

Formulation and Evaluation of Sunscreen Cream Based on Titanium Dioxide and Zinc Oxide Nanopowders Combined with Niacinamide

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Abstract: Excessive exposure to ultraviolet (UV) radiation can cause oxidative stress, erythema, photoaging, and other forms of skin damage, highlighting the need for sunscreen formulations that provide effective photoprotection and acceptable physicochemical characteristics. This study aimed to formulate and evaluate an oil-in-water (O/W) sunscreen cream containing titanium dioxide nanopowder, zinc oxide nanopowder, and niacinamide, and to determine the formulation with the best photoprotective performance. An experimental study was conducted using three cream formulations with different concentrations of titanium dioxide and zinc oxide nanopowders: F1 (2%:4%), F2 (4%:2%), and F3 (3%:3%), while niacinamide was maintained at 4% in all formulations. The formulations were evaluated for organoleptic properties, homogeneity, pH, viscosity, spreadability, adhesion, emulsion type, heating-cooling stability, Sun Protection Factor (SPF), Critical Wavelength (λ_c), and skin irritation. Quantitative data were analyzed using Two-Way Analysis of Variance (ANOVA). All formulations exhibited good physicochemical characteristics and remained stable during four weeks of storage without phase separation after six heating-cooling cycles. The SPF values of F1, F2, and F3 were 39.4, 37.0, and 40.4, respectively, indicating ultra-level UV protection. F3 demonstrated the highest SPF value and a Critical Wavelength of 383.35 nm, indicating broad-spectrum UV protection. Formulation variation significantly affected viscosity and spreadability ($p < 0.05$), whereas adhesion was not significantly affected ($p > 0.05$). No signs of skin irritation were observed for Formula 3 in the patch test. Therefore, F3 was selected as the optimal formulation based on its physicochemical characteristics, stability, SPF value, broad-spectrum UV protection, and skin tolerability.

Keywords: nanopowder, titanium dioxide, zinc oxide, niacinamide, sunscreen cream, SPF

Abstrak: Paparan radiasi ultraviolet (UV) secara berlebihan dapat menyebabkan stres oksidatif, eritema, penuaan dini, dan berbagai kerusakan kulit sehingga diperlukan sediaan tabir surya yang memberikan perlindungan efektif serta memiliki karakteristik fisik yang baik. Penelitian ini bertujuan memformulasikan dan mengevaluasi krim tabir surya tipe minyak dalam air (M/A) yang mengandung kombinasi nanopowder titanium dioxide, nanopowder zinc oxide, dan niacinamide, serta menentukan formula dengan aktivitas fotoprotektif terbaik. Penelitian eksperimental dilakukan menggunakan tiga formula krim dengan variasi konsentrasi nanopowder titanium dioxide dan nanopowder zinc oxide, yaitu F1 (2%:4%), F2 (4%:2%), dan F3 (3%:3%), sedangkan konsentrasi niacinamide dipertahankan sebesar 4% pada seluruh formula. Evaluasi meliputi organoleptik, homogenitas, pH, viskositas, daya sebar, daya lekat, tipe emulsi, stabilitas menggunakan heating-cooling test, nilai Sun Protection Factor (SPF), Critical Wavelength (λ_c), dan uji iritasi kulit. Data kuantitatif dianalisis menggunakan Two-Way Analysis of Variance (ANOVA). Hasil penelitian menunjukkan bahwa seluruh formula memiliki karakteristik fisik yang baik dan stabil selama empat minggu penyimpanan tanpa terjadi pemisahan fase setelah enam siklus heating-cooling. Nilai SPF F1, F2, dan F3 berturut-turut sebesar 39,4; 37,0; dan 40,4 yang termasuk kategori proteksi ultra. F3 memiliki nilai SPF tertinggi dan Critical Wavelength sebesar 383,35 nm yang memenuhi kriteria perlindungan spektrum luas. Variasi formula berpengaruh signifikan terhadap viskositas dan daya sebar ($p < 0,05$), tetapi tidak berpengaruh signifikan terhadap daya lekat ($p > 0,05$). F3 tidak menimbulkan tanda iritasi pada uji tempel sehingga dipilih sebagai formula terbaik berdasarkan karakteristik fisik, stabilitas, aktivitas fotoprotektif, dan tolerabilitas kulit.

