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Research article

Formulation of Sunscreen Lotion Combining Titanium Dioxide and Porang Starch (*Amorphophallus oncophyllus*) on Physical Properties and SPF Value

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Abstract

Porang tubers starch (*Amorphophallus oncophyllus*) has natural photoprotective properties due to its opacity, but its moderate SPF limits its efficacy as a standalone sunscreen agent. Titanium dioxide (TiO_2), an inorganic UV filter, is often added to enhance sunscreen performance. This study aimed to evaluate the impact of combining porang starch with varying concentrations of titanium dioxide on the SPF value and physical properties of sunscreen lotion. Porang starch was extracted and incorporated into lotion formulations with 0%, 5%, 10%, and 15% titanium dioxide. Each formulation was assessed for SPF (in vitro), pH, viscosity, spreadability, adhesiveness, organoleptic characteristics, and homogeneity. Statistical analysis was conducted using one-way ANOVA and Tukey HSD tests. Titanium dioxide significantly increased SPF values, with the highest value (34.69) observed in the 15% TiO_2 formulation. However, excessive TiO_2 reduced spreadability and homogeneity while increasing viscosity and adhesiveness. All formulations met pH safety standards (4.5–8.0). Formula 2 (10% TiO_2 + 15% porang starch) showed the most balanced profile in terms of efficacy and physical acceptability. The combination of porang starch and titanium dioxide have impacts SPF and physical characteristics of sunscreen lotions. Formula 2 was identified as the optimal formulation for further development as a natural-based sunscreen.

Keywords: Lotion, porang starch, physical evaluation, titanium dioxide, SPF, sunscreen

Introduction

Ultraviolet (UV) radiation from sunlight is a major environmental factor that can lead to various skin issues, ranging from premature aging to skin cancer. As a result, the use of sunscreen has become an essential protective measure. Recently, there has been a growing interest in utilizing natural ingredients in sunscreen formulations, driven by increasing consumer awareness of safety and sustainability [1][2]. Porang tubers (*Amorphophallus oncophyllus*), a plant widely cultivated in Indonesia, contains starch with notable opaque properties. This characteristic allows the starch to scatter and reflect light, making it a potential natural photoprotective agent in sunscreen products [3]. Previous studies have shown that sunscreen lotion containing 15% porang starch achieves an Sun Protection Factor (SPF) value of 17.231, which is classified as moderate protection [4].

However, in order to meet the market demand for sunscreens with higher protection levels, it is necessary to enhance SPF values by incorporating additional active ingredients [4]. Titanium dioxide (TiO_2), an inorganic UV filter, is widely used in sunscreen formulations due to its effectiveness in reflecting and scattering UV radiation [5][6]. It is also considered relatively safe and non-carcinogenic [5][7].

While titanium dioxide is commonly used as a UV filter, the synergistic effect of its combination with natural ingredients like porang starch on SPF value and physical properties of the formulation remains underexplored. Additionally, lotion was selected as the preferred dosage form for its light consistency, ease of application, and favorable spreadability, which contribute to user comfort [8][9]. This study aims to evaluated the effect of adding titanium dioxide on the SPF value and physical properties of sunscreen lotion formulated with porang tubers starch as the main active ingredient. The findings was expected to contribute to the development of safe, effective, and marketable natural-based sunscreen products.

Materials and Methods

Materials

The main active ingredients used in this study were porang tubers starch (*Amorphophallus oncophyllus*) from Banten, Indonesia and titanium dioxide (TiO_2) from PT. ROFA Laboratorium Center, Bandung. Other formulation components included glycerin (Brataco), stearic acid (Brataco), glyceryl monostearate (Ecogreen Oleochemical), vaseline (Rose Polymers), vitamin E (Ecogreen Oleochemical), citric acid (Brataco), potassium hydroxide (KOH) (Brataco), methylparaben (Brataco), and propylparaben (Brataco). Key instruments used included a UV-vis spectrophotometer (Shimadzu UV-1900), viscometer (Rheosys Merlin VR2), homogenizer (IKA T25 Ultra Turrax), microscope, pH meter (Ohaus), sonicator, analytical balance, oven, and various glassware.

