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Sunscreen activity of ethanol extract of mangrove fruit flour and its formulation in cream preparation

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Abstract

Increased exposure to ultraviolet (UV) radiation has raised public awareness of the importance of using effective and safe sunscreens, especially those derived from natural ingredients. Mangrove ecosystems are a source of natural bioactive compounds that have the potential to act as photoprotective agents because they contain flavonoids, tannins, and phenolics that can absorb ultraviolet (UV) rays and act as antioxidants. This study aims to evaluate the potential sunscreen activity of Brayo (*Avecenia marina*) and Lindur (*Bruguiera gymnorrhiza*) extracts in raw and cooked forms, and to develop a cream formulation with the best extract. Extraction was carried out by remaceration using 70% ethanol solvent. The Sun Protection Factor (SPF) value of each extract was determined using UV spectrophotometry. The extract with the highest SPF results was then formulated into a cream preparation with concentrations F1 (1%), F2 (3%), and F3 (9%). Each formula was tested for physical characteristics including pH, viscosity, adhesion, and spreadability, and tested for sunscreen activity in vitro. The results showed that raw Brayo had the highest SPF value (24.47 ± 0.58) and was included in the ultra protection category. Organoleptic and homogeneity tests showed that all formulas met the requirements. For pH and adhesion tests, formulas 3 and 4 met the requirements, while formula 1 did not. Viscosity and spreadability tests for all formulas met the requirements. the SPF value increased with increasing extract concentration. Thus, raw Brayo extract has the potential as a natural active ingredient in sunscreen cream formulations with high effectiveness. These findings indicate that raw Brayo extract has the potential as a natural sunscreen agent that can be formulated stably in cream form for skin protection applications against UV radiation.

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Keywords

Sunscreen, Mangrove, Cream, Extract, Brayo

Introduction

The skin is the largest organ in the human body, functioning as a protective barrier that separates the body's internal environment from the external environment, which is crucial for maintaining overall health [1]. Continuous exposure to ultraviolet (UV) rays can cause chronic skin diseases such as hyperpigmentation, skin aging, changes in the skin's immunological response, and, most seriously, skin cancer [2].

Sunscreen is a preparation that can protect the skin against UV radiation. The effectiveness of sunscreen preparations is based on the determination of the Sun Protection Factor (SPF), which indicates the ability of the sunscreen preparation to protect the skin from UV exposure [3]. Mangrove plant species such as Brayo fruit (*Avicennia marina*) and Lindur (*Bruguiera gymnorrhiza*) have the potential to be used as sunscreen preparations.

Mangroves are one of the most important ecosystems on earth because they have many benefits, such as storing carbon which helps reduce global warming, protecting the coast from waves and erosion, maintaining biodiversity by being a habitat for various animals, and supporting the production of fish and other marine products which are important for human life [4].

In previous research, it was stated that the extract of mangrove brayo fruit (Avicennia marina) from the infundation process has a high content of secondary metabolites such as tannins, saponins, and flavonoids so that it can be used as a natural antioxidant with an IC50 value of 38.53 ppm. Phenolic compounds, especially the flavonoid group, have the potential as a sunscreen because there is a chromophore group (conjugated single double bond) which has the ability to absorb ultraviolet rays, both UV A and UV B so that it can reduce the intensity of radiation that reaches the skin surface [5]. In addition, previous research showed that the methanol extract of lindur fruit has an IC50 value of 9.42 ppm [6].

The degree of processing of mangrove fruit, whether raw or cooked (boiled), can affect the natural active substances such as phenolics, flavonoids, and tannins. Raw mangrove fruit generally contains phenolic compounds and tannins bound in the form of polymer complexes. These compounds tend to have high antioxidant activity but low solubility. Meanwhile, when cooked mangrove fruit goes through a process, the chemical structure and bioavailability of these compounds can change through degradation or increased solubility. These compounds play an important role as natural antioxidants and also help protect the skin from UV exposure. This is the reason for conducting research related to the activity of raw and cooked mangrove fruit flour extracts [7].

