

## The effect of zinc oxide and *Curcuma heyneana* Val. combination on stability and sun protection factor (SPF) of lotion

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### ABSTRACT

Indonesian people risk-averse effects of Ultraviolet (UV) exposure, so they need skin protection agents. Zinc Oxide (ZnO) is an effective UV reflection, but at a concentration of more than 10%, it can cause skin irritation. Combining ZnO with other UV protective agents is necessary, so its concentration can be reduced but still effective. The flavonoid in *C. heyneana* has potential as a sunscreen because chromophore groups can absorb UV B and UV A light. This study aims to examine the effect of the combination of ZnO and *C. heyneana* on the stability and SPF value of lotion. The lotion is made in five variations of the composition of ZnO and *C. heyneana*, F1 of 100: 0%; F2 of 0:100%; F3 of 50:50%; F4 of 25:75%; and F5 of 75:25% respectively. Test parameters include organoleptic, pH, spreadability, viscosity, and SPF. The test is recorded before and after the cycling test. Statistical analysis using Paired Sample T-Test. The combination of ZnO and *C. heyneana* affected organoleptic, viscosity, spreadability, pH and SPF value. The higher concentration of ZnO will increase the viscosity, pH, and SPF but decrease the spreadability. The formula with a combination of ZnO and *C. heyneana* of 50%:50% is selected because it has an SPF value of 13.24 and; a viscosity of 48.67 d.Pas; pH of 7.1; and spreadability of 3.81 cm.

**Keywords:** lotion stability, cycling test, SPF, ZnO, *Curcuma heyneana* Val

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## INTRODUCTION

Indonesia is a country with a tropical climate that is located on the equator with a high intensity of UV light exposure. UVB and UVA light work synergistically, so protection is needed to reduce the adverse effects of UV radiation on the skin. UV light is harmful when exposed for a long time because it can cause skin cancer, eye damage such as cataracts and melanoma, premature skin aging, pigmentation, erythema, and damage to the immune system. The adverse effects of UV exposure depend on the intensity of the light, the sensitivity of the individual exposed, and the length and frequency. The World Health Organization (WHO) 2015 estimates the incidence of non-melanoma cancer will increase by 300,000 and melanoma by 4,500 due to the depletion of the ozone layer. In Indonesia, skin cancer is third rank after uterine and breast cancer. The percentage of skin cancer is 5.9-7.8% of all types of cancer per year. Therefore, the skin needs effective protection to reduce excessive sun exposure. The effects of sun exposure can prevent by using sunscreen. Sunscreen is an ingredient or preparation that contains substances that can absorb or reflect UV radiation, thereby reducing radiation energy that penetrates the skin (Maulida, 2015).

Sun Protection Factor (SPF) indicates the ability of sunscreen products to protect the skin from UV light exposure (Stanfield, 2001). SPF shows how long we are protected from UV light without getting burned. ZnO is an inorganic UV filter that can reflect or scatter UV light. The concentration range of ZnO required by the FDA (Food and Drug Administration) as sunscreen is 2% - 20% (Andre, 2013). The highest concentration of ZnO in cosmetic products is 10% (Martorano et al., 2010). Using a high concentration of ZnO causes skin irritation, so innovation is needed to reduce the concentration of ZnO in sunscreen products but still have optimal UV protection. The combination of organic UV filters and inorganic UV filters can provide a synergistic effect as UV protection (Amnuait & Boonme, 2013). *Curcuma heyneana* Val. has active ingredients, including starch, essential oils, resin, tannins, saponins, and flavonoids (Evizal, 2013). The flavonoids in *C. heyneana* have sunscreen potential because of the chromophore group (conjugated double bond). It can absorb UV B and UV A light (José et al., 2016). The starch of *C. heyneana* has potential as sunscreen due to the opaque properties of starch which is impermeable to light but can reflect UV light (Oktaviasari & Zulkarnain, 2017). Previous research reported that the SPF value of *C. heyneana* 1% w/w extract was 4.84 (moderate protection), 2.5% w/w was 8.76 (maximum protection), and 5% w/w was 18.86% (ultra-protection) respectively. According to research by (Wulandari et al., 2019), the SPF value of ZnO dispersed into hydrogel was 25 (medium UV protection). Combining the two ingredients is expected to work synergistically to optimize the SPF value of the preparation as a sunscreen.

