

A PILOT STUDY ON SUN PROTECTION FACTOR of ETANOL EXTRACT LOTION of PAPUAN GRAPE FRUIT (*Sararanga sinuosa* Hemsley.) AN OBSERVATIONAL STUDY

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ABSTRACT

Papuan grapes contain phenolic compounds, namely flavonoids and tannins, which have antioxidant potential. This study aims to determine the physical quality of Papuan grape ethanol extract lotion preparation and determine the SPF (Sun Protection Factor) value of Papuan grape ethanol extract lotion preparation. Physical quality evaluation includes organoleptic test, homogeneity, pH, adhesion, spreadability, emulsion type, centrifugation test, and SPF value by UV-Vis spectrophotometric method. The absorbance of the sample was read at a wavelength of 290-320 nm with an interval of 5 nm. The results of the study obtained lotion preparations of milk chocolate color, smells typical of the base, soft and not sticky, homogeneous, pH of the base, formula I, formula II and formula III are 7.3; 7.0; 7.0, and 7.0. The stickiness and spreadability of the lotion meets the requirements, the emulsion type is M/A, the centrifugation test does not occur separation and the SPF value on the base formulation, FI (0.5%); FII (1%); and FIII (1.5%) namely, respectively base (1); weak protection category, FI (7) medium protection category, FII (9); strong protection category and FIII (10); strong protection category. All formulas obtained good physical quality and potential as sunscreens.

KEYWORDS: Papuan grape, Lotion, *Sararanga sinuosa* Hemsley, SPF, Extract

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I. INTRODUCTION

Indonesia has abundant natural wealth and natural resources. Some regions in Indonesia are famous for their good natural resources. Papuan Province is one of them, Papuan has many endemic plants and one of the plants that has not been maximally utilized in the cosmetic field is Papuan grapes (*Sararanga sinuosa* Hemsley.).

The *Sararanga sinuosa* plant can be found in three regions, namely Yapen, Depapre, and Waropen. People usually consume the fruit as supplements and vitamins [1]. Papuan grape is a plant from the pandanaceae family which is only found in Papuan and the Philippines [2]. Papuan grape fruit extract contains secondary metabolite compounds, namely alkaloids, flavonoids, tannins, and saponins [3].

Flavonoids and tannins are phenolic compounds that have antioxidant potential and as natural sunscreens due to the presence of chromophore groups

that are able to absorb strong UV rays and protect plant tissues against damage due to solar radiation [4]. The chromophore group is a conjugated aromatic system that has the ability to absorb strong light in the UV wavelength range [5] Based on research [6] Papuan grape plants with antioxidant tests using the DPPH method (2,2-diphenyl-1-picrylhydrazyl) ethanol extract of Papuan grape fruit has an IC_{50} value of 5.41 ppm with an AAI value of 7.57 which means very strong antioxidant activity.

Naturally, the skin has a defense system against the toxic effects of sun exposure, such as sweat production, melanin formation, and stratum corneum thickening. However, when exposed to excessive sunlight, the epidermal tissue of the skin is unable to resist these negative effects, resulting in erythema and sunburn (sunburn) degenerative changes in the skin or premature aging and skin cancer. Various ways can be done to overcome the bad effects of sunlight, one of

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which is by using sunscreen preparations. Sunscreen is a cosmetic preparation used to reflect UV exposure or absorb it in order to reduce the amount of UV radiation that is harmful to the skin [7]. Sunscreen that is often used is lotion. Lotion preparations are widely used by the public because it is easier to apply to the skin, because the consistency is not too dense, so it can spread more evenly and quickly on a large surface of the skin [8].

The effectiveness of sunscreen is based on the measurement of SPF (Sun Protection Factor) value. SPF is a general indicator that explains the effectiveness of a product or substance that functions as a UV protector, where the SPF value of sunscreen is high, the ability to protect the skin from sunburn is also greater [9]. Based on previous research, the antioxidant activity of Papuan grapes is very strong with an AAI value of 7.57, then Papuan grapes can be utilized and developed into a sunscreen product in the form of lotion preparations. In order for sunscreen lotion products to be effective, research was conducted on the determination of the SPF (*Sun Protection Factor*) value of ethanol extract of Papuan grapes (*Sararanga sinuosa* Hemsley.) using UV-Vis spectrophotometric method.

