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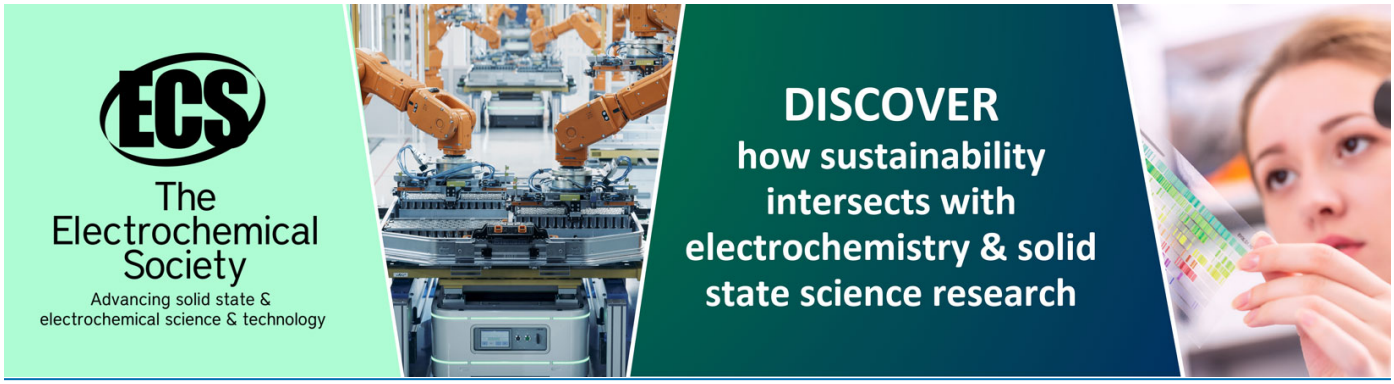

Physical Stability Test Sunscreen Gel Extracts Blackberry Fruit (*rubus fruticosus* l.)

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Abstract. Blackberry (*Rubus* sp.) fruit contains high levels of anthocyanins and other phenolic compounds, mainly flavonols and ellagitannins, which contribute to its high antioxidant capacity and other biological activities. Sunscreen preparations are cosmetic preparations that are used as protection to reduce the impact of sun exposure, whose formulations contain active ingredients to absorb or reduce sunlight, especially in areas exposed to ultraviolet rays and infrared waves. One of the potential natural ingredients for sunscreen is blackberry fruit (*Rubus* sp.). Contains flavonoid compounds that can prevent the harmful effects of UV rays. This study aims to determine the stability of the blackberry fruit extract sunscreen gel formula for 90 days of storage to determine the SPF value test results of blackberry fruit extract using UV-Visible. The research design is an experimental research conducted in a laboratory. The sample used in this study was Blackberry which was made into thick extract by maceration method. Furthermore, the thick extract obtained was made into a sunscreen gel and then tested for physical stability and the calculation of the SPF value. The sunscreen potency test is determined based on the method of calculating the percent erythema transmission value (%Te) and the percent transmission pigmentation (%Tp) as well as the Sun Protection Factor (SPF) value. On testing the SPF the average value on the test day 0, day 7, day 15, day 21, day 30, day 60, day 90, respectively 28,03±0,77; 28,28±0,16; 28,46±0,41; 28,66±0,59; 28,85±0,33; 29,04±0,11; 29.24±0,27. Percent of erythema (% Te) testing over 90 days was included in the total block category. Percent pigmentation (% Tp) testing for 90 days was included in the total block category and the SPF test for 90 days was included in the category ultra.

1. Introduction

Berries are well known for their properties that can improve the body's systems, antioxidants, vasodilators and antimicrobials [1]. Blackberry (*Rubus fruticosus* L.) is widely distributed in northern countries and has pharmacological activities, namely as an anticancer, antimicrobial, antioxidant, anti-dysentery, antidiabetic and anti-diarrheal [2].

Antioxidants are substances that can significantly inhibit or prevent oxidation caused by free radicals at small concentrations [3]. As for substances that can reduce the harmful effects of sunshine are sunscreens, sunscreens can ward off at least 85% of sunlight at a wavelength of 290-320 nm for UVB but can transmit light at a wavelength of more than 320 nm for UVA [4] Sunscreen is a cosmetic which physically or chemically can inhibit the penetration of UV rays into the skin [4]. Apart from chemical sunscreens that are also found in natural ingredients, sunscreens made from natural ingredients are widely used by people because they are considered safer. The ability to withstand UV

rays from sunscreens was assessed by the Sun Protection Factor (SPF) [6]. Sunscreen in gel preparations that are made must meet stability requirements including physical stability tests, physical stability tests are carried out to ensure the quality, safety and benefits of the gel, meet the expected specifications and are stable during storage [7]. Research on sunscreen gel formulation has been carried out in research on the Formulation and Activity Test of Sunscreen Gel from Blackberry Fruit Extract (*Rubus fruticosus* L.) In Vitro with UV-Visible Spectrophotometry [8]. Therefore, the researchers are interested in conducting research on physical stability tests on the sunscreen gel formulation of Blackberry fruit extract (*Rubus fruticosus* L.) with accelerated stability for 90 days of storage.

