

RESEARCH ARTICLE

Determination of Physical Stability Spray Sunscreen of Extract Wungu Leaf (*Graptophyllum pictum* (L.) Griff) with Varied Concentrations of Glycerine as A Humectant

Mayu Rahmayanti, Annisa Tri Maulidina, M. Bayu Firdaus Buana Putra

Maulana Malik Ibrahim State Islamic University of Malang,

Jl. Locari, Tlekung, Junrejo, Batu City, East Java 65151, Indonesia.

*Corresponding Author E-mail: mayu31@farmasi.uin-malang.ac.id

ABSTRACT:

Sunscreen is a preparation intended to counteract, reduce the effects, and protect the skin from ultraviolet (UV) radiation. Wungu leaf is one of the plants that contain antioxidants that can be used as a natural sunscreen preparation. In the manufacture of sunscreen, spray wungu leaf extract required additional materials in the form of humectants. Glycerin was chosen as a humectant with varying concentrations of 5%, 10%, and 15% in the formulation of a spray sunscreen which aims to improve the quality and maintain the stability of the resulting preparation. This study aims to determine the physical stability of the spray sunscreen preparation of wungu (*Graptophyllum pictum* (L.) Griff) leaf extract with varying concentrations of glycerin as a humectant. Spray sunscreen of wungu leaf extract was made in three formulas with variations in the concentration of glycerin used. The formulations that have been formulated are tested for physical stability by using the cycling test method for six cycles, and testing includes organoleptic, homogeneity, pH, viscosity, and diameter of the spray pattern. The organoleptic, homogeneity and pH tests of the preparations showed no significant changes after the physical stability test was carried out for six cycles with a significance pH value of 0.079 (<0.05). The resulting value still met the pH value criteria for topical preparations. The results of the viscosity test and spraying pattern showed an increase in the viscosity value and a decrease in the diameter of the spray pattern with a significant change with a significance of 0.01 (<0.05) and 0.008 (<0.05). However, this change still met the physical criteria for topical preparations. Based on the study's results, it can be concluded that the spray sunscreen preparation of wungu leaf extract (*Graptophyllum pictum* (L.) Griff) with variations in glycerin concentration has good physical stability.

KEYWORDS: Cosmetics, Cycling test, Glycerin, Spray sunscreen, Wungu leaves.

INTRODUCTION:

Sunscreen is a pharmaceutical product that protects the skin from exposure to ultraviolet (UV) radiation by forming a protective barrier on the skin surface^{1,2}. Today, many sunscreens on the market are in the form of creams and lotions, which has the drawbacks that it leaves an oily impression after wearing and most of them contain synthetic ingredients that are harmful if used in the long term^{3,4}.

In response to this problem, many studies have developed sunscreen preparations made from herbs and in various dosage forms. One of them is the development of a spray sunscreen which is considered more practical in its use. The type of system determined based on the physical, chemical, and pharmacological properties of the active ingredients and application sites⁵. Sunscreens with herbal ingredients are considered to have fewer unwanted side effects than sunscreens with synthetic ingredients, are non-toxic, and do not irritate the skin^{6,7,8}. Wungu leaves can be used in making herbal sunscreens. This plant is easy to find in Indonesia⁹. *Graptophyllum pictum* L. Griff contains various phytochemical compounds, one of which is antioxidants. Antioxidants are an essential compound in sunscreen preparations^{10,11,12}. Antioxidants can play a role in counteracting free radicals that can damage

tissues in the body^{13,14,15}.

The dosage form of sunscreen that is currently becoming a trend is spray sunscreen. This dosage form provides advantages in its use which is easily spread to the surface of the skin and has the same effectiveness as a lotion or cream^{15,16,17}. The manufacture of preparation required additional materials that have an essential role in the formulation of the sunscreen product, one of which is glycerin. Glycerin in topical preparations acts as a humectant that forms a layer on the skin's surface to protect the skin^{18,19,20}. In addition, glycerin can maintain the stability of the preparation in the long term and bind the components contained in the dosage form²¹. Currently, no scientific data and research have been found that observes the effect of glycerin concentration on the stability of spray sunscreen by utilizing wungu leaf extract as an active ingredient. Physical stability is one of the critical criteria in the manufacture of a cosmetic preparation. This study aims to determine the physical stability of the spray sunscreen preparation of wungu (*Graptophyllum pictum* (L.) Griff) leaf extract with varying concentrations of glycerin as a humectant.

