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Research Article

Optimization Formula and SPF Test of Lotion from Essential Oil of Patchouli Leaves (*Pogostemon cablin* Benth.)

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ABSTRACT

An increase in the ultraviolet (UV) index poses severe health risks by inducing harmful effects on the skin, such as sunburn, erythema, skin darkening, premature ageing, and skin cancer. These risks are mitigated through sunscreens with high Sun Protection Factor (SPF) values (>15). Natural sources of high SPF include patchouli leaf essential oil, which contains flavonoids. Flavonoid compounds with double bonds can absorb UV-A and UV-B rays, protecting against their harmful effects. The purpose of this study was to find the best formula and observe its impact on the physical characteristics of the preparation and SPF activity. Formula optimization used Design Expert software 13 with the simplex lattice design method. The optimized variables were TEA and stearic acid as emulsifying agents. The results of the optimum formula composition consisted of 4% TEA and 5% stearic acid. The optimum skin protection lotion preparation has a yellow color, a distinctive smell of patchouli oil, a semi-solid shape and a thick texture. The pH parameters of 7.7±0.09, adhesion of 2.34±0.06 seconds, spreadability of 6.85±0.02 cm and viscosity of 4503.3±219.39 Cp. Patchouli leaf essential oil and lotion have ultra-category SPF values with oil SPF value of 27.86±1.07 and lotion preparation of 27.65±1.75 preparation is also homogeneous with pH parameters of 7.7±0.09, adhesion of 2.34±0.06 seconds, spreadability of 6.85±0.02 cm and viscosity of 4503.3±219.39 Cp. Patchouli leaf essential oil and lotion have ultra-category SPF values with oil SPF value of 27.86±1.07 and lotion preparation of 27.65±1.75.

INTRODUCTION

The patchouli plant (*Pogostemon cablin* Benth.) is part of the Lamiaceae family of the Pogostemon genus. It is often used as a medicinal plant in conventional medicine (Rambe *et al*., 2022). The patchouli leaves contain phenolic compounds, flavonoids, alkaloids, saponins and triterpenoids (Widowati *et al*., 2019). Due to their conjugated single and double bonds, flavonoids can serve as sunscreen, absorbing ultraviolet (UV)-A and UV-B radiation (Nopiyanti and Aisiyah, 2020).

The antioxidant activity of patchouli oil, confirmed by the IC_{50} value, also showed good activity. Where patchouli leaf essential oil shows an IC_{50} value of 1.31µg/mL (Pandey *et al*., 2022), the antioxidant effect produced by phenolic compounds in plants can react with Reactive Oxygen Species (ROS), which can provide antioxidant effects and eliminate free radicals. These free radicals can be in the form of UV radiation on cells, especially skin cells, which can cause photooxidation reactions. This reaction can cause cancer, premature ageing, and skin darkening (Mohania *et al*., 2017). It can be applied in sunscreen preparations to utilize plant antioxidant activity to ward off UV radiation.

The lotion is a liquid emulsion consisting of an oil phase and a water phase stabilized by an emulsifier containing one or more active ingredients. An emulsifier is added to prevent the separation of the two phases (oil phase and water phase) (Lachman and Lieberman, 1994). Triethanolamine is widely used in topical pharmaceutical formulations, especially in the formation of emulsions. When mixed with fatty acids such as stearic or oleic acid, triethanolamine forms anionic soap with a pH of around eight. TEA-stearate salt can be used as an emulsifying agent to produce fine-grained and stable oil-in-water emulsions (Rowe *et al*., 2009).

The fatty acid that is suitable to be combined with TEA is stearic acid because stearic acid does not change color like oleic acid. This emulsifier (TEA-stearate) will cause the oil phase and water phase to combine to produce a homogeneous lotion preparation (Husni *et al*., 2021). The TEA concentration commonly used as an emulsifier is 2-4%, while the stearic acid used in topical preparations is 1-20% (Rowe *et al*., 2009).

Optimization is obtaining the best combination of a product or process under certain conditions. Various methods exist to process prediction data with data obtained through experiments for predetermined evaluation parameters. Design Expert software has multiple methods, including factorial design, response surface methodology (RSM), mixture and combined design (Hidayat *et al*., 2020).