Methods

Preparation of porang starch

Fresh porang tubers were washed, sliced, and soaked in an 8% NaCl solution (1:4 ratio of tubers to water) for 3–4 hours to reduce calcium oxalate content. The tubers were then blended, filtered, and the starch was allowed to settle for 6–24 hours. The precipitate was dried using a water bath at 50 °C for 6 hours followed by oven drying at 50 °C. The resulting starch was sieved using a 100-mesh sieve.

Formulation of sunscreen lotion

This lotion is formulated as an oil-in-water emulsion using the composition as shown in Table 1.

Table 1. Composition of sunscreen lotion

Phase	Materials	Composition (% b/b)			
		F1	F2	F3	F4
Active ingredients	Porang starch	15	15	15	15
	Titanium dioxide	0	5	10	15
Oil phase	Propyl paraben	0,05	0,05	0,05	0,05
	Glyceril monostearate	1,5	1,5	1,5	1,5
	Vaselin	4	4	4	4
	Stearic acid	1,5	1,5	1,5	1,5
	Vitamin E	1	1	1	1
Aqueous phase	Citric acid	1,5	1,5	1,5	1,5
	KOH	1	1	1	1
	Metyl paraben	0,20	0,20	0,20	0,20
	Glycerin	9	9	9	9
	Aquades ad	100	100	100	100

The aqueous phase (KOH, water, glycerin, methylparaben, citric acid) and the oil phase (glyceryl monostearate, stearic acid, vaseline, vitamin E, propylparaben) were heated separately to 80 °C. Titanium dioxide was added to the oil phase and homogenized. Both phases were then combined, and porang starch was added with continuous stirring to form a stable emulsion.

Evaluation of formulated sunscreen

The physical and functional properties of each sunscreen lotion formulation were evaluated through several tests. The organoleptic characteristics were first assessed by observing the color, odor, and texture of the preparation using sensory perception [10]. The homogeneity test was performed by visually examining a portion of the sample to determine the presence or absence of fine or coarse particles [10].

The pH test was conducted by diluting the sample to a 1% concentration in distilled water, after which the electrode of the pH meter was immersed into the solution. The pH value was recorded once the reading stabilized. According to the Indonesian National Standard (SNI 16-4399-1996), the acceptable pH range for sunscreen products is between 4.5 and 8.0 [10].

The spreadability test involved weighing 0.5 g of the sunscreen lotion and placing it at the center of a circular glass plate with scale markings. Another glass plate and a weight were placed on top of the sample for one minute, after which the spread diameter was recorded [11]. The adhesiveness test was carried out by weighing 1 g of the sample and placing it between two glass plates. The sample was pressed with a 1-kg weight for five minutes. After removing both the 80-g and 1-kg weights, the time required for the glass plates to separate was recorded [11].

The viscosity of each sunscreen formulation was measured using a Rheosys Merlin VR viscometer equipped with a cup-and-bob system. The instrument was operated under specific settings with a start speed of 1 rpm, an end speed of 60 rpm, eight measurement steps, a delay time of 5 seconds, and an integration time of 0.2 seconds.

The sun protection factor (SPF) value was determined in vitro using a UV–visible spectrophotometer. The sunscreen lotion containing porang starch was diluted to a 1% concentration in ethanol p.a. within a 10 mL volumetric flask. For comparison, stock solutions of porang starch (15% w/w) and titanium dioxide (15% w/w) were also prepared and subsequently diluted to 1% in ethanol p.a. Each solution was homogenized using a sonicator to ensure uniform dispersion. The absorbance of the prepared samples was then measured across wavelengths ranging from 290 to 320 nm, at 5 nm intervals [4].

Data analysis

The results obtained in this study were analyzed by comparing the significance values of the evaluations and interpreting the data descriptively to determine the optimal formulation. The evaluation test was replicated three times. Statistical analysis was performed using the one-way ANOVA (Analysis of Variance) method with SPSS for Windows version 25.0. Post Hoc analysis was then performed to identify formulations with significant differences [4].

