The Effectiveness Test of Sunscreen Cream with Raw Material of Coconut Oil and Active Ingredients of Bay (Eugenia polyantha Wight) Leaf Ethanol Extract and TiO$_2$

E Widiyati*, D Ratnawati, D Fitriani, S Wati

Chemistry Department, Mathematics and Natural Sciences Faculty, Bengkulu University, Bengkulu, Indonesia

*widiyati58@unib.ac.id

Abstract. A research on the effectiveness test of sunscreen cream made of coconut oil as raw material and active ingredients of bay (Eugenia polyantha Wight) leaf ethanol extract and TiO$_2$ has been conducted. Sunscreen cream was made by emulsifying the water phase (distilled water, glycerin, methyl paraben and triethanolamine = TEA) which has been heated to 70° C and oil phase (lanoline, cetyl alcohol, stearic acid, coconut oil and prophyl paraben) which has been heated to 70° C as well and it was added an active ingredient of TiO$_2$. The mixture was then added the bay leaf ethanol extract and was stirred, until a homogeneous cream was formed. The resulting cream was determined its properties such as pH, viscosity, absorption of ultraviolet (UV) radiation and sun protection factor (SPF). From the SPF value calculated, then the effectiveness of sunscreen creams could be determined. The results showed that the resulting cream have a pH of 7.55-7.75, viscosity of 4000-10800 cps. If TiO$_2$ is added to a cream containing 0.5% bay leaf ethanol extract, the SPF value of the cream will increase. If the concentration of TiO$_2$ added to the cream is increased, the SPF of the cream will also increase. Cream with bay leaf ethanol extract of 0.5% and 6% TiO$_2$, has an SPF value of 3.101 and is included in the low protection category of sunscreens.

1. Introduction

Currently, sunscreen cosmetics that function to protect the skin from sun exposure have been sold in the market. In general, sunscreens contain active ingredients in the form of organic, inorganic compounds or a mixture of both [1]. Besides the two active ingredients, compounds extracted from plants can also be used. One of the compounds derived from plants that can be added to the preparation of sunscreen cosmetics is coconut oil.

Coconut oil has several benefits, such as containing a high percentage of saturated fatty acids (93%) making it suitable for making cosmetics [2]. Coconut oil is composed of saturated fatty acids which contain C = O groups and unsaturated fatty acids which have C=C and C=O functional groups [3]. These groups can absorb ultraviolet (UV) radiation [4]. Coconut oil has an SPF value of 7,119, so it is recommended for use in sunscreens preparation [5].

Other natural ingredients that can be used as active ingredients for preparation of sunscreens are plants that contain flavonoids. The flavonoid class compounds have the potential as sunscreen, because they have a chromophore group that can absorb UV radiation from the sun so that it can protect the skin [6]. In order to develop the use of active ingredients derived from plants,
currently, a research on the preparation of sunscreens made of plant active ingredients containing flavonoids has been carried out [7,8,9].

One of the plants whose leaves contain flavonoids, so that it can be used as an active ingredient in sunscreen preparation is the bay plant (*Eugenia polyantha* Wight). Bay plants are plants whose leaves are used for cooking spices in everyday life. Bay leaves (Figure 1) contain secondary metabolites such as triterpenoids, polyphenols, alkaloids, tannins and flavonoids [10].

![Bay leaf](image)

**Figure 1.** Bay leaf

To determine whether a cosmetic preparation has activity as sunscreen, or a sunscreen that has high effectiveness is by determining the sun protection factor (SPF) value of the preparation. Sunscreen that has a high SPF value, it is expected that will also have a high ability to protect the skin from exposure to UV radiation [5]. In general, sunscreen cosmetics that contain active ingredients of plant extracts have a low SPF value, so their protection against UV radiation is also low. The determination of the SPF value can be done by using the spectroscopic method. The resulting UV radiation absorption value can be used to calculate the SPF of the preparation using the Mansur equation [11,12].

