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Research Article

Formulation of Spray Sunscreen with Variation Concentration of Wungu Leaf Extract

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Abstract

Sunscreen is applied to the skin to protect it from UV rays. One ingredient that can be made into sunscreen is wungu leaves (*Graptophyllum pictum* (L.) Griff). This research aims to determine the effect of varying concentrations of wungu leaf extract on the physical properties of spray sunscreen preparations. This research method is extraction, extract evaluation, formulation with varying extract concentrations (500 ppm, 1,000 ppm, 1,500 ppm), and physical evaluation of spray sunscreen. Data analysis uses linear regression equations. All formulas produce green, transparant, liquid, homogeneous with the distinctive smell of wungu leaf extract. The pH and viscosity test results meet the requirements for topical spray preparations with pH and viscosity values decreasing with slope values of -0.43 and -483.5 respectively. The spray pattern test results were oval to round in shape and spread with diameter values increasing with slope values at spray distances of 3, 5, 10 and 15 cm respectively by 0.55; 0.568; 0.6685; and 0.4865. Based on the research results, it can be concluded that variations in the concentration of wungu leaf extract affect the physical properties of spray sunscreen preparations in terms of pH, viscosity, and spray pattern diameter.

Keywords: Cosmetics, *Graptophyllum pictum,* Maceration, Regression correlation, Ultraviolet light

Formulasi *Spray Sunscreen* dengan Variasi Konsentrasi Ekstrak Daun Wungu

Abstrak

Sunscreen berfungsi untuk melindungi kulit dari paparan sinar UV. Salah satu bahan yang dapat dibuat menjadi sunscreen yaitu daun wungu (*Graptophyllum pictum* (L.) Griff). Penelitian ini bertujuan mengetahui pengaruh variasi konsentrasi ekstrak daun wungu terhadap sifat fisik sediaan *spray sunscreen*. Metode penelitian ini yaitu ekstraksi, evaluasi ekstrak, formulasi dengan variasi konsentrasi ekstrak (500 ppm, 1.000 ppm, 1.500 ppm), dan evaluasi fisik. Analisis data menggunakan persamaan regresi linear. Semua formula menghasilkan sediaan berwarna hijau, jernih, cair, homogen dan berbau khas ekstrak daun wungu. Hasil uji pH dan viskositas memenuhi syarat sediaan topikal spray dengan nilai pH dan viskositas menurun dengan nilai slope masing-masing -0,43 dan -483,5. Hasil uji pola penyemprotan berbentuk lonjong hingga bulat menyebar dengan nilai diameter meningkat dengan nilai slope pada jarak penyemprotan 3, 5, 10, dan 15 cm secara berurutan sebesar 0,55; 0,568; 0,6685; dan 0,4865. Berdasarkan hasil penelitian dapat disimpulkan bahwa variasi konsentrasi ekstrak daun wungu memengaruhi sifat fisik sediaan *spray sunscreen* pada pengujian pH, viskositas, dan diameter pola penyemprotan.

Kata Kunci: Kosmetik, *Graptophyllum pictum,* Maserasi, Korelasi regresi, Sinar ultraviolet

1. Introduction

The skin is one of the organs that comprise the integumentary system of the human body, and one of its functions is to protect the body from the sun.[1,2] The sun emits energy, which includes ultraviolet (UV) radiation such as UV A, UV B, and UV C. The formation of reactive oxygen species (ROS) is partially responsible for UV-induced skin damage.[3]

Sunscreen is a medicinal preparation whose purpose is to protect the skin from UV radiation. Until recently, many sunscreens were formulated with synthetic active chemicals that can have a harmful effect on the skin. Therefore, alternatives are required to replace synthetic active components with substances that possess the same efficacy with fewer negative effects.[4] Natural components can be used as safe materials.