Results and Discussion

Characterization of porang starch

Porang starch was extracted and characterized through organoleptic testing, microscopy, and iodine staining. Porang tubers was first analyzed at the Biology Laboratory of the Faculty of Science and Applied Sciences at Universitas Ahmad Dahlan before extraction. According to the analysis results in Letter No. 441/Lab.Bio/B/X/2023, the sample is identified as Amorphophallus oncophyllus.

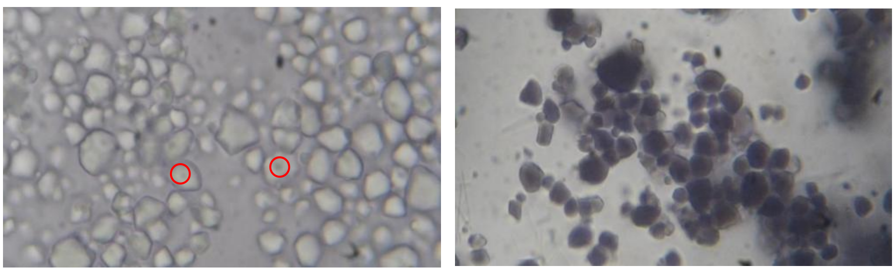
In the production of porang tubers starch, sodium chloride (NaCl) is added to reduce calcium oxalate crystals present in the porang tubers, which could irritate human skin [12]. The mechanism of calcium oxalate reduction using sodium chloride (NaCl) is because sodium chloride (NaCl) dissolves in water and undergoes ionization into Na+ and Cl- ions. The Na+ ions formed will bind with the oxalate in calcium oxalate [13]. Meanwhile, Cl- ions form calcium dichloride precipitates that are easily soluble in water [14]. The reaction is illustrated in Figure 1.

$$CaC_2O_4 + 2NaCl \rightarrow Na_2C_2O_4 + CaCl_2$$

Calcium oxalate ↓ (CaC₂O₄)   Sodium oxalate ↓ (Na₂C₂O₄)

Figure 1. Reaction of Sodium Oxalate Formation

Microscopically, the starch granules were irregularly shaped with a clear hilum, indicating a concentric amylose type. The hilum position was marked with a red circle in Figure 2a. This was in line with the research [15] which states that porang tubers starch was irregularly round in shape and has a concentric amylum type.



(a) (b)

Figure 2. (a) Observation of porang tubers starch and (b) Addition of starch with iodine reagent (1000x magnification)

In the qualitative starch test, starch was added to iodine reagent to confirm that the sample obtained was starch. After adding the iodine reagent, the starch changed color from white to purple-blue (Figure 2b). When reacted with iodine, the starch showed a dark blue coloration, confirming the presence of amylose. This was in line with the research [16], who noted that starch treated with iodine produces a blue-violet color. This occurs due to the bonding between spiral-shaped amylose and iodine.

Organoleptic properties and homogenity

The data of the physical properties test results for the preparation are shown in Table 3. All formulations (F0–F3) displayed a typical porang starch odor and were white to off-white in color. However, F3 exhibited a slightly coarse texture due to poor dispersion of titanium dioxide, indicating inadequate homogeneity. This may be attributed to the high concentration (15%) of titanium dioxide, which exceeded the solubilization capacity of the oil phase.

Table 3. Physical properties of sunscreen

Formula	Texture and Homogenity	pH Value	Viscosity (cPs)	Spreadability (cm)	Adhesiveness (s)
F0	Homogeneous and smooth texture	7.51 ± 0.13	13,066.12 ± 495.05	6.65 ± 0.06	2.89 ± 0.05
F1	Homogeneous and smooth texture	7.23 ± 0.06	37,304.17 ± 627.72	6.26 ± 0.07	3.04 ± 0.03
F2	Homogeneous and smooth texture	7.11 ± 0.02	47,176.90 ± 1191.51	6.13 ± 0.10	3.19 ± 0.06
F3	Not homogeneous and rough texture	6.99 ± 0.07	65,235.04 ± 615.29	5.82 ± 0.10	3.48 ± 0.06

Note : The test was carried out three times in replication. The table displays the data ± SD

pH measurement

The pH values of all formulations ranged from 6.99 to 7.51, which falls within the acceptable range for topical products (4.5–8.0) as per Indonesian National Standards (SNI). The data of pH was shown in Table 3. Increasing concentrations of titanium dioxide led to a gradual decrease in pH, likely due to the slightly acidic nature of titanium dioxide compared to porang starch. Despite these changes, all formulations remained within safe limits for skin application. Statistical analysis (One-way ANOVA, $p < 0.05$) confirmed significant differences in pH among all formulations.

