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Formulation and Evaluation of the Sun Protection Factor (SPF) of a Chemical Sunscreen Containing Molecularly Distilled Heavy Fraction Patchouli Oil

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Abstract: The Sun Protection Factor (SPF) indicates the level of protection that a sunscreen formulation provides against ultraviolet (UV) radiation. The heavy fraction patchouli oil obtained via molecular distillation contains 90.48% patchouli alcohol, which exhibits antioxidant activity. This study aimed to determine the SPF value of the heavy fraction patchouli oil and to evaluate the characteristics and SPF values of emulgel-based chemical sunscreens. Patchouli oil demonstrated an in vitro SPF of 37.63, classified as ultra-protection. Sunscreen formulations were prepared using the beaker method, with patchouli oil incorporated at varying concentrations, including 0% (control), 0.25% (F1), 0.5% (F2), and 1% (F3). The formulations were evaluated for physicochemical characteristics, stability, and skin safety. They exhibited an opaque white to ivory color, with a pH range of 5.2 to 6.4, viscosity between 2011 and 2924 cPs, spread ability of 4 to 6 cm, adhesion time of 5.38 to 8.28 seconds, and an oil-in-water (O/W) emulsion type. The SPF values of the sunscreen formulations ranged from 8.85 to 29.02, with F3 achieving an SPF of 29.02 and classified as ultra-protection. All formulations met the established quality standards, remained stable during storage, and caused no irritation in human volunteers. These findings indicate that the heavy fraction of patchouli oil has great potential as an active ingredient for chemical sunscreen products.

Keywords: Chemical sunscreen; Heavy fraction patchouli oil; Patchouli alcohol; Sun protection factor; UV radiation

Introduction

Repeated exposure to sunlight on the skin can cause structural changes both in the short and long term. Short-term exposure results in erythema, commonly known as sunburn, followed by melanocyte activation and increased melanin production (melanisation), leading to tanning. In the long-term, continuous exposure may cause permanent loss of skin elasticity and increase the risk of developing skin cancer, both melanoma and non-melanoma (Geoffrey et al., 2019).

The skin functions as the body's outermost defence barrier against various external disturbances and stimuli, including UV exposure. Although UV rays contribute to vitamin D synthesis and possess antibacterial properties, excessive exposure can be harmful. UV radiation is classified into UVA, UVB, and UVC (Sofia & Minerva, 2021), and prolonged exposure without protection can negatively impact the skin. It is estimated that unprotected skin can tolerate only about 10 minutes of direct sun exposure (Saputri et al., 2024). Therefore, it is important to protect the skin beyond its natural defence mechanisms. This protection may be achieved through physical means, such as wearing hats, umbrellas, or protective clothing, as well as chemical protection by applying sun protection products such as chemical sunscreens.

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Chemical sunscreen formulations can be enriched with natural ingredients that reflect or absorb UV rays, aiming to protect the skin from UV exposure (Avianka et al., 2022). UV exposure stimulates the formation of free radicals, leading to cellular damage and oxidative stress. One effective approach to mitigating these negative effects is the use of antioxidants (Gulhan et al., 2020). One potential source of antioxidants from Indonesia's flora is the patchouli plant (*Pogostemon cablin* Benth.), known for its essential oil production. Patchouli, a member of the Lamiaceae family, is widely recognized for its distinctive aroma and significant biological activities, including antioxidant properties (Geoffrey et al., 2019). A study by Sofia et al. (2021) and Antari (2023) reported that patchouli oil exhibits antioxidant activity with an IC_{50} value of 34.12 ppm, categorizing it as a highly potent antioxidant. The IC_{50} (Inhibition Concentration) represents the concentration required to inhibit 50% of free radical activity and is used to classify antioxidant activity (Saputri et al., 2024).

Topically applied antioxidants work by reducing damage caused by Reactive Oxygen Species (ROS), preventing skin tissue degradation, and supporting skin repair after UV exposure (Avianka et al., 2022). Continuous UV exposure has been shown to trigger oxidative stress, leading to skin cell damage. Therefore, incorporating antioxidants into sunscreen formulations can enhance their protective effect against sun damage (Gulhan et al., 2019). Patchouli oil, with its primary component, patchouli alcohol, exhibits strong antioxidant activity (Janet, 2023; Fields, 2020; Insuk et al., 2025), making it promising for development as an active ingredient in UV protection products.

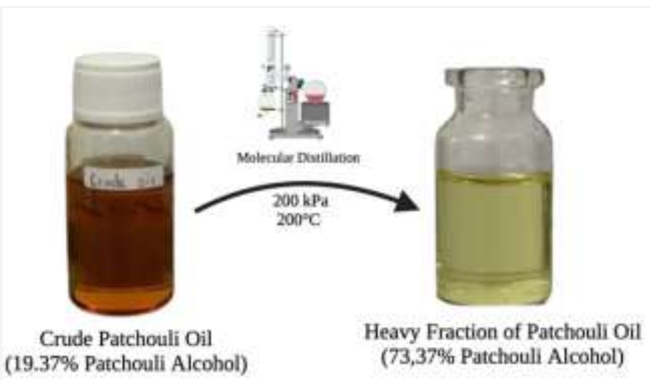

Figure 1. Transformation of crude patchouli oil into heavy fraction patchouli oil.