II. MATERIAL AND METHODS

A. Materials

Papuan grape fruit (*Sararanga sinuosa* Hemsley.), analytical scales, pH meter, UV-Vis spectrophotometer Shimadzu 1601, centrifugator, vacuum rotary evaporator, blender, water bath, hand mixer, stopwatch, food slicer, mesh no.80, glassware, glass funnel, glass jar, lotion container, filter paper, preparation glass, scales, glass plate, ethanol pro analysis, ethanol 96%, distilled water, concentrated H₂SO₄, K₂Cr₂O₇, Mayer reagent, Dragendroff reagent, Mg powder, HCl 2N, ethanol 96%, amyl alcohol, CHCl₃, anhydrous acetic acid, stearic acid, cetyl alcohol, liquid paraffin, triethanolamine, glycerin, DMDM Hydantoin.

B. Research Methods

Simplicia Preparation

Green-colored Papuan grapes (*Sararanga sinuosa* Hemsley.) were collected from Depapre, Jayapura Regency, Papuan. Furthermore, the Papuan grapes were wet sorted and then washed thoroughly with running water. The washed Papuan grapes were then weighed as much as 6 kg and dried. The dried simplicia of Papuan grapes was then dry sorted and mashed using a blender until it became powder and sieved using mesh no. 80.

Extract Preparation

The Papuan grape fruit simplicia powder weighed as much as 750 g and was soaked in 96% ethanol as much as 7.500 mL in a ratio of (1:5). The fine simplicia was then extracted by maceration method with remaceration every 1 x 24 hours for three days, with stirring every day. The extraction results were

then filtered to separate the filtrate and macerate using filter paper and funnel. The maserat was then concentrated using a *rotary evaporator* and water bath at 50°C to obtain a thick extract. The extract yield is then estimated using the formula:

$$\% \text{ extract yield} = \frac{\text{Weight of extract}}{\text{Weight of simplisia}} \times 100\%$$

Ethanol Free Test

The ethanol extract of Papuan grapes is tested for 96% ethanol-free using a qualitative test by adding 2 drops of concentrated H₂SO₄ and 1 mL of K₂Cr₂O₇ solution to the drip plate containing the extract and one drip plate as a control, the presence of ethanol content in the extract is indicated by a change in color from orange to bluish green [10].

Preparation of Papuan Grape Fruit Ethanol Extract Lotion

The formulation of M/A or A/M *lotion* of ethanol extract of Papuan grapefruit is based on research [11] by making modifications to the additional ingredient. This *lotion* preparation was made by mixing two phases, namely the oil phase consisting of stearic acid, cetyl alcohol, and liquid paraffin with the water phase consisting of triethanolamine, glycerin, and DMDM *hydantoin* dissolved in distilled water with stirring. Making *lotion* is done through 3 stages of work, first preparation A which is an oil base, then continued making preparation B which is a water base then finally mixing *lotion* with extract.

Table 1. Formulation of Papuann Grape Fruit Ethanol Extract Lotion

Material	Content (%b/v)			
	Base	FI	FII	FIII
Ethanol Extract of Papuan Grapes	-	0,5	1	1,5
Cetyl alcohol	0,5	0,5	0,5	0,5
Liquid paraffin	7	7	7	7
Stearic acid	5	5	5	5
Glycerin	5	5	5	5
Triethanolamine	1	1	1	1
DMDM <i>hydantoin</i>	0,2	0,2	0,2	0,2
Aquades add	100	100	100	100

Description: FI: Formula I; FII: Formula II; FIII: Formula III; Base: all material without extract.