Viscosity

Viscosity testing is conducted to determine the viscosity level of a preparation. The higher the viscosity value, the higher the viscosity level of a preparation [17]. The viscosity requirements in SNI 16-4399-1996 regarding sunscreen are that the viscosity of the formulation must be within the range of 2,000-50,000 cPs. Viscosity increased with higher concentrations of titanium dioxide. The data of viscosity was shown in Table 3. F0 had the lowest viscosity, while F3 exceeded the maximum standard (50,000 cPs), potentially affecting user comfort and spreadability. Viscosity values are closely related to adhesion and spreadability values because viscosity describes the ability of a preparation to flow [18]. Viscosity is inversely proportional to spreadability, meaning that as viscosity increases, spreadability decreases. Conversely, viscosity is directly proportional to adhesion, so as viscosity increases, adhesion also increases [19]. The increase in viscosity may be due to the solid particulate nature of titanium dioxide, which thickens the formulation. The viscosity value increases as the concentration of titanium dioxide in the preparation increases. According to [17]. The increase in viscosity is caused by a decrease in water content. Statistical analysis indicated significant differences between all formulations ($p < 0.05$).

Spreadability

The spreadability test was conducted to determine how widely the sunscreen could spread when subjected to pressure. The wider the spreadability, the greater the contact between the sunscreen and the skin [20]. A good spreadability value ranges between 5-7 cm [21]. The data of spreadability was shown in Table 3. Increasing the concentration of titanium dioxide can reduce the spreadability value. Spreadability was inversely related to viscosity. As viscosity increased, spreadability decreased [22]. F3, with the highest viscosity, exhibited the lowest spreadability, indicating difficulty in application. Based on statistical analysis of variances (ANOVA), a significance value of 0.000 ($p<0.05$) was obtained. This value indicates a significant difference between formulas.

The addition of titanium dioxide can reduce the spreadability value. The higher the concentration of titanium dioxide, the lower the spreadability value obtained. This results in a decrease in the spreadability of the formulation on the skin as the concentration of titanium dioxide in the formulation increases. Spreadability is related to viscosity. Higher viscosity values result in lower spreadability. This was because the thicker a preparation, the more difficult it was for particles to move, making it difficult to spread.

Adhesiveness

Adhesion testing was conducted to determine how long the formulation adheres to the skin surface. The longer the formulation adheres to the skin, the longer the sunscreen can protect the skin. A good adhesion standard is when the formulation can adhere to the skin surface for more than 4 seconds [23]. Based on Table 3, the adhesion strength that meets the established standard is formula 2 and formula 3.

After conducting a statistical analysis of variances (ANOVA), a significance value of 0.000 ($p < 0.05$) was obtained. This value indicates a significant difference between the formulas. Based on the statistical analysis, the addition of titanium dioxide tends to show a non-significant increase in adhesion strength. Adhesiveness was also related to viscosity. Higher viscosity makes the lotion thicker, thus it takes longer to adhere to the skin and was more difficult to remove [22]. However, as the concentration of titanium dioxide in the formulation increases, there wss a significant increase in the adhesion strength of the formulation. The higher the adhesion strength, the longer the formulation's ability to adhere to the skin surface.

In vitro SPF value

In vitro testing is one of the predictive SPF tests conducted prior to in vivo testing [16]. According to Indonesian National Standards (SNI), the minimum SPF value for sunscreen products is 4. Based on the results obtained as shown in table 3, all formulas meet the standards set by SNI. According to BPOM, SPF classification is as follows: low protection level ≥ 6 - < 15, moderate protection level with SPF ≥ 15 - < 30, and high protection level ≥ 30 - < 50. SPF data results can be seen in Figure 3.

