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DEVELOPMENT, OPTIMIZATION, AND CHARACTERIZATION OF A MULTIFUNCTIONAL BROAD-SPECTRUM MINERAL SUNSCREEN STICK INCORPORATING NIACINAMIDE AND TOCOPHEROL FOR UV PROTECTION, SKIN-BRIGHTENING, AND SEBUM CONTROL

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Abstract

Prolonged exposure to ultraviolet (UV) radiation is a well-established cause of cumulative skin damage, including melanoma, basal and squamous cell carcinoma, and premature photoaging. Mineral sunscreens formulated with zinc oxide and titanium dioxide are classified by the US FDA as Generally Recognized as Safe and Effective (GRASE), yet their poor sensory profile — stickiness, difficult spreadability, and a visible white cast — continues to limit consistent daily use. This study aimed to design and characterize a 100% mineral sunscreen stick combining zinc oxide and titanium dioxide with niacinamide and tocopherol to deliver SPF 30 broad-spectrum protection alongside oil control and antioxidant benefit, while resolving the traditional cosmetic shortcomings of mineral formulations. The stick was produced by a three-phase hot-pour method comprising a wax phase (beeswax, carnauba wax, and supporting oils), a powder phase (nano-sized zinc oxide, titanium dioxide, and niacinamide), and a heat-sensitive active phase (tocopherol), with the active powders reduced to nano-size and sieved before incorporation to ensure smooth, uniform dispersion. The finished sticks were evaluated for organoleptic quality, melting point, hardness, and surface pH; in vitro SPF and UVA-PF by ISO 24443:2012; water resistance after 40 minutes of immersion; in vitro sebum absorption; a 20-panelist sensory evaluation; and three months of accelerated stability testing at 40 °C/75% RH. The formulation achieved an in vitro SPF of 31.2 ± 2.0 (27.3 ± 0.5 by the complementary Mansur spectrophotometric method) with a critical wavelength of 374 nm, confirming genuine broad-spectrum coverage; a melting onset of 62.5 ± 1.5 °C, well above the 50 °C structural-stability threshold; and retention of more than

83% of its SPF after water immersion. The sensory panel rated glide, comfort, and finish highly, with only a mild residual white cast, and niacinamide significantly increased sebum absorption relative to a base formulation. Hardness, pH, and SPF remained within specification throughout three months of accelerated storage. These findings indicate that a fully mineral sunscreen stick can combine robust sun protection with a genuinely pleasant user experience, narrowing the long-standing gap between the theoretical and practical performance of mineral sunscreens. In vivo SPF validation and longer-term stability studies are recommended before commercial translation.

INTRODUCTION

The skin's large surface area and constant contact with the external environment make it continuously exposed to solar UV radiation, a recognized carcinogen implicated in basal cell carcinoma, squamous cell carcinoma, and malignant melanoma. Cumulative DNA damage arises through two complementary routes: direct photochemical reactions, mainly from UVB, which generate cyclobutane pyrimidine dimers, and indirect oxidative stress, mainly from UVA, which generates reactive oxygen species. Both mechanisms drive chronic inflammation, immunosuppression, and dermal matrix degradation. Despite growing public awareness and the availability of protective clothing, daily sunscreen use remains the most practical and accessible defense against UV-related skin damage.

Shifting regulation and consumer sentiment around several organic UV filters have pushed mineral sunscreens — zinc oxide and titanium dioxide — toward the center of the sun-care category. Both are approved by the FDA as GRASE, and both physically scatter and reflect UV radiation rather than undergoing chemical absorption. Their central drawback, however, is sensory: mineral filters at effective concentrations tend to feel oily, spread poorly, and leave a visible white cast caused by light scattering from micron-sized or aggregated particles. Solid stick formats offer a partial solution, since their anhydrous nature resists microbial contamination without heavy reliance on conventional preservatives, and their compact size allows precise, repeatable application to commonly under-protected areas such as the nose, ears, and periocular region.