Mangrove fruit has the potential to be developed into a cosmetic preparation that can protect the skin from UV radiation because it has an IC 50 value below 100 ppm, thus indicating high antioxidant activity. The ideal sunscreen cosmetic preparation can be formulated in the form of an oil-in-water (O/W) type cream because it is easy to use, has a simple formulation, and its ability as a skin protector that provides a comfortable feeling and can be spread evenly [8].

The advantages of cream preparations are that they are easy to spread evenly, easy to use, practical, easy to clean or wash, and not sticky when used. However, there are also disadvantages of cream preparations, such as they are easily damaged if stored in an inappropriate place, and their high-water content makes creams more susceptible to microbial contamination so they require preservatives [9].

Based on this background, a study will be conducted to determine the effectiveness of ethanol extract of mangrove fruit flour as an active ingredient in a cream preparation that is expected to have a high SPF value so that it can protect the skin from UV exposure. Although research on brayo and lindur mangrove fruit related to antioxidant activity has been extensive, its dosage form is still limited, so the author is interested in developing mangrove fruit extract in the form of a sunscreen cream preparation.

Method

Samples of brayo and lindur mangrove fruit flour (raw and cooked) were obtained from CV KeMANGI.

Extraction

The extract preparation in this study used the remaceration method with 70% ethanol solvent. 200 grams of the simplicia powder was weighed and then placed into a macerator jar, adding 600 mL of 70% ethanol solvent. The soaked extract was stirred occasionally, filtered every 24 hours for 3 days with the addition of the same solvent and amount as the initial method. The solvent of the extract obtained was evaporated using a rotary evaporator at a temperature of 50°C and then re-evaporated with a water bath to obtain a thick extract, then the yield was calculated.

Ethanol free test

Ethanol-free testing is carried out by adding 3 drops of concentrated acetic acid (CH_3COOH) and sulfuric acid (H_2SO_4) to the mangrove fruit flour extract, then heating it. The test result is negative if no ester aroma is detected [10].

Phytochemical screening

Phytochemical screening was performed using color reactions and precipitation. Screening was performed for phenolic compounds, flavonoids, tannins, alkaloids, saponins, and terpenoids.

Sunscreen activity test

The Sun Protection Factor (SPF) value of brayo and lindur extract was determined using a Shimadzu UV–Vis spectrophotometer. The extract and its fraction were each dissolved in ethanol at a concentration of 100 mg/mL and their absorbance was measured at a UV-B wavelength (290–320 nm) at 5 nm intervals. Ethanol PA was used as a blank solution. The SPF value was calculated using the equation:

$$SPF = CF\ x \sum_{290}^{320} EE(\lambda)\ x\ I\ (\lambda) x\ Abs\ (\lambda)$$

Where CF represents the correction factor, EE (λ) denotes the erythemal effectiveness of radiation at a specific wavelength λ, I(λ) is the solar intensity spectrum (considered constant), and Abs (λ) corresponds to the absorbance value of the sample at wavelength λ.

Sunscreen cream formula with ethanol extract of mangrove fruit flour

The cream formulation is designed for the extract with the highest sunscreen activity. The following is the cream formulation (Table 1).

Table 1. Mangrove fruit flour extract cream preparation formulation with the highest activity

Material	Formula (b/b) %		
	F1	F2	F3
Ethanol extract of mangrove fruit with the highest activity (gr)	1	3	9
Oil Phase			
Stearic acid	5.5	5.5	5.5
Cetyl alcohol	3	3	3
Water Phase			
Propylene glycol	7	7	7
TEA	1	1	1
Methyl paraben	0.2	0.2	0.2
Propyl paraben	0.018	0.018	0.018
Aquadest	Ad 100	Ad 100	Ad 100

Information: Formula 1: Cream preparation with an active extract concentration of 1%; Formula 2: Cream preparation with an active extract concentration of 3%; Formula 3: Cream preparation with an active extract concentration of 9%.