The lotion is easier to formulate than cream, has low viscosity, has shorter heating and cooling times, and has an O/W type emulsion base that is washed easily. ZnO and *C. heyneana* powder were sieved with a number 100 sieve because, generally, the powder must pass through a sieve with a particle size of 100 mesh to not irritate the skin. The particle size passing mesh 100 is 0.149 mm, while the estimated skin pore size is  $2 \times 10^{-5}$  mm -  $4 \times 10^{-5}$  mm so that it is ensured that the powder passing through mesh 100 does not irritate the pores. A good sunscreen cosmetic preparation is stable and meets the quality requirements for sunscreen cosmetic preparations, including organoleptic, pH, viscosity, and Sun Protection Factor (SPF). Quality requirements for sunscreen lotion preparations that apply in Indonesia refer to SNI 16-4399-1996. This study will examine the effect of the combination of ZnO and *C. heyneana* powder on the stability and SPF value of lotion.

## MATERIALS AND METHOD

### Materials

Rimpang Temugiring (*Cucuma heyneana* Val.) from CV. Herba Medika Tawangmangu, Central Java, Indonesia; Zinc Oxide grade 102 LLC 800HSA Kuraray Asia Pasific Singapore; Stearic Acid Dow Chemical Singapore; Cetyl alcohol Dow Chemical Singapore; Glycerin, Dow Chemical Singapore; Triethanolamine CAS 102-71-6, Kuraray Asia Pasific Singapore; liquid paraffine Kuraray Asia Pasific Singapore; phenoxyethanol (repacking by PT Nitra Kimia, Yogyakarta) Batch 028/SE/0817 No.17SE0121; all ingredients of lotion base are cosmetics grade; aquadest (repacking by

CV Agung Jaya, Surakarta). The main equipments in this research are UV-Vis spectrophotometer (GenesysTM, Japan), digital calipers (Krisbow, USA), incubator (Thermo Scientific Series 8000 WJ), glassware (Pyrex), and hotplate (thermo scientific), pH meter (ATC, China), Viscosimeter (Rion VT-04, Jepang), Oven (Mettler), refrigerator (SHARP, Jepang).

## Methods

### Sample Preparation

ZnO and *C. heyneana* powders were sieved using sieve number 100. The samples were tested for moisture content and powder flow rate. The 1.0 g of sample powders were weighed and then put in a moisture analyzer, then the percentage of moisture content was measured. The percentage of moisture content that meets the requirement is less than 10%. A sample powder of 100 g is passed through a funnel, and the time needed for all the powder to pass through the funnel is calculated. The flow time of good powder is less than 10 seconds.

**Table 1. The lotion formula using combination of ZnO and *C. heyneana***

Ingredients	Concentration (%)					
	Base	Formula 1 100% ZnO	Formula 2 100% <i>C. heyneana</i>	Formula 3 ZnO- <i>C. heyneana</i> 50: 50%	Formula 4 ZnO- <i>C. heyneana</i> 75: 25%	Formula 5 ZnO- <i>C. heyneana</i> 25: 75%
ZnO	0.0	2.0	0.0	1.0	1.5	0.5
<i>C. heyneana</i>	0.0	0.0	2.0	1.0	0.5	1.5
Glycerin	5.0	5.0	5.0	5.0	5.0	5.0
Cetyl Alcohol	2.5	2.5	2.5	2.5	2.5	2.5
Liquid Paraffin	7.0	7.0	7.0	7.0	7.0	7.0
Triethanolamine	1.0	1.0	1.0	1.0	1.0	1.0
Stearic Acid	2.5	2.5	2.5	2.5	2.5	2.5
Phenoxyethanol	0.2	0.2	0.2	0.2	0.2	0.2
Aquadest	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100

### Lotion formulation

Lotion Formula is developed from previous (Amatullah et al., 2017) research. The ingredients in Table 1 were weighed and heated separately until at a temperature of 70-80 °C. The mixture was continuously stirred. The oil phase, namely stearic acid, cetyl alcohol, and liquid paraffin, were melted in a water bath. In the aqueous phase, triethanolamine and glycerin dissolved in hot water and then stirred, at the last added phenoxyethanol. The mixture was homogenized, and ZnO and *C. heyneana* powder was added to the lotion base.

