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Stability of Ethanol Extract Sunscreen Spray Gel Formula Kalakai Leaves (*Stenochlaena palustris* (Burm F.) Bedd)

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ABSTRACT

There is evidence that an ethanol extract from the leaves of the kalakai tree (*Stenochlaena palustris* (Burm F.) Bedd.) may provide ultra-protective sun protection. The purpose of this study is to analyze the stability of a sunscreen spray gel composition that contains an ethanol extract of kalakai leaves. We used a rotary evaporator to concentrate the kalakai leaf ethanol extract until it was thick, after macerating the leaves in 70% ethanol solvent. A spray gel formulation containing EEKL was subsequently created, with EEKL concentrations of 5% (FI), 7.5% (FII), and 10% (FIII). The physical stability of the EEKL Spray gel formula was examined by six cycles of the cycling test technique in a climatic chamber with temperatures of 40°C ± 2°C and 4°C ± 2°C. Organoleptics, consistency, thickness, pH, adhesive spreading ability, and spray pattern are some of the tests that are conducted. Using a UV-Vis spectrophotometer set at 290-320 nm wavelength, the sunscreen test was conducted. A t-dependent test was used to examine the quantitative data on physical stability and sunscreen. No significant variations were seen throughout six storage cycles ($p>0.05$), indicating high physical stability, for the three EEKL Spray gel compositions (FI:5%, FII:7.5%, FIII: 10%). Formula III, which falls under the ultra protection category and had the highest average SPF value before and after the cycling test at 35.82 ± 0.50, exhibited no significant change ($P>0.05$) in the sunscreen activity during the six storage cycles, according to the results of the stability test of the EEKL Spray gel sunscreen activity in the three formulas.
Keywords: Spray gel; ethanol extract of *Stenochlaena palustris* (Burm F.) Bedd leaves; Physical stability; Sunscreen

INTRODUCTION

In addition to alkaloids and steroids, the secondary metabolites found in kalakai leaves (*Stenochlaena palustris* (Burm F.) Bedd) include flavonoids, which have anti-inflammatory, hydrolysis and oxidative enzyme inhibitory, and free radical scavenging and antioxidant properties (S Anggraeni & Erwin, 2015). The IC50 value of 143.1431 ppm indicates that the ethanol extract of kalakai leaves may inhibit free radicals (Rantia, 2018). The total flavonoid concentration of the ethanol extract of Kalakai leaves is 2.2159 ± 0.083%, according to study by (Syamsul, Hakim, & Nurhasnawati, 2019). In addition, studies conducted by (Puspita, Puspasari, Masykuroh, & Fitria, 2023) indicate that the ethanol extract of kalakai leaves acts as a sunscreen, boasting an SPF rating of 21.22, which falls within the category of ultra-protective sunscreen performance. Protecting skin from the sun's potentially damaging rays is the primary function of sunscreen. On the other hand, the active ingredient will stay in contact with the skin for a longer period of time if the sunscreen is applied topically rather than taken orally (Draelos & Thaman, 2006). The Spray gel formulation is one of these options; it has several benefits, such as being less contaminated with microorganisms, having a longer drug contact time than other preparations, being easier to use, and being able to be formulated into beautiful cosmetic products (Akbar, Hanik, Shabrina, & Zulfa, 2020)

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Product stability is an essential factor that must be considered in the quality of cosmetic preparations. Stability guarantees that pharmaceutical preparations' quality, purity, and strength are within the specifications applied until the time of use and storage (Kuncari, Iskandarsyah, & Praptiwi, 2014).

Temperature cycling test is a method developed to evaluate the stability of cosmetic preparations with variations in storage temperature within certain time intervals (CTFA, 2004). This method simulates temperature changes during product storage to test product stability (Suryani, Putri, & Agustyiani, 2017). A Spray gel preparation that is stable if the period of use and storage is within acceptable limits. This research aims to obtain the stable product formula of Spray gel sunscreen from ethanol extract of kalakai leaves (*Stenochlaena palustris* (Burm F.) Bedd).

Table I. Formula of Kalakai Leaf Ethanol Extract Spray gel

Bahan	Concentration (%)		
	FI	FII	FIII
Kalakai leaf ethanol extract	5	7.5	10
Propylene glycol	10	10	10
Carbopol 940	0.5	0.5	0.5
Triethanolamine	0.5	0.5	0.5
Methyl paraben	0.18	0.18	0.18
Propyl paraben	0.02	0.02	0.02
Aquadest ad	100 g	100 g	100 g

MATERIALS AND METHODS

Tools and materials

A variety of instruments were utilized in the research, including a mortar and stamper, an analytical balance, a stir bar, a hot plate (made by Maspion), aluminum foil, Pyrex beaker glass, Sharp refrigerator, a rotary evaporator (made by Heildolph), a UV-Vis spectrophotometer (made by Shimadzu), Pyrex measuring flask, Pyrex measuring cup, micropipette, dropper pipette, a stopwatch, and a spray bottle. Ingredients used in this study included kalakai leaves, distilled water, methyl paraben, propyl paraben, carbopol 940, triethanolamine, propylene glycol, and 96% ethanol, all of which are believed to be of pharmaceutical grade.