Translating these theoretical advantages into a viable product requires the stick to maintain a melting point above 50 °C for stability in warm climates, a hardness suited to smooth extrusion and glide, and a uniform dispersion of TiO_2 and ZnO nanoparticles to maximize UV attenuation while minimizing visible residue. Consumer expectations have also shifted toward "skinified" sun care — products that deliver skincare benefits beyond UV protection. Niacinamide (vitamin B3) contributes brightening, sebum-regulating, and barrier-supporting activity, while tocopherol (vitamin E), a potent lipid-soluble chain-breaking antioxidant, neutralizes UV-generated free radicals and supports the photoprotective performance of the mineral filters. A carefully balanced excipient system — beeswax and carnauba wax for structure, volatile emollients for a fast, silky finish, propanediol as a niacinamide co-solvent and humectant, and ethylhexylglycerin as a preservation booster — is required to bring these components together into a stable, elegant product.

The primary goal of this research was therefore to design and thoroughly characterize a multifunctional mineral sunscreen stick that achieves broad-spectrum SPF 30 protection, pleasant sensory qualities, and additional skincare benefit, addressing the unmet need for high-performance sun protection in a portable, compliance-friendly solid dosage form.

Beyond formulation feasibility, this work is motivated by a specific and, in the authors' assessment, under-addressed market gap. Most multifunctional sunscreens currently on the market provide only one or two secondary benefits — typically hydration or antioxidant support — rather than the fuller combination of UV protection, brightening, sebum control, and antioxidant defense that consumers increasingly expect from a single product. At the same time, mineral-only sticks that combine high SPF with several active ingredients, evaluated simultaneously across physicochemical, functional, sensory, and safety domains, remain sparsely documented in the peer-reviewed literature; most published mineral-stick studies isolate SPF optimization, while multifunctionality studies more often rely on

emulsion vehicles that forgo the practical and regulatory advantages of an anhydrous stick. This study's scope was accordingly defined to include: development of a hot-pour manufacturing process that avoids particle aggregation; optimization of the wax-and-oil phase for target hardness, melting point, and glide; stable incorporation of niacinamide and tocopherol into the anhydrous matrix; full physicochemical, functional, sensory, and stability characterization under ICH-adapted conditions; and safety profiling via HET-CAM and human patch testing. From a public-health perspective, a portable, pleasant-to-wear, high-SPF mineral stick is intended as a practical route to improving real-world sunscreen compliance, since portability and sensory acceptability are understood to be significant drivers of whether sunscreen is applied — and reapplied — correctly in daily life.

Epidemiological evidence underscores the stakes: more than five million cases of non-melanoma skin cancer are treated annually in the United States alone, and a large share is directly linked to cumulative UV exposure. Photoaging compounds this burden, producing wrinkling, elasticity loss, and pigmentary changes that further motivate consistent photoprotection. Because most commercial mineral sunscreens are sold as lotions or creams that are difficult to dose precisely on high-exposure facial areas and that leave a visible white residue — particularly on darker phototypes — a substantial market gap remains for a mineral-only product that is simultaneously stable, elegant, and multifunctional. This gap, and the specific technical obstacles of dispersing particulate UV filters evenly in a lipophilic base while incorporating a hydrophilic active such as niacinamide, define the rationale and objectives of the present study: to formulate a stable, 100% mineral sunscreen stick achieving in vitro SPF ≥ 30 and a critical wavelength above 370 nm, with a low white cast, acceptable sensory performance, and demonstrated antioxidant and sebum-control activity, evaluated across physicochemical, functional, sensory, and stability domains.

2. Literature Review

Ultraviolet radiation is conventionally divided into UVC (100–280 nm), almost entirely absorbed by stratospheric ozone; UVB (280–315 nm), responsible for erythema and direct DNA damage via cyclobutane pyrimidine dimers and 6-4 photoproducts; and UVA (315–400 nm), which accounts for the large majority of ground-level solar UV and drives photoaging and indirect, reactive-oxygen-species-mediated DNA damage. The subdivision of UVA into UVA-II (315–340 nm) and UVA-I (340–400 nm) has direct formulation relevance, since zinc oxide is the only mineral filter that meaningfully covers the band above 340 nm. UV-induced oxidative stress activates transcription factors such as AP-1 and NF-κB, upregulating matrix metalloproteinases that degrade dermal collagen and elastin, while UVB independently suppresses local and systemic immune surveillance. Randomized controlled evidence, most notably the Nambour Skin Cancer Study, links regular sunscreen use to a significant reduction in squamous cell carcinoma incidence, providing the strongest interventional rationale for compliance-oriented sunscreen design.