C. Physical Quality Evaluation of Lotions

Organoleptic test

Organoleptic tests are carried out by observing the organoleptic *lotion* preparation including color, aroma, and taste. In organoleptic testing, panelists are needed to assess the nature or quality of the preparation based on subjective impressions. The

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required panelists consisted of 20 people who had good enough sensitivity.

Homogeneity test

Homogeneity test is carried out using a glass object or glass plate with a certain amount of preparation when applied to a piece of glass or other suitable transparent material, the preparation must show a homogeneous arrangement and no coarse grains are visible [12] This test was replicated 3 times.

pH Test

The lotion preparation sample was made in a concentration of 1%, which was weighed as much as 1 gram and dissolved in distilled water. The electrode is dipped in the solution until the pH meter shows the pH price as constant. The number shown by the pH meter is the pH of the lotion preparation [13]. The stable reading is the pH of the lotion, which is expected to be the same as the pH of the skin which is 4.5-7.0. This test was replicated 3 times.

Adhesion Test

The lotion adhesion test is done by weighing the lotion preparation as much as 0.5 g placed on a glass object and then covered with another glass object and given a load weighing 500 g for 1 minute and then the two glass objects that have been attached to each other are mounted on a test device that is given a load weighing 65 g. The time required until the two glass objects can separate. The time required for the two glass objects to separate was recorded [14]. This test was replicated 3 times.

Spreadability Test

The lotion spreadability test is done by weighing the lotion preparation as much as 0.5 g and 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 a load weighing 50 g above the glass cover, is allowed to stand for 1 minute. Measurements were taken vertically and horizontally on the diameter pattern formed using a ruler. The same treatment was also repeated for the addition of a 100 g load. The results of the calculation of the average value of both were determined as the diameter of the spreadability. This test was replicated 3 times.

Emulsion Type Test

The lotion type test is carried out by the dilution method, by inserting a little lotion added to water and stirring. If a homogeneous emulsion is obtained again, then the emulsion is of type M/A. If the lotion is mixed with oil, it will cause the rupture of the emulsion so that an A / M type emulsion is obtained [15].

Centrifugation Test

A total of 2 g of sample was put into a test tube and capped after which it was put into a centrifuge and spun for 30 min at 3,000 rpm at room temperature. The lotion was evaluated for phase separation at the end of centrifugation [16].

D. Sun Protection Factor (SPF) Activity Testing

Taken 5 mg of Papuan grape fruit ethanol extract lotion, dissolved in 10 mL of ethanol p.a (concentration 500 ppm) until homogeneous from each concentration (base; 0.5%; 1%; 1.5%). Then put into a 10 mL volumetric flask and added ethanol p.a until the limit mark [17]. After that, a cuvette absorption curve with a length of 1 cm was made at a wavelength between 290 nm and 320 nm, and ethanol p.a was used as a blank. Absorbance readings were taken in the UVB wavelength range of 290 nm to 320 nm with a wavelength interval of 5 nm. The absorbance that appeared on the UV-Vis spectrophotometry was recorded. The SPF value was calculated using the Mansur formula:

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

Description: SPF: Sun Protection Factor; EE(λ): Erythema effect; I(λ): Solar intensity; Abs (λ): Absorbance of sunscreen; CF: Correction factor (10) [18].

Table 2. $KK \times I$ values at wavelengths of 290-320 nm

Wavelength (nm)	EE × I
290	0,0150
295	0,0817
300	0,2874
305	0,3278
310	0,1864
315	0,0839
320	0,0180

Table 3. Sunscreen Protection Level Based on Ultraviolet Index Value

SPF	Sunscreen Category	Protection
1-2	Weak protection	
3-7	Medium protection	
8-10	Strong protection	
≥11	Maximum protection	

Description: 1-2: No protection; 3-7: required protection 8-10: more protection; ≥11: extra protection [19]

E. Data Analysis

Data analysis in this study in organoleptic testing data is presented in the form of tables and descriptive narratives while testing the *Sun Protection Factor* (SPF) value is statistically