Kata kunci: nanopowder, titanium dioxide, zinc oxide, niacinamide, krim tabir surya, SPF

Introduction

Excessive exposure to ultraviolet (UV) radiation can cause various forms of skin damage. UVA radiation, which penetrates into the dermis, contributes to the generation of reactive oxygen species, oxidative stress, degradation of collagen and elastin, and photoaging, whereas UVB radiation is primarily associated with erythema and DNA damage. Therefore, the use of sunscreen is an important measure to protect the skin against both UVA and UVB radiation (Baki & Alexander, 2019).

Sunscreens may contain either organic or inorganic UV filters. Titanium dioxide and zinc oxide are widely used physical UV filters due to their good photostability and ability to provide protection against ultraviolet radiation. Titanium dioxide primarily provides protection against UVB and part of the UVA spectrum, whereas zinc oxide offers broader protection extending further into the UVA region. The combination of these two mineral filters has the potential to provide more comprehensive broad-spectrum photoprotection (Schneider & Lim, 2019).

The use of titanium dioxide and zinc oxide in nanopowder form has attracted considerable interest in sunscreen development. Smaller particle sizes can improve optical properties and help reduce the appearance of white cast compared with larger mineral particles. However, the incorporation of nanopowders also requires a formulation system capable of maintaining adequate dispersion, homogeneity, and stability. An oil-in-water (O/W) cream was selected because of its lightweight texture, ease of spreading, and suitability for topical application (Aulton & Taylor, 2022; Schneider & Lim, 2019).

In addition to physical UV filters, the incorporation of skin-care active ingredients may provide multifunctional benefits in sunscreen formulations. Niacinamide, the active form of vitamin B3, exhibits antioxidant and anti-inflammatory properties and contributes to the reduction of hyperpigmentation and the maintenance of skin barrier function. The combination of niacinamide with physical UV filters may therefore provide both photoprotection and skin-care benefits (Qin et al., 2024; Torres-Moral et al., 2024).

Although numerous studies have investigated titanium dioxide and zinc oxide as sunscreen agents, studies specifically combining titanium dioxide nanopowder and zinc oxide nanopowder with niacinamide in an O/W cream and evaluating the effects of their compositional variation on physical characteristics, stability, sun protection factor (SPF), and critical wavelength (λ_c) remain limited. Therefore, this study aimed to formulate and evaluate a sunscreen cream containing a combination of titanium dioxide nanopowder, zinc oxide nanopowder, and niacinamide, and to determine the formulation with the most favorable physical characteristics and photoprotective activity based on SPF and λ_c values.

Methods

This study employed a quantitative experimental design to formulate and evaluate an oil-in-water (O/W) sunscreen cream containing titanium dioxide nanopowder, zinc oxide nanopowder, and niacinamide. Titanium dioxide nanopowder and zinc oxide nanopowder were obtained from CV. Inovasi Teknologi Nano, Indonesia, while niacinamide was obtained from Arxada Chemical Singapore Pte. Ltd. The excipients used in the formulations included isopropyl myristate, stearic acid, cetyl alcohol, Tween 80, propylene glycol, glycerin, triethanolamine, phenoxyethanol, and distilled water. The instruments used included an analytical balance, magnetic stirrer, hot plate, Ultra-Turrax homogenizer, pH meter (Hanna® HI-2002, Romania), Brookfield viscometer (AMETEK®, USA), UV-Vis spectrophotometer (Shimadzu® UV-1900i, Japan), Particle Size Analyzer (Analysette® 22 NeXT Nano, Germany), refrigerator, and standard laboratory glassware.