Cream preparations

Each ingredient is weighed, for the oil phase using a porcelain cup (A) and the water phase using a porcelain cup (B). Then the water and oil phase cups are melted on a water bath at a temperature of 70°C, mix the two phases and then stir until homogeneous. After homogenous, mix the active substance of the ethanol extract of raw or cooked mangrove brayo fruit and stir again until everything is evenly mixed [11].

Physical characteristics test of sunscreen cream preparations

Some tests that will be carried out for cream preparations are:

1. *Organoleptic test*

Organoleptic testing is conducted using the five senses by assessing the shape, color, and odor produced by the preparation. Organoleptic will affect the comfort of use, therefore the resulting preparation is expected to provide a soft texture, attractive color, and pleasant odor [12].

2. *Homogeneity test*

A homogeneity test is performed to determine whether the ingredients used in a preparation are evenly mixed. A homogeneous preparation will help facilitate the active ingredients to be distributed throughout the base, thus optimally protecting the skin from sunlight [12]. A homogeneity test is performed by applying the preparation to a glass slide and then covering it with another glass slide. The requirement for homogeneity is that there are no coarse grains observed on the glass slide [13].

3. *pH test*

A pH test is performed to determine whether the preparation being made is acidic or basic. If the preparation is too acidic, it can cause skin irritation. However, if the preparation is too basic, it will make the skin dry. Therefore, it is necessary to match the preparation to the skin's pH. The pH requirement for facial skin is 4.5-8.0 [14].

4. Viscosity test

The viscosity test was conducted to determine the thickness of the cream preparation made, it is hoped that the preparation will be easy to apply to facial skin. The viscosity test of the preparation was conducted by measuring the cream made using a Brookfield Viscometer using spindle no. 7. The spindle was dipped into the cream that had been made. The viscosity requirements for cream preparations range from 2000-50,000 cPs [15].

5. Spread power test

The spreadability test is carried out to determine the ability of the preparation made to spread widely on the skin without requiring pressure when applied, the easier it is to apply, the greater the surface area of contact of the drug from the cream with the skin and the more optimal [12]. The spread ability test is carried out by weighing 0.5 grams of cream and then placing it in the middle of a glass object, covered with a glass object that has been weighed. It is left for one minute and then the diameter of the cream spread is measured. After that, a load of 50 grams is added every minute, then the diameter of the spread is measured to see the effect of the load on changes in the diameter of the cream spread [15].

6. Adhesion test

The adhesion test is carried out to determine how long the cream preparation can adhere to the skin, a good cream has high adhesion. The adhesion test is carried out by weighing 0.25 g of cream then spreading it evenly on one of the glass objects and then covering it with another glass object. Then it is pressed down with a 50 gram load for 5 minutes. The time is calculated from the application of the load and stopped when the glass object is released. The requirement for the adhesion of the cream preparation is not less than 4 seconds [15].

Data analysis

The data obtained included pH, viscosity, spread ability, adhesion, and SPF tests, and were processed using Excel. Quantitative research was analyzed using SPSS 26 with a 95% confidence level. If the results were homogeneous and normal, they were then analyzed statistically using the parametric one-way ANOVA test followed by a post-ANOVA test.

Results and Discussion

Results

This study used four samples raw brayo, boiled brayo, raw lindur, and boiled lindur flour. These four extracts were extracted with 70% ethanol using the remaceration method. The yields of each extract are shown in Table 2.

Table 2. Extract yield with 70% ethanol solvent

Mangrove flour	Rendemen (%)
Raw Brayo	60.37±0.23
Boiled Brayo	35.42±0.32
Raw Lindur	20.52±0.25
Boiled Lindur	55.73±0.22

The results showed significant variations in extract yield between brayo and lindur in both raw and cooked conditions. The highest yield was obtained in raw brayo at 60.37±0.23%, followed by boiled lindur at 55.73±0.22%, while the lowest yield was found in raw lindur at 20.52±0.25%. Meanwhile, boiled brayo produced a yield of 35.42±0.32%, lower than that of raw brayo. The resulting extracts were then subjected to phytochemical screening tests (Table 3). This test was conducted to determine the compound groups in each extract.