Wungu leaves (*Graptophyllum pictum* L.) are one of the many beneficial natural components. study revealed wungu leaves has antioxidant, anti-diabetic, anti-inflammatory, anti-fungal, anti-bacterial, and anti-viral properties.[5] Wungu leaves contain flavonoids and activity as antioxidants Antioxidants are substances that inhibit oxidation reactions by scavenging free radicals.[6] The effectiveness of wungu leaves is categorized as moderate to high depending on the fraction and concentration used.[7] According to Ibrahim et al. (2022), the ethanol extract of wungu leaves is suggested for use in sunscreen formulations due to its ability to withstand the 21-day storage test, that's pysical stability test with the cycling test at different temperature of ±4°C and ±40°C.[5] Poh Yen et al. (2018) observed that 200 ppm of ethanol extract of wungu leaves is highly effective in protecting against UV radiation of 13.423.[8]

Good physical properties of the preparation will affect its pharmacological effect. Spray preparations that have a pH that matches the pH of the skin so that they do not cause irritation, have good homogeneity, and a wide spray pattern will provide good pharmacological effects because the active substances can provide optimal activity. From previous studies (Martono et al., 2018; Ulandari & Sugihartini, 2020; Wulandari et al., 2022) variations in extract concentrations can affect the physical properties of the preparations in the form of organoleptic, pH, viscosity, spreadability, adhesion, and spraying patterns.[9,10,11]

The application of topical preparations on the skin surface which aims to protect the skin from sun exposure has been widely used by the public. The dosage form chosen in this study was a spray preparation. The spray dosage form was chosen because it has a liquid consistency so that it can be applied quickly and evenly to the skin surface, is easy to wash and absorbs to the skin, and is safe because of the low level of contamination.[12] In light of the explanation provided above, this study aims to determine the effect of the physical qualities of sunscreen spray formulation due to variation concentrations of wungu leaf extract (*Graptophyllum pictum* (L.) Griff).

2. Method

2.1. Tool

The research's instruments are rotary evaporator (Heidolph), oven (Memmert), moisture content analyzer, hotplate (Heidolph), analytical balance (Shimadzu), pH meter (ATC), ostwald viscometer, picnometer (Iwaki), glassware (Iwaki, Pyrex, and Herma), spatula, porcelain cup, aluminum foil (Klin pak), and spray bottle.

2.2. Materials

Wungu leaf powder simplicia from Karangpandan District, Karanganyar Regency, Central Java Province, Indonesia; ethanol 70% (Bratachem, Indonesia); methyl paraben (Bratachem, Indonesia); propyl paraben (Bratachem, Indonesia); glycerin (Bratachem, Indonesia); propylene glycol (Bratachem, Indonesia); butyl hydroxy toluene (BHT) (Bratachem, Indonesia); carbopol 940 (Bratachem, Indonesia); triethanolamine (TEA) (Bratachem, Indonesia); green color; and distilled water.

2.3. Methods

2.3.1. Preparation of Extract

The maceration process, which involves

soaking simplicia in a solvent appropriate for its active components for five days, was used to create the extract. an amount of 1,000 grams of the simplicia wungu leaf powder was soaked in a 1:5 mixture of 70% ethanol for five days and was stirred for 3 times a day. The extract was concentrated with a rotary evaporator at temperature 50°C for 1.5 hours at 275 mbar vacuum, resulting in a thick extract, resulting in a thick extract. In order to produce a dry extract, the viscous extract was then heated to 40 ± 2°C in an oven.

2.3.2. Extract Evaluation

a. Organoleptic

The scent, color, and texture of the extract were all visually observed.

b. Yield

The yield is calculated based on the ratio of the final weight (weight of extract produced) to the initial weight (weight of simplicia) multiplied by 100%.

$$\% \text{ Yield} = \frac{\text{Extract weight}}{\text{Simplicia weight}} \text{ x } 100\%$$

c. Water content

Using moisture content analyzer (MCA) to determine the water content, place 1 gram of extract on the instrument, and run the instrument at 40°C. The water content results will be automatically read.

2.3.3. Formulation

Wungu leaf extract was used to prepare three different recipes for spray sunscreen, each having three replications and three different extract concentrations. Dissolve the Wungu leaf extract in distilled water until dissolved. Carbopol 940 is mixed in a portion of distilled water and stirred until homogeneous, then added TEA, stirred until homogeneous (mixture A). Aquades, methyl paraben, propylene glycol, glycerin, and propyl paraben are heated in a container at ±70°C while stirring (mixture B). Mixture A is mixed with mixture B and stirred using a magnetic stirrer at 1,200 rpm for 2 minutes. Wungu leaf extract and green color were added to the mixture and then stirred until homogeneous. The homogeneous preparations were put in a spray container and then tested for the physical characteristics.