Three sunscreen cream formulations were prepared with different concentrations of titanium dioxide and zinc oxide nanopowders: F1 (2%:4%), F2 (4%:2%), and F3 (3%:3%), while the concentration of niacinamide was maintained at 4% in all formulations. The creams were prepared using a hot emulsification method. The oil and aqueous phases were heated separately to 60–70°C and subsequently emulsified by gradually combining the two phases under homogenization. Titanium dioxide and zinc oxide nanopowders were incorporated into the formulation during the homogenization process. Niacinamide was added after the temperature of the formulation had decreased to approximately 40–45°C, followed by further homogenization until a homogeneous cream was obtained.

The formulations were evaluated for organoleptic characteristics, homogeneity, pH, viscosity, spreadability, adhesion, emulsion type, and physical stability. Organoleptic characteristics, homogeneity, emulsion type, and heating–cooling stability were evaluated once for each formulation, whereas pH, viscosity, spreadability, and adhesion were measured in triplicate ($n = 3$) for each formulation at each observation time during the four-week storage period. Physical stability was evaluated using a heating–cooling test consisting of six cycles, in which the formulations were stored at $4 \pm 2^\circ\text{C}$ for 24 h and subsequently at $40 \pm 2^\circ\text{C}$ for 24 h.

The Sun Protection Factor (SPF) was determined in vitro using a UV-Vis spectrophotometer at wavelengths ranging from 290 to 320 nm at 5-nm intervals according to the Mansur method. SPF measurements were performed in triplicate ($n = 3$) for each formulation, and the results were expressed as mean values. The formulation exhibiting the highest SPF value was subsequently evaluated for Critical Wavelength (λ_c) over the wavelength range of 290–400 nm.

The skin irritation test was performed using a closed patch test on four healthy panelists following ethical approval and obtaining informed consent. The skin was observed for signs of

erythema, edema, itching, and burning sensation. Data on pH, viscosity, spreadability, and adhesion were analyzed using Two-Way ANOVA at a 95% confidence level ($p < 0.05$) to determine the effects of formulation, storage time, and their interaction. Data obtained from organoleptic evaluation, homogeneity, emulsion type, physical stability, SPF, Critical Wavelength (λ_c), and skin irritation testing were analyzed descriptively.

Results and Discussion

Characterization of Active Ingredients

The physicochemical characteristics of the active ingredients used in the sunscreen cream formulations are presented in Table 1. Titanium dioxide and zinc oxide nanopowders appeared as fine white, odorless powders, whereas niacinamide was obtained as a white crystalline powder with a characteristic odor. The solubility and pH characteristics of the three active ingredients were consistent with the specifications of the materials used for topical formulations. The pH values of titanium dioxide, zinc oxide, and niacinamide were 6.32, 6.01, and 6.23, respectively, indicating that the active ingredients possessed suitable physicochemical characteristics for incorporation into topical preparations.

Table 1. Physicochemical Characteristics, Particle Size (D95), and Sun Protection Factor (SPF) of Active Ingredients

Parameter	Titanium Dioxide	Zinc Oxide	Niacinamide
Appearance	Fine white powder	Fine white powder	White crystalline powder
Odor	Odorless	Odorless	Characteristic Odor
Solubility	Insoluble in water and ethanol	Insoluble in water and ethanol	Soluble in water and ethanol
pH	6.32	6.01	6.23
D95 (nm)	43.79 ± 3.39	91.48 ± 10.10	-
SPF	38	39	-

Particle size analysis showed that titanium dioxide had a D95 value of 43.79 ± 3.39 nm, while zinc oxide had a D95 value of 91.48 ± 10.10 nm. These results indicate that both physical UV filters were within the nanoparticle size range. The relatively small particle size may facilitate more uniform distribution of the particles on the skin surface and contribute to improved interaction with ultraviolet radiation. The use of nanopowders may also reduce the whitening effect commonly associated with conventional physical UV filters containing larger particles.

Preliminary photoprotective evaluation showed that titanium dioxide and zinc oxide exhibited SPF values of 38 and 39, respectively. These results indicate that both nanopowders possess high UV-protective potential and are suitable candidates for use as physical UV filters sunscreen formulations. Their photoprotective activity is associated with the ability of titanium dioxide and zinc oxide to scatter and absorb ultraviolet radiation. Therefore, the particle size characteristics and preliminary photoprotective activity of both nanopowders support their incorporation as active ingredients in sunscreen cream formulations.