2. Methods

2.1. Tools and Materials

The tools used in this research are Spatel, Watch Glass, Spreadability Test Glass, Stirring Rod, Beaker Glass, Oven, Erlenmeyer, Pipette, Water bath, Porcelain Cup, Measuring cup, Climatic chamber, UV-Visible Spectrophotometer (Thermo Scientific Evolution 201), Viscometer (Lamy Rheology First Touch 15.04T), pH meter (NeoMet pH-240L GJ-7726), Homogenizer (WiseTis HG-15D), Analytical balance (Adam PW254). The research materials used were Blackberry extract (*Rubus fruticosus* L.), Karbopol Blackberry extract (*Rubus fruticosus* L.), Karbopol 940, TEA, Nipagin, Nipasol, Glycerin, H_2SO_4, HCl, $FeCl_3$, Fragrance, Aquades, methanol (1.060009.2500).

2.2. Location and Time of Research

This research is an experimental research, which was conducted at the Laboratory of Preparation Technology, Faculty of Pharmacy, University of Buana Perjuangan Karawang in January 21 - May 21, 2020.

2.3. Experimental procedure

Experimental procedures include determination, preparation of raw materials, formulation of preparations, test of physical stability of preparations and test of SPF values.

2.4. Determination

Determination was carried out to ensure the correctness of the plants used in this study, this determination was carried out at SITH Labtek XI, ITB Bandung.

2.5. Phytochemical Screening

Phytochemical screening includes alkaloid test, flavonoid test, saponin test, tannin test and triterpenoid and steroid test.

2.6. Material Preparation

The part of the Blackberry (Rubus fruticosus L.) used was ripe fruit with a blackish purple color and made a thick extract using the maceration method.

2.7. Dosage Formulations

The formula of the preparation uses the active substance of Blackberry fruit ethanol extract, Karbopol 940 (gelling agent), glycerin (humectant), Triethanolamine (neutralizing agent), Nipagin (preservative), Nipasol (preservative), and aquadest (solvent).

2.8. Physical Stability

Physical stability tests carried out included organoleptic, preparation pH, viscosity, dispersion, and SPF value test.

2.9. Organoleptic
Organoleptic observations were carried out by observing changes in shape, color, and odor of the Blackberry fruit extract (*Rubus fruticosus* L.) sunscreen gel. The gel is usually clear with a semi-solid concentration [9].

2.10. pH
Observations were made on the gel preparation by dipping the pH meter into the gel preparation that had been made and then recorded. The pH of the skin is 4.5-6.5 [10].

2.11. Viscosity
The viscosity test is carried out to determine the thickness of the preparation that affects the time of use on the skin. A good viscosity of gel preparations is 2000 - 4000 Cps. [11].

2.12. Dispersibility
A total of 0.5 grams of the gel sample was placed on a round glass with a diameter of 15 cm, the other glass was placed on top of the gel and left for 1 minute. The scatter diameter is measured. After that, 150 grams of additional load were added and allowed to stand for 1 minute and then measured the diameter of the preparation. Good gel dispersion is 5-7 cm [11].

2.13. SPF Value Test
The SPF value is calculated by first calculating the area under the absorption curve (AUC) from the absorption value at a wavelength of 29-400 nm with 5nm intervals. The AUC value is calculated using the following formula (Chaims, 2004).
AUC = (Aa+Ab)/5x dPb-a
Information:
Aa = Absorbance at wavelength a nm
Ab = Absorbance at wavelength b nm
dPb-a = the difference between wavelengths a and b
The total AUC value is calculated by adding up the AUC value for each wavelength segment. The SPF value of each concentration is determined using the following formula: (Chaims, 2004).
Log SPF = AUC/(λn-λ1)

Information:
λn = The largest wavelength
λ1 = the smallest wavelength

3. Result And Discussion
The formulation of the Blackberry extract gel used a formula with a concentration of 1% Blackberry fruit extract.

