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FORMULATION OF SUNSCREEN CREAM OF PARIJOTO FRUIT EXTRACT (*Medinilla speciosa* Blume) AND IN VITRO SPF VALUE TEST

FORMULASI KRIM TABIR SURYA EKSTRAK BUAH PARIJOTO (*Medinilla speciosa* Blume) DAN UJI NILAI SPF SECARA IN VITRO

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ABSTRACT

Sunscreen preparations are cosmetic preparations used as a protection to reduce the impact of sun exposure whose formulations contain active ingredients to absorb or diffuse sunlight, especially in areas of ultraviolet and infrared wave emissions. One of the potential natural ingredients for a sunscreen is parijoto fruit (Medinilla speciosa Blume). It contains flavonoid compounds that are able to prevent the harmful effects of UV rays. The objective of this research is to find out the formula of sunscreen cream of parijoto extract that meets the good physical quality of cream and to find out the result of SPF value test of parijoto fruit extract as sunscreen cream preparation in Vitro. The design of the study was experimental research conducted in the laboratory. The sample used in this research was parijoto made into thick extract by maceration method. Further, the viscous extract obtained was made to be a sunscreen cream and then tested either its physical evaluation or calculation of SPF value. The results of this study indicate that the preparation of sunscreen cream of parijoto fruit extract included in the extra protection category with the value is 6.66 and can be made into the good and stable preparations. The sunscreen cream of parijoto fruit extract has good physical properties and also has activity as UV protection in vitro.
Keywords: *cream, parijoto fruit (Medinilla speciosa Blume), SPF, sunscreen*

ABSTRAK

Sediaan tabir surya adalah sediaan kosmetika yang digunakan sebagai salah satu perlindungan untuk mengurangi dampak paparan sinar matahari. Formulasinya mengandung zat aktif untuk menyerap atau menyebarkan sinar matahari terutama daerah emisi gelombang ultraviolet dan inframerah. Salah satu bahan alam yang memiliki potensi sebagai tabir surya adalah buah Medinilla speciosa (Medinilla speciosa Blume) yang mengandung senyawa flavonoid yang mampu mencegah efek berbahaya dari sinar UV. Tujuan dari penelitian ini adalah untuk mengetahui formula sediaan krim tabir surya ekstrak buah Medinilla speciosa yang memenuhi persyaratan mutu fisik krim yang baik serta mengetahui hasil uji nilai SPF secara in vitro ekstrak buah Medinilla speciosa. Sampel yang digunakan pada penelitian ini yaitu buah Medinilla speciosa yang dibuat menjadi ekstrak kental dengan metode maserasi. Ekstrak kental yang diperoleh kemudian dibuat menjadi sediaan krim tabir surya kemudian dilakukan uji evaluasi fisik dan perhitungan nilai SPF. Hasil penelitian ini menunjukkan bahwa sediaan krim tabir surya esktrak buah Medinilla speciosa termasuk dalam kategori proteksi ekstra dengan nilai 6,66 serta dapat dibuat menjadi sediaan yang baik dan stabil. Krim sunscreen ekstrak buah Medinilla speciosa mempunyai sifat fisik yang baik dan juga memiliki aktivitas sebagai perlindungan sinar UV secara in vitro.
Kata kunci: *krim, buah parijoto (Medinilla speciosa Blume), SPF, tabir surya*

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INTRODUCTION

Indonesia is a country with high exposure of sunlight. Human beings need sunlight to create vitamin D which is very useful to the bones. However, if an individual is excessively exposed to sunlight, it can cause the skin epidermis layers unable to guard from the generated negative effect ranging from light dermatitis to the skin cancer (Chiari et al., 2014).

One of the chemical protections which can be taken to help reduce the effect of sunlight exposure is using the cosmetic preparation of the sunscreen by applying it before being exposed to the sunlight. The sunscreen cream can absorb at least 85 % of the sunlight at the wavelength of 390-320 nm for UVB, while for UVA can absorb the light at the wavelength of 320 (Suryanto, 2012).