Wati (2020) has been conducted a research on the preparation and characterization of sunscreen creams with the active ingredient of bay leaf ethanol extract. The results showed that the cream containing 0.5% extract had a low SPF value of 1.016, so it was not yet effective as a sunscreen [13]. To increase the activity of sunscreen creams that contain plant extract ingredients, inorganic sunscreen active ingredients such as TiO$_2$ can be added [1]. Based on the literature search, until now there has been no research or study on the effectiveness of sunscreen cream with active ingredients mixture of bay leaf ethanol extract and TiO$_2$. Therefore, it is necessary to conduct research on the effectiveness test of sunscreen cream made of coconut oil as raw material and a mixture of bay leaf ethanol extract and TiO$_2$ as active ingredients.

2. **Research Methods**

2.1 **Apparatus and Materials**

The research apparatus were laboratory glassware (such as measuring cups, beaker glass, measuring flasks, measuring pipettes, stirring rods, glass funnels and watch glasses), dark glass bottles, electric stoves, digital scales, UV-Vis spectrophotometers (Hitachi U-2900), rotary evaporator, cream container and filter paper.

The research materials were bay leaves, technical ethanol, virgin coconut oil, lanolin, cetyl alcohol, stearic acid, glycerin, triethanolamine (TEA), isopropanol (p.a), TiO$_2$, methyl paraben (p.a), propyl paraben (p.a) and distilled water (aquadest).
2.2 Work procedures

2.2.1 Preparation of Bay Leaf Ethanol Extract. Bay leaves were dried by air drying (without direct sun heating). After that, as much as 350 g of dried bay leaves were put into a dark bottle and added with technical ethanol, until all the bay leaves were submerged. The bay leaves were macerated for 4-7 days, and it were stirred occasionally. The filtrate containing the extract was separated and the dregs of the bay leaves were remacerated until a clear filtrate was obtained. The filtrate containing the extract was concentrated with a rotary evaporator to separate the solvent. The thick extract obtained was used as an active ingredient in making cosmetic creams.

2.2.2 Preparation of cream with the active ingredients of bay leaf ethanol extract and TiO$_2$ in various concentrations (2; 4 and 6%). In this study, 5 types of cream were made, namely sample 1, 2, 3, 4 and 5 (the cream formula was in Table 1). The sample 1 was made by the water phase (16 g glycerin, 1.5 g TEA, 0.4 g methyl paraben and 140.5 g distilled water) was put in a 500 mL beaker, then heated on an electric stove until it reached a temperature of 70°C. The oil phase (20 g coconut oil, 16 g stearic acid, 2 g lanolin, 2 g cetyl alcohol and 0.04 g prophyl paraben) was put in a 500 mL beaker then heated until it reached a temperature of 70°C, as well. Furthermore, the water phase was poured into the oil phase gradually while stirring until a semi-solid preparation was formed. The mixture was stirred until homogeneous and reached a temperature of approximately 35°C.

| Table 1. Formula of cream |
|---------------------------|
| Materials                 | 1  | 2  | 3  | 4  | 5  |
| Oil Phase :               |    |    |    |    |    |
| Stearic Acid (g)          | 16 | 16 | 16 | 16 | 16 |
| Coconut Oil (g)           | 20 | 20 | 20 | 20 | 20 |
| Cetyl Alcohol (g)         | 2  | 2  | 2  | 2  | 2  |
| Lanolin (g)               | 2  | 2  | 2  | 2  | 2  |
| Prophyl paraben (g)       | 0.04 | 0.04 | 0.04 | 0.04 | 0.04 |
| Water Phase :             |    |    |    |    |    |
| Glycerol (g)              | 16 | 16 | 16 | 16 | 16 |
| Aquadest (g)              | 142.06 | 141.06 | 137.06 | 133.06 | 129.06 |
| TEA (g)                   | 1.5 | 1.5 | 1.5 | 1.5 | 1.5 |
| Methyl paraben (g)        | 0.4 | 0.4 | 0.4 | 0.4 | 0.4 |
| Actives ingredient :      |    |    |    |    |    |
| Bay leaf extract (g)      | -  | 1  | 1  | 1  | 1  |
| TiO$_2$ (g)               | -  | -  | 4  | 8  | 12 |

The sample 2 was made by using the same procedure for making sample 1, but after the cream was formed, it was added 1 g of bay leaf extract. The mixture was stirred until a homogeneous form was formed. The preparation of samples 3, 4 and 5 were carried out in the same procedure as preparation of cream 2, but TiO$_2$ (the amount of TiO$_2$ could be seen in Table 1) was added to the heated oil phase. The creams formed were determined its properties such as pH, viscosity, UV radiation absorption and SPF value.