Physical Stability Test
The physical stability test of the gel preparations was carried out by storing at 40 ° C with a relative humidity of 75% for 3 months using an oven. Stability testing was carried out for 90 days or 3 months, evaluated at 0, 7, 15, 21, 30, 60 and 90 days.

Organoleptic Test

Table 1. The result organoleptic test

Organolepic	Day test-						
	0	7	15	21	30	60	90
Shape	Semi	semi	semi	semi	semi	semi	semi
Color	Solid Red Brownish	solid Red brownish	solid Red brownish	solid Red brownish	solid Red brownish	solid Red brownish	solid Red brownish
Smell	typical weak berry	typical weak berry	typical weak berry	typical weak berry	typical weak berry	typical weak berry	typical weak berry

Table 1 states that the organoleptic test for the gel preparation did not show a significant change at each test and it can be said that the gel preparation is stable.

Ph

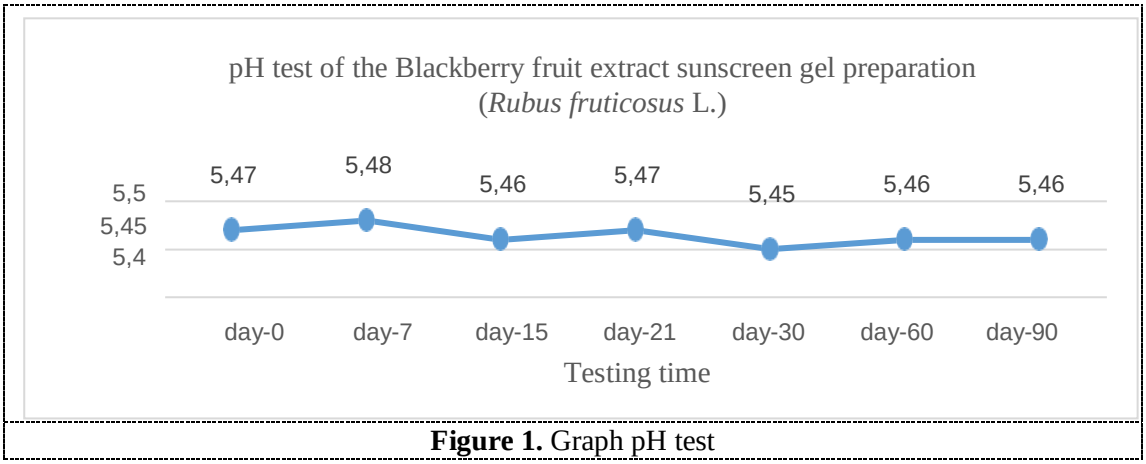

Figure 1. Graph pH test

In Figure 1, it states that the pH test for the gel preparation did not show a significant change in each test and it can be said that for the pH test using a pH meter for the sunscreen gel preparation of Blackberry extract (*Rubus fruticosus* L.) was stable for 90 days of storage.

Viscosity Test

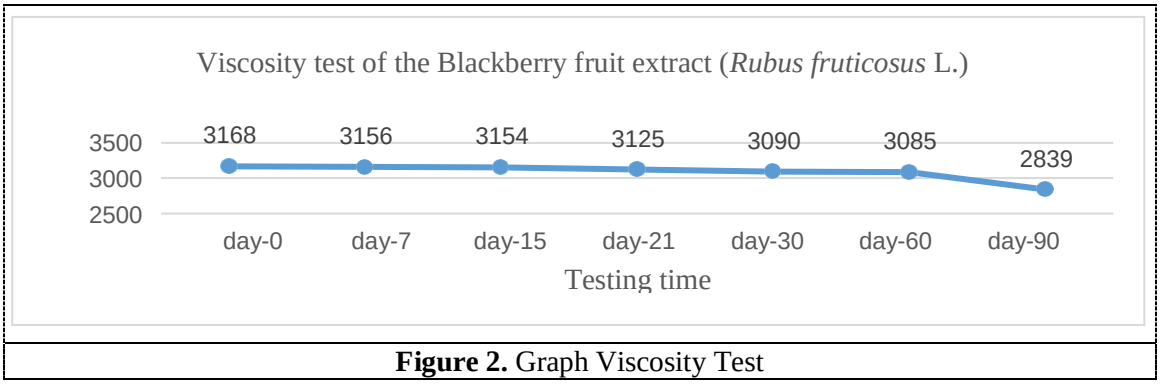

Figure 2. Graph Viscosity Test

In Figure 2 it states that based on the results of the viscosity measurement on the 0-90 day test, it has decreased. This can be due to the gel preparation showing its characteristics, namely synersis, which is the process of releasing the entangled liquid in the gel so that it allows the liquid to move towards the surface, therefore the preparation has decreased viscosity but is still in a good characteristic range (2000-4000cPs). The decrease in gel thickness can also be caused by external factors such as temperature and storage method.

