway bark extract affects the SPF value and can potentially be a sunscreen with moderate to ultra protection category.

KEYWORDS: Akway Bark; Sun Protection Factor; Lotion; West Papua

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I. INTRODUCTION

Sunlight exposure in tropical countries has a high intensity; sunlight can have beneficial and detrimental effects on the human body, depending on the length and frequency of exposure, the sun's power, and the exposed individual's sensitivity (Damogalad, 2013). Sunlight has benefits in synthesizing vitamin D and killing bacteria. Excessive UV exposure can increase free radicals and can cause damage to the skin, such as dry skin, sunburn, redness, irritation, premature aging, and increase the risk of skin cancer (Lann et al., 2016).

Prevention of the adverse effects of sun exposure can be minimized by using preparations applied to the skin, one of which is a lotion preparation with an SPF value. The ability of a preparation to protect the skin is based on the determination of the Sun Protection Factor (SPF) value. Based on the Food Drug Administration (2013), SPF values can be categorized as follows: minimal protection (SPF 2-4), medium protection (SPF 4-6), extra protection (SPF), maximum protection (SPF 8-15) and ultra protection (SPF >15). The higher the SPF value of a sunscreen preparation, the better its protective ability (Widyawati et al., 2019).

Bonnina et al. (1996) reported that using antioxidants in sunscreen preparations can increase photoprotective activity by absorbing excessive UV rays. Akway wood (Drymis piperita Hook f.) is an endemic plant of Papua, which contains flavonoids with high antioxidant activity. This is based on research conducted by Pratiwi et al. (2021), which reported that the results of phytochemical screening of the ethyl acetate fraction of akway bark contained flavonoid, alkaloid, terpenoid, and quinone compounds. Ethanol extract of stem bark has a solid antioxidant content with an IC50 value of 25.6 ppm, and akway leaves have a strong

antioxidant content with an IC50 value of 50.95 ppm (Marpaung, 2008).

The results showed that, the ethanol extract of akway bark had a tonic effect on mice that were tranquilized (Pratiwi et al, 2018), the ethyl acetate extract of akway bark had a stimulant effect on mice that were tranquilized (Pratiwi and Simaremare, 2020), akway bark fraction has anti-inflammatory activity tested on male mice (Pratiwi and gunawan, 2021) and ethanol extract of akway bark has cytotoxic activity using the BSLT method (Pratiwi et al, 2022).

Another study also conducted by Apriliani (2018), showed that extracts and fractions of akway bark have the highest antioxidant activity content, namely, 9.93 ppm and 9.07 ppm. Previous research by Nishanthini et al. (2012) reported that secondary metabolites such as flavonoids and phenols are antioxidants and can ward off free radicals. Flavonoids have the ability as a sunscreen due to the presence of chromophore groups (conjugated single, double bonds) that can absorb UV rays, thus reducing their intensity on the skin (Shovyana & Zulkarnain, 2013). Aside from being an antioxidant, the flavonoid content in akway bark can also be utilized as an active substance in sunscreens.

Sunscreen is a skincare cosmetics designed to reduce the harmful effects of UV exposure by inhibiting the penetration of UV rays into the skin. Sunscreen can be made in various pharmaceutical preparations, including a lotion preparation. The lotion was chosen because it is easy to apply to the skin, and its consistency is not too dense, so it is easy to spread evenly and quickly on a large skin surface. Based on the above studies, it is necessary to research and determine the SPF value of lotion preparations of ethanol extract of akway bark using UV-Vis spectrophotometry.

II. RESEARCH METHODS

A. Tools

Glassware, sieve, stir bar, vial bottle, filter paper, pipette, analytical balance, mortar, stamper, water bath, rotary evaporator, thermometer, vial bottle, desiccator, watch glass, glass jar, 15 cm glass plate, object glass, weighing scale (5g, 10g, 50g, 100g, 500g).

B. Material

Akway (Drymis piperita) bark simplisia extract, stearic acid, sulfuric acid, distilled water, ethanol p.a, ethanol 96%, potassium dichromate, triethanolamine, propyleneglycol, cetyl alcohol, liquid paraffin, phenoxyethanol, DMDM hydantoin, cherry blossom fragrance and a market lotion preparation with SPF.

C. Extract Preparation

400 g of powder was weighed and then extracted by maceration using 96% ethanol as much as 2 L for three days, stirring once every 24 hours, then remacerated. After that, the macerate is filtered and collected to evaporate the solvent using a rotary vacuum at 50 ° C to obtain a thick extract.