Mixture Design is an optimization method used for components that change in a formulation proportionally to each other. One of the mixture methods is simplex lattice design (SLD), which is used to obtain the best formula for a mixture of components with the total number of different components having to be 1 (100%). This method's material components used for optimization consist of at least two materials (Hidayat *et al*., 2020). Optimization of TEA and stearic acid as emulsifiers in sunscreen lotion preparations was carried out to determine the suitable composition to obtain a formula that had good organoleptic, homogeneity, pH, adhesion, spreadability, viscosity.

METHODS

Study design

This study used an experimental method of mixing design to find the optimum composition of triethanolamine and stearic acid as emulsifying agents.

Variable

The independent variables in this study were the concentration of TEA and stearic acid. The dependent variables were the lotion's pH, adhesion, spreadability, and viscosity.

Instruments and Materials

The equipment used in this research was analytical scales (Ohaus, USA), mortar, stamper, hotplate (Thermo Scientific, USA), porcelain spoon, spatula, spatula, glass stirrer, scale ruler, lotion container, glass object, pH meter (Martini MI 150, US), viscometer (Brookfield Ametek, USA), tissue, scissors, ultraviolet visible (UV-Vis) spectrophotometer (Thermo Scientific, USA), glass beaker, measuring, petri dish, stirring rod, adhesion test equipment, and weights.

The material used for research was patchouli leaf essential oil (Tetesan Atsiri Indonesia, Bogor), which already has a Certificate of Analysis (CoA). The other materials were stearic acid (Camden-grey, USA)., cetyl alcohol (Sisco research laboratories, India), triethanolamine (Alpha Chemika, India), paraffin liquid, glycerin (Avantor Performance Material, USA), distilled water, methyl paraben and propyl paraben (Yokkaichi factory, Japan) with pharmaceutical grade and ethanol grade pro analysis (Brataco, Tangerang).

Optimization Lotion Formula

The simplex lattice design method was used in this study to predict the composition of Triethanolamine and stearic acid as an emulsifier in the formulation of patchouli leaf essential oil sunscreen lotion. The stearic acid range refers to research by (Iryani *et al*., 2021), namely in the range of 5 -7%, and the triethanolamine (TEA) range (Rowe *et al*., 2009), which is in the range of 2-4%. The responses used in the optimization stage are pH, adhesion, spreadability, and viscosity. Based on the TEA and stearic acid formula obtained through simplex lattice design optimization, a lotion formulation for patchouli leaf essential oil was created, which refers to the formulation of Ratnasari *et al*. (2023).

Formulation Lotion

The process of making lotion begins with weighing the ingredients. The manufacturing process is similar to creating an emulsion of water and oil phases. Materials that include the water phase include glycerin, methylparaben, TEA, and distilled water heated on a hotplate at 70ºC – 75ºC. Ingredients in the oil phase include stearic acid, paraffin, propylparaben and cetyl alcohol, which are melted on a hotplate at 70ºC – 75ºC while stirring. Then, the oil phase is added to the water phase while stirring to form an emulsion base. Once cool, the patchouli leaf essential oil is mixed and stirred until homogeneous (Iskandar *et al.*, 2021).

Organoleptic Test

Organoleptic tests are typically done by directly observing the lotion formulation's surface texture and physical appearance, including color, aroma and consistency (Ambari and Suena, 2019).

Homogeneity Test

The lotion was smeared on the slide for testing. The homogeneity of the preparation can be seen based on the absence of coarse grains or lumps in the preparation (Nisa *et al*., 2021).

pH measurement

The pH measurement test was done with a pH meter by dipping the pH meter stick into the lotion preparation. The pH results from the lotion will appear on the pH meter-monitor screen. If we refer to SNI number 16-4399-1996, the pH value of lotion that meets the criteria must match the skin's pH, namely between 5 and 8.

Adhesion Test

Lotion weighing as much as 0.1 g was placed in the middle of the glass object and covered with another glass object. A 50 g weight was placed on a covered glass object for 5 minutes. The end of the cover glass object and the bottom end of the glass object are connected to a clamp on the adhesion test tool, and then the load support is removed. The time the two glass objects are separated from the test equipment is recorded as the sticking time of the preparation (Pujiastuti *et al*., 2019).

Spreadability Test

Testing the spreadability of patchouli oil lotion was done by taking a small sample of the preparation and placing it in the middle of a petri dish. Another round glass was placed on top of the lotion preparation and a weight of 50 g added for 1 minute. Next, the weight was changed to 100 g and left for another 1 minute. The same steps were repeated with weights of 150 g and 200 g, and then the diameter of the distribution was recorded. The spreadability of lotion preparations is seen from the distribution width scale of the preparation, where the spreadability requirement is 5-7 cm based on SNI number 16-4399-1996.