2.2.3 Determination of the cream pH. The amount of 5 g of cream was dissolved in distilled water until it reached a volume of 50 mL. The pH of the solution was determined using a pH meter that had been calibrated with a buffer solution of pH 4 and pH 7. The pH determination was carried out 3 times, and the resulting pH value was the average pH value read on a pH meter.
2.2.4. **Determination of the cream viscosity.** The determination of the cream viscosity was carried out by using a Brookfield viscometer, spindle 7, and 50 rpm. The viscosity value was calculated by multiplying the number shown on the instrument with the value shown in the table.

2.2.5. **Determination of the Ultraviolet (UV) radiation absorption of the cream.** The amount of 50 mg of cream was put into a 50 mL measuring flask and it was added isopropanol as a solvent until the limit mark. The resulting solution was determined for its UV absorption at a wavelength of 290-320 nm with 5 nm intervals, using a UV-Vis spectrophotometer. The absorbance obtained was used to determine the SPF value of the cream.

2.2.6. **Calculation of SPF cream.** The UV radiation absorption obtained was used to calculate the SPF value of each cream according to the Mansur method. The Mansur equation (1) was:

\[
\text{SPF spectrophotometric} = \frac{320}{290} \cdot \sum EE(\lambda) \cdot I(\lambda) \cdot \text{Abs}(\lambda)
\]

Note: CF = correction factor = 10, EE = erythema effect spectrum, I = sun intensity spectrum, Abs = absorbance of sunscreen products [14]. The normal values for EE x I for the calculation of the SPF were presented in Table 2.

| Wavelength (nm) | EE x I     |
|----------------|------------|
| 290            | 0.0150     |
| 295            | 0.0817     |
| 300            | 0.2874     |
| 305            | 0.3278     |
| 310            | 0.1864     |
| 315            | 0.0839     |
| 320            | 0.0180     |
| **Total**      | **1**      |

From the SPF value obtained, it could be determined the effectiveness of each cream produced. According to the FDA, based on the SPF value, the effectiveness of sunscreens could be categorized into: maximum protection level (SPF value > 50), high protection (SPF value 30-50), medium protection (SPF value 15-30) and low protection (SPF value 2-15) [15].

3. **Results And Discussion**

3.1 **The resulting of bay leaf extract**

In this study, the extract of bay leaves was made by maceration using ethanol as a solvent. Photo of bay leaves is presented in Figure 1. The ethanol solvent was chosen because it is polar, so it is hoped that the active ingredients that are polar, such as flavonoids, can be extracted. Preliminary test results for the presence of flavonoid compounds in the bay leaves show a positive (+) test. A positive test is indicated by a change in the color of the bay leaf extract from yellowish to purple red, after the extract was added with concentrated HCl reagent and Mg Tape [16]. This means that the bay leaves contain flavonoids (Figure 2).
Flavonoids are secondary metabolite compounds, in their structure have a chromophore group that can absorb UV radiation so that they have the potential as sunscreen [6]. From 340 g of dried bay leaves, after being macerated with ethanol solvent, then evaporated with a rotary evaporator to evaporate the solvent, obtained concentrated bay leaf extract as much as 30.25 g or 8.89%. The resulting extract is a thick green liquid (Figure 3). The extract obtained is used as an active ingredient in the preparation of sunscreen creams.

3.2 Cream with active ingredients of bay leaf extract and TiO$_2$

In this study, 5 creams (samples 1-5) were made with different concentrations of active ingredients. The resulting cream is semi-solid, with a soft texture. Sample 1 is the basic cream (without active ingredients) as a comparison, is white. Sample 2 is a cream with the active ingredient 1% bay leaf extract, greenish yellow. Samples 3, 4 and 5 were creams with the active ingredient 1% bay leaf extract and TiO$_2$ with different concentrations, namely 2; 4 and 6%. The more TiO$_2$ is added to the cream containing bay leaf extract, the lighter the cream color (Figure 4).