D. Ethanol Free Test

Performed by adding two drops of H_2SO_4 (sulfuric acid) and 1 mL of potassiumdichromate into 1 g of akway bark extract, the presence of ethanol in the extract is indicated by a color change from orange to bluish-green (Harbone, 1987).

E. Lotion Making

Lotions were made according to the formulation listed in Table 1. The oil phase ingredients (stearic acid, cetyl alcohol, liquid paraffin, and phenoxyethanol) were placed in a glass beaker. The aqueous phase (triethanolamine, propylene glycol, DMDM hydantoin, and distilled water) was put in a glass beaker. The oil and water phases were heated separately in a water bath 70-75ºC. After everything was melted, the oil phase was poured slowly into the heated mortar while stirring, followed by pouring the water phase while still mixing until a lotion mass was formed. Then, add akway bark extract and fragrance to the mixture while stirring until homogeneous and put in a container.

Table 1. Formulation of Akway Bark Ethanol Extract Lotion Formulation

Ingredients	Formula (%) (b/v)			Ingredient Function
	FI	FII	FIII	
Ethanol extract of bark akway	0,5	1	1,5	Active substance
Triethanolamine	2	2	2	Emulsifiers
Stearic acid	10	10	10	Emulsifiers
Propylene glycol	5	5	5	Humectant
Cetyl alcohol	1	1	1	Thickener
Liquid paraffin	1,5	1,5	1,5	Emolient
Phenoxyethanol	0,5	0,5	0,5	Preservatives
DMDM hydantoin	0,5	0,5	0,5	Preservatives
Fragrance cherry blossom	Six drops	Six drops	Six drops	Fragrance
Aquadest add	100	100	100	Carrier

Description:

F I: Formulation of lotion with 0.5% concentration of ethanol extract of akway bark

F II: Formulation of lotion with a concentration of ethanol extract of akway bark 1.0%

F III: Lotion formulation with 1.5% concentration of ethanol extract of akway bark

K (-): Negative control (lotion base)

K (+): Positive control (market lotion preparation)

F. pH testing

This pH testing is done using a universal pH indicator. The lotion preparation is weighed as much as 0.5 grams diluted with distilled water; the pH paper is allowed to stand

in the lotion solution until it changes color. The color that appears matches or is compared to the standard color on the universal indicator pH package. The stable reading result is the pH of the lotion, which is expected to be the same as the skin's pH, which is 4.5-7.0 (Sipahelut, 2020). This test was replicated three times.

G. Physical Quality Test

Organoleptic Testing: Organoleptic testing involves using the five human senses (30 panelists) to observe color, aroma, and texture changes.

Emulsion Type Test: Several lotion preparations are placed on filter paper; if the filter paperbecomes wet, the emulsion produced is oil in water type (O/W). If oil stains appear on the filter paper, the emulsion is water in oil (W/O).

Homogeneity Testing: This homogeneity test uses a glass object with a certain amount of preparation. When applied to a piece of glass or other suitable transparent material, the preparation must be homogeneous, and no coarse grains are visible. This test was replicated three times.

Adhesive Power Testing: This lotion adhesion testing is done by weighing the lotion preparation, as much as 0.5 grams, placed on a glass object, then covered with another glass object, and given a load weighing 500 grams for 1 minute. The two glass objects that have been attached are mounted on a test device that is given a load weighing 65 grams. The time required for the two glass objects to separate was recorded . This test was replicated three times.

Spreadability Testing: Testing the spreadability of lotion is done by weighing the lotion preparation as much as 0.5 grams, placing it in the middle of a glass plate with a diameter of 15 cm, and above the preparation, placing another glass plate as a cover. Then, given a load weighing 50 grams above the glass cover, it was allowed to stand for 1 minute. Using a ruler, measurements were taken vertically and horizontally on the diameter pattern formed. The same treatment was also repeated for the addition of 100 grams and 150 grams. The calculation results of the average value of both are determined as the diameter of the spreadability. This test was replicated three times.

H. Protective Power

The protective power test assesses whether the lotion preparation base can protect the skin from external influences. The longer the time required for the PP indicator to react with KOH, the better the protection power of the lotion produced.

I. Stability Test

The formula is then poured into a container and stored for 21 days at room temperature. The lotion, including organoleptic, pH, homogeneity, spreadability, and adhesion tests, are evaluated.