Dispersibility Test

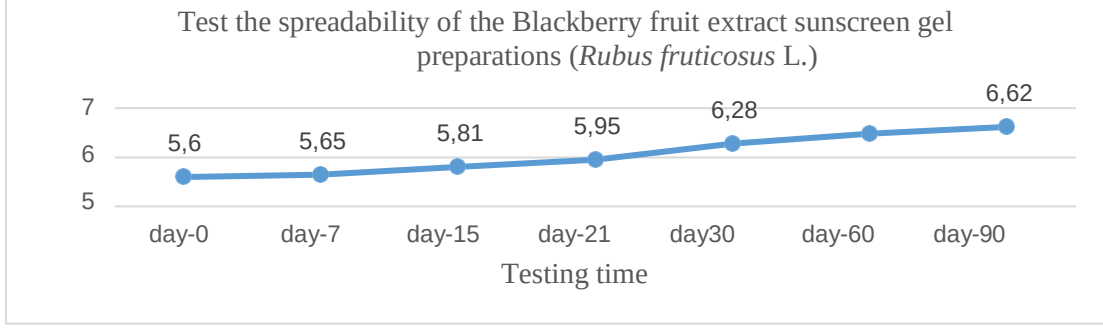

Figure 3. Graph Dispersibility Test

Figure 3 states that the results of the measurement of the dispersion power of the Blackberry extract (*Rubus fruticosus* L.) sunscreen gel, indicate that the gel has an increase in the diameter of the dispersion power.

SPF VALUE TEST

Percentage of Erythema Transmission

The Sun Protection Factor (SPF) value test was carried out in vitro using a UV-Visible Spectophotometer and the absorbance value was measured. The 10 g sample was dissolved in methanol up to 100 ml, then put in a 1 ml pipette into the cuvette and then the percent erythema transmission and pigmentation transmission percent (% Tp) were measured.

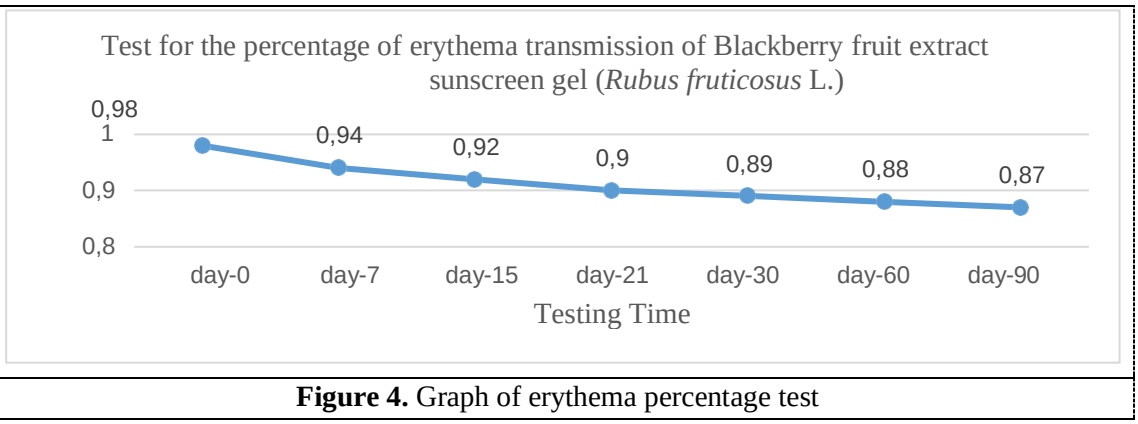

Figure 4. Graph of erythema percentage test

In Figure 4, it is stated that the test results for the percent erythema transmission (% Te) show that during the 90-day test, the percentage of erythema decreased, which means that the total flux transmitted by the sunscreen is getting less. This is because the gel experiences synergy. The value of% erythema as a whole did not change significantly and was still in the range % erythema <1% and included in the total block category.