Viscosity Test

Testing the viscosity of the lotion preparation was done by placing 120 grams of the preparation into a container and then measuring the viscosity using a Brookfield Ametek viscometer. Measurements started by installing spindle number 64 by turning the spindle lock clockwise. Viscosity measurements were recorded from the most extended number that frequently appeared on the viscometer screen. A suitable viscosity value based on SNI number 16-4399-1996 ranges from 2000-50,000 cP.

Verify optimum formula

The optimum formula generated by the Design Expert software is formulated into a lotion preparation using the same method as the previous eight runs. The optimum formula lotion was then tested for its physical properties and compared with the predicted physical properties values from the expert design software. The physical property parameters used include pH, spreadability, viscosity and stickiness.

Lotion Stability Test

One way to speed up the evaluation of preparation stability is by centrifugation testing. This mechanical test was done by inserting the lotion sample into a centrifuge tube. The centrifuge was used to centrifuge the sample for 30 minutes at a speed of 5000 rpm. Once finished, the product was observed whether separation occurs (Febrianto *et al*., 2021).

SPF Value Measurement

Patchouli leaf essential oil was tested by diluting the oil to a concentration of 5000 ppm. An amount of 0.05 g of essential oil was measured and then dissolved in 10 ml ethanol pro analysis. Meanwhile, when measuring lotion preparations, each lotion formula containing patchouli leaf essential oil (*Pogostemon cablin* Benth.) was weighed as much as 1 gram and dissolved in 10 ml of ethanol p.a. The SPF value was then measured using a UV-Vis spectrophotometer at a wavelength of 290-320 nm with 5 nm intervals (Ratnasari *et al*., 2023).

Data analysis

Evaluation of the physical properties of the preparation, including organoleptic and homogeneity tests, were evaluated descriptively. Meanwhile, from the verification tests that were done for the optimal formula that was found, the predicted response from the simplex lattice design method is seen and then compared statistically with the actual response using a one sample T-test with an accuracy of 95%. In analyzing the SPF value, the Mansur method was used as follows (Ratnasari *et al*., 2023):

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

Information:
CF : Correction Factor (=10)
EE : Erythemal effect spectrum
I : Solar Intensity spectrum
Abs : Absorbance of sunscreen product

RESULTS AND DISCUSSION

Physic evaluation of lotion

Based on the results in Table 2, it is known that all lotion formulas have the same distinctive smell, namely the smell of patchouli leaf essential oil. Apart from that, the similarity between the formulas can also be found in observing the color, where all the lotions produced are yellow, which comes from the color of the essential oil of patchouli leaves. There was no difference in color due to the same concentration of essential oils in all formulas.

The results of organoleptic testing on texture observations obtained varying results, where increasing the concentration of stearic acid caused the consistency of the lotion to become thicker. Runs 4 and 5 have the highest stearic acid concentration, resulting in a thick preparation. In the homogeneity test, homogeneous results were obtained for all runs made because there were no coarse grains on the glass object.

Based on the pH test data in Table 3, it can be seen that all runs meet the requirements for lotion pH. In topical preparations, pH is related to the feeling when applied; a pH that is too acidic or alkaline will irritate the skin, so it is necessary to match the lotion preparation with the skin's pH. The adhesion test results showed that eight runs had 2-6 seconds adhesion values.

Table 1. Patchouli Leaf Essential Oil Sunscreen Lotion Formula

Material (%)	Run							
	1	2	3	4	5	6	7	8
Patchouli oil	5	5	5	5	5	5	5	5
Stearic acid	5	6.5	5	7	7	5.5	6	6
Cetyl Alcohol	3	3	3	3	3	3	3	3
Paraffin liquid	8	8	8	8	8	8	8	8
Glycerin	8	8	8	8	8	8	8	8
TEA	4	2.5	4	2	2	3.5	3	3
Propyl Paraben	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Methyl Paraben	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Aquadest add	100	100	100	100	100	100	100	100

Table 2. Organoleptic Results

Run	R1	R2	R3	R4	R5	R6	R7	R8
Aroma	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil	Typical of patchouli oil
Form	Semi-solid	Semi-solid	Semi-solid	Semi-solid	Semi-solid	Semi-solid	Semi-solid	Semi-solid
Texture	A little thick	Thick	A little thick	Very thick	Very thick	Thick	Thick	Thick
Color	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow	Yellow