Physicochemical Evaluation of Sunscreen Cream



Figure 1. (a) Representative appearance of sunscreen cream formulations (F1, F2, and F3) after preparation, and (b) representative homogeneity of the sunscreen cream formulation.

Three sunscreen cream formulations, namely F1, F2, and F3 were prepared using different concentrations of titanium dioxide and zinc oxide nanopowders. The organoleptic and homogeneity characteristics of the formulations are presented in Figure 1. All formulations exhibited a white appearance with a smooth semisolid consistency and showed no visible phase separation after preparation. Differences in the concentrations of titanium dioxide and zinc oxide did not produce noticeable changes in the physical appearance of the formulations.

The homogeneity evaluation demonstrated that all formulations had a uniform distribution of ingredients, with no visible coarse particles or aggregates. These findings indicate that the emulsification process was able to produce creams with good distribution of the nanopowders throughout the cream matrix. Homogeneity is an important characteristic of sunscreen preparations because uniform distribution of the active ingredients is required to achieve relatively consistent exposure of the skin surface to the photoprotective agents during application.

Table 2. Physicochemical Properties of Sunscreen Cream Formulations During Storage

Parameter	F1	F2	F3
Ph	5.01–5.29	5.01–5.55	4.84–5.83
Viscosity (cP)	4693–6083	4837–6453	5230–6203
Spreadability (cm)	5.03–6.17	5.03–6.10	5.83–6.23
Adhesion (s)	3.44–3.70	3.27–3.68	3.41–3.58
Emulsion type	Oil-in-water (O/W)	Oil-in-water (O/W)	Oil-in-water (O/W)
Heating-cooling stability	Stable (Cycle 1–6)	Stable (Cycle 1–6)	Stable (Cycle 1–6)
Phase separation	None	None	None

The physicochemical characteristics of the three formulations during storage are presented in Table 3. The pH values of F1 ranged from 5.01 to 5.29, F2 from 5.01 to 5.55, and F3 from 4.84 to 5.83. All values were within the physiological skin pH range of 4.5–6.5, indicating that the three formulations possessed pH values suitable for topical application. The changes in pH during storage were relatively small and did not cause the values to fall outside the

acceptable range for topical preparations. Such variations may be associated with interactions among formulation components and changes within the emulsion system during storage.

The viscosity values of F1 ranged from 4693 to 6083 cP, F2 from 4837 to 6453 cP, and F3 from 5230 to 6203 cP. Differences in viscosity among the formulations indicate that variations in nanopowder composition may influence the rheological characteristics of the creams. During storage, viscosity showed changes that may be associated with structural rearrangement within the emulsion network following preparation. Despite these changes, all formulations retained characteristics suitable for topical application and showed no evidence of physical emulsion breakdown.

The spreadability values ranged from 5.03 to 6.17 cm for F1, 5.03 to 6.10 cm for F2, and 5.83 to 6.23 cm for F3. All formulations were within the 5–7 cm range used as an acceptance criterion for semisolid topical preparations. Appropriate spreadability indicates that the cream can be readily distributed over the skin surface. Changes in spreadability during storage also showed a relationship with viscosity, whereby a decrease in viscosity tended to be accompanied by an increase in spreadability. This relationship indicates that the consistency of the formulation influences the ease of cream application and distribution on the skin.

The adhesion values of F1 ranged from 3.44 to 3.70 s, F2 from 3.27 to 3.68 s, and F3 from 3.41 to 3.58 s. These results indicate that the three formulations were able to maintain contact with the skin surface during the test period. Adequate adhesion is important for topical sunscreen preparations because sufficient contact with the skin surface may help maintain the formulation at the application site during use.

The methylene blue test confirmed that all three formulations were oil-in-water (O/W) emulsions. This emulsion type is suitable for sunscreen cream preparations because the aqueous external phase generally provides a less greasy sensory characteristic and facilitates removal with water. The heating–cooling test showed that all three formulations remained stable throughout six cycles without phase separation. These findings indicate that the emulsion systems were able to maintain their physical characteristics when subjected to alternating temperature conditions.

