



Effect of Addition Virgin Coconut Oil (VCO) on Physical Stability and SPF Value of Sunscreen Cream Combination of Zinc Oxide and Titanium Dioxide

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Abstract

Virgin Coconut Oil (VCO) is one of the natural ingredients used as antioxidants because of the high saturated fatty acids that cause VCO to be resistant to oxidation. In this research, a combination of zinc oxide (ZnO) and titanium dioxide (TiO₂) sunscreen cream formulation was carried out with the addition of VCO as an antioxidant. The purpose of this study was to determine the effect of adding VCO to physical stability and SPF values of sunscreen cream combination of zinc oxide (ZnO) and titanium dioxide (TiO₂). Sunscreen cream made 5 formulas, namely F1 (7% VCO), F2 (8% VCO), F3 (ZnO + TiO₂), F4 (ZnO + TiO₂ + VCO 7%) and F5 (ZnO + TiO₂ + VCO 8%). The results showed that sunscreen cream formulas 1,2,4, and 5 had good physical-chemical characteristics and were stable during the storage period. While formula 3 produces white cast and is unstable in storage with temperature changes. The formula for sunscreen cream that produces the best SPF value is F5 (ZnO + TiO₂ + VCO 8%) with the ultra protection type.

Keywords: VCO, SPF value, sunscreen, Zinc Oxide (ZnO), Titanium Dioxide (TiO₂)

Introduction

Exposure to UV rays for a long time can cause skin redness, form wrinkles on the skin, premature aging, skin damage, and can cause skin cancer. To protect against the dangers caused by solar radiation on the skin, one of them can be used cosmetic sunscreen (sunblock) (Nole and Johnson, 2004). There are two types of sunscreen preparations: chemical sunscreens that absorb UV radiation and physical sunscreens that reflect and block UV rays. Physical sunscreens such as zinc oxide (ZnO) and titanium dioxide (TiO₂) have many advantages, such as non-irritating and skin sensitization, limited skin penetration, and broad-spectrum protection (Antoniou *et al.*, 2008). However, the opaque nature of ZnO and TiO₂ and the particle size of the powder can affect the appearance of the skin when worn (Gadri and Iwo, 2012).

Current sunscreen preparations cannot protect the skin optimally without antioxidant compounds. Reactive Oxygen Species mediate most of the damage caused by exposure to UV A rays (ROS) produced after exposure to UV A and UV B, which is the cause of erythema

(sunburn). The addition of natural antioxidants into sunscreen preparations is highly recommended because antioxidants can detect the level of ROS production during UV irradiation (Montesano *et al.*, 2006).

Virgin Coconut Oil (VCO) is one of the most widely used natural antioxidants. The high level of saturated fatty acids it contains makes VCO resistant to oxidation. Saturated fats do not have double carbon bonds that easily separate and form free radicals. Saturated fat tends to be more stable under various conditions (Alamsyah, 2005). A previous study of VCO with a concentration of 8% added to a combination sunscreen of octyl methoxycinnamate and titanium dioxide obtained an ultra protection type (Sihombing, 2016). At the same time, the VCO concentration of 7% is an emulsion with good stability (Mu'awanah and Bambang, 2014).

From the potential of virgin coconut oil as an antioxidant, this study will combine virgin coconut oil with concentrations of 7% and 8% with zinc oxide and titanium dioxide in sunscreen preparations and measure the SPF value using photoprotective properties parameters.

Materials and Methods

Materials

The materials used in this study were Zinc Oxide (ZnO) (Merck), Titanium Dioxide (TiO₂) (Merck), Virgin Coconut Oil (VCO) (Herba bagoes), stearic acid, stearyl alcohol (Bratachem), TEA, glycerin (Bratachem), nipagin (Bratachem), nipasol (Bratachem), 70% ethanol and aquadest.