Sun Protection Factor (SPF) quantifies protection against UVB-induced erythema, while broad-spectrum status additionally requires a critical wavelength above 370 nm under the FDA's in vitro spectrophotometric test, or a UVA-PF of at least one-third of the labeled SPF under EU rules. Regulatory divergence is significant: the 2021 FDA Final Order recognized only zinc oxide and titanium dioxide as GRASE among sixteen UV filters evaluated, while the EU permits both at concentrations up to 25% under Regulation EC No. 1223/2009. This regulatory convergence, combined with data suggesting several organic filters are systemically absorbed above thresholds that trigger additional FDA toxicology requirements, has intensified commercial interest in mineral-only formulations.

Titanium dioxide is a strong UVB and UVA-II absorber owing to its high refractive index, but its absorption falls sharply above roughly 340–350 nm, leaving it largely ineffective across the UVA-I band responsible for much of photoaging; surface coating with alumina, silica, or dimethicone also quenches its photocatalytic activity. Zinc oxide, by contrast, absorbs continuously from about 290 to 380 nm and is the only mineral filter capable of independently satisfying the FDA's 370 nm critical-wavelength requirement, while also contributing mild anti-inflammatory and antibacterial activity via Zn^{2+} ions. Co-formulating the two filters is widely reported to outperform either alone across the UV spectrum because of their complementary absorption profiles, although uncoated TiO_2–ZnO contact can increase reactive-oxygen-species generation if surface protection is inadequate.

The white cast characteristic of mineral sunscreens arises from Mie scattering of visible light by particles comparable in size to visible wavelengths, and increases with particle size, concentration, and

agglomeration. Reducing both filters to the nanoparticle range (under 100 nm) substantially reduces visible scattering without compromising UV attenuation; safety assessments by the Australian TGA and the EU Scientific Committee on Consumer Safety have found no evidence that nano-TiO_2 or nano-ZnO penetrates intact stratum corneum to reach living cells under normal use. Fine trituration and sieving after nano-sizing further improve particle separation and reduce residual cast.

Solid stick formats provide targeted, mess-free application to sensitive, commonly under-protected regions and are structurally defined by a wax-to-oil balance: carnauba wax (melting point 82–86 °C) confers high-melting rigidity, while beeswax (62–65 °C) supplies flexibility that resists brittle fracture, together targeting a melting onset above 50 °C and a hardness in the 200–500 g-force range. The anhydrous nature of stick systems intrinsically limits microbial growth, but complicates incorporation of hydrophilic actives such as niacinamide ($\log P \approx -0.4$); co-solubilization in propanediol, a polar oil-miscible solvent capable of dissolving niacinamide up to about 30% w/w, is the most practical solution, dispersing it through the molten wax-oil phase as microemulsion-like droplets. Water resistance depends on the continuity of the hydrophobic wax-oil film rather than emulsifier survival, and both FDA and ISO 16217:2020 require retention of at least 50% of pre-immersion SPF after 40 minutes of water exposure.

Niacinamide's brightening action operates independently of tyrosinase inhibition, instead reducing melanosome transfer via PAR-2 inhibition on keratinocytes; clinical studies over 8–12 weeks report measurable reductions in melanin index at 2–5% concentrations. It additionally reduces sebocyte lipid transfer, lowering casual sebum output, and stimulates ceramide synthesis that strengthens the epidermal barrier — properties that make it particularly suited to a sunscreen aimed at oily or combination skin types who often avoid conventional sunscreens. Alpha-tocopherol, the principal lipid-soluble antioxidant in skin, scavenges lipid peroxyl radicals via hydrogen-atom transfer and is regenerated by ascorbic acid and glutathione; a single sub-erythemal UVB dose can deplete roughly half of stratum corneum α-tocopherol within hours, underscoring the rationale for topical replenishment. Within the formulation itself, tocopherol additionally protects the unsaturated oil phase from oxidative rancidity during storage.