Medinilla speciosa plant is one of the particular plants that is found in Colo village in Kudus District, Central Java which has not been explored concerning its benefits. Therefore, the extract of *Medinilla speciosa* was chosen as the material used for sunscreen cream formulation. The *Medinilla speciosa* fruit contains some phenolic compounds, among others, the flavonoids, saponins, and cardenolin (Tussanti et al., 2014). Flavonoid has been identified to be functional as the antioxidant. The flavonoid can prevent the harmful effect or UV rays or can reduce the skin distraction (Mokodompit et al., 2013). The flavonoid compound found in the *Medinilla speciosa* fruit has been known to be able to give protection against the exposure of the sunlight hence it was determined to be the background of this research, concerning the preparation formulation of the sunscreen cream of the *Medinilla speciosa* extract. The solvent used in the formulation of *Medinilla speciosa* extract was the ethanol 70% because it had some advantages. Some of them were that it was very effective in producing optimal active ingredients, and also the polar solvent such as ethanol was the more effective solvent that could be used for the natural antioxidant extraction. It was chosen as the cream preparation because of its spreading ability which was good for the skin, easy to wash with water, and delivers the good medicine (Voight, 1994). To identify the effectiveness for the preparation of sunscreen cream based on *Medinilla speciosa* ingredient, it was necessary to test the physical quality and in vitro testing of the SPF values of the sunscreen cream preparation which was carried out using the spectrophotometry of UV-VIS.

The aim of this research is to identify the formula of the preparation of the sunscreen cream which was determined from the extract of *Medinilla speciosa* to able to fulfill the physical qualification of the cream and to identify the result of the SPF value test in vitro contained in the extract of *Medinilla speciosa* fruit for the preparation of sunscreen cream.

METHODS

Instrumentations and Materials

The instrumentations used in this research were waterbath, chemical glass, pH indicator, spreadability test apparatus, porcelain cup, drop pipette, cream pot, oven, filter paper, analytical scales, Brookfield viscometer, spectrophotometry UV-Vis. The materials used in this research were, the *Medinilla speciosa* fruit, aquadest, ethanol 70% (technical), cream base which includes cetyl alcohol (Brataco Chemical, Indonesia), mineral oil (technical), tween 80 (technical), glycerin (Brataco Chemical, Indonesia), span 80 (technical), methyl paraben (technical), propyl paraben (technical), and stearate acid (technical).

The Collecting and Identification of the Plant

The selected *Medinilla speciosa* fruit had the specification of which had purplish pink color. The selected *Medinilla speciosa* fruit was sorted first to clean it from the dust, dirt and the insects so that it was free from the pollutants which could reduce its quality. Then, the *Medinilla speciosa* fruit was separated from its stalk. Next, the *Medinilla speciosa* fruit was chopped up to make the dry process easy which was done in the oven at the temperature of 50°C until it become dried simplicia (Wulandari et al., 2017).

The Extract Production

The extract of *Medinilla speciosa* fruit was made using the maceration method, that is, the dried *Medinilla speciosa* fruit was extracted using ethanol 70% as long as 3 x 24 hours. The result of extraction, then was evaporated using the waterbath so that the viscous extract could be obtained (Sharon et al., 2013).

The Characterization of the Extract

The characterization of the extract was seen by using the organoleptic, measuring the extract pH, and by testing the phytochemical compound content.

Organoleptic Properties

Organoleptic properties of extract was tested by using the human senses, starting from the shape, smell, and color.

pH

The pH of the extract was measured using pH indicator, that is, by immersing the indicator into the extract of *Medinilla speciosa* fruit. Then, the change of the color was observed and adjusted to the color spectrum in that indicator.

The Phytochemical Content Testing

Preparation of phytochemical test solution

As many as 0.5 gram of ethanol extraction of the *Medinilla speciosa* fruit was dissolved with the 50 mL of methanol, then they were shaken until they were homogeneous. Next, they were divided into three test tubes (Artini et al., 2008).

The Examination of Flavonoids

It took 1 mL of test solution; it was added with little powder of Mg and 1 mL of concentrated HCl, then, they were shaken. The positive testing was marked by the formation of the red color, pink, or purple (Marliana and Saleh, 2011).