J. Determination of SPF Value

A total of 1 g of each lotion sample of ethanol extract of akway bark (Base, F1, F2, F3) and K+) was weighed and dissolved in 5 mL of ethanol p.a, then filtered with filter paper into a 10 mL volumetric flask, added ethanol p.a until the limit mark and homogenized. After that, the diluted solution was taken as much as 2 mL and put into a 10 mL volumetric flask, and ethanol p.a was added until the limit mark was homogenized. Put into a vial bottle. Then, a test absorption curve was made in a cuvette with a length of 1 cm at a wavelength of 290-320 nm, using ethanol p.a as a blank. A total of 4 mL of sample was put into the cuvette. The SPF value was calculated using the Mansur.

$$_{\text{Spectrophotometric}}\ \text{SPF} = \text{CF} \times \sum_{290}^{320} \text{KK}(\lambda) \times I(\lambda) \times Abs(\lambda)$$

Description:

CF : correlation factor (10)
EE : erythema efficiency
I : simulated spectrum of sunlight
Abs : absorption value read

III. RESULTS AND DISCUSSION

A. Extraction Result

The extraction process of Akway bark (Drymis piperita Hook f.) in this study used the maceration method because this method is carried out by immersion and without heating to reduce the decomposition of active compounds (Nainggolan et al., 2018). Ethanol 96% was chosen as a solvent because it is more selective, safe, volatile, not quickly overgrown with fungi and bacteria, and increases membrane permeability so that absorption is good and ethanol is polar so that it can attract polar compounds such as flavonoids, saponins and others (Liswandari et al., 2018). The yield results of akway bark extract obtained the following results:

Table 2. Yield of Akway Bark Extract (Drymis piperita Hook .f)

Weight (g)	**Yield**	
Simplisia	Extract	(%)
400	117.1	29.3

B. Eethanol Free Test

The results of ethanol-free testing on akway bark extract can be seen in the following table:

Table 3. Ethanol Free Test Results

Sample	**Work steps**	**Test results**	**Description**
Extract	1 mL $K_2Cr_2O_7$ +2 drops H_2SO_4	Fixed color chocolate	Ethanol-free

The results of the ethanol-free test show that there is no more ethanol in the akway bark extract; this is because

there is no color change in the sample when tested with potassium dichromate ($K_2Cr_2O_7$) and sulfuric acid (H_2SO_4). The ethanol-free test is carried out to free the extract from ethanol so that it does not affect the lotion preparation process (Prasongko, 2020).

C. Stability Test Results

Organoleptic Testing: Organoleptic testing determines the quality of lotion preparations based on color, aroma, and texture. According to 30 respondents who have filled out questionnaires on day 0 and day 21. Organoleptic testing of each lotion formula of ethanol extract of akway bark, the results are obtained in the following table:

Table 4. Organoleptic evaluation results of lotion formula of ethanol extract of akway bark

Formula	Organoleptic Test	Day-0	Day-21
FI	Color	Brown	Brown
	Aroma	Typical fragrance	Typical fragrance
	Texture	Soft and Non-Sticky	Soft and Non-Sticky
FII	Color	Milk Chocolate	Milk Chocolate
	Aroma	Typical fragrance	Typical fragrance
	Texture	Soft and Non-Sticky	Soft and Non-Sticky
FIII	Color	Milk Chocolate	Milk Chocolate
	Aroma	Typical fragrance	Typical fragrance
	Texture	Soft and Non-Sticky	Soft and Non-Sticky

The results of the organoleptic evaluation showed color differences produced between FI, FII, and FIII, namely FI is brown, while FII and FIII have the same color, namely milk chocolate; differences influence this in the concentration of the active substances used, namely FI (0.5%), FII (1%), and FIII (1.5%). The typical fragrance aroma dominates the lotion aroma because the fragrance material used to manufacture this lotion preparation formula is cherry blossom. The lotion preparation has a soft and non-sticky texture; this is related to mixing the components of the lotion-forming ingredients well.

In the stability test on day 21, organoleptically, there were no changes in color, aroma, or texture, which means that the lotion preparation was still stable from day 0 to day 21.

Emulsion TypeTest: The results of the lotion emulsion type can be seen in the table below.

Table 5. Emulsion Type Test ResultsFormula Type

Formula	Emulsion Type (Day 0)	Emulsion Type (Day 21)
F I	O/W	O/W
F II	O/W	O/W
F III	O/W	O/W
K (-)	O/W	O/W

Description:

K (-): Negative control (Lotion base), M/A: Oil in water

The results of testing the emulsion type of lotion preparations in FI, F II, and F III have an oil-in-water emulsion type (M/A), indicated by wet filter paper after placing thelotion. This is because the amount of oil phase used is smaller than the water phase, so the oil phase will dissolve evenly into the water and form an oil-in-water phase assistedby emulgators (Sirajuddin et al., 2013). This emulsion is easy to rinse with water, spreads quickly, and is not sticky (Daud, 2016).