Crude patchouli oil contains approximately 19.37% patchouli alcohol, whereas the minimum content required by the Indonesian National Standard (SNI 2006) is 30%. Fractionation via molecular distillation separates patchouli oil into light and heavy fractions (Rachmatillah et al., 2021; Firmansyah et al., 2023). The light fraction has a lower patchouli alcohol content, while the heavy fraction contains a higher concentration (Putri & Mahfur, 2023; Anggraini et al., 2025; Suci et al., 2025). The molecular distillation process can significantly increase the patchouli alcohol concentration to as high as 73.37% (Figure 1) (Matsui et al., 2009), which enhances both antioxidant and potential photoprotective effects. Given its high patchouli alcohol content, heavy fraction patchouli oil is expected to produce a sunscreen product with a high SPF value.

Based on these considerations, this study aimed to determine the SPF of heavy fraction patchouli oil and to develop a chemical sunscreen emulgel containing varying concentrations of this active ingredient. The formulations were further evaluated for their SPF performance, physicochemical characteristics, stability, and dermatological safety.

Method

Study Design and Setting

This study employed an experimental laboratory design to evaluate the Sun Protection Factor (SPF) of molecularly distilled heavy fraction patchouli oil and its incorporation into a chemical sunscreen formulation. The research was conducted at the Pharmaceutical Technology Laboratory and Microbiology Laboratory of Universitas Syiah Kuala from January to June 2024. The study was divided into three main phases: Characterization of the heavy fraction patchouli oil using GC–MS analysis, determination of the SPF value of heavy fraction patchouli oil using spectrophotometric analysis, and formulation and evaluation of an emulgel-based chemical sunscreen containing varying concentrations of patchouli oil (0%, 0.25%, 0.5%, and 1%). The sunscreen formulations were prepared using the beaker method and were subjected to comprehensive physicochemical, stability, and safety assessments. Heavy fraction patchouli oil, containing 90.48% patchouli alcohol, was used as the active ingredient. Sunscreen formulations were prepared in four variations: control (F0, 0% patchouli oil), F1 (0.25%), F2 (0.5%), and F3 (1%). The formulations consisted of lanolin, castor oil, squalane, butylene glycol, carbomer, glycerin, and xanthan gum, cocamidopropyl betaine, 1% sodium hyaluronate, sodium PCA, phenoxyetanol, KOH 10%, and distilled water. The emulgel formulation was developed using a gel matrix combined with an oil-in-water (O/W) emulsion system, ensuring ease of application and enhanced skin adherence.

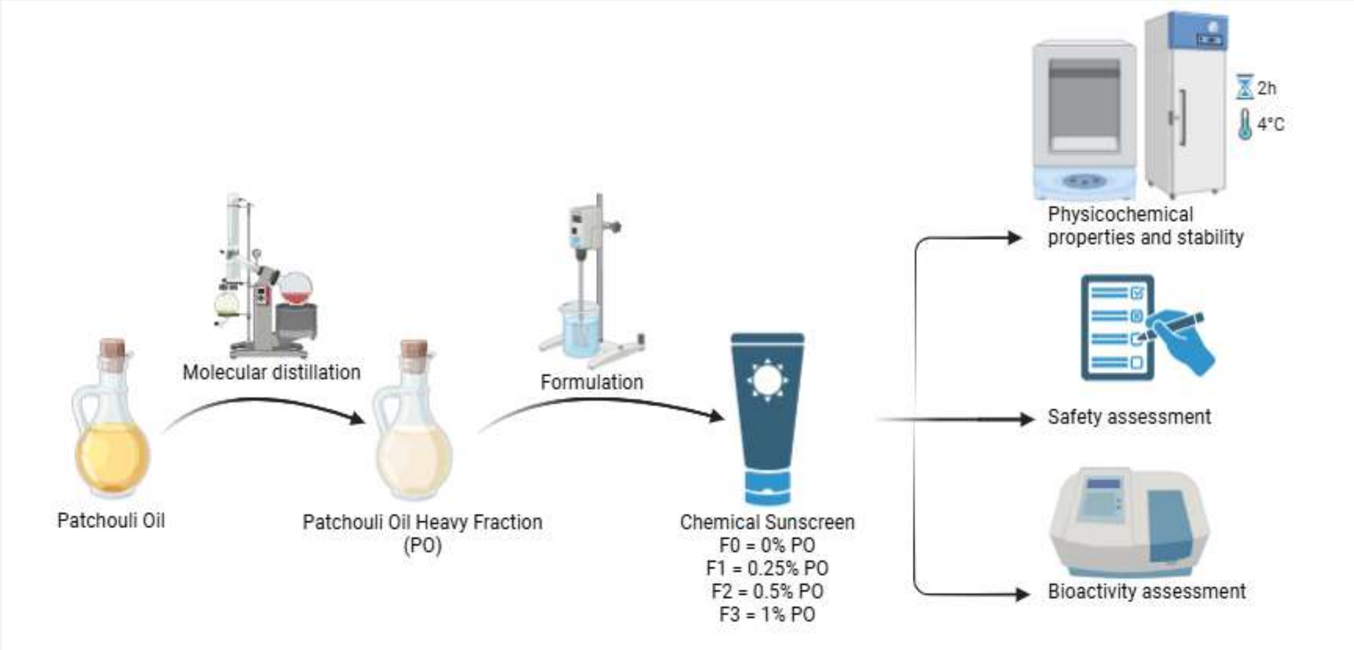

Figure 2. Study design of heavy fraction patchouli oil loaded chemical sunscreen.