Formulation Sunscreen Cream Combination of Zinc Oxide and Titanium Dioxide

The manufacture of sunscreen cream begins with the manufacture of the oil phase. Stearic acid, cetyl alcohol, and nipasol were melted on a hot plate at 70°C. Then TEA, glycerin, and nipagin were dissolved in distilled water on a hotplate (Heildolph Germany MR Hei-Tec) at 70°C. The spreadability test was carried out by placing 1 gram of cream between 2 glass plates. The top glass plate was weighed first, then placed on top of the cream, and left for 1 minute. It was given 50 grams of additional load, left for 1 minute, and the spread diameter was measured. Then the load with a maximum weight of 150 grams was added, and the diameter of the spread was measured again (Swastika and Purwanto, 2013).

The next step is zinc oxide, titanium dioxide, and VCO are added to the oil phase according to the formulation in each formula. After being homogeneous, the water phase is added to the oil phase little by a little while stirring until a creamy mass is formed. The design of the sunscreen cream formula can be seen in Table 2.

Tabel 2 Formula of Sunscreen Cream Combination of Zinc Oxide and Titanium Dioxide

No.	Material Name	F1%	F2%	F3%	F4%	F5%
1	Zink oksida	-	-	7%	7%	7%
2	Titanium dioxide	-	-	4%	4%	4%
3	VCO	7%	8%	-	7%	8%
4	AsamStearat	10%	10%	10%	10%	10%
5	StearilAlkohol	2 %	2 %	2 %	2 %	2 %
6	Gliserin	5 %	5%	5%	5 %	5 %
7	TEA	2%	2%	2%	2%	2%
8	Nipagin	0,1 %	0,1 %	0,1 %	0,1 %	0,1 %
9	Nipasol	0,18%	0,18%	0,18%	0,18%	0,18%
10	Aquadest	Add	Add	Add	Add	Add
		100%	100%	100%	100%	100%

Evaluation of the Physical-Chemical Characteristics of Formula Sunscreen Cream

Organoleptic Test

Organoleptic observations aimed to see the physical characteristics of the preparations that were observed visually, including color, shape, odor, and application to the skin (Elyaet *al.*, 2013).

Homogeneity Test

A homogeneity test was conducted to determine the homogeneity of sunscreen cream preparations. The homogeneity test was carried out by applying the preparation on a piece of glass, then visually observing the presence or absence of coarse material (Ditjen POM, 1979).

pH

Test the pH value to determine that the sunscreen cream preparation has a pH by the expected specifications. One gram of cream sample was dissolved in 10 ml of distilled water, then tested using a pH meter (Senzpal Tester). The skin has good pH criteria ranging from 4.5 to 6.5, which is safe to use not irritate the skin (Wasitaatmaja, 1997).

Viscosity Test

Viscosity test was carried out using a viscometer (Brookfield Cone and Plate, model: DV-III ULTRA CPE), the appropriate spindle was installed on the tool then immersed in the preparation to a certain extent, the tool was turned on at a speed of 2, 4, 10, 20 rpm, then the speed was reversed successively (Reiger, 2000).

Stability Test

Room Temperature Stability

A room temperature stability test was carried out to determine the stability of the preparation in storage at room temperature ($25\pm 2^{\circ}\text{C}$). The preparations were stored at room temperature for 6 weeks, and the changes were observed every two weeks (Fauzy, 2012).

Freeze-thaw cycle

A freeze-thaw test was carried out by storing sunscreen cream samples in an oven at $40\pm 20^{\circ}\text{C}$ for 24 hours, then transferred to a refrigerator at $4\pm 20^{\circ}\text{C}$ for 24 hours (1 cycle). The stability test of the preparation was carried out for six cycles or 12 days of testing (Fauzy, 2012).

Storage Time Stability

Storage time stability test was carried out by centrifugation method. The preparation was rotated at a speed of 3750 rpm for 5 hours because the results were equivalent to the effect of gravity for one year (Fauzy, 2012).

Spreadability Test

The spreadability test was carried out by placing 1 gram of cream between 2 glass plates. The top glass plate was weighed first, then placed on top of the cream, and left for 1 minute. It was given 50 grams of additional load, left for 1 minute, and the spread diameter was measured. Then the load with a maximum weight of 150 grams was added, and the diameter of the spread was measured again (Swastika and Purwanto, 2013).