The examination of Saponin

It took as many as 1 mL of test solution; it was poured into the test tube, then, was shaken strongly for 10 seconds. The formation of the foam about 1-10 cm high which was stable for no less than 10 minutes, indicated the existence of the saponin. On the addition of 1 drop of HCl 2N, the foam did not disappear (Artini et al., 2008).

The examination of Tannin

It took 1 mL test solution and was poured into the test tube and was added with 3 drops of $FeCl_3$ 1%. The sample contained tannin if the color changed to be blackish green (Arief et al., 2017).

Formulation of Sunscreen cream of *Medinilla speciosa* fruit Extract

The cream was made by modified formula from Hastuti (2016) and can be seen in Table I. The phase of oil was heated in the waterbath at the temperatures of 65-75ºC. At the same time, in the different way, the phase of water was heated in the waterbath at the temperatures of 65-75ºC. The phase of oil was poured into the mortar while being stirred. The phase of water was added to the phase of oil in the condition of being heated, drop by drop while it was continuously stirred. The cream was cooled while being stirred until it was homogeneous.

The evaluation of the physical properties of cream

Organoleptic

The organoleptic test was conducted using the five senses, starting from the shape, the smell, and the color. The parameter of quality of the physical properties of cream was that there were no changes in the form, color, and the smell since the beginning of the production, storage, up to the usage. The organoleptic compared the sunscreen cream base with the sunscreen *Medinilla speciosa* fruit extract base.

pH

The pH of the preparation was measured using the pH indicator by immersing the pH indicator into the sunscreen cream preparation. Then, the color change was observed and adjusted to the color spectrum in the indicator tool. The pH of the sunscreen cream *Medinilla speciosa* fruit extract should also be compared with the sunscreen cream base.

Viscosity

The test was conducted using the Brookfield viscometer and utilizing 64 spindles. Afterward, the cream was placed in a container, then, the spindle which had been installed was pulled down until the spindles was immersed.

Spreadability

The cream was placed on the glass plate and was left alone for 1 minute, then, the diameter of the cream spread was measured. Next, the load was added by 50 mg. It was left alone for 1 minute, then, diameter of the cream spread was measured. That same thing should be done again and again until the constant diameter of the cream spread was obtained. (Rindiyantoko and Hastuti, 2017).

Homogeneity

The homogeneity test was conducted by smearing the preparation to the surface of the object glass, then, it was spread to the other object glass to find the homogeneous surface. The cream could be said homogeneous if the particle structure did not cause to clot or was not mixed (Wulandari et al., 2017).

Freeze-Thaw Cycling

The freeze-thaw test was conducted by keeping each of the cream formula in the storage in the temperatures of -10°C and 30°C for 24 hours in 3 cycles. The cream that passed through the freeze-thaw was observed organoleptically and was identified whether the change of the phase occurred (Yuliani et al., 2016).

The Determination of the SPF (Sun Protection Factor) Value

The determination of the effectiveness of the sunscreen was carried out by determining the SPF value in vitro using the spectrophotometer UV-Vis. The cream was diluted in 4000 ppm by taking out 0.1 gram of the *Medinilla speciosa* fruit extract being dissolved in the 96% ethanol as many as 25 mL and being mixed up until it became homogeneous (Mokodompit et al., 2013). Before the spectrophotometer was calibrated using ethanol 96%, by way of 1 mL of ethanol was poured into the cuvette in the wavelengths between 290-320 nm, utilizing the ethanol 96% as the blank. Then, the absorption average (Ar) was determined in the interval of 5 nm. The result of the absorbance was recorded, then, the SPF value was calculated by applying the following formula (Rauf et al., 2017):

$$AUC = \left(\frac{Aa + Ab}{2}\right)(dPa - b)$$

$$\Delta AUC = AUC_1 + AUC_2 + AUC_3 + AUC_4 + AUC_5 + AUC_6$$

$$\text{Log SPF} = \left(\frac{\Delta AUC}{\lambda_n - \lambda_1}\right) x\ 2$$

SPF = antilog SPF

With Aa: Absorbance in wavelength a nm; Ab: Absorbance in wavelength b nm; dPa-b: the difference between wavelengths a and b; λn: the biggest wavelength (320 nm); λ1: the smallest wavelength (290 nm); AUC: Area Under Curve; ΔAUC: Total AUC.