GC–MS Characterization Method of Heavy Fraction Patchouli Oil

The GC–MS analysis of the oil fraction was performed on a Shimadzu GC-2010 Plus system equipped with a TG-5MS capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness). Helium was used as the carrier gas at a constant flow rate of 1.35 mL/min, and 1 µL of the sample was injected. The oven temperature was programmed from 60°C (held for 4 min) to 150°C (held for 4 min), then increased to 250°C, with a total run time of 35 minutes. Compound identification was achieved by comparing mass spectra with reference data available in the Chromeleon software library.

Determination of SPF Value of Heavy Fraction Patchouli Oil from Molecular Distillation

A total of 0.05 g of oil was dissolved in 96% ethanol in a 10 mL volumetric flask to obtain a solution with a concentration of 5000 ppm. The solution was then serially diluted to obtain concentrations of 4000 ppm, 500 ppm, 100 ppm, and 50 ppm. The SPF activity of patchouli oil was tested to determine its photoprotective potential against UV radiation. Patchouli oil solutions at various concentrations were prepared and analysed. Absorbance was measured using a UV-Vis spectrophotometer at wavelengths of 290–320 nm at 5 nm intervals, with 96% ethanol as the blank. The SPF values for each concentration were then calculated using the Mansur equation.

$$SPF = CF \sum_{290}^{320} \mathrm{EE}\,(\lambda) \mathrm{x}\, \mathrm{I}\,(\lambda)\ x\ Abs\,(\lambda) \quad (1)$$

Where:

CF	:	Correction factor
EE(λ)	:	Erythemal effectiveness of radiation at wavelength (λ)
I(λ)	:	Intensity of the solar radiation spectrum
Abs(λ)	:	Absorbance value of the sample

Preparation of Patchouli Heavy Fraction I Chemical Sunscreen

The formulation of the chemical sunscreen was developed using heavy fraction patchouli oil, which had been previously tested and proven to have an SPF value classified in the ultra-protection category, demonstrating its potential as a strong photoprotective active ingredient. To optimise its topical performance, the oil was incorporated into an emulgel system. The composition of the base formulation (F0) and active formulations (F1-F3) with increasing concentrations of patchouli oil are presented in Table 1.

The formulations were prepared by dispersing carbomer (0.8% w/w) in distilled water and allowing it to hydrate, followed by neutralisation with 10% KOH to form the gel base. The oil phase (patchouli heavy fraction, lanolin, castor oil and squalane) was heated to 70 °C and gradually incorporated into the gel under homogenisation to obtain the emulgel. After cooling below 40 °C, the remaining excipients, including glycerin, butylene glycol, sodium hyaluronate, sodium PCA, xanthan gum, cocamidopropyl betaine and phenoxyethanol, were added, and the pH was adjusted to 4.5–6.5. The 0.8% carbomer refers to the actual polymer content and falls within the typical range (0.5–2.0% w/w) required to form a stable emulgel base.

Table 1. Formulation of Chemical Sunscreen

Ingredients	Concentration (%w/w)				Function
	F0	F1	F2	F3	
Heavy fraction of patchoil	0	0.25	0.5	1.00	Active ingredient
Lanolin	0.9	0.9	0.9	0.9	Emollient
Castor oil	0.6	0.6	0.6	0.6	Emulsifier
Squalane	1.2	1.2	1.2	1.2	Emollient
Butylene glycol	4.00	4.00	4.00	4.00	Co-solvent
Carbomer	0.8	0.8	0.8	0.8	Gel base
Glycerin	4.00	4.00	4.00	4.00	Humectant
Xanthan gum	0.1	0.1	0.1	0.1	Thickener
Cocamidopropyl betaine	0.1	0.1	0.1	0.1	Surfactant
1% sodium hyaluronate	2.00	2.00	2.00	2.00	Humectant
Sodium PCA	0.50	0.50	0.50	0.50	Humectant
Phenoxyetanol	1.00	1.00	1.00	1.00	Preservative
KOH 10%	0.01	0.01	0.01	0.01	pH neutralizer
Distilled water	Ad 100	Ad 100	Ad 100	Ad 100	Solvent

Investigation of Physicochemical Properties

Organoleptic test

Organoleptic characteristics (form, color, and odor) of the emulgel were evaluated with 30 healthy panellists (Resende et al., 2009; Lee et al., 2020).

Homogeneity test

The homogeneity test was conducted by applying 0.5 g of the emulgel formulation onto an object glass, followed by visual observation of the mixture of components in the formulation. The preparation was considered homogeneous in the absence of visible coarse particles or clumps (Lee et al., 2020; Utami & Lubis, 2024).

pH Test

The test was conducted by dissolving 0.1 g of the emulgel in 10 mL of distilled water. The electrode was then immersed in a beaker containing the emulgel formulation, and the pH value was recorded using a pH meter (Nafissa et al., 2023).