Homogeneity Testing: Homogeneity testing is done to determine whether the active substance in the lotion is evenly mixed with lotion-forming ingredients so that the active substances contained in the lotion preparation can be spread evenly and cause appropriate and maximum therapeutic effects. The average results of the homogeneity evaluation of the lotion formula of ethanol extract of akway bark can be seen in the following table:

Table 6. Results of homogeneity evaluation of lotion formula of ethanol extract of akway bark

Formula	Homogeneity (+/-)		Stability (Yes/No)
	Day ke-0	Day ke-21	
FI	+	+	Yes
FII	+	+	Yes
FIII	+	+	Yes

Description:

+ : Homogeneous

-: Not Homogeneous

The research results on the lotion preparation formula show the absence of granules on the glass object. Based on SNI 16-4399-1996, the lack of spots indicates a homogeneous preparation composition, so the lotion preparation has met the SNI. Therefore, it can be said that the lotion preparation already has good homogeneity properties.

Factors that affect the lotion preparation are homogeneous due to the addition of emulsifying agents to the preparation, which is assisted by constant stirring, and the appropriate temperature, which will accelerate the formation of emulsions. The temperature must be relevant when mixing the lotion ingredients because it can affect the compaction too fast. Prolonged stirring can expand the contact area with increasing stirring speed, thus increasing the homogeneity of a mixture.

The observation of stability inhomogeneity on day 21 showed no change, so it can be concluded that the lotion preparation is still stable from day 0 to day 21.

Adhesion Testing: Adhesive power testing determines the length of time the lotion sticks to the skin. A good lotion can guarantee adequate contact time with the skin to achieve its intended use, but it should not be too sticky (Sipahelut, 2020).

Table 7. Results of the evaluation of the stickiness of the lotion formula of ethanol extract of akway bark

Formula	Average Adhesion Test (sec) ± SD		Terms (Betageri and Prabhu, 2002)
	Day-0	Day-21	
FI	7,8 ± 0,4	8,1 ± 0,4	2 - 300 sec
FII	4,6 ± 0,3	4,9 ± 0,3	
FIII	2,2 ± 0,3	2,6 ± 0,4	

The results of the average evaluation of the adhesion of each lotion preparation formula of ethanol extract of akway bark on day 0 obtained FI (7.8 seconds), FII (4.6 seconds), and FIII (2.2 seconds), then on day 21 received the results of FI (8.1 seconds), FII (4.9 seconds), and FIII (2.6 seconds). Reasonable adhesion time requirements are 2 - 300 seconds. The results of the average time off object glass lotion adhesion test meet the requirements of good adhesion time because the results obtained are more than 2 seconds, so it can be concluded that the results of testing the adhesion of lotion sedan formula ethanol extract of akway bark meet the requirements.

Based on the adhesion test obtained, each formula has a different adhesion. This is due to the difference in viscosity of each formula, which occurs due to variations in the concentration of other active substances.

Spreadability Testing: Scatterability testing aims to determine the ability of lotion to spread when applied to the skin. Good-quality lotions must have sufficient spreadability so that their active substances can be dispersed evenly and provide maximum therapeutic effect. The greater the spreadability of the preparation, the faster the release of the desired therapeutic effect in the skin.

Table 4. 6 Results of the evaluation of the spreadability of the lotion formula of ethanol extract of akway bark

Average Spreadability Test (cm) ± SD			
Formula	Load (150 grams)		Terms
	Day-0	Day-21	
FI	6,1 ± 0,1	6 ± 0,1	5 - 7 cm
FII	8,4 ± 0,1	8,1 ± 0,1	
FIII	9,6 ± 0	9,4 ± 0	

Based on the results of the evaluation of the average spreadability of the lotion preparation formula of ethanol extract of akway bark, the spreadability of lotion with a load of 150 grams on day 0 is obtained, namely, FI = 6.1 cm, FII = 8.4 cm, FIII = 9.6 cm, and on day 21 with the same load, namely, FI = 6 cm, FII = 8.1 cm, FIII = 9.4 cm.

D. pH testing

Testing the preparation's pH aims to determine whether it is alkaline or acidic, **ensuring** its safety and that it does not cause irritation to the skin.