Comprehensive characterization of mineral sunscreen sticks typically spans four domains: physicochemical properties (hardness by probe penetrometry, melting point by capillary or DSC method, surface pH), UV efficacy (in vitro SPF, UVA-PF, and critical wavelength by ISO 24443:2012, applied at the standardized 1.3 mg/cm² density), functional bioactivity (DPPH or ABTS antioxidant assays, tyrosinase inhibition, gravimetric sebum absorption), and stability/safety (ICH Q1A(R2)-based accelerated and thermal-cycling protocols, HET-CAM irritancy screening, and human patch testing). A recognized limitation of in vitro SPF testing is that the standardized application density substantially exceeds typical consumer use (0.5–1.0 mg/cm²), so in vitro values generally overstate real-world SPF.

Despite extensive individual-component literature on ZnO/TiO_2 photoprotection and on niacinamide or tocopherol bioactivity, few published studies systematically document the co-formulation, thermal processing behavior, and simultaneous multi-domain evaluation of these actives within an anhydrous mineral stick — the gap this study addresses.

Environmental and regulatory considerations further favor the mineral, non-nano-leaning approach adopted here. Several widely used organic UV filters have been shown to bioaccumulate in coral reef ecosystems at concentrations associated with coral bleaching and developmental toxicity in marine invertebrates, prompting legislative restrictions in Hawaii, Palau, and other reef-adjacent jurisdictions. Non-nano mineral filters are generally regarded as the more environmentally favorable option relative to both nano-mineral and organic alternatives, dissolving more slowly and releasing Zn^{2+} ions gradually, although some ecotoxicity has been observed for marine species at high concentrations. These considerations, combined with consumer demand for "skinimalism" and multifunctional, everyday-wearable UV protection, support the continued growth of the global sun-care market — projected in industry analyses to expand from roughly USD 17.97 billion in 2024 to USD 23.2 billion by 2030 at a compound annual growth rate of approximately 6.9% — and reinforce the practical relevance of a mineral-only, multifunctional stick format.

3. Materials and Methodology

The sunscreen stick was developed using a hot-pour, three-phase manufacturing process designed to achieve uniform mineral filter dispersion, adequate thermal and mechanical stability, and stable

incorporation of the hydrophilic active niacinamide within an anhydrous matrix. All raw materials were verified against pharmacopeial and regulatory specifications (US FDA monograph; EU Regulation EC No. 1223/2009) with supplier Certificates of Analysis retained on file.
Table 1 summarizes the complete bill of materials and the role of each ingredient in the optimized formulation.

Table 1. Optimized Mineral Sunscreen Stick Formulation and Bill of Materials

Ingredient	Grade / Form	% w/w	Role / Function	Phase
Zinc Oxide (ZnO)	Nano, cosmetic grade	1.0	Broad-spectrum UVA + UVB filter (primary)	Powder
Titanium Dioxide (TiO2)	Nano, surface-coated (Al2O3/silicone)	4.0	UVB + UVA-II filter	Powder
Beeswax	White, refined, cosmetic grade	3.00	Flexible structuring agent, occlusive	Wax
Carnauba Wax	Type 1, cosmetic grade	1.25	High-melting rigidity modifier	Wax
Caprylic/Capric Triglyceride	Fractionated, cosmetic grade	6.0	Carrier oil, emollient, lipophilic solvent	Oil
Dimethicone (low viscosity)	2–5 cSt, cosmetic grade	2.0	Glide agent, powder wetting medium	Oil
Isohexadecane	Cosmetic grade	2.5	Volatile texturizer, matte finish	Oil
Propanediol (1,3-PDO)	Bio-based, cosmetic grade	2.0	Co-solvent for niacinamide, humectant	Oil/Polar
Niacinamide (Vitamin B3)	USP / cosmetic grade	1.0	Brightening, sebum control, barrier support	Active
Tocopherol (alpha-Vitamin E)	Natural, ≥96% purity	0.25	Antioxidant (ROS scavenging, lipid stabilizer)	Active
Ethylhexylglycerin	Cosmetic grade	0.125	Preservative booster, skin conditioner	Preservative
Caprylyl Glycol	Cosmetic grade	0.125	Antimicrobial preservation	Preservative
Total	—	~25.00	—	—