Viscosity and Flow Properties Test

The viscosity test was conducted using a Rheometer RM200 Plus cone-plate type with a measurement system of cP 4220 and a shear rate of 60 s^{-1}. The test was performed by placing 0.5 g of each sample onto the plate, and the resulting viscosity values were recorded. The acceptable viscosity range for the emulgel formulation is between 2,000 and 50,000 cPs. Each formulation was tested in triplicate (Muhammad et al., 2022).

The viscosity test also allows for determining the flow properties of the emulgel, as viscosity affects its movement and flow behavior. The flow properties were assessed by varying the shear rate at 20 s^{-1}, 40 s^{-1}, 60 s^{-1}, 80 s^{-1}, and 100 s^{-1}, then recording the shear stress values and plotting a curve between shear rate and viscosity (Laksmono et al., 2023; Viena et al., 2020).

Spreadability Test

The spreadability was evaluated by placing 0.5 g of the emulgel formulation between object glasses and applying weights of 50 g, 100 g, 150 g, and 200 g. The diameter of spreading was measured after one minute. These varying loads were selected to simulate different pressures during topical application and to assess the ease of spreading on the skin surface. An ideal spreadability for emulgel formulations ranges between 5 and 7 cm (Firmansyah et al., 2023). Each formulation was tested in triplicate.

Adhesion Test

This test was conducted by placing 0.5 g of the emulgel formulation between two object glasses and applying a 200 g weight for 5 minutes. Afterwards, the two adhered glass slides were separated, and the detachment time was recorded. An adhesion time of at least 4 seconds is considered acceptable for topical formulations (Halid et al., 2023; Nurfitri & Endriyatno, 2023; Ramba et al., 2023). Each formulation was tested in triplicate.

Emulsion Type Test

The test was conducted using the methylene blue staining method. A 0.5 g sample of the emulgel was mixed with methylene blue, and the dispersion pattern was observed. This emulsion-type test was performed to determine whether the emulgel formed was oil-in-water (O/W) or water-in-oil (W/O). Uniform dye dispersion indicated an oil-in-water (O/W) emulsion, whereas dye aggregation suggested a water-in-oil (W/O) system (Iriani et al., 2022).

Stability Test of Chemical Sunscreen Formulation

Centrifugation Test

The stability test was conducted using a centrifuge to observe phase separation in the emulgel formulation.

A total of 1 g of emulgel was centrifuged at 5000 rpm for 20 minutes at room temperature, and any phase separation in each formulation was observed (Pratiwi, 2023).

Accelerated Stability Test

The chemical sunscreen samples were stored at 40°C ± 2°C in an incubator for 24 hours, then transferred to a refrigerator at 4°C ± 2°C for another 24 hours (one cycle). This test was conducted for a total of seven cycles (Rimba et al., 2023). Observations included organoleptic evaluation, homogeneity, pH, and viscosity tests.

Real Time Test

The chemical sunscreen samples were stored at 30°C ± 2°C with 75% ± 5% relative humidity (RH) (protected from direct sunlight) for three months and in an incubator at 40°C ± 2°C for three months (Wulandari et al., 2023). Organoleptic evaluation, homogeneity, pH, and viscosity were assessed at 1-month, 2-month, and 3-month intervals.

Pour Plate Method for Determining Total Plate Count

A total of 15-20 mL of agar media, with a temperature not exceeding 48°C, was poured into a Petri dish with a diameter of 85-100 mm. Then, 1 mL of the prepared initial suspension was inoculated into the agar media in the Petri dish by pouring and gently swirling or tilting it to ensure even dispersion. The media was then allowed to solidify at room temperature (Larasati et al., 2023).

Spread Plate Method for Determining Yeast and Mold Count

A total of 15-20 mL of agar media, maintained below 48°C, was poured into a Petri dish with a diameter of 85-100 mm. The agar media was then allowed to cool and solidify in an incubator. Subsequently, at least 0.1 mL of the prepared initial suspension was spread evenly on the surface of the solidified media (Chandra et al., 2022).

Enrichment Method for Determining Microbial Contamination Levels

This method was used to detect microbial contaminants, including *Staphylococcus aureus, Pseudomonas aeruginosa,* and *Candida albicans*. A defined amount of the sample was inoculated into a non-selective liquid medium containing neutralising and/or dispersing agents, followed by incubation at an appropriate temperature for a specific duration. A designated volume of the enrichment suspension was then inoculated onto a non-selective agar medium and incubated at a suitable temperature for a defined period. Bacterial growth was detected and reported as the presence or absence of aerobic mesophilic bacteria per sample of the product (Rakhma et al., 2020).

Safety Assessment of Chemical Sunscreen

The irritation test on the chemical sunscreen formulation was conducted on six volunteers (Shintyawati et al., 2024). With ethical clearance No.113031241024. Participants were eligible if they provided informed consent after receiving an explanation of the study, were male or female aged 20-30 years, had no history of skin allergies, and agreed to complete the antioxidant formulation irritation test throughout the study period. The irritation test was performed using an open patch test, where the formulation was applied to a specific area (2.5×2.5 cm) on the inner forearm, left exposed, and any reactions were observed. The application site was monitored at 0, 24, 48, and 72 hours.