### SPF test using spectrophotometer UV-VIS method

The samples test includes a solution of ZnO, *C. heyneana*, and lotion diluted up to 2000 ppm. The absorbance of sample solutions was then measured using spectrophotometer UV-VIS at a wavelength of 290-320 nm with a wavelength interval of 5 nm. The absorbance value is then calculated using the Mansur method. Where CF is the correction factor, EE is the erythema effect spectrum, I is a sunlight intensity spectrum, and Abs is the absorbance of the sample. The value of  $EE \times I$  is constant to 1 (Mansur, 1986). The SPF value test is carried out before and after the cycling test procedure.

$$\text{SPF} = \text{CF} \times \sum \text{EE} (\lambda) \times \text{I} (\lambda) \times \text{Abs} (\lambda) \quad (1)$$

### **Stability test of lotion**

The stability test was carried out using the cycling test method for six cycles. The samples were stored at 4 °C and 40 °C for 24 hours, respectively. Parameters observed in the test were: a) Organoleptic test: Observing the samples includes odor, consistency, and color of the lotion preparation. b) pH test: The electrode of the pH meter was dipped in the lotion until submerged and steady for reading. The results of the pH measurement of the preparation will meet the skin pH criteria at an interval pH value of 4.5-8 (SNI 16-4399-1996). c) Spreadability test: Lotion is weighed at 0.5 g, then placed on the test equipment and covered with glass. The equipment is then given a load of 150 g, wait for 1 minute until constant, and record the diameter of the lotion that spreads. d) Viscosity test: Viscosity was measured with a viscosimeter. The lotion is placed into the container, and the spindle number 1 is installed until submerged into the lotion. The viscosimeter is turned-on, and the viscosity value is recorded until it stops stable. The number shown at the viscometer is in dPas (1 d.Pas = 1 poise).

### **Data Analysis**

The data obtained were analyzed using the IBM SPSS Statistics 23 program. The normality of the data was tested using the Shapiro-Wilk test method. The effect of the ZnO and *C. heyneana* combination was analyzed using the one-way ANOVA test. Paired Sample T-Test analysis to determine the effect of the cycling test on lotion preparations.

## **RESULT AND DISCUSSION**

Plant identification is carried out at the Plant Systematics Laboratory, Biology Faculty, Universitas Gadjah Mada, Yogyakarta. Plant identification aims to ensure the botanical identity of the plants used in the study. The identification results state that the plant used in the study is a *Curcuma heyneana* Val species with a test number of 014950/5. Tb/II/2021. The organoleptic result of *C. heyneana* powder is orange color, homogeneous powder size, aromatic odor, and bitter taste.

The moisture content test aims to provide a minimum limit of water content in the material. The higher water content is easier to contaminate with fungi and molds, so it can reduce the quality of the powder during storage. The moisture content also depends on the drying time; a longer drying time will lower the moisture content. Moisture content in powder should not be more than 10%. Good moisture content is between 1-5%. The test result of moisture content of *C. heyneana* powder is 5.5%, and ZnO is 7.48%.

The SPF value test aims to determine the SPF value contained in the active substances of *C. heyneana* and ZnO powder. Sun protection factor (SPF) is a universal indicator that describes the effectiveness of UV protector products. The in vitro method used in determining SPF value in lotion preparations is Spectrophotometer UV-VIS, calculating the absorption value at a wavelength of 290-320 nm. The result of measuring the SPF value of *C. heyneana* and ZnO powder is 7.3 and 15.02, respectively. According to the ability as sunscreen, *C. heyneana* has an extra protection category, and ZnO has an ultra-category. Sunscreen products in Indonesia must meet the minimum requirements of SPF value of 4 according to Indonesian Standard Product by SNI 16-4399-1996. ZnO can absorb the solar spectrum and light quantum, and *C. heyneana*'s flavonoid that has chromophore groups can absorb UV B and UV A light.

### **Lotion formulation**

The oil-in-water (o/w) type of emulsion is chosen for lotion preparation. The o/w type lotion will be used on the skin with a wide distribution area, thus providing optimum effect. In addition, it can increase the concentration gradient of the active substance that penetrates the skin, thereby

increasing skin adsorption. It is because the amount of oil phase that is dispersed is smaller than the water phase, thus forming an oil-in-water emulsion with the help of an emulsifier (Ermawati, 2017).