Table 3. Verification of Optimum Lotion Formula

Characteristic test	Result test			Average±SD	Prediction value
	R1	R2	R3		
pH	7.6	7.74	7.78	7.7 ± 0.09	7.57
Adhesive test	2.32	2.41	2.28	2.34 ± 0.06	2.4
Spread test	6.87	6.85	6.82	6.85 ± 0,02	6.87
Viscosity	4430	4750	4330	4503.3 ± 219.39	4384.58

According to SNI standard 16-4399-199616-4399-1996, the value of good adhesion is ≥ 4 seconds, but in other studies, namely in the research conducted by Utami and Sari (2022), the value of good adhesion for lotion preparations is no more than 4 seconds. Therefore, based on the research by Utami and Sari (2023), several runs, specifically R1, R3, R6, R7, and R8, fall within the required adhesion range of 2-4 seconds, while R2, R4, and R5 exhibit adhesion values exceeding 4 seconds.

The spreadability data of lotion preparations showed that the eight runs tested had good spreadability according to the requirements, namely 5-7 cm (Utami and Sari, 2022). The ability of the lotion base and active substances to spread over the skin's surface to provide a therapeutic effect is influenced by the extent of distribution when the lotion is applied. The viscosity test shows varying viscosity values, ranging from 4520 Cp to 9600 Cp. However, all variations in viscosity values still meet the range of viscosity requirements for lotion preparations. Increasing the concentration of stearic acid provides an increase in viscosity values.