Heavy Metal Contamination Test

The heavy metal contamination test was conducted to detect the presence of heavy metals such as As, Cd, Pb, and Hg. The analysis was conducted using the wet digestion method. For As, Cd, Pb, and Hg, 0.15 grams of the sample was weighed and placed into a quartz tube, followed by the addition of 3 mL of concentrated HCl and 1 mL of 30% v/v H_2O_2. The tube was sealed and left to stand for 15 minutes before undergoing digestion in a microwave digestion system. After digestion, the sample was cooled to room temperature, diluted with 20 mL of deionized water, rinsed (including the inner walls and cap), filtered using Whatman No. 1 filter paper, and the filtrate was collected in a 50 mL volumetric flask. The solution was then diluted with deionized water up to the calibration mark. Analysis was performed using ICP-OES, followed by concentration calculations using the following formula (Rachbini et al., 2020).

$$\frac{Cu}{Bu} \times \frac{1}{1000} \times F \quad (2)$$

Where:

Cu = Concentration of As, Cd, Pb, and Hg (μg/L) in the calibration curve sample
F = Volume of the test solution (mL)
Bu = Weight of the sample (g) in the test solution

Sun Protection Factor Activity

A total of 50 mg of the chemical sunscreen formulation was dissolved in 96% ethanol in a 10 mL volumetric flask, resulting in a solution with a concentration of 5000 ppm. The solution was then diluted to obtain concentrations of 4000 ppm, 500 ppm, 100 ppm, and 50 ppm. The absorbance was measured using a UV-Vis spectrophotometer at wavelengths ranging from 290 to 320 nm at 5 nm intervals, with 96% ethanol used as the blank. The SPF value was then calculated using the Mansur equation (Eq. 1).

Statistical Analysis

The results were expressed as mean ± standard deviation (SD) and analysed using ANOVA in SPSS version 29. When significant differences were observed ($P < 0.05$), post hoc testing was conducted to determine differences between groups.

Results and Discussion

GC–MS Analysis of Heavy Fraction Patchouli Oil

The GC–MS analysis of heavy fraction patchouli oil revealed two main constituents, including patchouli alcohol and azulene (Table 2, Figure 3). These results confirm the compositional concentration pattern reported for molecularly distilled fractions (Rachbini et al., 2020; Badan Pengawas Obat dan Makanan., 2024). Patchouli alcohol was identified as the major component (90.48%) of the heavy fraction. It exhibits antioxidant and anti-inflammatory activities by enhancing endogenous enzymes such as superoxide dismutase (SOD) and glutathione peroxidase (GSH-Px), while suppressing inflammatory cytokines including IL-6, IL-10, and TNF-α. It also prevents photoaging by inhibiting matrix metalloproteinases and maintaining skin elasticity (Waglewska & Bazylinska, 2022), which contributes to SPF performance. In addition, azulene (9.45%) has been investigated as a photosensitizer in photodynamic therapy, exhibiting anti-inflammatory properties that further support its potential contribution to SPF enhancement (Rahmayanti et al., 2021). Therefore, these compounds demonstrate the potential of the heavy fraction as an active ingredient in sunscreen formulations.

Table 2. Major Volatile Compounds in Heavy Fraction Patchouli Oil Determined by GC-MS

Retention Time (min)	Compound Name	Area (%)
22.52	Azulene	9.45
26.33	Patchoule Alcohol	90.48

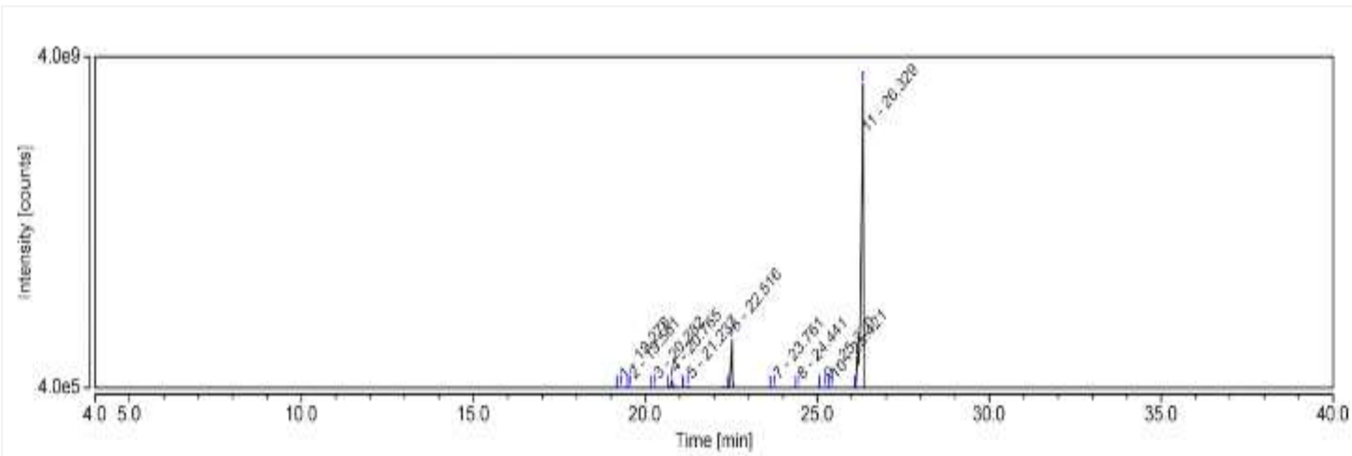

Figure 3. GC-MS chromatogram of heavy fraction patchouli oil

Sun Protection Factor of Heavy Fraction Patchouli Oil

The test was conducted by measuring the absorbance of the sample using a UV-Vis spectrophotometer at wavelengths ranging from 290 to 320 nm. The calculated SPF value of the heavy fraction of patchouli oil is presented in Table 3.