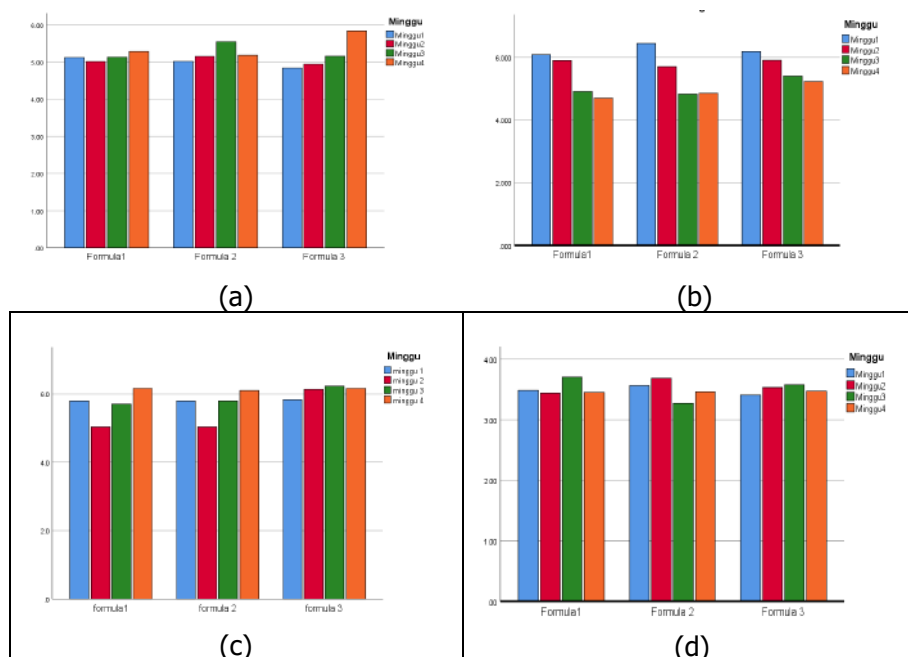


Figure 2. Changes in physicochemical properties of sunscreen cream formulations during four weeks of storage. (a) pH, (b) viscosity, (c) spreadability, and (d) adhesion.

Changes in pH, viscosity, spreadability, and adhesion during four weeks of storage are presented in Figure 2. Overall, the three formulations showed relatively small changes in physicochemical characteristics during the observation period. No physical changes indicative of emulsion instability were observed. These results demonstrate that all formulations were able to maintain acceptable physicochemical characteristics throughout the evaluated storage period.

Statistical Analysis of Physicochemical Properties

Table 3. Summary of Two-Way ANOVA Results for Physicochemical Properties of Sunscreen Cream Formulations

Parameter	Formula (p)	Storage time (p)	Interaction (p)
pH	0.761	0.014	0.077
Viscosity	<0.001	<0.001	<0.001
Spreadability	0.001	<0.001	0.014
Adhesion	0.886	0.623	0.017

The effects of formulation and storage time on the physicochemical characteristics of the sunscreen creams were analyzed using Two-Way ANOVA, and the results are presented in Table 4. For pH, formulation variation did not significantly affect the pH values ($p = 0.761$), whereas storage time had a significant effect ($p = 0.014$). The interaction between formulation and storage time was not statistically significant ($p = 0.077$). These findings indicate that changes in pH were more strongly associated with storage time than with formulation variation, although the observed changes remained within the acceptable range for topical application.

Viscosity was significantly affected by formulation, storage time, and the interaction between formulation and storage time, with $p < 0.001$ for all three factors. These findings indicate that both formulation composition and storage duration contributed to changes in the viscosity of the creams. The significant interaction indicates that the changes in viscosity during storage did not occur in the same pattern for all formulations. Nevertheless, these changes did not result in loss of the physical characteristics of the creams.