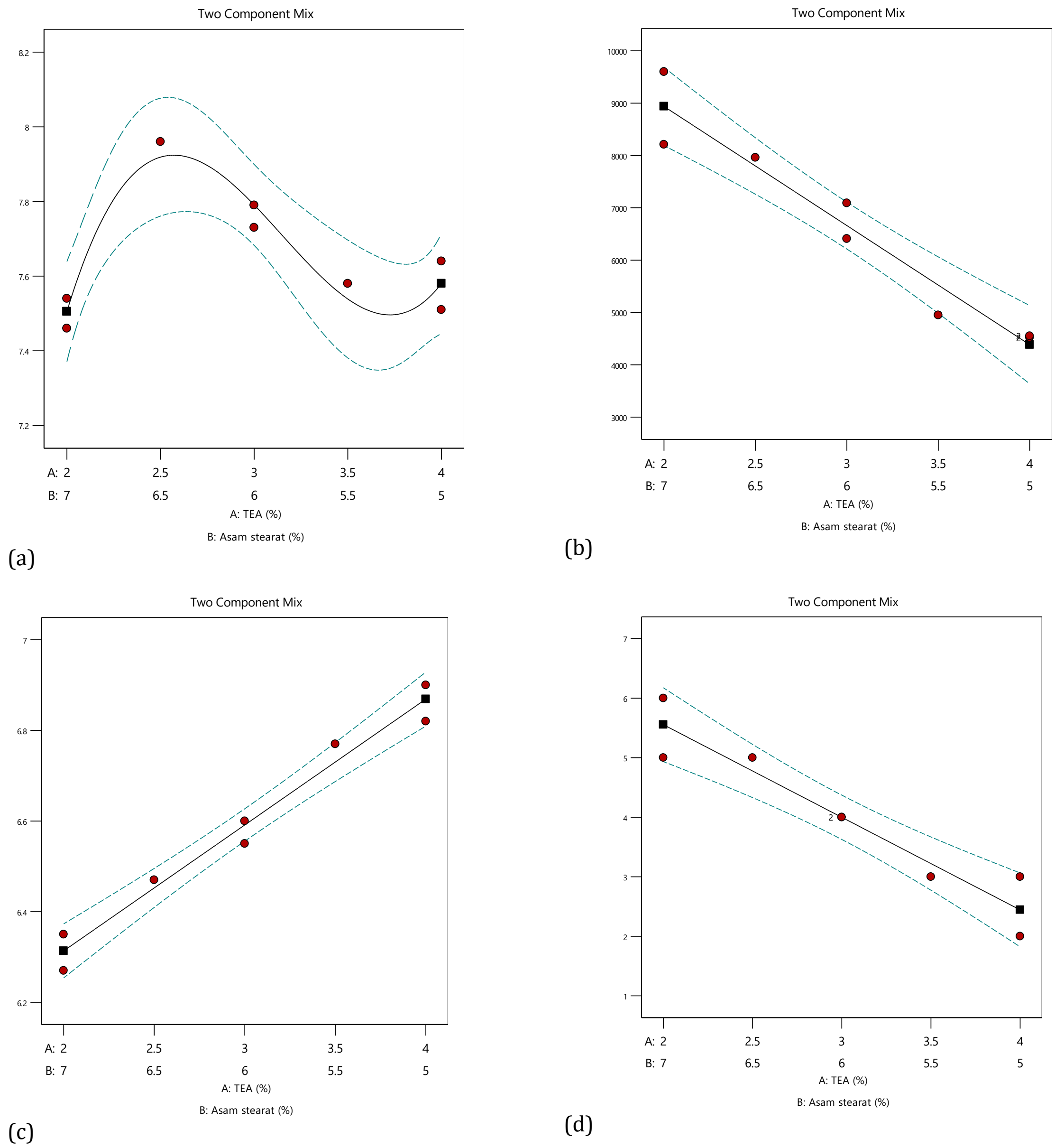

Figure 1. Contour Plot Graph (a) pH (b) Viscosity (c) Spreadability (d) Adhesion.

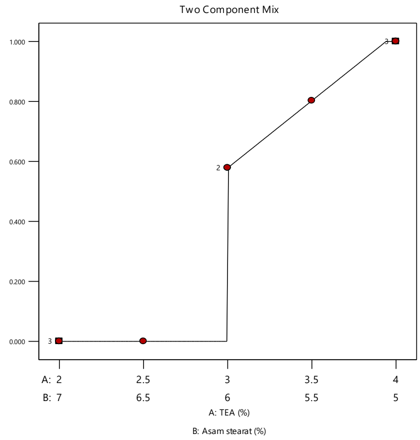

Figure 2. Preparation of Desirability Curve for Formulation.

Based on the contour plot graph in Figure 1, the results of optimization with simplex lattice design for the pH parameter using a cubic model, while for the adhesiveness, spreadability, and viscosity parameters using a linear model. The model was selected because it showed a significant p-value (0.05) and a lack of fit value that was not significant (>0.05).

Analysis of pH data produces a counterplot graph in Figure 1 with the following equation:

Y=7.58(A)+7.51(B)+0.9929(AB)-2.23(AB(A-B))………………………………………………………...(2)

Information:
Y = pH value
A = TEA composition
B = stearic acid composition

From equation 2, it can be said that the increased use of stearic acid and triethanolamine alone can increase the pH of the lotion preparation produced. The positive values of both components in the equation indicate this result. Meanwhile, the TEA component exerts the most significant influence, which has a better similarity value than stearic acid.

The interaction between stearic acid and TEA can also increase the pH value of the lotion preparation, as indicated by a positive coefficient. However, the more complex TEA and stearic acid interaction is shown to involve a negative interaction difference between the two. A negative coefficient indicates that an inappropriate ratio of TEA and stearic acid concentrations in the formulation can reduce the pH value of the lotion preparation. This can be seen in R7 and R8, which have a concentration of 6% stearic acid and 3% TEA, which can reduce the pH value of the preparation.

Analysis data of adhesion produces a counterplot graph in Figure 1 with the following equation:

Y=2.44(A)+5.56(B).. (3)

Information:
Y = adhesion value
A = TEA composition
B = stearic acid composition

From equation 3, it can be said that increasing the concentration of stearic acid and TEA influences the increase in adhesion, as indicated by the positive values of the two components in the equation. Meanwhile, the stearic acid component exerts the most significant influence with an equation value greater than TEA.

Analysis data of the spread test produces a counterplot graph in Figure 1 with the following equation:

Y=6.87(A)+6.31(B)... (4)

Information:
Y = spread test value
A = TEA composition
B = stearic acid composition

From equation 4, it can be said that increasing the concentration of stearic acid and TEA increases the spreadability of the resulting lotion preparation. The positive values of both components in the equation indicate this. Meanwhile, the TEA component exerts the most significant influence, which has a more excellent similarity value than stearic acid.

The data analysis of viscosity produces a counterplot graph in Figure 1 with the following equation:

Y=4384.58(A)+8937.92(B)....................................... (5)
Information:
Y = viscosity value
A = TEA composition
B = stearic acid composition

From equation 5, increasing the use of stearic acid and triethanolamine alone can increase the viscosity of the lotion produced. The positive values of both components in the equation indicate this. Meanwhile, the stearic acid component exerts the most significant influence with an equation value greater than TEA.