Table 3. Calculation of the SPF Value of Heavy Fraction Patchouli Oil

Wavelength (λ)	EE X I (λ)	Absorbance	EE x I x A x CF
290	0.015	3.745	0.056175
295	0.0817	3.754	0.306701
300	0.2874	4	1.1496
305	0.3278	3.648	1.1958144
310	0.1864	3.905	0.727892
315	0.0839	3.25	0.272675
320	0.018	3.045	0.05481
Total (SPF Value)			37.63668

EE = Erythemal Effectiveness at wavelength λ; I= Solar Intensity at wavelength λ; CF= Correction Factor

The heavy fraction of patchouli oil tested in this study demonstrated significant photoprotective potential, with a Sun Protection Factor (SPF) value of 37.63 at a 0.5% concentration, categorising it as providing ultra protection. Based on this finding, a sunscreen formulation was developed with varying concentrations of 0.25%, 0.5%, and 1% to evaluate its photoprotective efficacy and physical characteristics. The 0.5% concentration was chosen as a reference due to its proven optimal protection, while the 1% concentration was included to assess whether increasing the patchouli oil content further enhances SPF values significantly. The 0.25% concentration was considered to explore effectiveness at a lower range. The maximum concentration was limited to 1% based on formulation stability and physical characteristics.

The heavy fraction of patchouli oil contains bioactive compounds such as patchouli alcohol and sesquiterpenes, which have a strong aroma and high volatility (Tang et al., 2024). Using concentrations above 1% may result in an overpowering fragrance and compromise formulation stability. Therefore, the selected concentration variations in this study aim to optimise photoprotective effectiveness while ensuring the stability and user comfort of the resulting sunscreen.

Physicochemical Evaluation of the Chemical Sunscreen

Physicochemical evaluation was conducted to ensure its quality, stability, and safety of the formulations (Tables 4, 5, and 6). All sunscreen formulations exhibited a homogeneous semisolid consistency (Figure 4). The presence of cocamidopropyl betaine (CAPB) contributed to this uniformity by reducing surface tension and improving emulsion stability, resulting in consistent formulations (Pratasik et al., 2024).

Differences were observed in color and aroma characteristics as the concentration of patchouli oil increased in each formulation. Formula F0 had a slightly translucent white color with a characteristic fatty odor, while F1 appeared white with a distinct patchouli oil scent. Formula F2 had an off-white color with a patchouli oil aroma, whereas F3 exhibited an ivory-white shade with the most dominant patchouli oil scent.

(a) (b) (c) (d)

Figure 4. (a) = control chemical sunscreen formulation; (b) = chemical sunscreen formulation with 0.25% po concentration; (c) = chemical sunscreen formulation with 0.5% po concentration; (d) = chemical sunscreen formulation with 1% po concentration

Table 4. Physicochemical Evaluation of Chemical Sunscreen Formulation

Evaluation				Freshly prepared
	F0	F1	F2	F3
pH	6.4 ± 0.04*	6.3 ± 0.04*	5.7 ± 0*	5.6 ± 0.04*
Viscosity (cPs)	2491 ± 54	2459 ± 24	2373 ± 16	2356 ± 90
Adhesion	8.25 ±0.21	7.3 ± 0.14	6.2 ± 0.16	5.38 ± 0.13
Spreadability	4.5 ± 0.04	5,2 ± 0,04	5.7± 0.04	5.9 ± 0.04

Data are presented as mean ± SD (n = 3). F0 = 0% PO; F1 = 0.25% PO; F2 = 0.5% PO; F3 = 1% PO; *p < 0.05 compared to F0.

Table 5. Thermodynamic Stability Study

Evaluation				Freeze-Thaw
	F0	F1	F2	F3
pH	6.1 ± 0.0	5.5 ± 0.08	5.3 ± 0.18	5.2 ± 0.12
Viscosity (cPs)	2767 ± 68	2327 ± 16	2179 ± 30	2089 ± 22
Adhesion	8.2 ± 0.13	7.3 ± 0.20	6.2 ± 0.10	5.27 ± 0.15
Spreadability	4.3 ± 0.04	5.0 ± 0.09	5.2 ± 0.04	5.6 ± 0.04

Data are presented as mean ± SD (n = 3). F0 = 0% PO; F1 = 0.25% PO; F2 = 0.5% PO; F3 = 1% PO

Table 6. Three-Month Stability Evaluation

Evaluation	Three months (30 ± 20C)				Three months (40 ± 20C)			
	F0	F1	F2	F3	F0	F1	F2	F3
pH	5.6±0.04	5.8±0.04	5.7± 0.08	5.2± 0.12	6.1±0.04	5.8±0.04	5.3± 0.18	5.3± 0.18
Viscosity (cPs)	2781±4.18	2645±6.54	2633.3±16	2292.3±3.6	2292±3.6	2306±1.88	2430±3.26	2011±12.3