Cetyl alcohol, as a stiffening agent, will increase viscosity. Cetyl alcohol works by incorporating the oil phase into small droplets and is carried into larger droplets. Cetyl alcohol also acts as a co-surfactant because it helps solubilize the oil phase into droplets containing the aqueous phase. Cetyl alcohol is a fatty alcohol group, where 3-OH groups will form hydrogen bonds with water, and the fatty part will bind to oil. Cetyl alcohol at a concentration of 2-5% has a function as an emulsifying agent and is stable in acids and bases conditions (Rowe, 2009). A combination of cetyl alcohol and stearic acid in a 1-20% concentration will produce a stable emulsion. Stearic acid and cetyl alcohol are solid, so they need to be melted to facilitate mixing at a temperature of 70 °C. The temperature above the melting point of cetyl alcohol and stearic acid so that the oil phase does not accelerate and solidifies when poured into the aqueous phase. Stearic acid will react with triethanolamine to form triethanolamine stearate, which is a soap (anionic surfactant). Triethanolamine stearate functions as an internal emulsifier so that the oil phase and water phases are miscible. Triethanolamine stearate is a soap monovalent that forms an oil-in-water-type lotion (Anief, 2000).

Glycerin functions to increase viscosity and form hydrogen bonds with water so that it can inhibit evaporation from emulsion preparations. Glycerin also acts as a co-solvent in emulsion preparations (Rowe, 2009). Glycerin functions as a humectant in retaining moisture so that the lotion preparation is still moist during storage. Glycerin as humectants can increase the adsorption of water from the dermis to the epidermis to inhibit skin hydration and absorb water from the skin's external environment. Glycerin is an emollient that provides a moisturizing effect on the skin and improves skin elasticity skin flexibility. Phenoxyethanol functions as a preservative to minimize bacterial and fungal contamination.

### Stability test

The lotion has changed color, odor, and consistency after the cycling test. Test results are presented in Figure 1. The lotion base has milky white color, the formula with 100% ZnO has milky white color, the formula with 100% *C. heyneana* has a brown color, and the formula with ZnO-*C. heyneana* of 50:50% has a cream color formula with ZnO-*C. heyneana* of 75:25% has a pale cream color and the formula with ZnO-*C. heyneana* of 25:75% has a pale brown color. The concentration of ZnO causes the color of the lotion to become white, with a stiffer texture. Lotions on the 12th-day have change inconsistency to become runnier and begin to separate especially at formula 3, 4 and 5. It is necessary to shake it, so the lotion re-unites. Phase separation occurs because the active ingredient is starch powder and ZnO powder which are insoluble in water and affect the force gravity. Particles that have a lower density will rise to the surface. The separation is called creaming, is reversible, and can be removed by shaking (Ali, 2013). The higher concentration of *C. heyneana* affects the color of the lotion darker. The organoleptic observations concluded that the concentration of *C. heyneana* and zinc oxide could affect the organoleptic properties of a lotion.

According to SNI 16-4399-1996, the lotion viscosity requirement is between 2000 and 50,000 cP. The higher viscosity value affects the viscosity of the preparation, so it has a long sticking time. In contrast, the low viscosity value affects the viscosity of preparation more dilute and has a fast attachment time. A concentration of ZnO can increase viscosity, and it is because ZnO is an insoluble material, so the lotion preparation becomes stiffer, thicker, and higher the viscosity response (Rosyidi et al., 2018). Lotion preparation with a 100% concentration of *C. heyneana* powder has a higher viscosity than a lotion with 100% ZnO. Because starch is insoluble in water, the starch has soft amorphous powder properties (Jain et al., 2014). Interaction between ZnO and *C. heyneana* powder shows a tendency to increase the viscosity of lotion preparation (Table 2).



**Figure 1.** the result of organoleptic test of lotion before and after cycling test. Formula 0 is lotion base; Formula 1 is 100% ZnO; Formula 2 is 100% *C. heyneana*; Formula 3 is combination of ZnO-*C. heyneana* 50:50%; Formula 4 is combination of ZnO-*C. heyneana* 75:25%; and Formula 5 is combination of ZnO-*C. heyneana* 25:75%

**Table 2.** The result of stability test of lotions before and after cycling test

Formula	Viscosity (d.Pas)		pH value		Spreadability (cm)		SPF value	
	before	after	before	after	before	after	before	after
ZnO powder							15.02	
<i>C. heyneana</i> powder							7.30	
Lotion base	26.67±1.53	24.33±1.15	6.90±0.10	6.97±0.06	5.97±0.17	6.28±0.16	1.57±0.12	1.20±0.11
F1 (100% ZnO)	26.67±2.89	23.33±5.77	6.70±0.17	7.43±0.49	4.72±0.15	4.94±0.06	12.25±0.27	7.43±0.03
F2 (100% <i>C. heyneana</i> )	43.00±3.00	24.67±4.16	6.40±0.10	6.67±0.12	5.65±0.22	5.83±0.26	6.77±0.04	3.53±0.12
F3 (50: 50% ZnO- <i>C. heyneana</i> )	48.67±1.15	15.00±3.00	7.10±0.26	7.47±0.12	3.81±0.09	6.89±0.11	13.24±0.20	8.33±0.03
F4 (75: 25% ZnO- <i>C. heyneana</i> )	50.67±0.58	9.33±1.15	7.27±0.21	7.53±0.12	3.99±0.15	7.09±0.13	9.94±0.06	6.20±0.06
F5 (25: 75% ZnO- <i>C. heyneana</i> )	49.00±3.61	10.67±1.15	7.40±0.26	7.67±0.25	3.93±0.08	6.57±0.13	8.01±0.04	6.20±0.06