The measurement of SPF values in vitro using spectrophotometry is influenced by several factors such as the use of excipients, concentration, solvent interactions, and interactions between components in the formulation, which can affect the absorbance value [24]. The high predicted SPF values for the active ingredients porang tubers starch and titanium dioxide may be due to the absence of factors such as additives in the testing [25].

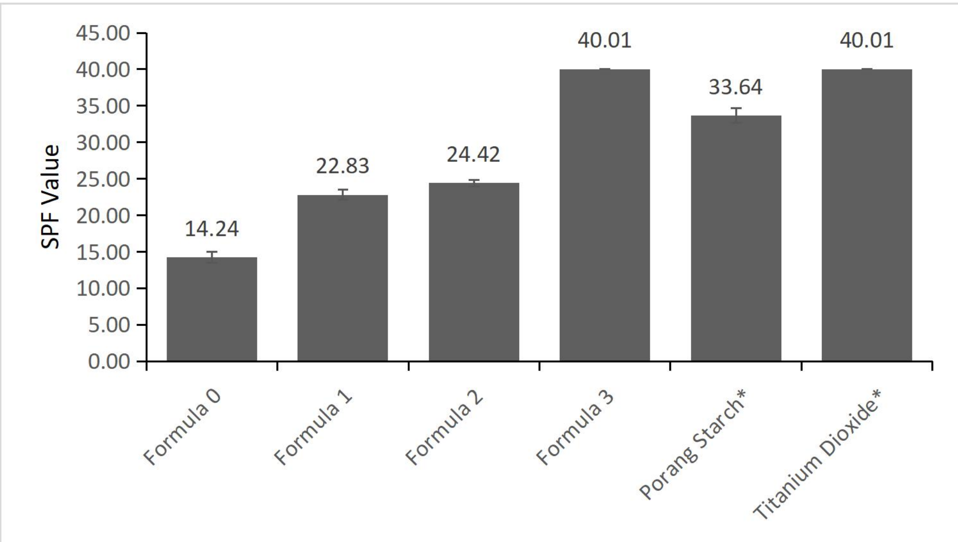


Figure 3. SPF value with in vitro method

The SPF value increased significantly with higher concentrations of titanium dioxide. F0 (porang starch only) exhibited an SPF of 17.231, which is classified as moderate protection. Formulations containing titanium dioxide showed a progressive enhancement in SPF. However, titanium dioxide, as a single substance, could also provide a high SPF value. When measuring SPF, porang starch and titanium dioxide were not formulated as a complete formula, thus the high SPF results from porang starch and titanium dioxide cannot be used as a direct comparison.

Based on statistical analysis using the one-way analysis of variances (ANOVA) method, the results showed a significance $p < 0.05$, which means there was a significant difference in SPF between formulas. The results showed that Formula 0 showed a significant difference with Formula 1, Formula 2, and Formula 3. Formula 1 showed a significant difference with Formula 0, Formula 2, and Formula 3. Formula 2 showed a significant difference with Formula 0, Formula 1, and Formula 3. Formula 3 showed a significant difference with Formula 0, Formula 1, and Formula 2. These results indicate that there is a significant difference between formulas. This can be interpreted that each additional concentration of titanium dioxide shows a significant increase in SPF. The increase in the SPF value in the preparation indicates that the preparation's ability to protect the skin will last longer [26].

Conclusion

Titanium dioxide significantly affected the SPF and physical properties of the sunscreen lotion. Increasing its concentration enhanced SPF, viscosity, and adhesiveness, improving skin retention, but reduced spreadability and slightly lowered pH—yet all values remained within acceptable limits. Based on characterization and statistical analysis, Formula F2 (15% porang starch and 10% titanium dioxide) was identified as the optimal formulation, meeting all standards for homogeneity, SPF, pH, viscosity, spreadability, and adhesiveness. Although F3 exhibited a higher SPF, it was excluded due to poor homogeneity. Thus, F2 represents the most suitable natural-based sunscreen candidate for further development.

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Declarations

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Conflict of interest	:	All authors declare that there are no conflicts of interest with respect to this manuscript.
Ethics Declaration	:	All authors declare that the research work has fulfilled all relevant ethical guidelines including statements of approval/consent.
Additional information	:	This article is original and has not been published elsewhere.

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