2.3.4. Physical Properties of Preparations Evaluation

a. Organoleptic Test

The shape, color, odor, and transparency of the finished preparations were evaluated visually.[4,13]

b. Homogeneity Test

The homogeneity test was carried out to evaluate the mixing of the ingredients in the preparation by spraying it on a flat glass surface and covering it with another layer of glass. The preparation is stated to be homogeneous, namely the absence of large particles on the glass layer.[4,14]

c. pH test

Table 1. Formula sunscreen spray

Ingredients	Formula		
	F1	F2	F3
Wungu leaf extract	500 ppm	1000 ppm	1500 ppm
Methyl paraben	0,027 g	0,027 g	0,027 g
Propyl paraben	0,013 g	0,013 g	0,013 g
Glycerin	15 mL	15 mL	15 mL
Propylene glycol	10 mL	10 mL	10 mL
BHT	0,01 g	0,01 g	0,01 g
Carbopol 940	0,06 g	0,06 g	0,06 g
TEA	0,05 mL	0,05 mL	0,05 mL
Green color	q.s	q.s	q.s
Aquadest ad	100 mL	100 mL	100 mL

Using a digital pH meter, pH levels were measured. The pH meter is calibrated using an acidic solution (pH of 4.01), a neutral solution (pH of 6.86), and an alkaline solution (pH of 9.18), then submerged in the sample,, and the result are automatically displayed on the pH meter.

d. Viscosity Test

The viscosity test was carried out with the help of a pycnometer to measure the density of the preparation being tested and an Ostwald viscometer to determine the flow time of the test preparation.15,16,17 Viscosity is calculated using the equation:

$$\eta = \eta o \frac{t \,.\, \rho}{to \,.\, \rho o}$$

Information:
η = viscosity to be determined
ηo = viscosity of water
t = flow time to be specified
to = water flow time
ρ = density to be specified
ρo = density of water

e. Spray Pattern Test

Spraying the solution onto mica plastic at distances of 3, 5, 10, and 15 cm served as the test's target surface.13,18 Spray three times (triplicate) at each distance, notice the pattern that develops, and calculate the diameter of the pattern.[18]

2.3.5. Data analysis

Testing the relationship between the causal factor variables (x) and the outcome variable (y) during the analysis of the study's data using linear regression equations in the Software Microsoft Excel.

3. Results

The obtained extract was in the form of a dry extract with a distinctive green hue and odor. The extraction of 1,000 grams of wungu leaf simplicia resulted in 480.97 grams of dry extract with a yield of 40.89 %, and the water content was 4.53%.

Organoleptic and homogeneity evaluation results can be shown in Table 2, there were no variations in the outcomes of the organoleptic and homogeneity test of the wungu leaf extract (*Graptophyllum pictum* (L.) Griff) spray sunscreen formulation using different extract concentrations of 500 ppm, 1,000 ppm, and 1,500 ppm.

As the concentration of wungu leaf extract in a spray sunscreen increased (500 ppm, 1,000 ppm, 1,500 ppm), the pH value decreased, as shown in Figure 1.

The viscosity of spray sunscreen with varied extract concentrations (500 ppm, 1,000 ppm, and 1,500 ppm) decreased as the extract concentration increased, as can be seen in Figure 2.

The results of the spraying pattern for each formula and distance are distributed in an oval to round pattern. Tabel 3 and Figure 3 Depicts the diameter of the spraying pattern as a consequence of the measurements.

