Original Article

Sunscreen cream formulation from a combination of propolis extract and titanium dioxide

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ABSTRACT

Excessive exposure to sunlight can increase the risk of skin damage. This study aimed to produce a cosmetic product, namely sunscreen cream, using active ingredients of propolis extract and TiO2 to protect against the harmful effects of UV radiation. Propolis is beneficial as an antioxidant, while TiO2 is used to enhance protection against UV radiation. The sunscreen cream was formulated in four variations of TiO2 concentration, namely Formula (control); Formula 1 (10%); Formula 2 (2%); and Formula 3 (0%). The physical and chemical properties of the sunscreen cream were observed, and stability testing was conducted using the *Freeze-Thaw* method. The observations included organoleptic, homogeneity, cream type, pH, spreadability, adhesiveness, viscosity, and Sun Protection Factor (SPF) values. The results showed that the variations in TiO2 concentration did not have any effect on adhesiveness but had an effect on pH, spreadability, and viscosity. The test results revealed the SPF values of the formulations as follows; Formula 0 (5.663); Formula 1 (18.386); Formula 2 (9.466); and Formula 3 (8.583), and the value decreased after the stability testing using the *Freeze-Thaw* method conducted.

Keywords: Cream, Sunscreen, Propolis, Titanium dioxide

Introduction

Continuous exposure to ultraviolet radiation from the sun can have negative effects on the skin, such as erythema, pigmentation, photosensitivity, and long-term effects such as premature aging. Sunscreen preparations are recommended to prevent or minimize the risk of negative effects caused by ultraviolet radiation on the skis [1].

The skin naturally protects itself and its underlying organs from the harmful effects of ultraviolet radiation by producing the pigment melanin, which reflects sunlight [2]. However, continuous production of melanin can result in black spots on the skin [3]. Therefore, skin protection is necessary through the use of topical sunscreen. Topical sunscreens are divided into two types: physical and chemical sunscreens. Physical sunscreens, also known as sunblocks, work by reflecting ultraviolet radiation and can protect the skin more quickly [4]. Chemical sunscreens work by absorbing ultraviolet radiation and take about 15-20 minutes to work in protecting the skin. Examples of physical sunscreen ingredients are titanium dioxide (TiO_2), zinc oxide (ZnO), kaolin, talc, and magnesium oxide (MgO). Examples of chemical sunscreen ingredients are benzophenone-3, avobenzone, octyl methoxycinnamate, and octyl salicylate [5]. Sunscreen products are expressed in terms of their Sun Protecting Factor (SPF) value. The SPF value indicates how long a sunscreen product can protect or block ultraviolet radiation that can cause skin damage [6].

This study used propolis extract as an active ingredient. Propolis is a bioactive compound found in honey bee nests. Propolis contains an important chemical compound called flavonoids [7]. Flavonoids have antioxidant activity that has the potential to be used as a sunscreen. The greater the antioxidant activity, the higher the Sun Protecting Factor (SPF) value [8]. Antioxidants function to protect the body from damage caused

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by free radicals such as UV rays. Antioxidants act as hydrogen donors or as acceptors of free radicals, thus delaying the initiation stage of free radical formation [9]. Propolis extract with a concentration of 16% can produce an SPF value with moderate protection [10]. In addition to propolis, TiO_2 also has activity as a sunscreen. TiO_2 can reflect high-energy UV rays, thus providing maximum protection to the skin [11].

Materials and Methods

Instruments

The instruments used were beakers (pyrex), analytical scales (Precisa), a pH meter (Eutech Cybersan), a viscometer (Rhion VT-04), a set of adhesive strength testing equipment, a *hot plate* (Maspion), a *water bath*, a refrigerator (Sharp), microscope (Nikon Eclipse E100), UV-VIS spectrophotometry (Genesys™), and other supporting instruments.

Materials

The materials used were Propolis extract, TiO_2 Anatase Grade ka-100, Propylene Glycol USP, Stearic acid, *Cera alba*/Beeswax, *Vaseline alba*, Triethanolamine, Methylparaben, Distilled water, Concentrated HCl, Mg metal powder, technical grade methanol, and 70% technical grade alcohol.