Cream Type Test

The cream type test was carried out by staining method using methylene blue reagent. Weighed as much as 0.1 grams of cream, then dripped with methylene blue, then observed under a microscope. Including o/w type cream if methylene blue is evenly dispersed (Swastika and Purwanto, 2013).

Determination of SPF Value

The SPF value of sunscreen cream was determined was carried out in vitro using UV-Vis spectrophotometry. 50 mg of cream was weighed and dissolved with 70% ethanol p.a in a 50 ml volumetric flask to obtain a concentration of 1000 ppm (stock solution), then 1 ml of stock solution was taken into a 50 ml volumetric flask and dissolved in 70% ethanol. Then the absorption value was measured in the wavelength region of 290-320 nm with 5 nm intervals using a UV-Vis spectrophotometer (Shimadzu UV-1800) to obtain the SPF value. The absorbance data obtained were entered in the SPF equation and used a predetermined constant. The formula for determining the SPF value is as follows:

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

CF : Correction factor = 10

EE : Erythematous effect spectrum

I : Intensity of the solar spectrum at a wavelength

Abs : Absorbance of sunscreen products

Result and Discussion

The results of the organoleptic test on cream preparations showed that the five cream formulations had a white color; 1, 2, 4, and 5 formulas had a distinctive smell of VCO. While in Formula 3, there is no smell, this is because, in formula 3, there is no addition of VCO. The appearance on the skin after applying cream on F3 leaves a white cast or white mark. This is due to the rigid nature of zinc oxide and titanium dioxide, which do not penetrate light. Whereas in formulas 1, 2, 4, and 5, the appearance of the cream is even with skin color.

The homogeneity test results showed that all sunscreen cream formulas were homogeneous. This is based on the absence of coarse grains in the sunscreen cream preparation when tested. The pH test results can be seen that the pH values of the five formulas have good pH values and are suitable in the pH range that is suitable for the skin, namely 4.5-6.5. A pH value that is too acidic will cause skin irritation, and if it is too alkaline, it will cause scaly skin (Purnamasari, 2012). The results of the pH test can be seen in Table 3. The results of the viscosity test that have been carried out, the average results of the five formulas can be known if the five sunscreen cream formulas have good viscosity values and follow a predetermined standard range, namely in the field of 4000-40,000 cps. Viscosity is one of the factors related to the convenience of using cream. The cream should not be too hard or too runny because it relates to the therapeutic effect and comfort of the cream when used (Shovyanat et al., 2013). The results of the viscosity test can be seen in Table 3.

The stability test results of sunscreen cream preparations at room temperature ($25 \pm 2^\circ\text{C}$), which were carried out for six weeks, showed no organoleptic changes and good homogeneity at the 2nd week, 4th week, and 6th week. It is based on the absence of changes in color, odor, texture, appearance on the skin, and homogeneity. The results of the organoleptic test carried out

for six cycles of the freeze-thaw test found that there were no organoleptic changes, including color, odor, shape, or physical condition of the preparation and appearance on the skin when applied, and homogeneity in formulas 1, 2, 4 and 5. 3 there is a change in shape and uniformity. The dosage form of formula 3 in the fifth cycle of the cream began to clot and dry. Meanwhile, in the sixth cycle, formula 3 cream became stiff and inhomogeneous, indicated by the presence of a few coarse grains.

The results of the pH stability test for six cycles showed that the initial pH of the preparation and after the freeze-thaw test was carried out at room temperature, high temperature, and the low temperature had a pH value that was suitable for the skin, which was in the range of 4.5-6.5. The cream formula added with VCO tends not to change the pH value for six cycles and has good stability. This is possible because of the nature of fatty acids with medium and long chains, which are relatively stable at high and low temperatures and do not oxidize quickly (Mu'awanah and Bambang, 2014).