4. Discussion

The maceration extraction method was

Table 2. Organoleptic and homogeneity of sunscreen spray

Formula	Organoleptic				Homogeneity
	Shape	Color	Odor	Clarity	
F1A	Liquid	Green	Extract scent	Transparent	Homogeneous
F1B	Liquid	Green	Extract scent	Transparent	Homogeneous
F1C	Liquid	Green	Extract scent	Transparent	Homogeneous
F2A	Liquid	Green	Extract scent	Transparent	Homogeneous
F2B	Liquid	Green	Extract scent	Transparent	Homogeneous
F2C	Liquid	Green	Extract scent	Transparent	Homogeneous
F3A	Liquid	Green	Extract scent	Transparent	Homogeneous
F3B	Liquid	Green	Extract scent	Transparent	Homogeneous
F3C	Liquid	Green	Extract scent	Transparent	Homogeneous

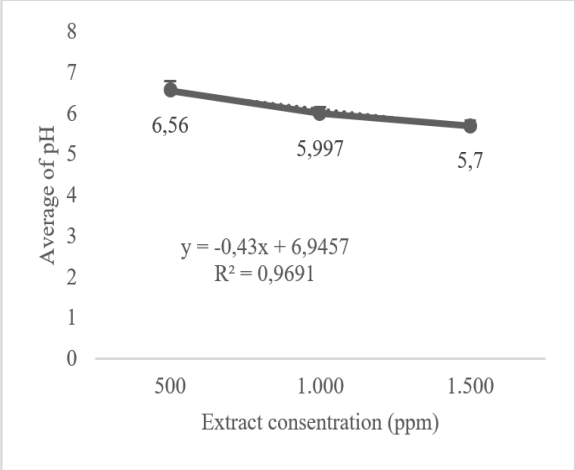


Figure 1. Correlation regression graph of extract concentration with pH

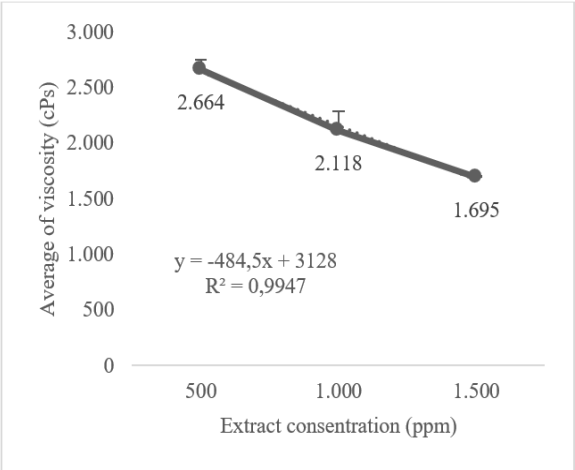


Figure 2. Correlation regression graph of extract concentration with viscosity

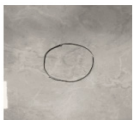
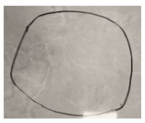
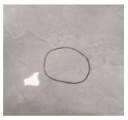
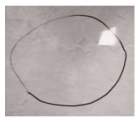
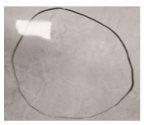
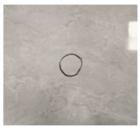
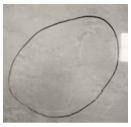
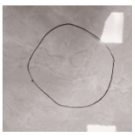
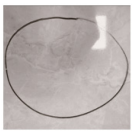
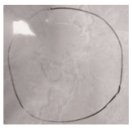
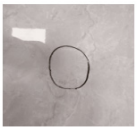
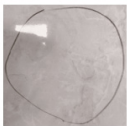
used to get wungu leaf extract (*Graptophyllum pictum* (L.) Griff). The advantage of maceration extraction is the instrument used is simple, namely only requiring a soaking vessel, without heating so as to avoid the risk of damage to the thermolabile compounds in the simplicia.[19] The results of the water content of the Wungu leaf extract also meet the maximum water content standard of 10% in Wungu leaf extract, and the yield achieved meets the standards for wungu leaf extract yield, which is at least 9.3%.[20] The yield results are comparable to those of Wijaya and Utami (2018), who used 100.03 grams of powdered simplicia wungu leaves and 600 milliliters of 96% ethanol solvent in maceration to obtain a yield of 30.09 %.[21] In the findings of a study conducted by Wulandari et al. (2022) using 1,500 grams of wungu leaf powder and 70% ethanol (1:10), the yield was 11.16 percent.[11] The discrepancy in extract yield probably due to the size of the simplicia, with smaller simplicia yielding more extracts, as well as by variations in the amount of solvent employed during extraction.[12]