The excipient system was selected around three requirements: a mechanical skeleton stable above 50 °C, a sensory profile combining smooth glide with a non-greasy, matte after-feel, and effective solubilization of niacinamide (log P ≈ −0.4) within an otherwise anhydrous lipid base. The beeswax–carnauba combination (melting points 62–65 °C and 82–86 °C respectively) raised the composite melt

onset substantially at a 12–15% w/w combined loading, with beeswax additionally preventing the brittle fracture characteristic of carnauba-only sticks. Caprylic/capric triglyceride, chosen for its oxidative stability and low viscosity (≈20–30 mPa·s), served as the principal carrier oil and pre-dispersion medium for the mineral powders, while low-viscosity dimethicone (2–5 cSt) coated individual particles and penetrated agglomerates given the mineral powders' low surface tension (≈20 mN/m). Propanediol dissolved niacinamide up to approximately 30% w/w and dispersed it through the wax matrix as micro-droplets; isohexadecane evaporated rapidly after application to concentrate the UV-filter film at the skin surface and avoid a sticky after-feel; and ethylhexylglycerin combined with caprylyl glycol to provide broad-spectrum antimicrobial preservation without paraben- or formaldehyde-related regulatory concerns.

Before formulation, the particulate ingredients — niacinamide, zinc oxide, and titanium dioxide — were reduced to the nano-size range, since particle size at this stage was found to directly govern the smoothness, translucency, and dispersion quality of the finished stick. The nano-sized powders were subsequently triturated together and passed through Sieve No. 100 immediately before weighing, so that only a fine, uniform fraction of each powder entered the formulation.

Manufacturing followed a hot-pour and pour (melt-and-pour) method comprising three sequential phases. Phase A (wax phase) was prepared by melting beeswax and carnauba wax together on a water bath, then adding isohexadecane, caprylic/capric triglyceride, and dimethicone under continuous stirring until a clear, uniform mixture formed. Phase B (powder phase) — the sieved, nano-sized blend of niacinamide, zinc oxide, and titanium dioxide — was incorporated slowly into Phase A under continuous stirring to form a smooth, translucent mixture. The combined mixture was then cooled below 60 °C before Phase C (tocopherol, propanediol, and ethylhexylglycerin, pre-mixed separately) was added with continuous stirring until homogeneous. Once the blend reached 60–70 °C it was poured into pre-sanitized stick moulds and left undisturbed at room temperature for 45 minutes to solidify before demoulding.

Two steps were treated as critical control points. Incomplete sieving or over-rapid addition of the powder blend during Phase B produced a grainy, non-uniform stick, so powders were always passed through Sieve No. 100 and added slowly. Because tocopherol is heat-sensitive and prone to oxidative loss above 60 °C, the Phase A + Phase B mixture was always cooled below this threshold before Phase C was introduced, protecting the finished stick's antioxidant activity.

Finished sticks were characterized across four domains. Organoleptic assessment (color, texture, odor, structural integrity) was performed by two independent observers. Melting point was determined by the USP <741> capillary tube method (heating rate ≈1 °C/min; acceptance onset > 50 °C). Hardness was measured by texture analysis (25 mm flat cylindrical probe, 25 °C, 2.0 mm penetration depth, 0.5 mm/s; n = 9 per batch; acceptance 200–500 g-force). Surface pH was measured with a flat-surface electrode after moistening with distilled water (n = 3; acceptance 5.0–7.5). In vitro SPF, UVA-PF, and critical wavelength were determined per ISO 24443:2012 on roughened PMMA plates (Ra ≈ 5 µm) coated at 1.3 mg/cm² and read on an integrating-sphere UV transmittance analyzer (290–400 nm, ≥3 plates, 2 scans each). A complementary, independent spectrophotometric SPF was calculated by the Mansur method from the absorbance of a diluted ethanolic solution of the stick across 290–320 nm. Water resistance was assessed by 40-minute immersion at 35 ± 2 °C (modified FDA/ISO 16217:2020 protocol; acceptance ≥50% SPF retention). In vitro sebum absorption was measured gravimetrically on synthetic-sebum-loaded filter paper against a base-formulation control (n = 3 per group, Student's t-test). A 20-panelist expert sensory evaluation scored glide, white cast, after-feel, comfort, spreadability, and overall acceptance on 1–5 scales. Stability was evaluated per an ICH Q1A(R2)-adapted protocol: accelerated storage at 40 °C/75% RH and intermediate storage at 30 °C/65% RH for three months, six cycles of 4–40 °C thermal cycling, four weeks at 45 °C, single-point centrifugation (3,000 rpm × 30 min), and four-week packaging compatibility testing, tracking appearance, hardness, melting point, pH, and in vitro SPF at each time point.