Data are presented as mean ± SD (n = 3). F0 = 0% PO; F1 = 0.25% PO; F2 = 0.5% PO; F3 = 1% PO

The physical appearance of the formulations remained unchanged after stability testing, with no alterations in color, aroma, or texture observed across all four formulations. Changes in color and odor are often triggered by oxidation due to storage conditions (Guenther, 2019). Phenoxyethanol acts as a stabilizer by inhibiting the oxidation of active ingredients, thereby helping to maintain formulation stability and extend product shelf life (Putra et al, 2014). These findings indicate that the formulations were stable in terms of organoleptic properties.

The results showed that formulations containing patchouli oil had a pH range of 5.2 to 6.4, which complies with the standard for topical formulations (pH 4.5–6.5) (33). The addition of patchouli oil contributes to a decrease in pH due to the weak acidic nature of essential oils, particularly patchouli alcohol (Dewi & Saptarini, 2023). Stability testing revealed pH fluctuations after the cycling test, attributed to the breakdown of unsaturated fatty acids (Hardiansyah & Mawarni, 2021), and the oxidation of active compounds (Gard et al., 2002) However, real-time stability testing showed that all formulations remained within the safe pH range and complied with the Indonesian National Standard (SNI), confirming their pH stability.

An increase in patchouli oil concentration resulted in a decrease in viscosity. Differences in viscosity values between formulas occurred due to the increased interaction between the gel base polymers and the oil, reducing the formulation's thickness (Suciyani et al., 2020). The viscosity of the formulations before and after stability testing remained within the acceptable range for emulgel formulations, which is 2000–4000 cPs (Garg et al., 2002).

The chemical sunscreen formulations containing patchouli oil exhibited non-Newtonian pseudoplastic flow behavior, where viscosity decreased under applied stress (Figure 5) (Hasbiah et al., 2023). This characteristic ensures that the formulation remains thick in its container but spreads easily when applied, forming a uniform thin layer on the skin (Lumayung et al., 2023). Such properties are highly desirable in semisolid formulations like sunscreen, as they enhance user comfort, ensure even distribution on the skin, and improve UV protection effectiveness.

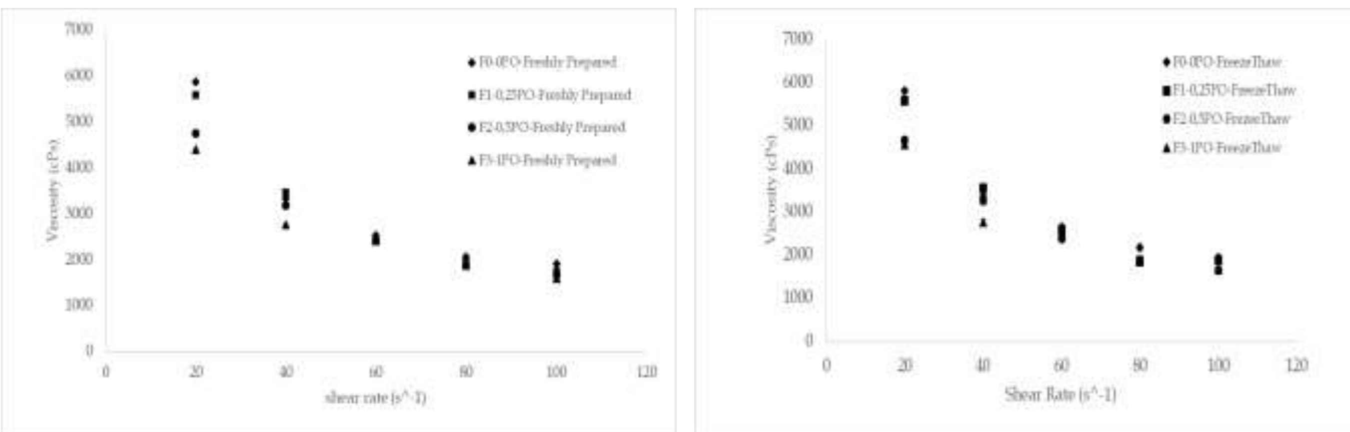

Figure 5. Rheogram of chemical sunscreen formulations, F0 (0% PO), F1 (0.25% PO), F2 (0.5% PO), and F3 (1% PO) in freshly prepared (right) and after cycle test (left). Data are presented as mean ± SD (n=3), where n represents the number of samples

The spreadability and adhesion tests were conducted to evaluate the physical characteristics of the formulation on the skin (Handayani et al., 2015). The results showed that spreadability increased as viscosity decreased, with formulations containing higher concentrations of patchouli oil exhibiting greater spreadability. Meanwhile, adhesion tended to decrease with increasing patchouli oil content, as the formulation became less viscous. After stability testing, no significant changes were observed, indicating that the formulation maintained good stability in terms of spreadability and adhesion.