Qualitative test for flavonoid compounds

The sample was weighed for 0.5 grams and was dissolved in 2 mL of methanol, and heated. 3 mg of magnesium metal and a few drops of concentrated HCl were added. The appearance of a reddish-orange to reddish-purple color indicates the presence of flavonoids. Meanwhile, the appearance of yellow-orange color indicates the presence of flavones, chalcones, and aurones [12].

Design formula

Table 1. Formulation of Propolis- TiO_2 Sunscreen Cream Material Function BB Cream Formula (%b/b)

Material	Function	BB Cream Formula (%*b*/*b*)			
		F1	F2	F3	F4
Propolis extract	Active substance	0	16	16	16
TiO_2	Active substance	2	10	2	0
Propylene glycol	Humectant	8	8	8	8
Stearic Acid	Emulgator	15	15	15	15
Cera alba	Cream base	2	2	2	2
Vaselin alba	Cream base	8	8	8	8
Triethanolamine	Emulgator	1.5	1.5	1.5	1.5
Methylparaben	Preservative	0.83	0.83	0.83	0.83
Aquadest (ad)	Solvent	100	100	100	100

The making process

The oil phase consisting of stearic acid, beeswax, and vaseline alba was prepared and mixed until getting homogenous. Separately, the water phase consisting of propylene glycol, triethanolamine, methylparaben, and distilled water was prepared and mixed until getting homogenous [13]. Then, the oil phase was slowly added to the water phase while stirring until a homogenous cream was formed. Afterward, TiO_2 was added according to the respective formula, followed by the addition of propolis extract. The mixture was stirred until a homogenous cream was formed.

Testing of physical and chemical properties of sunscreen cream

Organoleptic test

An organoleptic test was carried out to physically examine the cream by observing its form/consistency, color, and odor [14].

Homogeneity test

A homogeneity test was performed by weighing 0.1 grams of cream and then applying it to a glass slide until a thin layer was formed and then covered with another glass slide [15]. The observation was conducted using a microscope at a magnification of 40x10. The test result is considered homogeneous if it shows a homogeneous arrangement and no coarse particles are visible.

Cream-type test

The cream-type test was conducted by weighing 0.2 grams of the cream and then diluting it with 20 mL of water. If the cream can be mixed with water, it is included in the m/a cream type, and if the cream cannot be mixed with water, it is included in the a/m cream type [16].

pH test

The pH test was performed using a pH meter. Before its use, the pH meter was calibrated using a standard buffer. After that, the pH meter was dipped into the cream to be tested [15].

Spreadability test

The spreadability test was conducted by placing 1 gram of cream into the center of a petri dish and covering it with another petri dish, then allowing it to stand for 1 minute. The spread of the cream was measured using a ruler. The measurement was repeated with a load of 50 grams, 100 grams, and 150 grams [17].

Adhesion test

The adhesion test was carried out by placing 0.5 grams of the cream on a glass slide and covering it with another glass slide. Then the glass slides were pressed using a 1 kg weight for 5 minutes. Then, the glass slide was mounted on a testing apparatus that has been given an 80-gram weight, then the time for the sample to detach from the glass slide was recorded [17].

Viscosity test

The viscosity test was carried out using a viscometer. The rotor was fully immersed in the cream. The viscometer was turned on so that the needle automatically moved to the right to indicate the viscosity value. We waited till the needle stabilized and stopped moving [18].

The freeze-thaw stability test

The freeze-thaw test was carried out by placing the sample at a temperature of (4 ± 2oC) for 24 hours, then it was followed by placing the sample at room temperature for 24 hours (1 cycle). Freeze-thaw testing was carried out for 3 cycles and observed for any physical and chemical changes in the preparation at the beginning and end of the cycle [19].

The sun protecting factor (SPF) test

The determination of SPF value was carried out using a UV-VIS spectrophotometer. The cream was diluted to 2000 ppm. From each cream formula, a 0.2-gram sample was taken and dissolved in 10 mL of distilled water, then 1 mL of the sample solution was taken and dissolved in 10 mL of distilled water. Each sample solution was measured for its absorbance at a wavelength of 290-320 nm with an interval of 5 nm. Distilled water was used as a blank in the measurement [1].