The effectiveness of the sunscreen cream preparation based on the SPF values were presented in Table II.

Table I. Cream Formula

Composition	Quantity (%)
Extract	500 mg
Cetyl alcohol	4
VCO	10
Tween 80	2,204
Glycerin	10
Span 80	2
Methyl paraben	0,2
Propyl Paraben	0,1
Stearate Acid	3,796
Aquadest	Add 100

Source: (Hastuti, 2016)

Table II. Effectiveness of the Sunscreen Cream Preparation

SPF	Category
2-4	Minimum Protection
4-6	Medium Protection
6-8	Extra Protection
8-15	Maximum Protection
≥15	Ultra-Protection

Table III. The Result of Characterization of *Medinilla speciosa* fruit Extract

Type of Characterization	Result
Organoleptic	
Color	Dark Brown
Form	Concentrated Extract
Smell	Particular Extract
pH	4
Phytochemical Test	
Flavonoids	+
Saponin	+
Tannin	+

Table IV. The Result of characterization test of the Physical properties of cream

Characterization of physical properties of cream	Base sunscreen cream	*Medinilla speciosa* fruit Extracted cream
Organoleptic		
Color	White	Brown
Form	Cream, smooth, not sticky	Cream, smooth, not sticky
Smell	Particular base	Particular extract
pH	5	5
Homogeneity	Homogeneous	Homogeneous
Viscosity (cP)	Average of SD 3451.13 ± 41.491	Average of SD 578.15 ± 24.614
Spreadability	Average of SD 1.09 ± 22.275	Average of SD 1.03 ± 14
Freeze-thaw	-	+

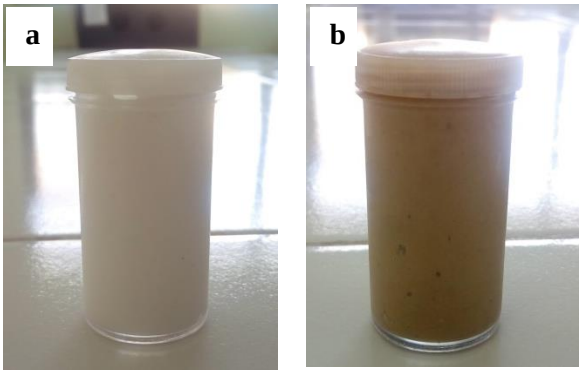

Figure 1. Physical appearance of Sunscreen cream Medinilla speciose fruit extract (a) and base sunscreen cream (b)

RESULTS AND DISCUSSION

The Rendemen Result of *Medinilla speciosa* fruit Ethanol Extract

The production process of *Medinilla speciosa* fruit extract was done using maceration method applying the solvent of ethanol 70%. The maceration was conducted as long as 3 x 24 hours by stirring occasionally and was strained using the filter paper, then, the dregs was macerated by applying the same solvent until it produced clear macerate. The liquid extract obtained from the maceration result was, then, concentrated by using the waterbath at the temperature of 40ºC so that the concentrate extract was gained. From the result of the extraction, it was obtained 10.95% macerates. The obtained extract with the bigger solubility in the water was compared with the solubility in the oil.

Characterization of the Extract

The characterization of extract can be showed by Table III. According to Wachidah (2013), the total of flavonoids grades of the *Medinilla speciosa* fruit was as many as 164 mg RE/g of extract. This flavonoids compound was exactly used as the active content of the sunscreen.

The Cream Formulation

The cream formula was the formula by Hastuti (2016), which was modified with which the applied base was cetyl alcohol, mineral oil, tween 80, glycerin, span 80, methyl paraben, propyl paraben, and stearate acid. Those which were included in the water phase were the tween 80, glycerin, methyl paraben, aquadest, and *Medinilla speciosa* fruit extract. While the ingredients that were included in the oil phase were span 80, cetyl alcohol, mineral oil, propyl paraben, and stearate acid.