MATERIALS AND METHODS:

Materials:

The materials used in this research are Dry extract of wungu leaves (*Graptophyllum pictum* (L.) Griff) from Karanganyar, Central Java, Indonesia, methyl paraben, propyl paraben, glycerin, propylene glycol, BHT, carbopol 940, triethanolamine (TEA), ethyl vanillin, green dye, and aquadest. The tools used in this research are rotary evaporator (Heidolph), analytical balance (Shimadzu), oven (Memmert), refrigerator (Sharp), Ostwald viscometer, pH meter (ATC), magnetic stirrer, glassware (Pyrex, Iwaki, and Herma), spatula, porcelain cup, filter cloth, aluminum foil, and spray bottle.

Methods of Formulation of Spray Sunscreen Wungu Leaf Extract:

The formulation of spray sunscreen was made by dispersing 0.06g of carbopol 940 with 1/3 hot distilled water (temperature 70°C) for 30 minutes in container A. Then stirred until homogeneous and added TEA. Methylparaben, glycerin, propylene glycol, BHT, and propylparaben were mixed in distilled water at a temperature of 70°C in container B. Then, the mixture of materials in container B with the remaining distilled water was put in container A and stirred until homogeneous. Dissolve the extract with 70% ethanol, then mix it in the preparation mixture and homogenize it with a magnetic stirrer on a hotplate for 2 minutes at a speed of 1200rpm. The preparation is put in a spray bottle, and replicated each formula in 3 replications with variations in glycerin concentrations of 5%, 10%, and 15%.

Physical Stability Test of Spray Sunscreen Wungu Leaf Extract:

The physical stability test was carried out using the cycling test method by placing the preparation in a cold room (4±2°C) and a higher temperature room (40±2°C) for 48hours each, counted as one cycle. This test was carried out for six cycles and observed changes in organoleptic, homogeneity, pH, viscosity, and spray patterns.

Organoleptic Test:

Direct visual observation carried out organoleptic tests, including the dosage form's color, odor, clarity, and texture.

Homogeneity Test:

The homogeneity test was carried out by observing all completely dissolved materials.

pH test:

The pH test was carried out using a pH meter with the required range of 4.5 - 7.

Viscosity Test:

The viscosity test measured the density and flowed time of each preparation and distilled water using a pycnometer and Ostwald viscometer. Density is calculated using equation (i). The flow time is calculated using equation (ii).

$$P = \frac{m}{V} \dots\dots\dots (i)$$

ρ = density of the sample
 m = weight of the stock
 v = volume of the pycnometer

$$\eta = \eta_o \frac{t \cdot \rho}{t_o \cdot \rho_o} \dots\dots\dots (ii)$$

η = viscosity of the sample
 η_o = viscosity of water
 t = stock flow time
 t_o = water flow time
 ρ = density of the sample
 ρ_o = density of water

Spray Pattern Test:

The spray pattern test was carried out by spraying the preparation on mica plastic at a distance of 3, 5, 10, and 15 cm. The determination of the sample is repeated three times. Then, measured the diameter spray of the sample.

RESULT:

Organoleptic Test:

Based on the cycling test results for six cycles, there were no significant changes in the color, shape, odor, and clarity of each dosage formula.

Table 1: Organoleptic data of spray sunscreen [n=3]

Formulate	Cycle-0				Cycle-6			
	Color	Fragrant	Shape	Clarity	Color	Fragrant	Shape	Clarity
F1A	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F1B	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F1C	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F2A	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F2B	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F2C	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F3A	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F3B	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant
F3C	Green	Vanilla	Liquid	Transparant	Green	Vanilla	Liquid	Transparant

Homogeneity Test:

Based on the test for six cycles, the particles in formulas 1, 2, and 3 were dissolved entirely with no accumulation in each formula after six environmental cycles.