The absorbance appearing on the UV-VIS spectrophotometry was recorded. The SPF value was calculated using Mansur's formula [20]:

$$SPF = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times abs(\lambda) \quad (1)$$

Where:

CF : Correction Factor (10)

EE : Erythema Effect

I : Simulation Spectrum of Sunlight

Abs: Absorbance Value

Results and Discussion

Qualitative test for flavonoid compounds

The testing of secondary metabolites in this research aims to determine the content of flavonoid compounds in propolis extract. Flavonoid compounds contained in an extract act as antioxidants. The antioxidant content in sunscreen preparations can increase photoprotective effectiveness and protect the skin from damage caused by UV exposure [21]. The results of the flavonoid compound test on propolis extract showed a color change from dark brown to reddish-orange. From these test results, it can be concluded that propolis extract positively contains flavonoid compounds.

The results showed a pale yellow color when the propolis extract was mixed with methanol; shows a clear brown color when the propolis extract is mixed with methanol and heated, the result is the same when magnesium is added; and showed a red-orange color when the propolis extract was added with methanol, magnesium powder and HCl.

Testing the physicochemical properties of sunscreen

Organoleptic test

The organoleptic test of propolis extract sunscreen cream aims to determine the characteristics of the resulting product. The result of organoleptic test can be seen in **Table 2**.

Table 2. Results of organoleptic observations of sunscreen cream before and after the *Freeze-Thaw* Test.

Formula	Organoleptic	Freeze-Thaw Test	
		Before (4°C)	After (25°C)
0	Color	White	White
	Odor	Typical cera alba	Typical cera alba
	Consistency	Slightly runny	Slightly runny
1	Color	Cream	Slightly thicker cream
	Odor	Typical Propolis	Typical Propolis
	Consistency	Slightly firmer	Slightly firmer
2	Color	Thick cream	Brownish cream
	Odor	Typical Propolis	Typical Propolis
	Consistency	Slightly more liquid than Formula 1	Slightly more liquid than Formula 1
3	Color	Brownish cream	Yellowish-brown
	Odor	Typical Propolis	Typical Propolis
	Consistency	Slightly more liquid than Formula 2	Slightly more liquid than Formula 2

The organoleptic observations of the sunscreen cream preparation were carried out before and after the *Freeze-Thaw* test to determine whether there were any changes in the physicochemical properties of the sunscreen cream during storage. Based on the organoleptic observations of the sunscreen cream preparation, it can be concluded that the *Freeze-Thaw* test only affected the color change of the cream

Homogeneity test

The homogeneity test on the cream aimed to determine whether all components in the cream are evenly mixed [22]. A homogenous cream indicates that all ingredients in the preparation are evenly mixed, thus not irritating when applied to the surface of the skin. Based on the homogeneity test observation, it is observed that all formulas have good homogeneity.

Cream-type test

The cream-type test aimed to determine the type of cream made, whether it is a/m or m/a [23]. Cream-type testing was performed by using the dilution method, which was by diluting the cream sample in distilled water. Cream that dissolves when diluted with distilled water is included in the m/a type, while cream that does not dissolve belongs to the a/m type [24]. In the cream type test, it was observed that the cream sample dissolved in distilled water, so it can be concluded that this sunscreen cream has a m/a type.

pH test

The pH test on cream aimed to determine the acidity level of a sample to ensure that its pH falls within a good range for topical products, making it safe to use and not irritating to the skin. The safe pH range for the skin is between 4.5 - 8 [25]. The properties of topical products that are increasingly basic or acidic when in contact with the skin cause the skin to have difficulty neutralizing, resulting in tired, dry, cracked, sensitive, and easily infected skin reactions [3]. The result of the pH test can be seen in **Figure 1**.

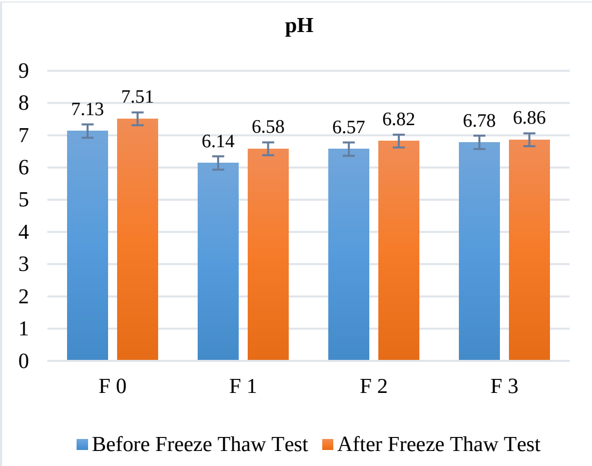

Figure 1. Observation of pH test of sunscreen cream.