Spray sunscreen including wungu leaf extract in each of formulas 1, 2, and 3 resulted in a transparent, green, liquid formulation with a distinct odor. In agreement with the findings of Puspita et al. (2020) on organoleptic observations of spray gel preparations with variations of extract 1, 2, and 3%, all formulations exhibited the same color, smell, and consistency.[22]

Homogeneity test was carried out by observing the preparation that is sprayed on a layer of glass. The purpose of this test was to know the mixing of each ingredient in the manufacture of a perfectly mixed sunscreen spray.[4,9,23] The uniform distribution of the active ingredient inside the preparation can also be ensured by the preparation's homogeneity.[9] Spray sunscreen wungu leaf extract (*Graptophyllum pictum* (L.) Griff) with varied concentrations provided in formulas 1, 2, and 3 had a homogenous mass This is due to the fact that creating sunscreen spray requires the preparation of the proper ingredients as well as frequent stirring. The research by Puspita et al. (2020) on the observation of the homogeneity of spray gel preparations with variations of extracts 1, 2, and 3% indicated all homogeneous formulations were characterized by the uniform distribution of all particles.[22]

The pH test was conducted with a digital pH meter that had been calibrated using an acidic solution (pH of 4.01), a neutral solution (pH of 6.86), and an alkaline solution (pH of 9.18). The purpose of the pH test is to determine the acidity or alkalinity of a substance. The results of each preparation's pH indicate a level that can be tolerated by the skin when applied topically. If the pH of a topical medication falls between 4.5-7.0, it is considered to meet the standards, that's because if the pH of the preparation is too acidic it will cause skin irritation whereas if the pH of the preparation is too alkaline it can cause scaly skin.[4,13,23,24] As the concentration

Table 3. Spraying pattern of sunscreen spray

Formula	Spray pattern at 3 cm	Spray pattern at 5 cm	Spray pattern at 10 cm	Spray pattern at 15 cm
F1A				
F1B				
F1C				
F2A				
F2B				
F2C				
F3A				
F3B				
F3C				

of wungu leaf extract in a spray sunscreen increased (500 ppm, 1,000 ppm, 1,500 ppm), the pH value decreased. According to the research of Puspita et al. (2020), the drop in pH or the more acidic the preparation is due to the flavonoid chemicals included in the extract, which are weak acids, causing the pH of the preparation to fall with increasing extract concentrations.[25] Wungu leaves also contain acids that can affect pH preparations, including formic acid, palmitic acid, myristic acid.[26,27]

Based on the graph of the correlation regression show a decreasing line so that it can be explained that the higher the concentration of the extract used, the lower the pH. Based on the data obtained, the regression equation is y = -0.43x + 6.9457 and the correlation coefficient (r) is 0.9844 which means the relationship between the two variables x (extract concentration) and y (pH value) falls into the category very strong. This is because a correlation exists between concentration and resultant absorbance if the correlation

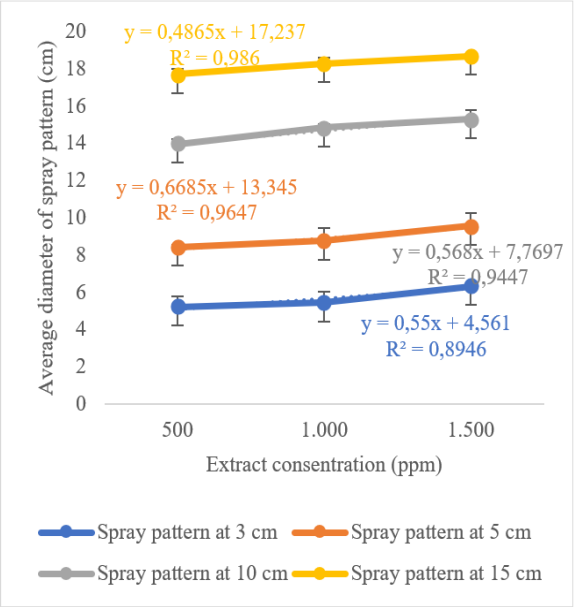


Figure 3. Correlation regression graph of extract concentration with the diameter of the spray pattern