The test using methylene blue as an indicator showed that all formulations produced a uniformly blue coloration without clumping, confirming that the formulation is an oil-in-water (O/W) emulsion. O/W emulsions have water as the dispersing phase and oil as the dispersed phase (Rustiani et al., 2021). Aligning with the characteristics of the emulgel used in this formulation. Previous research by Yetkin et al. (2022) demonstrated that carbomer 940 at concentrations of 0.5–2% results in an O/W emulgel type. This finding is consistent with the present formulation, where the O/W type is desirable due to its ease of application, better user comfort, and simple removal with water, enhancing its usability.

The stability test was performed using a centrifuge at 5000 rpm for 30 minutes, simulating the gravitational effects of one year of storage (Rustiani et al., 2021). This evaluation is crucial to assess the impact of gravitational forces on the stability of the emulgel base (Feng et al., 2014). Results indicated that none of the formulations exhibited phase separation before or after the cycling test, demonstrating good physical stability and structural integrity under extreme storage conditions.

Safety Assessment of Chemical Sunscreen

Irritation test

The results showed that none of the volunteers experienced any signs of irritation, such as erythema or edema, after applying the chemical sunscreen formulation. Throughout the observation period, the volunteers' skin remained in normal condition without any signs of sensitivity or adverse effects. This finding confirms that the sunscreen formulation containing heavy fraction of patchouli oil is non-irritating and dermatologically safe.

Test for Microbial and Heavy Metal Contamination

The microbiological contamination analysis showed that the chemical sunscreen formulation meets ISO standards, with a total plate count (≤10^1 CFU/g) and yeast-mold counts within safe limits. Pathogenic microorganism tests (*Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Candida albicans*) returned negative results, indicating no contamination. This success is attributed to the use of phenoxyethanol as a preservative. The heavy metal contamination test also confirmed the formulation's safety, with arsenic (As), cadmium (Cd), and mercury (Hg) being undetected, while lead (Pb) was present at a very low level (0.0937 µg/g), significantly below the maximum allowable limit. Overall, these results confirm that the formulated chemical sunscreen is safe for use, meeting microbiological quality and heavy metal safety standards.

Table 7. Test Results for Microbial and Heavy Metal Contamination in Formulation

Parameter	Result	Requirement	Reference
Total plate count	≤ 10ˆ 1 Kol/g	≤ 10^3 Koloni/ g	ISO21149: 2017
Yeast and mold count	≤ 10ˆ 1 Kol/g	≤ 10^3 Koloni/ g	ISO 16212:2017
S. aureus	Negative/0,1 g	Negative	ISO 22718:2015
P. aeruginosa	Negative /0,1 g	Negative	ISO 22717:2015
Candida albicans	Negative /0,1 g	Negative	ISO 18416:2015
PK As	Negative	Max 5 ppm	ACM THA 05
PK Cd	Negative	Max 5 ppm	ACM THA 05
PK Hg	Negative	Max 1 ppm	ACM THA 05
PK Pb	0.0937 µg/g	Max 20 ppm	ACM THA 05

Sun Protection Factor Activity of Chemical Sunscreen

SPF measurement is the primary method for determining the effectiveness of sunscreen formulations. A higher SPF value reflects greater sunscreen's protection against UV radiation. The SPF values of the chemical sunscreen formulations containing heavy fraction of patchouli oil are presented in Table 8.

Table 8. SPF Values of Heavy Fraction Patchouli Oil Chemical Sunscreen Formulation

Formulation	SPF Value	Protection Category
F0	8.85	Extra protection
F1	12.78	Maximum protection
F2	16.24	Ultra protection
F3	29.02	Ultra protection

F0 = 0% PO; F1 = 0.25% PO; F2 = 0.5% PO; F3 = 1% PO

Based on the SPF results, formulation F3 exhibited the best performance with an SPF value of 29.02. The SPF values of formulations F1, F2, and F3 increased as the concentration of patchouli oil in the chemical sunscreen formulation increased. This trend indicates that a higher concentration of patchouli oil leads to a higher SPF value. According to SPF classification, formulation F3, with the highest SPF value, falls into the ultra-protection category, demonstrating the most effective UV protection compared to other formulations at the same concentration. The high SPF activity in F3 is likely attributed to the active compound patchouli alcohol and a formulation composition that optimally enhances UV absorption. Additionally, the possible synergy between active ingredients and excipients in the formulation may further contribute to its enhanced photoprotective properties.

Conclusion

Natural compounds with antioxidant activity, such as patchouli oil, have the potential to be used as active ingredients in sunscreen formulations to enhance protection against UV exposure. Based on the findings, the heavy fraction of patchouli oil exhibited an SPF value of 37.63, with patchouli alcohol (90.48%) identified as the most abundant compound, indicating its potential as a photoprotective agent. The formulated chemical sunscreen had an ivory-white color, a pH ranging from 6.4 to 5.2, viscosity between 2924 and 2011 cPs, a spreadability of 4–6 cm, and an adhesion time of 5.38–8.28 seconds. The formulation was identified as an oil-in-water (O/W) emulgel, which is suitable for topical application. Among the three developed formulations, F3 demonstrated the highest SPF activity at 29.02, placing it in the ultra-protection category, highlighting its strong potential as an effective sunscreen product.

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Author Contributions

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Conflicts of Interest

The authors declare that there is no conflict of interest.

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