Spreadability was also significantly affected by formulation ($p = 0.001$), storage time ($p < 0.001$), and the interaction between formulation and storage time ($p = 0.014$). These results indicate that spreadability was influenced by formulation composition as well as changes in the formulation during storage. The changes in spreadability were consistent with changes in viscosity, as formulations with lower viscosity tended to exhibit greater spreadability. This finding indicates that the rheological characteristics of the cream contribute to its ease of application and distribution on the skin.

In contrast, formulation variation did not significantly affect adhesion ($p = 0.886$), and storage time also had no significant effect ($p = 0.623$). However, the interaction between formulation and storage time was statistically significant ($p = 0.017$). This finding indicates that adhesion was generally maintained during storage, although changes in adhesion in certain formulations were influenced by the combined effects of formulation composition and storage duration. Overall, the statistical analysis demonstrated that the three formulations maintained acceptable physicochemical characteristics throughout the evaluation period.

Photoprotective Activity

Table 4. Sun Protection Factor (SPF) and Broad-Spectrum Evaluation of Sunscreen Cream Formulations

Formulation	SPF Value	Protection Category	Critical Wavelength (λ_c) (nm)	Broad-spectrum
Base (F0)	1.52	No protection	–	No
F1	39.4	Ultra protection	–	–
F2	37.0	Ultra protection	–	–
F3	40.0	Ultra protection	383.35	Yes

The photoprotective activity of the three sunscreen cream formulations was evaluated using the Mansur spectrophotometric method, and the results are presented in Table 5. The cream base without active ingredients (F0) exhibited an SPF value of 1.52, whereas F1, F2, and F3 produced SPF values of 39.4, 37.0, and 40.0, respectively. All three formulations containing titanium dioxide nanopowder, zinc oxide nanopowder, and niacinamide exhibited SPF values above 30 and were classified as providing ultra protection according to the classification applied in this study. F3 exhibited the highest SPF value of 40.0, followed by F1 at 39.4 and F2 at 37.0.

The differences in SPF values among the formulations indicate that variations in the ratio of titanium dioxide and zinc oxide nanopowders may influence the photoprotective performance of the creams. F3, which contained a balanced concentration of titanium dioxide and zinc oxide, produced the highest SPF value among the formulations. This finding suggests that the combination of the two physical UV filters at this composition provided the highest UVB photoprotective performance under the experimental conditions.

Further evaluation of broad-spectrum protection was performed for F3 by determining its critical wavelength (λ_c). F3 exhibited a λ_c value of 383.35 nm. This value exceeded the 370 nm threshold used as the criterion for broad-spectrum protection. Therefore, F3 met the broad-spectrum criterion, indicating that the formulation provided protection extending across both UVB and UVA regions based on the SPF and critical wavelength evaluations.

Skin Irritation Test

A skin irritation test was performed using the closed patch method on four healthy volunteers to evaluate the tolerability of the sunscreen cream. The results are presented in Table 6. Neither the cream base nor F3 produced signs of skin irritation in any of the panelists following the observation period. No erythema, edema, itching, or other visible skin reactions were observed, and all observations were therefore classified as negative for irritation.

The absence of visible irritation indicates that Formula 3 was well tolerated by the skin under the experimental conditions. The pH of F3, which ranged from 4.84 to 5.83, was also within the physiological skin pH range. In addition, the use of titanium dioxide and zinc oxide as inorganic UV filters supports the topical characteristics of the formulation. Based on these findings, F3 demonstrated satisfactory physicochemical characteristics, photoprotective activity, and skin tolerability, indicating its potential for further development as a sunscreen cream.

Conclusion

This study successfully formulated sunscreen creams containing titanium dioxide and zinc oxide nanopowders in combination with niacinamide. All three formulations demonstrated acceptable physicochemical characteristics, including good appearance and homogeneity, skin-compatible pH, suitable viscosity, spreadability, and adhesion, as well as stability during the heating-cooling test. Among the tested formulations, F3 showed the highest SPF value of 40.0 and a critical wavelength of 383.35 nm, meeting the criterion for broad-spectrum protection. F3 also showed no signs of skin irritation under the experimental conditions. Further studies are recommended to evaluate long-term stability, in vivo photoprotective efficacy, and consumer acceptability to further support the development of the formulation.

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