The Evaluation of the Physical properties of cream

The result of characterization test of the Base Sunscreen cream and *Medinilla speciosa* fruit extracted cream are presented in Table IV and Figure 1.

The organoleptic test was conducted by observing the cream visually on the form, color, and the smell which was meant to see the physical appearance of a preparation. Then, the base cream was compared with the extracted cream. The result showed that the base cream was white while the extracted cream was brown because there was extract addition which was dark brown in color.

The measurement of pH was implemented using the pH indicator by way of immersing the pH indicator into the cream preparation, then, the color was checked with the color spectrum in the indicator tool (Mailana et al., 2016). The result of pH examination indicated that the comparation between the base cream and the extracted cream had same pH score, that is, 5. The score of pH base cream preparation and the extracted cream was still in the range of normal skin pH, that is, between 4 – 6 (Zulkarnain et al., 2015). Hence, that cream was classified safe if it was applied on the skin.

The objective of homogeneity test is to observe and identify the mixing of the ingredients of the cream preparation (Setyowati et al., 2013). The homogeneity of cream preparation was tested using the object glass by way of smearing the cream on the glass and the existence of coarse grain was observed. The result of the homogeneity test to the base cream and extracted cream indicated good result, that is, the cream was dispersed evenly and there was no particle clod which could be observed visually.

The viscosity is a statement on the endurance of a liquid to flow. The higher is the volume of the preparation, its viscosity is also higher, hence, the preparation will get more stable because the movement of the particle is likely difficult as the preparation gets thicker (Mailana et al., 2016).

The result showed that the viscosity between the base cream and the extracted cream had fulfilled the standard. According to Gozali et al. (2009), the ideal viscosity score of the cream is more than 5000 cP. While according to the Indonesian Nasional Standard SNI 16-4399-1996 about the quality standard of the sunscreen cream, the good viscosity of the preparation ranges between 2000 - 50.000 cP.

The spreadability test is used to identify how wide the cream can spread on the skin. The bigger the spreadability of the cream, the more active substance of the cream can be delivered into the skin layer (Voight, 1994). The obtained result of the spreadability test was, then, examined by T-test using the SPSS and the result of the T-test was Sig. 0.001 < 0.05 which could be defined that there was difference, as the result of the spreadability test, between the base sunscreen cream and sunscreen cream from the *Medinilla speciosa* fruit extract.

Freeze-thaw test was conducted to identify the stability of the physical properties of cream. The test can be seen by the absence or presence of phase separation during the storage in the extreme temperature, i.e. -10°C dan 30°C. The testing was done in three cycles. The results obtained were that the base cream did not undergo a phase change during 3-cycled storage. In contrast, in Medinilla fruit extract, cream extract undergoes phase changes in the second cycle. This can be caused by the addition of *Medinilla speciosa* fruit extract which causes physical properties of cream instability.

Determining the SPF Value

The tests of the UV light treatment for *Medinilla speciosa* fruit extract cream in vitro were carried out using a UV-Vis spectrophotometer in the range of wavelengths between 290-320 nm. The wavelength is included in the wavelength for UV A which can continue light on the 320 nm and UV B wavelengths that can absorb sunlight in the wavelength of 290-320 nm (Suryanto, 2012). Then, the value of SPF cream Medinilla specimen fruit extract was 6.66 which was categorized as extra protection. The SPF value determines the ability of sunscreens to protect the skin and prevent sun exposure. The higher the SPF value on sunscreen preparations, the better the ability of protection (Rahmawanty and Fadhillaturrahmah, 2014). Sunscreen preparations can be said to provide protection if they have an SPF value of at least 2 and a good sunscreen assessment category if sunscreen preparations have an SPF value above 15 (Rosniah et al., 2016)

CONCLUSION

The formula for the preparation of sunscreen cream based on *Medinilla speciosa* fruit extract meets the physical properties of good cream. The test results of in vitro SPF value of Medinilla specimen fruit extract sunscreen cream were 6.66%. It is categorized as extra protection.

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