From the results of the centrifugation test that have been carried out, it is known that there is no phase separation in the five formulas, either with the addition of VCO or not. The principle of centrifugation is based on the separation of two or more components with different densities using centrifugal force. The centrifugation test is also a method used to predict the shelf life of preparations (Mu'awanah and Bambang, 2014).

All sunscreen cream formulas have good dispersion values, which are in the range of 5-7cm. Spreadability is inversely proportional to viscosity. The greater the viscosity of preparation, the smaller the spreadability of the preparation. Creams with good spreadability will provide convenience when applied (Naibahoet al., 2013). The results of the dispersion test can be seen in Table 3.

The results of the cream type test showed that the cream made was mint cream in water. This was known after the addition of methylene blue. The cream preparation turned blue. The blue color indicates when methylene blue has been evenly dispersed on the entire surface of the sunscreen cream preparation. Oil-in-water cream has less sticky properties, easy to wash, and creates a comfortable and cool feeling when used, so it is widely preferred.

Tabel 3.Result of pH, Viscosity and Spreadability Test

Formula	pH	Viscosity	Spreadability
F1	5,74	8875	6,77
F2	5,67	7304	6,87
F3	5,94	12950	5,43
F4	5,84	11808	6,37
F5	5,77	12310	6,53

Determination of SPF Value

Sun Protection Factor (SPF) was determined in vitro using a UV-VIS spectrophotometer. The cream was dissolved in ethanol, then measured, and the absorbance was obtained at a wavelength of 290nm-320nm. The results of the SPF value in formula 1 (7% VCO) obtained an SPF value of 3.517, which is included in the type of minimal protection. The SPF value of formula 2 (VCO 8%) received an SPF value of 3.975 and is included in minimal protection. Formulas 1 and 2 do not contain active ingredients (ZnO and TiO₂). The addition of Virgin Coconut Oil (VCO) which also functions as an antioxidant, can inhibit damage to the skin due to free radicals (Nelson *et al.*, 2003). Formula 3 (ZnO+TiO₂) and formula 4 (ZnO+TiO₂+VCO7%) obtained SPF values of 10,063 and 13,672, which are included in the type of extra protection. The SPF value in formula 5 (ZnO+TiO₂+VCO8%) received an SPF value of 16.0132 and is

included in the ultra protection type. These results also indicate that the greater the concentration of VCO added to the sunscreen cream preparation, the greater the SPF value of the practice. The results of the SPF test can be seen in Table 4.

The SPF value of this preparation is mainly intended as protection against UV B and not explicitly intended as protection against UV A. UV B is included in the group of harmful rays because it can cause damage faster and easier than UV A. The amount of UV B rays received by the earth is 10% less than UV A, but more erythema production is caused by UV B. Most UV B is absorbed by the epidermis and can stimulate the highest melanogenesis (Willis and Cylus, 1977). The addition of VCO as an antioxidant can also prevent aging, skin wrinkling, and erythema and avoid the mediation of skin damage due to UV rays (Liliana, 2007).

Table 4 Result of calculation SPF value

No.	Formula	SPF	Protection Type
1	F1 (VCO 7%)	3,517	Minimal
2	F2 (VCO 8%)	3,975	Minimal
3	F3 (ZnO+TiO ₂)	10,063	Ekstra
4	F4 (ZnO+TiO ₂ +VCO 7%)	13,672	Ekstra
5	F5 (ZnO+TiO ₂ +VCO 8%)	16,0132	Ultra

Conclusion

Variations in VCO concentrations of 7% and 8% affect sunscreen cream's physical characteristics and value. Cream with the addition of VCO does not cause white cast, is homogeneous, and the pH value remains stable both at room temperature, high temperature, and low temperature and provides better dispersion. Sunscreen cream with the addition of 8% produces the best physical and chemical characteristics. Judging from the spreadability of the cream, which has the most excellent value and remains stable in every condition. The highest SPF value is sunscreen cream F5 (ZnO+TiO₂+VCO 8%) of 16.01

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