3.3 pH of cream

Determining the pH of the cream needs to be done because the cream produced is expected to be used on the skin surface so that the pH must match the skin pH. According to SNI (Standar Nasional Indonesia = Indonesian National Standards), the pH of sunscreens is 4.5 - 8. The results of pH determination in this study are presented in Figure 5.
From Figure 5, it can be seen that the resulting creams have a pH of 7.61 to 7.7. The pH results are still in accordance with the pH of sunscreen cream according to SNI.

**Figure 5.** The pH of cream with ingredients of 0.5% bay leaf extract and TiO$_2$ in varied concentration

Note:
Sample 1 = cream without active ingredient
Sample 2 = cream with 0.5% extract of bay leaf
Sample 3 = cream with 0.5% extract of bay leaf and 2% TiO$_2$
Sample 4 = cream with 0.5% extract of bay leaf and 4% TiO$_2$
Sample 5 = cream with 0.5% extract of bay leaf and 6% TiO$_2$

3.4 Viscosity of cream

According to the SNI standards (16-4399-1996), a sunscreen preparation must have a viscosity of 2000-50000 cps. The results of the viscosity determination in this study are presented in Table 3. From Table 3, it can be seen that the resulting cream has a viscosity of 4000 – 10800 cps, so it is still in accordance with SNI standards (16-4399-1996).

**Table 3.** The viscosity of cream with ingredients of 0.5% bay leaf extract and TiO$_2$ in varied concentration

| No | Sample  | Viscosity (cps) |
|----|---------|-----------------|
| 1  | Sample 1 | 4000            |
| 2  | Sample 2 | 4800            |
| 3  | Sample 3 | 10800           |
| 4  | Sample 4 | 6400            |
| 5  | Sample 5 | 6000            |

3.5 UV Radiation Absorption of Cream Containing Bay Leaf Extract and TiO$_2$

To find out whether the cream produced has sunscreen activity, it can be seen from the calculated SPF value. To calculate the SPF value, UV radiation absorption data at a wavelength of 290-320 nm, with 5 nm intervals are needed. The results of the absorbance determination of the cream are presented in Table 4. From Table 4 it can be seen that, sample 1 is a cream without active ingredients, resulting in the absorption with the lowest intensity, when compared with the other 4 samples. Sample 2 is a cream containing 0.5% bay leaf extract. With the addition of the active ingredient in the form of bay leaf extract, there is an increase in the absorption intensity. This is because the leaves contain secondary metabolites, one of which is flavonoids, as shown in the preliminary test. Samples 3, 4 and 5 are
creams containing 0.5% bay leaf extract in which TiO$_2$ is added with different concentration (2; 4 and 6%).

**Table 4.** UV radiation absorption of cream at wavelength of 290-320 nm (5 nm interval)

| No | Wave length (nm) | UV Radiation Absorption of Sample 1 | UV Radiation Absorption of Sample 2 | UV Radiation Absorption of Sample 3 | UV Radiation Absorption of Sample 4 | UV Radiation Absorption of Sample 5 |
|----|-----------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|
| 1  | 290             | 0.014                               | 0.061                               | 0.133                               | 0.200                               | 0.346                               |
| 2  | 295             | 0.010                               | 0.053                               | 0.126                               | 0.194                               | 0.330                               |
| 3  | 300             | 0.008                               | 0.048                               | 0.123                               | 0.190                               | 0.315                               |
| 4  | 305             | 0.008                               | 0.044                               | 0.119                               | 0.187                               | 0.306                               |
| 5  | 310             | 0.007                               | 0.040                               | 0.116                               | 0.184                               | 0.302                               |
| 6  | 315             | 0.007                               | 0.037                               | 0.115                               | 0.183                               | 0.302                               |
| 7  | 320             | 0.007                               | 0.035                               | 0.113                               | 0.182                               | 0.303                               |