III. RESULTS AND DISCUSSION

A. Results of Extraction

Table 1. Extraction results of *Camposperma brevipetiolatum* Volkens

Sampel	Simplici a Weight	Volum e of	Extract Weight	Extrac t Yield
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	(g)	Solvent (mL)	Obtained (g)	(% b/v)
<i>Camposperma brevipetiolatum</i> Volken	750	7.500	51.5	10.3

The extraction operation is carried out using cold extraction, namely the maceration method, as given in table 1. The maceration method of the simplicia of *Camposperma brevipetiolatum* Volken as much as 750 g was soaked three times in 24 hours in 96% ethanol as much as 7.5 L and agitated once every 24 hours before filtering with filter paper. The filtrate was then evaporated in a rotary vacuum evaporator at 50°C until a thick extract was obtained.

B. Ethanol Free Results

Results Ethanol-free testing obtained brownish-orange results which means the extract is free of ethanol.

C. Physical Quality Evaluation of Lotion Results

The results of the organoleptic evaluation carried out with a questionnaire filled out by 20 panelists showed differences in the color

Organoleptic Results

The results of the organoleptic evaluation carried out with a questionnaire filled out by 20 panelists showed differences in the color produced and differences in color density. One sign of a chemical reaction to the ingredients mixed is a change in color, so the results obtained change the color of milk chocolate. This can be influenced by the different concentrations of extracts used as 0.5 g, 1 g, and 1.5 g for each formula.

The extract is blackish brown, so that when mixed with the base (white) the resulting color is milk chocolate. The color of the preparation is also rather shiny or shiny which comes from the constituent ingredients of the *lotion*, namely stearic acid, based on the *handbook of pharmaceutical excipients* mentioned stearic acid can give a distinctive shine to the *lotion*.

The odor in the *lotion* is dominated by the odor of the base because the *lotion* preparation formulation does not use fragrance ingredients. The *lotion* preparation has a soft and non-sticky texture, this is related to the presence of a good mix of *lotion-forming* material components, namely cetyl alcohol to increase the stability of the emulsion, liquid paraffin as an *emollient*, glycerin as a *humectant* and *emollient*. *Emollient* is a softener, which with the addition of *emollient* substances can make the skin feel comfortable, not dry, and not greasy [20].

Homogeneity Results

The homogeneity test was carried out in two ways, namely, with a glass plate or glass object and observation through a microscope. The results of the homogeneity check of the *lotion* preparation are on average homogeneous.

pH Results

pH testing is very necessary in topical preparations because the skin has a sensitivity to acidity. If the preparation is too acidic it will cause irritation, while if the preparation is too alkaline it can cause scaly skin [21]. The measurement results show that the pH of the preparation is 7.3 for the base and pH 7 for FI, FII, and FIII, which is neutral. This is in accordance with the pH of the skin 4.5-7.0 [22]. The addition of extract concentration in each formulation causes a decrease in pH in the *lotion preparation* formulation. This is because of the phenol content that decomposes in polyphenol compounds contained in the ethanol extract of Papuan grapes. The decomposition that occurs causes the amount of H⁺ to increase with the addition of extracts to the *lotion* so that the pH decreases. However, the pH value is still within the ideal pH range. The pH value obtained is not far away because there is no difference in the concentration of additional ingredients and there is only a difference in extract concentration, but the preparation is safe to use. Based on SNI 16-4399-1996 the pH in *lotion* is between 4.5-8.0. So that the *lotion preparation* that has been made meets the quality requirements of SNI.

Adhesion Results

Testing the adhesion of the preparation aims to determine the ability of the *lotion* to adhere and coat the surface of the skin in order to function optimally. Preparations that can adhere well to the skin will also optimize use avoid repeated use and save usage [23]. The average adhesion test results can be seen in the table below. From the results of the average evaluation of the adhesion of the base and the three *lotion* formulas presented, the adhesion of the base *lotion* (5.38), FI (3.81), FII (3.79), and FIII (3.15) was obtained. Good adhesion time requirements are in the range of 2-300 seconds [24].