Table 3. Phytochemical screening of ethanol extracts

Compounds	Raw Brayo	Boiled Brayo	Raw Lindur	Boiled Lindur
Phenolic	+	+	+	+
Flavanoids	+	+	+	+
Tannin	+	+	+	+
Alkaloids	+	+	+	+
Saponins	+	+	+	+
Terpenoids	+	+	+	+

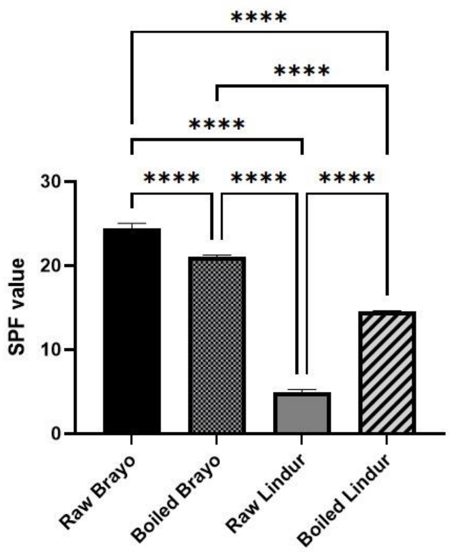

Figure 1. SPF value from raw brayo, boiled brayo, law lindur, and boiled lindur using four sample, n=5, ns = not significant, **** = < 0.0001

Sunscreen testing was performed by calculating the sun protection factor (SPF). The results of the sunscreen activity test for each extract were obtained using the Mansur method (Figure 1). The extract with the highest activity (raw brayo) was then made into a cream preparation. The cream was made using three formulas (Figure 2). The obtained cream preparation was then subjected to characteristic tests. The results of the characteristic tests are shown in Table 4.

Figure 2. Cream from Extract Raw Brayo

Table 4. Physical characteristics test cream from extract raw brayo

Physical Characteristics Test	Formula 1	Formula 2	Formula 3
Organoleptic			
Color	Brownish white	Light brown	Dark brown
Smell	No smell	No smell	No smell
Form	Cream	Cream	Cream
Texture	Gentle	Gentle	Gentle
Homogeneous	Homogeneous	Homogeneous	Homogeneous
pH	6.84±0.14	6.10±0.34	5.52±0.14
Viscosity (cP)	2673±66.58	3527±77.67	4256±61.10
Sticking power (second)	5.25±0.32	4.17±0.17	4.52±0.20
Spreadability (cm)	5.58±0.06	5.56±0.28	5.50±0.19
SPF	1.84	3.05	6.56

Table 4 shows that sunscreen cream preparations containing mangrove fruit extract at concentrations of 1%, 3%, and 9% have a cream form with a soft, odorless, and homogeneous texture in all formulas. The color of the preparation increases from brownish white in formula 1 to dark brown in formula 3 as the extract concentration increases. The pH value of the cream preparations ranges from 5.52 ± 0.14 to 6.84 ± 0.14. Formula 1 has the highest pH value, while formula 3 shows the lowest pH value.

The viscosity value increased with increasing extract concentration, namely from 2673 ± 66.58 cP in Formula 1 to 4256 ± 61.10 cP in Formula 3. The results of the adhesion test showed that all formulas had an adhesion time of more than 4 seconds, with the highest value obtained in Formula 1 (5.25 ± 0.32 seconds) and the lowest in Formula 2 (4.17 ± 0.17 seconds). The spreadability value was relatively uniform in all formulas, ranging from 5.50 ± 0.19 cm to 5.58 ± 0.06 cm.

The Sun Protection Factor (SPF) value of the cream preparation increased with increasing extract concentration. Formula 1 showed an SPF value of 1.84, Formula 2 3.05, and Formula 3 6.56. Statistical analysis using a one-way ANOVA test showed that variations in extract concentration had a significant effect on the SPF value of the cream preparation ($p < 0.05$).