Table 2: Homogeneity data of spray sunscreen [n=3]

Formulate	Cycle-0	Cycle-6
F1A	Homogeneous	Homogeneous
F1B	Homogeneous	Homogeneous
F1C	Homogeneous	Homogeneous
F2A	Homogeneous	Homogeneous
F2B	Homogeneous	Homogeneous
F2C	Homogeneous	Homogeneous
F3A	Homogeneous	Homogeneous
F3B	Homogeneous	Homogeneous
F3C	Homogeneous	Homogeneous

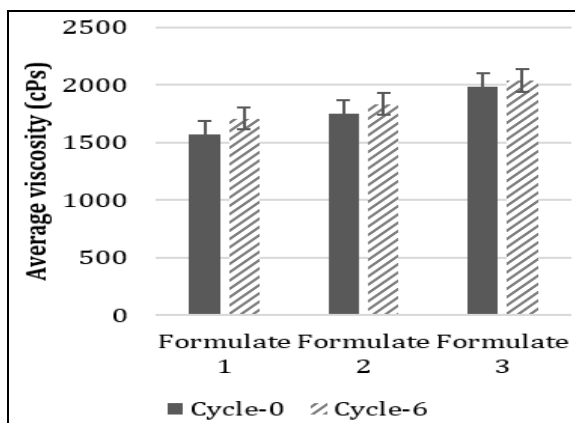


Figure 2. Viscosity of spray sunscreen [n=3]

pH test:

Observational data show no significant change in the pH value between the preparations in the 0th and 6th cycles. The pH of each preparation is within range 4,5 - 7.

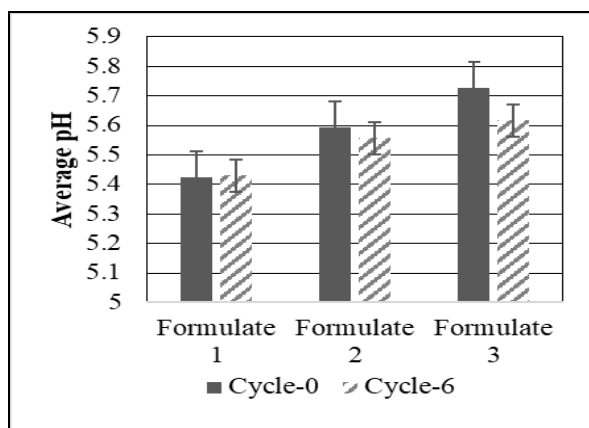


Figure 1. pH of spray sunscreen [n=3]

Viscosity Test:

All preparations have an increase in the viscosity value and there was no significant change in the viscosity of the preparation during the 0th and sixth cycles on statistical data.

Spray Pattern Test:

The results of this study show variations in diameter of spray pattern in each formula and no significant changes of the diameter on statistical data.

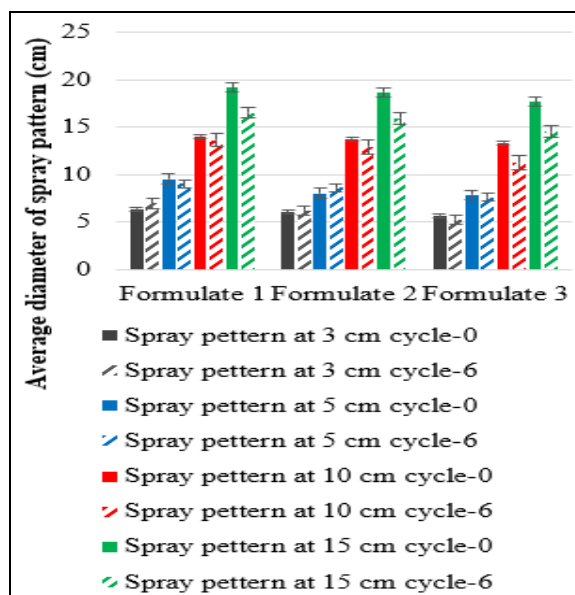


Figure 3. Spray pattern diameter of spray sunscreen [n=3]

DISCUSSION:

Organoleptic Test:

The organoleptic test compared the preparation's color, shape, odor, and clarity during the 0th and 6th cycles (Table 1) and there were no significant changes in each dosage formula. These results are from research conducted by Maharani et al. (2021), who showed no change in the preparation after stability testing was carried out for up to 21 days (4 cycles)²². Research conducted by Faizah and Sutningsih (2019) showed no color, odor, or shape change after six storage cycles²³. Spray sunscreen of wungu leaf extract (*Graptophyllum pictum* (L.) Griff) has stable organoleptic characteristics and meets the requirements, namely in liquid form, no phase separation, has a color according to the active ingredient, and does not have a pungent odor²⁴.