ZnO can increase the pH value of the preparation because of the reaction between ZnO and water in a lotion base. ZnO ionizes (in low concentration) to Zn<sup>2+</sup> and combines with OH<sup>-</sup> ions to form Zn(OH)<sub>2</sub>. Zn(OH)<sub>2</sub> is a weak base compound that causes the pH of the preparation to increase (Wardhani, 2006). The effect of 100% *C. heyneana* powder resulted in a lower pH because *C. heyneana* was lower than the pH of 100% ZnO (Yustin & Wijayanti, 2018), so it is possible to lower the pH of the preparation. Lotion with a combination of ZnO- *C. heyneana* as active ingredients (at F3, F4, and F5) have a high pH. In this study, during the cycling process, the lotions have a changed pH value after the cycling test. Measurement was carried out using a pH meter. The change in the pH value may be affected by components in the formula. There are degraded by high temperatures during

manufacture or storage that produce acids or bases. The changes in pH are caused by environmental factors such as temperature. The concentration of ZnO- *C. heyneana*, and the combination of ZnO- *C. heyneana* affect the pH value. Paired sample test analysis by comparing the value of the result before and after the cycling test. pH test at formula 3 show that the sig value of 0.123 ( $p > 0.05$ ), it can be concluded that the cycling test affects pH.

The spreadability test of the lotion aims to determine the ability of the lotion to spread and the ease of application to the skin. Good dispersion causes the active substance to contact and absorb into the skin quickly. The diameter of the spreadability that meets the requirement for semisolid preparations is 5-7 cm. Influencing factors of spreadability are the viscosity of the preparation, duration of pressure, and temperature at the site of action. The high viscosity value impacts decreasing the dispersion value, as well as otherwise (Garg et al., 2010). The addition of ZnO composition, *C. heyneana* powder, and combinations of ZnO- *C. heyneana* in the preparation affects the lower diameter of dispersion. Lotion with an active ingredient combination of ZnO- *C. heyneana* has the highest viscosity. The results of the one-way ANOVA test show the sig value of 0.000 ( $\text{sig} > 0.05$ ). The interpretation of the results shows the differences in dispersion value between formulas because it contains different concentrations of active ingredients. These results mean the concentration of ZnO, *C. heyneana*, and the combination of ZnO- *C. heyneana* affects the dispersion. The sig value of 0.041 ( $p > 0.05$ ) at the cycling test means correlation, so it is concluded that it affects the lotions' spreadability. Formula 4 is the most high spreadability value after cycling test.

Factors that affect the results of the SPF value include solvent, sunscreen combination, sunscreen concentration, emulsion type, effect, and interactions of the components of the preparation base (esters, emollients, and emulsifiers), interactions of the lotion base with the skin, additions of the active ingredient, and the pH system. These factors can increase or decrease UV absorption ability in sunscreen products (Yulianti et al., 2015). Based on the results of the SPF test, the SPF value of lotion preparations using active ingredients of ZnO, *C. heyneana*, and the combination of ZnO- *C. heyneana*, have different SPF values before and after the cycling test in each formula. Extreme temperatures cause a decrease in the SPF value due to the hydrolysis process of ZnO and *C. heyneana*, which causes sunscreen's ability. The difference in the SPF value in each formula is due to the difference in ZnO and *C. heyneana* concentration.

## CONCLUSION

The combination of ZnO and *C. heyneana* affected organoleptic, viscosity, spreadability, pH, and SPF value. The higher concentration of ZnO in combination with *C. heyneana* will increase the viscosity and pH but decrease the spreadability. The formula with a combination of ZnO- *C. heyneana* of 50%:50% is selected because it meets the requirement of sunscreen product by SNI 16-4399-1996. The lotion has an SPF value of 13.24, viscosity of 48.67 dPas; pH of 7.1; and spreadability of 3.81 cm.

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