Table 8. Results of pH evaluation of lotion formula of ethanol extract of akway bark

Formula	Average pH Test ± SD		Stability (Yes/No)	Terms (SNI)
	Hari ke-0	Hari ke-21		
FI	6 ± 0	6 ± 0	Yes	4,5 - 8
FII	6 ± 0	6 ± 0	Yes	
FIII	7 ± 0	7 ± 0	Yes	

The average evaluation results of pH in the lotion preparation formula of ethanol extract of akway bark are FI (6), FII (6), and FIII (7); seen from the data obtained, the pH value of the lotion preparation is still by the pH of the skin which is 4.5 - 7, so the lotion is safe to use. Based on SNI 16-4399-1996, the pH of semisolid preparations is in the range of 4.5 - 8, so the lotion preparation made has met the quality requirements of SNI. If the lotion preparation has a pH of 9-14, it will cause skin peeling, while if it has a pH of 1-4, it will irritate the skin.

The stability of lotion preparations in the pH test on day 21 did not show any changes in each formula, so it can be said that the lotion preparation formula is still stable from day 0 to day 21.

E. Protection Power Testing

Protection power testing is done to determine the ability of protection or protection against external influences such as dust, pollution, and sunlight that can reduce the effectiveness of lotions. This protection power test uses phenolphthalein (PP) and KOH 0.1 N as indicators.

Table 9. Protective power evaluation results of akway bark extract lotion formula

Formula	Protection Time Day- 0					
	15 sec	30 sec	45 sec	60 sec	3 minutes	5 minutes
FI	-	-	-	-	-	-
FII	-	-	-	-	+	+
FIII	-	-	-	+	+	+
Formula	Protection Time Day- 21					
	15 sec	30 sec	45 sec	60 sec	3 minutes	5 minutes

FI	-	-	-	-	-	+
FII	-	-	-	+	+	+
FIII	-	+	+	+	+	+

F. **Determination of SPF Value**

SPF value testing aims to determine the ability of akway bark extract lotion preparation to absorb radiation that hits the skin. This test was conducted in vitro using the spectrophotometric method at a wavelength of 290-992.

320 nm (UV B spectrum); this is because the effects of UV B exposure can penetratethe outermost layer of the skin (epidermis), causing burning on the skin and causing erythema (redness). The results of the SPF value of ethanol extract of akway bark canbe seen in Table 6 below.

Table 9. SPF value results

Formula	Mean value SPF ± SD	Category
F I	5.20 ± 0.00	Medium
F II	11.52 ± 0.00	Maximum
F III	16.00 ± 0.03	Ultra
K (-)	0.00 ± 0.00	-
K (+)	29.00 ± 0.01	Ultra

Based on Table 5, the SPF measurement results of akway bark extract lotion in FI has an SPF value of 5.20 ± 0.00 including the medium protection category that protects the skin from UV B exposure for 52.0 minutes; FII with SPF 11.52 ± 0.00 maximum protection category that can protect the skin for 115.2 minutes (1 hour 55 minutes); and FIII with SPF 16.00 ± 0.03 belongs to the ultra protection category which can protect the skin for 160 minutes (2 hours) from UV B exposure, while the test carried out on the base (negative control) has no effect as a sunscreen with the results obtained 0.00 ± 0.00 because it only consists of carrier ingredients without the addition of akway bark extract. This is different from the SPF value of the positive control, which is a sunscreen with an ultra protection category (SPF 29.00 ± 0.01); this isbecause the active substance of the preparation is a combination of several active substances, such as phenylbenzimidazole sulfonic acid, butyl methoxydibenzoylmethane, titanium dioxide, and octocrylene. The division of SPF values according to the FDA isas follows: SPF 2-4 (minimal protection), SPF 4-6 (extra protection), SPF 8-15 (maximum protection), and SPF >15 (ultra protection).

CONCLUSIONS

Based on the results of the study, it can be concluded that as follows:

1. The physical quality of the lotion preparation of ethanol extract of akway bark Formulation I, II, and III meets the requirements of the physical quality test of semi-solid preparations.
2. Akway bark ethanol lotion Formulations I, II, and III are stable based on organoleptic test, emulsion type, homogeneity, adhesion, spreadability, and protective power.
3. Ethanol extract lotion preparations with varying concentrations, namely FI (0.5%), FII (1.0%), and FIII (1.5%), have the potential as sunscreens with an average SPF value of FI (5.20) in the medium protection category, FII (11.52) in the extra protection category, and FIII (16.00) in the ultra protection category.

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