Method

Kalakai Leaf Ethanol Extract Spray gel Preparation

To begin, dissolve Carbopol 940 in hot water until it is fully dissolved. Then, add Triethanolamine (TEA) to the mixture to create a translucent gel mass. Mix propylene glycol with methyl and propyl parabens until they dissolve. Then, mixed until homogeneous in a large mortar, the ethanol extract of kalakai leaves and the remaining distilled water were added. Then, the preparation is stirred until homogeneous, and the preparation is put into a spray bottle.

Evaluation of Physical Stability of Kalakai Leaf Ethanol Extract Spray gel

The cycle test technique was used to conduct the stability test. Six cycles were used to complete the cycling test. The mixture is allowed to cool to about 4°C for one day, after which it is taken out and let to cool to around 40°C for another day. One cycle is used to compute this procedure. Over the course of six cycles, researchers monitored organoleptic parameters, homogeneity, pH, viscosity, adhesive spreadability, and spraying pattern after each treatment.

Testing of sunscreen activity of ethanol extract kalakai leaves Spray gel formula

Finding the SPF value in vitro using UV-Vis spectrophotometry is the standard procedure for assessing the activity of sunscreens. To make the spray gel, weigh out 0.1 gram of kalakai leaf ethanol extract FI (5%), FII (7.5%), and FIII (10%). In a 5 mL measuring flask, combine 5 mL of 96% ethanol and stir until the mixture is uniform. First, 96% ethanol was used to calibrate the UV-Vis spectrophotometer. Using a 96% ethanol blank, an absorption curve was measured in a cuvette at a wavelength ranging from 290 to 320 nm. According to Damogalad et al. (2013), the SPF value was determined by recording the absorbance values of each concentration. A six-cycle cycling test was used to assess the Spray gel formulation both before and after storage.

RESULTS
DISCUSSIONS

Since ethanol is polar, it is believed that all kinds of flavonoids may be extracted from kalakai leaves. A 70% ethanol solvent is used for this purpose. According to the study conducted by (Syamsul et al., 2019), a total flavonoid content of 2.2159 ± 0.083% was obtained by extracting kala kai leaves with 70% ethanol. According to (Leba, 2017), extraction is a method of chemical separation that involves obtaining the desired chemical content by use of an acceptable solvent. To make the chemical composition more usable and extend its storage life, extraction aims to remove as much of it as feasible. In this study, the maceration technique is used for extraction. Maceration was selected due to its ability to effectively extract chemicals and to halt the breakdown of heat-sensitive compounds (Murbantan, Mustafa, Rosjidi, & Saputra, 2010).

There are three different concentrations of kalakai leaf ethanol extract that were used to prepare the spray gel: 5% (FI), 7.5% (FII), and 10% (FIII). A combination of Carbopol 940 (a gel

(a) (b)

Figure 1. Visualization of Spray gel (a) before temperature cycling test and (b) after temperature cycling test of several formulas. FI formula Spray gel EEKL 5%, FII formulaSpray gel EEKL 7.5% and FIII formula Spray gel EEKL 10%

Table II. Organoleptic Examination Results of EEKL Spray gel

	Organoleptic	**FI (5%)**	**FII (7,5%)**	**FIII (10%)**
Before cycling test	Color	Dark Brown	Dark Brown	Dark Brown
	Typical kalakai smell	Yes	Yes	Yes
	Tekstur	Thick	Thick	Thick
After cycling test	**Organoleptik**	**FI**	**FII**	**FIII**
	Color	Dark Brown	Dark Brown	Dark Brown
	Typical kalakai smell	Yes	Yes	Yes
	Tekxture	Thick	Thick	Thick

Table III. Homogeneity Test

Temperature cycling test	**Homogeneity**		
	FI (5%)	**FII (7,5%)**	**FIII (10%)**
Before	Homogeneous	Homogeneous	Homogeneous
After	Homogeneous	Homogeneous	Homogeneous