The observation of the pH test on the sunscreen cream sample above shows that the pH of the sunscreen cream meets the safe pH range for the skin. In the observation of pH on the control formula and other formulas, a decrease in pH was observed, which was due to the acidic nature of the added propolis extract in the cream [26]. In addition, the addition of variations in TiO_2 concentration also affects the decrease in pH in each formula, which is because TiO_2 tends to be acidic [27]. From the observation results, it can be seen that before and after the *Freeze-Thaw* test, each formula showed an increase in pH after the test. This is because the temperature change during storage can affect the pH of the sunscreen cream.

The spreadability test

The spreadability test on the cream aims to determine the cream's ability to spread on the skin surface or is easy to apply. Good spreadability affects the speed of active ingredients absorbed by the skin. The higher the spreadability, the easier the cream is applied to the skin, so that the contact area between the drug and the skin becomes larger [22]. The requirement for good spreadability for topical preparations is around 5-7 cm [22]. The result of the spreadability test can be seen in **Figure 2**.

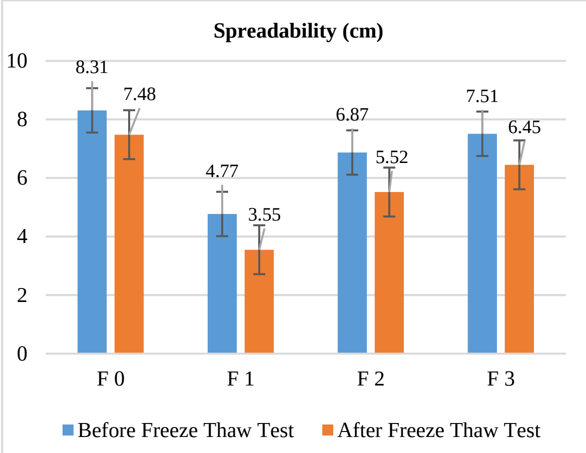

Figure 2. The spreadability test of sunscreen cream formula.

In the spreadability test, it can be seen that the spreading ability of the sunscreen cream before and after the *Freeze-Thaw* test meets the good spreading range for topical products which is around 5-7 cm. Based on the results, it can be concluded that there is a difference between the spreadability test results before and after the *Freeze-Thaw* test. This was because temperature changes during storage affected the spreadability of the sunscreen cream.

Adhesion test

The adhesion test on a cream preparation aims to determine how long the cream can stick to the surface of the skin. The longer the adhesion time, the longer the preparation contacts the skin. A longer adhesion time allows for complete absorption of the active ingredients by the skin. The appropriate adhesion time for topical products should not be less than 4 seconds [28]. Topical products with low adhesion time are less effective in delivering active ingredients to the skin. The result of adhesion test can be seen in **Figure 3**.

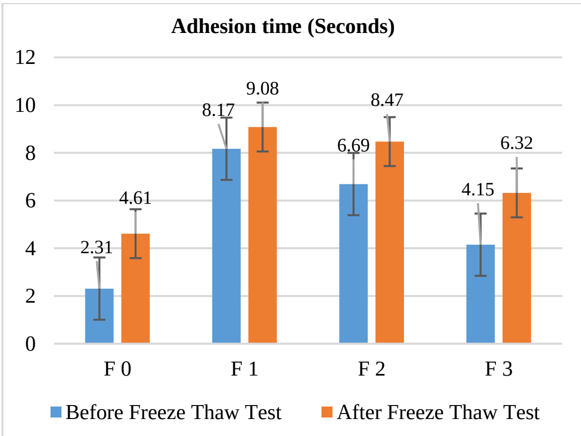

Figure 3. Adhesion test of sunscreen cream formulation.

In the adhesion test, it was found that all formulas met the standard for good adhesion. Based on the results of the test, it can be concluded that there is a difference between the results of the adhesion test before and after the *Freeze-Thaw* test. This was because temperature changes during storage affected the duration of adhesion of the sunscreen cream.

Viscosity test

The viscosity test on cream aims to determine the level of thickness of a made preparation. The higher the viscosity value, the more difficult it is to flow or the greater its resistance to the skin [29]. The viscosity requirement for good semisolid products is between 50-1000 dPas with an optimum value of 200 dPas [30]. The lower the viscosity of the topical product, the greater its spreading power on the skin which is inversely proportional to its adhesion. The lower the viscosity, the lower the adhesion [22]. The result of the viscosity test can be seen in **Figure 4**.