Discussion

The high yield of raw brayo indicates that the material is still rich in compounds that are readily soluble in solvents, such as simple carbohydrates, phenolic compounds, flavonoids, and other secondary metabolites. In the raw state, the cell structure is relatively intact and the water content is higher, facilitating solvent penetration and the diffusion of target compounds into the solvent during the extraction process [16,17].

The decrease in yield in boiled brayo is thought to be due to the aging process, which triggers the degradation or transformation of certain bioactive compounds due to enzymatic activity and respiration. Furthermore, cooking can reduce easily extractable polar components and increase volatile compounds lost during processing, thus reducing extract yield [18,19].

Conversely, in lindur, the ripening process significantly increased the yield, from 20.52±0.25% in the raw state to 55.73±0.22% in the cooked state. This increase is thought to be related to changes in cell wall structure during cooling, such as tissue softening, cellulose and hemicellulose degradation, and the hydrolysis of complex polysaccharides into simpler, more soluble compounds. These changes increase tissue permeability and facilitate the release of soluble compounds into the solvent, thereby increasing extract yield [20, 21].

The differences in yield patterns between brayo and lindur indicate that the characteristics of the raw materials, their chemical composition, and their response to the ripening process significantly influence extraction efficiency. Factors such as water content, the degree of tissue lignification, and the stability of secondary metabolites also determine the amount of extract produced. Therefore, selecting the ripeness level of the material is a crucial factor in optimizing the extraction process to achieve optimal and consistent yield, particularly in research focused on the utilization of natural bioactive compounds.

Phytochemical screening results showed that the extracts of raw brayo, mature brayo, raw lindur, and mature lindur contained phenolic compounds, flavonoids, tannins, alkaloids, saponins, and terpenoids. The presence of these secondary metabolite groups indicates strong biological potential, especially in supporting sunscreen activity. Phenolic and flavonoid compounds are known to have a conjugated double bond system and chromophore groups that can absorb ultraviolet (UV) radiation in the UVA and UVB ranges, thus acting as natural UV filters [22,23]. In addition, the antioxidant activity of phenolic compounds, flavonoids, and tannins plays an important role in neutralizing free radicals formed due to UV exposure, thereby preventing skin cell damage and premature aging [24].

Tannin and terpenoid compounds are also reported to contribute to photoprotection through UV absorption and anti-inflammatory activity, while alkaloids provide supporting effects through antioxidant activity and photostability [25]. Saponins, despite having limited UV absorption capacity, play a role in increasing the stability and homogeneity of preparations, thus supporting the effectiveness of other active compounds in sunscreen formulations. Thus, the presence of these secondary metabolites synergistically supports the potential of brayo and lindur extracts as active ingredients in natural sunscreens.

The results of the sunscreen activity test showed that raw brayo extract had the highest SPF value (24.47±0.58), followed by boiled brayo (21.06±0.20), boiled lindur

(14.53±0.09), and raw lindur (4.92±0.35). Based on the classification of sunscreen effectiveness, SPF values 15–30 are categorized as medium to high protection, while SPF <10 is categorized as low protection [25,26]. Thus, raw brayo and boiled brayo have strong potential as sources of natural sunscreen active ingredients, while mature lindur shows medium potential and raw lindur is relatively low.

The high SPF value of raw brayo is thought to be related to its optimal content of phenolic and flavonoid compounds. These compounds have a conjugated double bond system capable of absorbing ultraviolet radiation, particularly UV-B, thus contributing significantly to sunscreen activity [23]. The ripening process is known to cause partial degradation of bioactive compounds due to enzymatic activity and oxidation, which explains the decreased SPF value of boiled brayo compared to raw brayo.

In the case of lindur, the low SPF value in the raw state indicates limited UV protection. However, the increased SPF value in boiled lindur indicates that the ripening process may trigger the formation or increase of certain compounds, such as phenolic transformation products or pigments, which contribute to UV absorption. This phenomenon aligns with reports that certain ripening or processing processes can enhance specific bioactivities, including sunscreen activity, although the effect is highly dependent on the type of ingredient and its chemical composition [27].