Table IV. Results of pH measurements

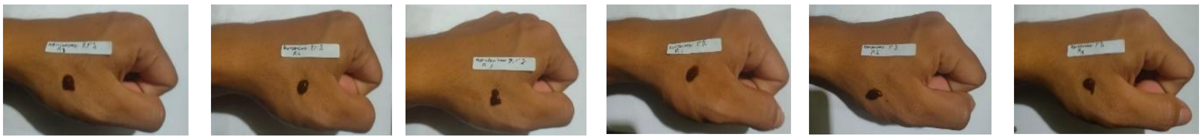
Temperature cycling test	**pH value**		
	FI (5%)	**FII (7,5%)**	**FIII (10%)**
Before	5.54 ± 0.08	5.61 ± 0.01	5.54 ± 0.05
After	5.56 ± 0.12	5.70 ± 0.02	5.57 ± 0.11

Table V. Viscosity Measurement Results

Measurement	**Viscosity (dPas)**		
	FI (5%)	**FII (7,5%)**	**FIII (10%)**
Before temperature cycling test	17.33 ± 0.57	14.33 ± 0.57	09.00 ± 0.01
After temperature cycling test	16.85 ± 0.27	14.20 ± 0.57	08.55 ± 0.57

Table VI. Results of EEKL Spray Spray gel pattern inspection

Measurement	**Average weight/spray (grams)**		
	FI (5%)	**FII (7,5%)**	**FIII (10%)**
Before temperature cycling test	0.098 ± 0.57	0.112 ± 0.67	0.142 ± 0.60
After temperature cycling test	0.121 ± 0.27	0.135 ± 0.57	0.174 ± 0.27



FI FII FIII FI FII FIII
(a) (b)

Figure 2. Visualization of the adhesive spread of Spray gel (a) before the temperature cycling test and (b) after the temperature cycling test for several formulas. FI formula Spray gel EEKL 5%, FII formula Spray gel EEKL 7.5% and FIII formula Spray gel EEKL 10%

Table VII. SPF Value of Kalakai Leaf Ethanol Extract Spray gel

Measurement	SPF Value		
	FI (5%)	**FII (7,5%)**	**FIII (10%)**
Before temperature cycling test	22.01 ± 0.20 (Ultra Protection)	23.34 ± 0.25 (Ultra Protection)	35.82 ± 0.50 ((Ultra Protection))
After temperature cycling test	21.30 ± 0.27 (Ultra Protection)	22.75 ± 0.57 (Ultra Protection)	34.52 ± 0.27 (Ultra Protection)

forming), triethanolamine (a base), propylene glycol (a humectant), methyl and propyl parabens (a preservative and a solvent, respectively) create the Spray gel formulation. Following this, the sunscreen spray gel made from an ethanol extract of kalakai leaves (*Stenochlaena palustris* (Burm F.) Bedd.) was tested for its physical and chemical stability, which included organoleptic, homogeneity, pH, adhesive spreadability, viscosity, spray pattern, and sunscreen activity.

The development of spray gel preparations can increase effectiveness and make use easier (Salwa, Kadir, & Sulistyowati, 2020) Research states that Spray gel preparations have advantages including being practical in use and stable in storage (Akbar et al., 2020). In addition, based on research by (Akbar et al., 2020) it is stated that the spray gel formulation of soybean seed ethanol extract (*Glycine max*) has activity as a sunscreen cosmetic preparation.

The physical stability test of Kalakai leaves ethanol extract Spray gel included observing organoleptic parameters, homogeneity, pH, viscosity, sticky spreadability, and spraying pattern. Observing the form/texture, color, and aroma is what the organoleptic test is all about. As part of the organoleptic test, the spray gel is examined visually, olfactorily, and texturally (Ansel, 1989). Organoleptic evaluations in Figure 1 and Table II provide identical visual, olfactory, and textural characteristics. The active ingredient, ethanol extract from kalakai leaves, gives the spray gel its dark brown hue and pungent aroma. Spray gel preparations like this one result in a thick texture. The goal of the organoleptic test is to find a spray gel formulation that meets the intended aesthetic standards in terms of color, scent, and texture. After all, this is a spray gel formulation, so it needs to be visually appealing and easy to apply. Preparations made using any of these three spray gel compositions maintain their organoleptic properties regardless of the temperature cycling test circumstances.

The requirement for a homogeneous preparation is that it must not contain coarse materials that can be touched. Homogeneity examination aims to see the particle distribution of the preparation (Armadany Fery Indradewi, Hasnawati, 2021). Based on Table 3, examining the homogeneity of Spray gel preparations using glass preparations from the three formulas shows that each preparation is homogeneous and has particles evenly distributed before and after the temperature cycling test. The results are shown like that because there are no coarse grains in all preparation formulas, and they show a homogeneous composition or no lumps in the preparation.