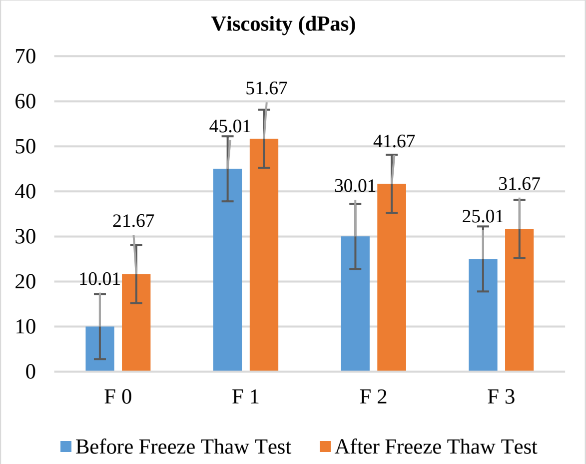

Figure 4. Sunscreen cream viscosity test.

In the test results, it was found that the viscosity of the sunscreen cream increased with the addition of propolis extract. This is because the added extract is a slightly thick liquid extract. The addition of TiO_2 concentration also affects the increase in product viscosity. From the test results, it was found that the higher the concentration of TiO_2 added, the higher the viscosity of the sunscreen cream. However, the sunscreen cream in each formula has not yet met the range of good viscosity values. This could be due to several factors such as the duration of stirring, the stirring speed, and the temperature during the preparation of the preparation [31]. Based on the test results, it can be concluded that there is a difference between the viscosity test results before and after the *Freeze-Thaw* test. This was because temperature changes during storage affected the viscosity of the sunscreen preparation.

Sun protection factor (SPF) test

The active skin protection of a sunscreen product is indicated by the Sun Protection Factor (SPF). The SPF value indicates how long a sunscreen product can protect or block ultraviolet rays that can cause skin damage [6]. The SPF value indicates how long a sunscreen product can protect the skin without causing damage when exposed to sunlight. The higher the SPF value of a sunscreen product, the better its protective activity is against the skin [32]. The result of Sun Protection Factor (SPF) test can be seen in **Figure 5**.

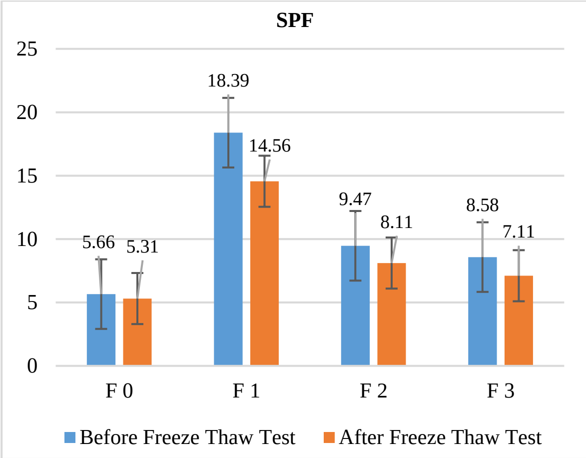

Figure 5. Sun Protection Factor (SPF) testing of sunscreen cream.

In the results of the above SPF value test, it can be seen that the addition of propolis extract in the sunscreen cream can increase the SPF value, as well as the addition of different concentrations of TiO_2 can result in different SPF values for each formula. The SPF value test results before and after the *Freeze-Thaw* test for each formula have relatively high protection. Based on the test results, it can be concluded that there are differences between the SPF test results before and after the Freeze-Thaw test. This was because temperature changes during storage affected the SPF value of the sunscreen cream formulation.

Conclusion

Variations in TiO_2 concentration have UV-protective activity with SPF values of Formula 0 at 5.66, Formula 1 at 18.39, Formula 2 at 9.47, and Formula 3 at 8.58. These SPF values decreased after the *Freeze-Thaw* test. The sunscreen cream cosmetic preparation with active ingredients of propolis extract and TiO_2 met the requirements for organoleptic, homogeneity, cream type, pH, spreadability, and adhesion tests. However, in the viscosity test, it did not meet the range of good cream requirements.

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Conflict of interest: None

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Ethics statement: None

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