Determination of Optimum Formula

The optimum formula for this research was determined using Design Expert Version 13 software, using the simplex lattice design method. Optimization is then carried out using the numerical type because there are only two components to be optimized. To obtain the optimum formula, the researcher must determine the desired goal for each response with a significant value. Parameters of good lotion physical properties are used as targets. In testing pH and spreadability, "in range" was chosen with a range according to the pH range in testing patchouli leaf essential oil lotion because all test values were still within the required range. Meanwhile, the target for adhesive strength is made "in range" with a range of 2-4, where the lower limit adjusts the lowest adhesive strength value from the tests carried out. Meanwhile, the upper limit of 4 adjusts the range required for adhesion power, namely no more than 4 seconds, so it is hoped that the optimum formula can have adhesion power that meets the requirements.

The viscosity target is made into a minimized with a 4520 – 9600 Cp range. This considers the optimum lotion consistency, which is expected not to be too thick. Based on the physical properties' tests obtained, increasing the concentration of stearic acid will make the dosage too dense, making it challenging to apply. The viscosity value of an excellent lotion preparation ranges from 2000 - 50,000 Cp (Utami and Sari, 2022).

The optimal formula value obtained with a TEA concentration of 4% and stearic acid of 5% shows the program's ability to fulfil the desired final product in the software. A desirability value close to one indicates the possibility of getting the desired response value is better. The results of the optimum desirability formula in this study are shown in Figure 2.

The optimum formula recommended by simplex lattice design was then formulated with three replications. The actual physical property test results for the four responses in pH, viscosity, spreadability, and adhesion were then compared with those predicted by the simplex lattice design. The optimum formula verification results can be seen in Table 3.

The actual test data were then tested for normality using the Shapiro-Wilk test. The test results for all responses show a significance value of >0.05, which means all data were normally distributed. These results can be continued with the one sample T-Test test on actual test data with simplex lattice design prediction data.

The one sample T-test aims to determine whether a significant difference exists between the sample test results and the predicted results from the simplex lattice design. The results of the one-sample T-test for all responses have a p-value greater than 0.05, which means that the lotion showed conformity with the predictions of the simplex lattice design, indicating that the design is appropriate and accurate for formula development.

(a) (b) (c)

Figure 3. Lotion Centrifugation Test Results (a) Replication 1 (b) Replication 2 (c) Replication 3.

Stability test

A centrifugation test was conducted to see the phase separation that occurred in the lotion. Based on this test, the stability of the emulsion will be seen by observing the phase separation after centrifugation. In testing the optimum formula with a ratio of 4% TEA and 5% stearic acid, there was no separation between the three replications (Figure 3). Centrifugation done at a speed of 5000 rpm within 30 minutes showed no separation between the water and oil phases. Apart from that, preparation incompatibilities such as cracking or creaming were also not found in preparations that had been centrifuged. The effect of centrifugal force of 5000 rpm in 30 minutes is equivalent to the gravitational force received by the test preparation for a year.

Sun Protection Factor (SPF) test

Based on the results of measurements on patchouli leaf essential oil with a concentration of 5000 ppm, the SPF result was 27.86, included in the ultra-protection category. Patchouli leaf essential oil is then formulated into a lotion with a concentration of 5% combined with other lotion ingredients. Measuring the SPF in the lotion then obtained an SPF value of 27.65 (ultra protection), which was then carried out with a one sample T-test to see whether there was a significant difference in the SPF value of the oil before and after formulation.

Testing, which began with the Shapiro-Wilk normality test, showed that the data were normally distributed with p=0.14 (>0.05), then continued with the one sample T-test, which showed a value of p=0.170 (>0.05). This result means there is no significant difference between the SPF value of patchouli leaf essential oil before formulation and the SPF of patchouli leaf essential oil after formulation.

According to the FDA, a compound can be said to have maximum protection against UV-B rays if the SPF value is in the range 2-4, while the range 4-6 has moderate protection, 6-8 extra protection, 8-15 maximum protection and a value >15 indicates ultra protection (Cahyani *et al.*, 2021).

CONCLUSIONS

The optimal concentration of TEA and stearic acid in the lotion preparation of patchouli leaf essential oil was obtained by comparing the 4% TEA and 5% stearic acid concentration. Testing of physical properties shows the influence of TEA and stearic acid concentrations on the physical properties of pH, adhesion, spreadability, and viscosity pH, viscosity, spreadability and stickiness. The SPF value of patchouli leaf essential oil is 27.86±1.07, and after it was formulated into a lotion, the SPF value decreased to 27.65±1.75.

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