Despite six cycles of temperature cycling, the pH of the spray gel preparation did not vary significantly ($p>0.05$) throughout the test. Based on Table IV, the findings of the temperature cycling test show that the pH value of the spray gel remains steady before and after the test, which means that it fits the skin pH criteria, which are 4.5-6.5 (Tranggono & Latifah, 2007). It is believed that scaly skin or irritation might result if the preparation's pH is beyond the skin's pH interval. But if it's more acidic than the skin's pH, it can make the skin feel slick, dry out rapidly, and reduce its suppleness (Dureja, Kaushik, Gupta, Kumar, & Lather, 2005).

One way to find out how thick a liquid is is to do a viscosity test. In order to facilitate spray application, the resultant spray gel formulation has a low viscosity value (Salwa et al., 2020).

The viscosity of the spray gel did not change before or after the temperature cycling test ($p>0.05$), according to the results in Table V, which were obtained by measuring the three different spray gel preparation formulas using a Rion VT-06 viscometer with viscometer stirrer number 2. The resulting gel is in the stable preparation category. The three Spray gel formulas' viscosity still meets the 500-5000 cPs range or 5-50 dPas. If the viscosity is less than 500 cP, it will cause dripping too quickly when sprayed from the applicator. If the viscosity is more than 500 cP, it will cause the particle size of the sprayed preparation to become irregular and large so that it is less spread over the surface of the skin or mucous membrane (Shafira, Amila, & Lestari, 2015).

One way to judge a spray applicator is by looking at the pattern it produces. A number of factors, including the properties of the formula, impact the spraying patterns. Both the spraying distance and the preparation's viscosity affect the variation in spraying patterns. The ratio of the preparation's spraying pattern diameter to the spraying distance is directly proportional. The spray pattern that is produced is proportional to the distance. An extended and spreading pattern is produced by the spray by all three formulations. The temperature cycling test revealed that the delivery weight of the preparation for each spray of three different spray gel formulae increased (see Table VI). This happens because the spray gel's viscosity drops during the temperature cycling test, making the prepared gel more fluid and simpler to spray. The spray gel preparation's stability was observed before and after the temperature cycling test, as shown by the delivery weight of each spray ($p>0.05$), so from the stability test process the three spray gel formulas had a stable delivery weight for each spray. This shows that the applicator used in the spray gel preparation is effective in delivering reproducible amounts with each spray (Suyudi, 2014).

The adhesive spreadability test results of the three formulas in Figure 2, both before and after the temperature cycling test, show that the ethanol extract of kalakai leaves Spray gel preparation can stick when sprayed on the skin on the upper arm for 10 seconds. It can form a layer that sticks to the skin and does not flow. Based on the research results, the ethanol extract of kalakai leaves Spray gel preparation meets the requirements for Spray gel preparations, produces good physical properties, and can be applied to the skin.

The SPF values of the Spray gel range from 21.30 ± 0.27 (Ultra Protection) to 35.82 ± 0.50 (Ultra Protection), according to the findings of the SPF value tests in Table VII. There is a direct correlation between the concentration of extract in the Spray gel formula and the SPF value, which rises with increasing concentration (both before and after the cycling test circumstances). According to the study (Damogalad, Edy, & Supriati, 2013), the SPF value is directly proportional to the concentration of the extract. Although the Spray gel SPF value decreased following the cycling test, there was no statistically significant difference between the two sets of data ($p>0.05$). The ultra-protection category had the highest average SPF value, 35.82 ± 0.50, both before and after the cycling test, specifically in Formula III.

Sunscreen activity, as measured by the SPF value, is affected by concentration in this research. The UV absorption of sunscreens may be affected by this issue. various concentrations of extracts absorb various types of UV radiation, as shown by the fact that absorbance rises with increasing concentrations. The ethanol extract of kalakai leaves (*Stenochlaena palustris* (Burm F.) Bedd) has sunscreen properties because kalakai leaves contain a relatively high amount of flavonoids, namely 2.2159 ± 0.083% (Syamsul et al., 2019). Due to their chromophore group, which can absorb both UV A and UV B rays, flavonoids show promise as a sunscreen (Shovyana & Zulkarnain A, 2013).

CONCLUSION

The findings of the physical stability test are supported by the study that was conducted. Since there were no discernible changes over time, the three kalakai leaf spray gel formulations (FI:5%, FII:7.5%, and FIII:10%) demonstrate excellent physical stability. The final product, after six storage cycles ($P>0.05$), has the desired properties: a thick, uniform texture, a dark brown aroma, and it satisfies the specifications for pH, viscosity, spray pattern, and adhesive spreadability.

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