Homogeneity Test:

The homogeneity test was carried out by observing the presence or absence of materials not completely mixed in the preparation during the 0th and 6th cycles. The particles in formulas 1, 2, and 3 were dissolved entirely with no accumulation in each formula after six environmental cycles (Table 2). That follows research conducted by Iswandana and Sihombing (2017), which showed that after 12 weeks of storage, all preparations remained homogeneous, with no clumping, and the preparations remained physically stable²⁵. So it can be said that the spray sunscreen preparation of wungu leaf extract (*Graptophyllum pictum* (L.) Griff) has a stable homogeneity of the preparation after the test and meets the requirements, such as no insoluble particles^{26,27}.

pH test:

Based on observational data, the pH of each preparation in cycle 0 and cycle 6 had a pH between 5.31 - 5.85 with a significance value (Figure 1) for each formula 1, 2, and 3, respectively, 0.593; 0.053; and 0.145 ($P > 0.05$) which indicated that there was no significant change in the pH value between the preparations in the 0th and 6th cycles. These results suggest that all formulations of spray sunscreen preparations of wungu leaf extract meet the pH value of topical preparations, namely 4.5 - 7^{26,27}. The results of this study follow the results of research conducted by Ariyanti et al. (2022), where there was no change in the pH of the preparation after four storage cycles there was no significant change²⁸.

Viscosity Test:

The results of the viscosity test observations are shown in Figure 2. There is an increase in the viscosity value of all preparations with a significance value in formulas 1, 2, and 3 after the cycling test for six cycles of 0.43; 0.49; and 0.126 ($P > 0.05$), which indicated that there was no significant change in the viscosity of the preparation during the 0th and sixth cycles. Observational data

showed that the viscosity of the entire formula was 1546.84 - 2091.01 Cp. This value indicates that all formulations of spray sunscreen preparations meet the criteria for good viscosity, namely the viscosity value in the range of 1080 to 5000 Cp²⁹. These results follow the research conducted by Sumule et al. (2020), which stated that there was an increase in viscosity on the second day to the fourth week of storage, from 353.33 dPas to 383.33 dPas³⁰. This change is due to the difference in the concentration of glycerin in each formula. Glycerin acts as a humectant that can maintain the stability of the preparation in the long term and bind the components contained in the preparation²¹. Glycerin can increase the viscosity of preparation by binding to water, thereby increasing the molecular size, which also affects the increase in resistance to flow³⁰.

Spray Pattern Test:

The results of the pattern observation show the cycles in Figure 3, which indicate variations in each formula, both the 0th and 6th cycles. The difference is due to the influence of the spraying distance. The farther the spraying distance, the wider the resulting diameter³¹. These results have similarity to the results from Anindhita and Oktaviani's (2020) research, where the average spraying diameter at a distance of 3cm, 5cm, 10 cm, and 15cm is 2.57cm, 2.87cm, and 3.67cm, respectively, 4.57cm, and 6.13cm. The results of this study indicate that the significant value in formula 1 with spraying distances of 3cm, 5cm, 10cm, and 15cm is 0.180; 0.285; 0.276; and 0.109 ($P > 0.05$)³². The significance of formula 2 with spraying distances of 3 cm, 5cm, 10cm, and 15cm is 0.180; 0.180; 0.109; and 0.109 ($P > 0.05$). The significance of formula 3 with spraying distances of 3 cm, 5 cm, 10 cm, and 15 cm are 0.276; 0.655; 0.102; and 0.109 ($P > 0.05$). The significance values in formulas 1, 2, and 3 indicated no significant change in the spray diameter during the 0th and 6th cycles. Besides being influenced by the spraying distance, this difference is also influenced by an increase in viscosity. The increase in viscosity causes the pressure to spray the preparation to be greater, making it difficult for the preparation to be sprayed from the device³¹.

CONCLUSION:

The study results concluded that the spray sunscreen preparations with varying concentrations of glycerin (5%, 10%, and 15%) were physically stable.

CONFLICT OF INTEREST:

The authors have no conflicts of interest regarding this investigation.

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