In the data obtained as the extract increases the adhesion obtained decreases, this can be due to the high water content in the Papuan grapefruit extract, it is known that the water content of Papuan grapes is 86.88 g when compared to other types of fruit such as avocado, durian, soursop, langsung, papaya, rambutan, and salak, the water content of Papuan grapes is much higher [25]. So as the concentration of extract increases, the viscosity decreases and the adhesion test also decreases. However, the average release time of the object glass *lotion* adhesion test still meets the requirements of a good adhesion time because the average results obtained are more than 2 seconds, so it can be concluded that the results of the adhesion test of each formula of Papuan grape fruit ethanol extract *lotion* are qualified.

Spreadability Results

The spreadability test was carried out with the aim of seeing the ability to spread the preparation *lotion* at the time of application on the skin surface. *Lotions* are expected to spread easily when applied without requiring significant pressure. The greater the diameter value of the spreadability, the greater the surface area

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that can be reached by the *lotion*. The results of the spreadability test can be seen in the table below.

Table 4. Results of the *Lotion* Spreadability Test

Spreadability test mean (cm) ± SD					
Load (gram)	Formula				Conditions (Grag, et al., 2002)
	Base	FI	FII	FIII	
0	6.37 ± 0.32	6.47 ± 0.15	6.70 ± 0.26	7.40 ± 0.10	5.00-7.00 cm
50	6.17 ± 0.06	7.13 ± 0.12	7.30 ± 0.20	7.53 ± 0.06	
100	6.43 ± 0.12	7.40 ± 0.10	7.57 ± 0.06	7.60 ± 0.10	

From the results of the evaluation of the average spreadability of the three *lotion* formulas presented, it was found that the spreadability with the addition of 0, 50, and 100 g load had different spread results in each formulation, this stated that the addition of Papuan grape ethanol extract concentration had the effect of increasing the spreadability of the *lotion* while reducing the viscosity of the preparation. The spreadability of semi-solid preparations is around 5-7 cm [26], so the spreadability of Papuan grape fruit ethanol extract lotion of each formula is good because it is in the range of *lotion* spreadability according to requirements.

Emulsion Type Results

Based on the data obtained, the average emulsion type is M/A. The emulsion type of the preparation is also caused by the use of emulgators that tend to be more soluble in water, namely triethanolamine [27]. In addition, it can also be caused by the solvent used in the manufacture of this *lotion* which is distilled water [14].

Centrifugation Results

This test was conducted to determine the stability of the *lotion* after shaking at high speed using a centrifuge. Based on the data obtained, the average centrifugation test showed that the base, FI, FII, and FIII did not occur phase separation. So that the Papuan grape fruit ethanol extract *lotion* formula is qualified.

D. Sun Protection Factor (SPF) Value Determination Results

Measurement of the effectiveness of sunscreen preparations is based on the determination of the *Sun Protection Factor* (SPF) value which describes the ability of sunscreen products such as *lotion* preparations. The results of the calculation of the SPF (*Sun Protection Factor*) value of ethanol extract of Papuan grapes (*Sararanga sinuosa*) at several concentration variations can be seen in the table below.

Table 5. SPF Value Results

Formula	Mean SPF value ± SD	Category
Base	1 ± 0.03	Weak Protection
F I (0.5%)	7 ± 0.02	Medium Protection
F II (1%)	9 ± 0.01	Strong Protection
F III (1.5%)	10 ± 0.02	Strong Protection

Based on the data obtained, it can be concluded that the SPF values of the base formulation, FI (0.5%); FII (1%); and FIII (1.5%) are, respectively base (1); weak protection category > FI (7) medium protection category > FII (9); strong protection category > FIII (10); strong protection category. Based on the SPF value, it can be seen that the SPF with good protection is at a high concentration of FIII (1.5%) which is 10. The SPF value of the *lotion* formula of ethanol extract of Papuan grape fruit increases with increasing concentration of the extract, this indicates that there is a synergistic relationship between the concentration of the extract and the SPF value of the *lotion*.