The intensity of UV radiation absorption from sample 3 was higher than samples 2 and 1. From Table 4 it can be seen that the more TiO$_2$ concentration is added to the cream containing 0.5% bay leaf ethanol extract, the absorbance value also increased. Sample 5 is a sample containing 6% TiO$_2$ which has the highest absorption value compared to other samples. This condition indicates that the addition of TiO$_2$ affects the absorbance value of UV radiation cream with the active ingredient of bay leaf extract. The higher the TiO$_2$ concentration added, the higher the UV radiation absorption intensity from the cream. TiO$_2$ concentration can affect the absorbance value of the cream, because this compound is an inorganic sunscreen.

### 3.6 Cream SPF Value and Cream Effectiveness

The results of UV radiation absorbance determination are used to calculate the SPF value of the cream, using the equation developed by Mansur. The SPF calculation results of cream are presented in Table 5. From Table 5, it can be seen that cream containing only 0.5% of bay leaf ethanol extract has the lowest SPF value of 0.423. If this value is compared with the level of sunscreen protection based on the SPF value according to the FDA [15], sample 2 cannot yet be categorized as sunscreen. If 2% TiO$_2$ is added to sample 2, the SPF value of the cream (sample 3) will increase to 1.165, however this cream also cannot be categorized as having sunscreen protection. If the TiO$_2$ concentration was increased to 4% (sample 4) and 6% (sample 5), the SPF values of the cream also increased to 1.820 and 3.104. Cream with a concentration of 0.5% bay leaf extract and 6% TiO$_2$ is a cream that has the highest SPF value and is included in the low protection sunscreen.

**Table 5.** SPF value of cream

| Sample      | SPF   |
|-------------|-------|
| 1 (cream 1) | 0.080 |
| 2 (cream 2) | 0.423 |
| 3 (cream 3) | 1.165 |
| 4 (cream 4) | 1.820 |
| 5 (cream 5) | 3.101 |

The relationship between TiO$_2$ concentration and SPF value is presented in Figure 6. From Figure 6 it can be seen that the higher the TiO$_2$ concentration added to the cream with the active ingredient 0.5% bay leaf ethanol extract, the SPF value also increased. In this study, sample 5 was the cream that had the highest SPF value of 3.101. The cream is included in the low protection sunscreen.
In order for sunscreen preparations to have a high protective power against exposure to UV radiation, the sunscreen must also have a high SPF value. In this study, to increase the SPF value of cream, it could be done by increasing the concentration of the active ingredient, bay leaf ethanol extract, or the concentration of TiO$_2$ or both.

![Figure 6. Curve of the effect of TiO$_2$ concentration on SPF value of creams containing 0.5% bay leaf extract](image)

Figure 6. Curve of the effect of TiO$_2$ concentration on SPF value of creams containing 0.5% bay leaf extract

From Figure 6, it can be concluded that to get the high effectiveness of sunscreen creams that contain bay leaf extract, it can be done by increasing the concentration of TiO$_2$ added to the cream. According to the FDA, the maximum concentration of TiO$_2$ that can be added as an active ingredient in sunscreens is 25%. From the research results, it can be seen that if too much TiO$_2$ is added to the cream, the resulting cream will have a thick texture and if it is applied to the skin, it will turn white so that people do not like it. Therefore, it is necessary to develop a formula by varying the ratio of bay leaf extract and the right TiO$_2$ so that a sunscreen cream with high effectiveness and good efficacy is produced.

4. Conclusion
Creams made of coconut oil as raw material and active ingredients of bay leaf ethanol extract and TiO$_2$ have been successfully prepared. The resulting creams have a pH of 7.61 – 7.77, and a viscosity of 4000 - 10800 cps. The SPF value of the creams produced were 0.423 - 3.101. TiO$_2$ concentration affect SPF of cream. If the concentration of TiO$_2$ is increased, the SPF of cream will also increase. Cream with an active ingredient of 0.5% bay leaf ethanol extract and 6% TiO$_2$ has an SPF value of 3.101 and is included in a sunscreen with low effectiveness.

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