Overall, the differences in SPF values between brayo and lindur, as well as between raw and boiled varieties, confirm that the type of ingredient and maturity level play a significant role in determining the effectiveness of natural sunscreens. Brayo extract, especially in its raw form, has better prospects for development as an active ingredient in natural sunscreen cosmetics. Its high content of phenolic and flavonoid compounds not only acts as a UV absorber but also provides antioxidant effects that can protect the skin from oxidative stress caused by sun exposure.

The results of the physical characteristics evaluation showed that all cream formulas with varying extract concentrations met the basic requirements for topical preparations, characterized by good homogeneity, a soft texture, and no odor. Darkening of the color with increasing extract concentration is a common phenomenon in natural-based formulations and is related to the increasing content of phenolic and flavonoid compounds, which have natural brown to dark pigments [28]. This finding is in line with previous reports which stated that polyphenol compounds contribute significantly to the color intensity of herbal cosmetic preparations [29].

The pH value of the cream preparation is within the physiological pH range of facial skin, making it safe for topical use. The decrease in pH that occurs with increasing extract concentration is thought to be related to the weak acidic nature of the phenolic and tannin compounds [30]. This is consistent with previous studies that reported that in various topical formulations based on polyphenol-rich plant extracts, increasing the extract concentration caused a decrease in pH without exceeding the skin's tolerance limit [31].

The increase in viscosity at higher extract concentrations indicates that the extract contributes to the internal structure of the emulsion. The interaction between the hydroxyl groups of the phenolic compounds with the water phase and the emulsifier components is thought to play a role in increasing the viscosity of the system [32]. Although the viscosity increased, it remained within the recommended range for cream formulations, thus not hindering topical application. Appropriate viscosity is important for maintaining the physical stability and skin contact time of the formulation.

Spread ability generally shows an inverse relationship with viscosity, where increasing viscosity has the potential to reduce the preparation's ability to spread. However, the results of this study show that the spread ability value is relatively constant despite the increase in viscosity. This indicates that the formulation system remains in good rheological balance, so that the increase in viscosity is not large enough to inhibit deformation and spreading of the cream during application. The presence of humectants and emulsifiers in the formulation is thought to play a role in maintaining the preparation's flow properties, so that spread ability is maintained despite the increase in viscosity [33]. The adhesion strength showed results that still met the criteria for a topical preparation. This indicates that the viscosity changes in the range of values obtained are still within the functional range for a topical cream preparation. This condition is advantageous because the increase in viscosity during the residence time of the preparation does not reduce the ease of application and comfort of use [34].

The SPF value of the cream preparation increased with increasing extract concentration, indicating a dose-response relationship between the amount of extract and photoprotective activity. This activity is associated with the presence of phenolic and flavonoid compounds, which have a conjugated double bond system that allows them to absorb ultraviolet radiation, particularly in the UV-B region [35]. However, the SPF value of the cream preparation was lower than the SPF value of the extract before formulation. This decrease is a common phenomenon in natural-based sunscreen formulations and may be caused by the dilution effect of the active ingredient, light scattering by the emulsion matrix, and limited diffusion of the UV-absorbing compound to the surface of the preparation [36]. Several studies have shown that the formulation environment can reduce the UV absorption efficiency of the active compound even though its basic activity remains intact [37].

Overall, the data show that increasing the extract concentration can enhance sunscreen activity without degrading the main physical characteristics of the preparation. This indicates that mangrove fruit extract has potential as a natural sunscreen active ingredient, although further formulation optimization is needed to improve the efficiency of photoprotective activity transfer from the extract to the cream preparation.

Conclusion

Raw brayo had the highest yield and SPF activity compared to boiled brayo, raw lindur, and bailed lindur. Correlation analysis results showed a linear relationship between extract yield and sunscreen activity. The raw brayo extract formulated as a cream met the physical characteristics requirements. Furthermore, the extract's sunscreen activity was higher than that of the cream.

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