Percentage of Pigmentation Transmission

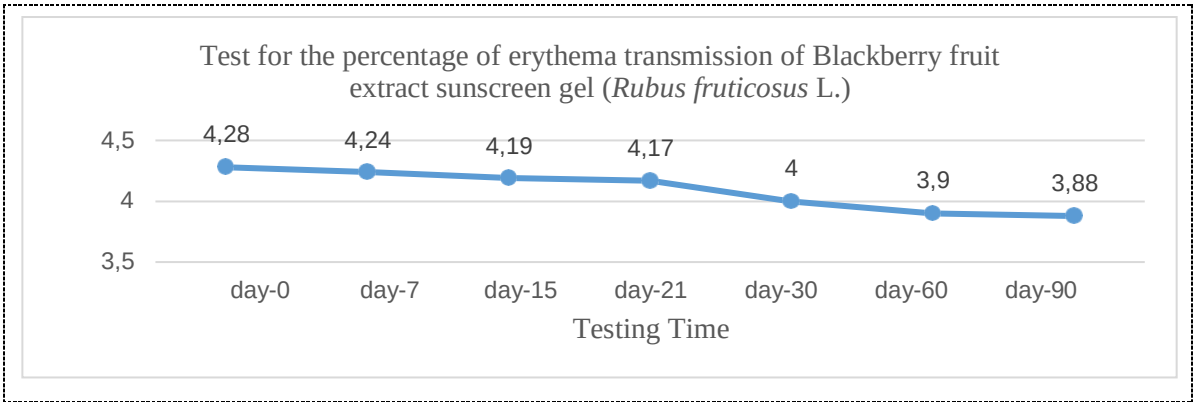

Figure 5. Graph of pigmentation percentage test

Figure 5 states that the results of the percent pigmentation test (% Tp) show that during the 90-day test, the pigmentation percent decreased, which means that the total flux continued by the sunscreen was getting smaller. This is because the gel experiences synergy. The overall pigmentation% value did not change significantly and was still in the range % 3-40% pigmentation and included in the total block category.

SPF Value Test

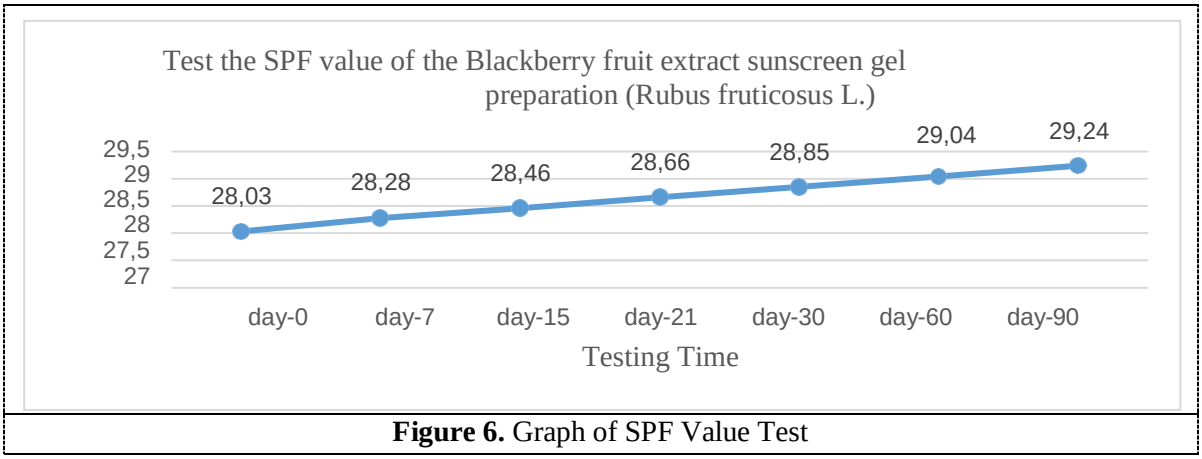

Figure 6. Graph of SPF Value Test

Figure 6 states that the results of measuring the SPF value carried out on the sunscreen gel preparation of Blackberry extract (Rubus fruticosus L.) stored for 90 days showed an increase in the SPF value. This is due to the fact that the gel can experience synergy, namely the occurrence of water discharge from the preparation so that it tends to remove water from the preparation (Kuncari et al, 2014). This resulted in decreased viscosity and increased levels measured (Martin et al, 1993). The SPF value resulting from testing for 90 days did not show a significant change so that the gel was still in the ultra and stable category during storage.

Data Analysis

Data analysis using Kruskal wallis analysis states that if the Asymp.Sig value <0.05, there is a difference from the tested viscosity so that the viscosity test decreases due to syneresis.

4. Conclusion

After doing research, physical stability test for 90 days of storage in an oven at a temperature of 40 ℃ and humidity <75% sunscreen gel extract of Blackberry (*Rubus fruticosus* L.) was stable during storage.

Suggestion

It is necessary to carry out chemical stability tests and long term stability tests at storage of 2 years 30 ± 2 ℃ with a humidity of 75 ± 5%.

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