The effectiveness of sunscreen compounds is defined as the effectiveness in absorbing UV rays in the wavelength range of 290-320 nm without causing

interference that will reduce its efficiency or that will cause toxicity or irritation. SPF values are obtained from absorbance measurements at UVB wavelengths of 290-320 nm based on the Mansur equation. UV light consists of UVA (320-400 nm), UVB (290-320 nm), and UVC (200-290 nm). In determining the SPF value, the wavelength absorbance measurement used is in the UVB spectrum (290-320 nm) because the effect of UVB exposure causes *burning* (like *burning*) on the skin, causing erythema (reddish rash). *Sun Protection Factor* (SPF) is a universal indicator that explains the effectiveness of a product or substance that is a UV *protector* and can show how many times a person's skin protection is doubled so that it is safe to be under sun exposure without experiencing erythema. Generally, the ability of normal human skin to survive in the sun

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without using sunscreen is approximately 10 minutes (SPF value increases tenfold the skin's resistance to sun exposure) so, when someone uses a sunscreen product with SPF 15, 15×10 minutes = 150 minutes (2 and a half hours), meaning that the sunscreen product is reused every 2 and a half hours [28].

Papuan grapes contain phenolic bioactive compounds (flavonoids and tannins) that have antioxidant and photoprotective activities. Antioxidant activity is influenced by the presence of flavonoid compounds contained in the extract [29]. The compound has potential as a high sunscreen due to the presence of chromophore groups (conjugated single double bonds) which are able to absorb UV rays thereby reducing their intensity on the skin. Tannin compounds function as sunscreens by helping to absorb and suppress UV radiation (hydroxyl radicals) by donating *reducible* hydrogen atoms so as to get protection against UV rays [30].

Table 6. One-wayANOVA test data analysis results

ANOVA					
SPF Value Result					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	146.745	3	48.915	4096.829	.000
Within Groups	.096	8	.012		
Total	146.841	11			

Then the *One-way* ANOVA test obtained a significance value of $0.000 < 0.05$, which means that the average SPF value of the *lotion* formula of ethanol extract of Papuan grapes can be said to be significantly different. so that the next step can be done, namely the *post-hoc* test to determine the results of SPF values in which groups are significantly different. Further tests in this study using the *Games Howell test (post-hoc)* this test was carried out because the homogeneity test obtained results that were not significant or not homogeneous.

After the *Games Howell test (post-hoc)*, the results of the data in each *lotion* formula of ethanol extract of Papuan grapes are in different subset columns (different notations) so that it can be said that there is a significant difference in SPF value in each *lotion* formula. So, the difference in sample concentration affects the SPF value, the higher the sample concentration, the higher the SPF value produced.

IV.CONCLUSION

The results of physical quality evaluation in the

E. Data Analysis

The results of the analysis with statistical tests were carried out to determine the difference in SPF values between each formula of Papuan grape ethanol extract *lotion*. The first step was a normality test in the form of the *One-Sample Kolmogorov-Smirnov Test* obtained a significance value of $0.200 > 0.05$ which means the data is normally distributed. The third step is the homogeneity test in the form of the *Levene statistic*, the significance value is $0.003 < 0.05$, which means that the data is not homogeneous. The homogeneity test is not an absolute requirement in the *One-Way* ANOVA test, even though the assumptions in the homogeneity test are not met or are not significant. *One-way* ANOVA testing can still be done, provided that the data used is normally distributed.

form of organoleptic test, homogeneity test, pH test, adhesion test, spreadability test, emulsion type test, and centrifugation test on Papuan grape ethanol extract lotion preparations meet the physical quality requirements of semi-solid preparations. Based on the data obtained, it can be concluded that the SPF values of the base formulation, FI (0.5%); FII (1%); and FIII (1.5%) are, respectively, base (1); weak protection category > FI (7) medium protection category > FII (9); strong protection category > FIII (10); strong protection category. Based on the SPF value, it can be seen that the SPF with good protection is at a high concentration of FIII (1.5%), namely 10.

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