coefficient is close to 1.[28] While the value of the coefficient of determination (R^2) is 0.9691 which means that the contribution of the influence of Wungu leaf extract to the test results of the pH value has a percentage of 96.91% while the remaining 3.9% is influenced by other factors. As evidenced by the value of b = -0.43, the relationship between the two variables is negative. This indicates that the pH decreases by 0.43 parts per addition of a 1 part concentration of wungu leaf extract (*Graptophyllum pictum* (L.) Griff) According to the research of Wulandari et al. (2022), a base without extract has a pH of 7.48, whereas the addition of wungu leaf extract at concentrations of 12.5% and 25% decreases the pH to 7.15 and 7.04, respectively, indicating that the pH value decreases with increasing concentrations of wungu leaf extract.[11]

Viscosity test is designed to determine the viscosity of a preparation, which can influence whether or not the preparation is easily applied to the skin's surface.[23] The viscosity values of each formula 1, 2, and 3 met the spray gel viscosity requirements of 500-5,000 cPs, as determined by this research.[18] The viscosity of spray sunscreen with varied extract concentrations (500 ppm, 1,000 ppm, and 1,500 ppm) decreased as the extract concentration increased. According to Latif et al. (2020), the decrease in viscosity may be attributable to flavonoid components in the ethanol extract of wungu leaf.[29]

According to the graph of the correlation regression show a decreasing line so that it can be explained that the higher the concentration of the extract used, the lower the viscosity results. Based on the data obtained, the regression equation is y = -484.5x + 3128 and the correlation coefficient (r) is 0.9973 which means the relationship between the two variables x (extract concentration) and y (viscosity value) is included in the very strong category because it is close to 1. While the value of the coefficient of determination (R^2) is 0.9947 which means that the contribution of the influence of Wungu leaf extract to the test results of the viscosity value has a percentage of 99.47% while the remaining 0.53% is influenced by other factors. As evidenced by the value of b = -483.5, the relationship between the two variables is negative. This indicates that the viscosity was decreased by 483.5 parts for each addition of 1 part concentration of wungu leaf extract (*Graptophyllum pictum* (L.) Griff). The outcomes are in line with the research of Wulandari et al. (2022), which found that the viscosity of the preparation greatly decreased with the addition of 12.5% and 25% wungu leaf extract.[11]

The spray pattern test was conducted on mica plastic media with spray distances of 3,

5, 10, and 15 centimeters. To achieve more precise findings, each distance was sprayed three times (triplicate).[18,30] The results of the spraying pattern for each formula and distance are distributed in an oval to round pattern. Depicts the diameter of the spraying pattern as a consequence of the measurements. The viscosity and spraying distance of the preparation affect the difference in the diameter of the spray pattern. The greater the viscosity of the preparation, the smaller the spray diameter, whereas the spray distance is directly proportional to the spray diameter, i.e. the greater the spraying distance, the larger the spray diameter of the preparation.[4]

The graph of the correlation regression show an increasing line so that it can be explained that the higher the concentration of the extract used, the larger the diameter of the resulting spray pattern. Based on the data obtained, the results of the correlation coefficient (r) at all spraying distances are close to 1, which means that the relationship between the two variables x (extract concentration) and y (spray pattern diameter value) falls into the very strong category. The positive relationship between the two variables is revealed by the values of b at spraying distances of 3, 5, 10, and 15 cm, b= 0.55, b = 0.568, b = 0.6685, and b = 0.4865, respectively. This implies that at each spraying distance of 3, 5, 10, and 15 cm, the diameter of the spray pattern decreases by 0.55, 0.568, 0.6685, and 0.4865 parts, respectively, for each addition of 1 part of the concentration of wungu leaf extract (*Graptophyllum pictum* (L.) Griff). According to the research conducted by Martono & Surhayani (2018), raising the concentration of ethanol extract causes the spray pattern's diameter to expand.[9]

5. Conclusion

The experiment reveal that the three formulas met the requirements for the physical properties of sunscreen spray preparations. The different concentration of wungu leaf extract in spray formulation affect the physical characteristic of sunscreen, as measured by pH, viscosity, and spray pattern diameter.

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