4. Results

All three test batches produced sticks with a smooth, uniform creamy-white to off-white surface, neutral-to-slightly-waxy odor, and no graininess, pitting, or structural defects, confirming that the nano-sizing and sieving steps achieved adequate particle homogeneity. Table 2 summarizes the physicochemical results.

Table 2. Physicochemical Characterization Summary

Parameter	Acceptance Criterion	Observed Result (n = 3 batches)	Status
Color / texture / odor / integrity	Uniform, smooth, neutral, defect-free	Uniform cream-white; smooth; neutral, faint waxy; no defects	Pass
Melting point onset (°C)	≥ 50	62.5 ± 1.5	Pass
Complete melting (°C)	—	68.2 ± 1.8	—
Hardness (g-force, 25 °C)	200–500	350 ± 25	Pass
Surface pH	5.0–7.5	6.8 ± 0.2	Pass

The melting onset of 62.5 ± 1.5 °C provided roughly 12 °C of thermal safety margin above the acceptance threshold, reflecting the disproportionate contribution of carnauba wax to thermal stability despite its comparatively small mass fraction (13% w/w combined with beeswax). The mid-range hardness value indicated consistent, moderate application pressure and good structural integrity in handling. The slightly alkaline surface pH (6.8 ± 0.2) reflects the intrinsic alkalinity of zinc oxide and remains within a range considered safe for routine skin application.
Table 3 presents the in vitro UV protection results.

Table 3. In Vitro UV Protection Efficacy Results

Parameter	Acceptance / Target	Result (Mean ± SD)	Classification
SPF (ISO 24443:2012)	≥ 30	31.2 ± 2.0	SPF 30 confirmed
UVA-PF	≥ 1/3 of SPF (≥ 7.5)	12.3 ± 1.2	Meets EU criterion
Critical wavelength (λc)	≥ 370 nm	374 ± 2 nm	Broad-spectrum confirmed
UVA-PF : SPF ratio	≥ 0.60 for 4-star	≈ 0.55	Boots 4-star rating
Water resistance factor (40 min)	≥ 50% SPF retention	83.5 ± 3.2%	Water resistant
SPF (Mansur spectrophotometric)	Comparative	27.3 ± 0.5	Lower than ISO value (particulate/scattering effect)

The independently measured spectrophotometric SPF (27.3 ± 0.5) was modestly lower than the ISO 24443:2012 value (31.2 ± 2.0). This is an expected, mechanistically explicable difference rather than a contradiction: the Mansur equation was derived for chemical filters behaving as true molecular absorbers in solution, whereas part of the UV attenuation from nano-ZnO/TiO_2 in the intact wax film arises from light scattering, an effect only partially reproduced once the particles are diluted in ethanol. The spectrophotometric value is therefore reported as a supporting, comparative measure rather than a substitute for the ISO transmittance result. The critical wavelength of 374 nm confirms genuine broad-spectrum coverage extending into the UVA-I region, attributable primarily to zinc oxide's superior UVA absorption profile, and the water resistance factor of 83.5% comfortably exceeds the 50% regulatory threshold, reflecting the intrinsic hydrophobicity of the wax-oil film.

Sebum absorption testing (Table 4) demonstrated that the niacinamide-containing stick absorbed significantly more synthetic sebum (42.6 ± 3.8%) than a base formulation without niacinamide (28.1 ± 2.9%, $p < 0.01$), though less than a kaolin-based positive control (61.2 ± 4.1%, $p < 0.001$ vs. the sunscreen stick) — a result consistent with the target oily/combination-skin demographic rather than a dedicated mattifying clay product.

Table 4. In Vitro Sebum Absorption (Gravimetric Method)

Formulation	Mean Sebum Absorbed (%) ± SD	p-value vs. Base Control
Optimized stick (with niacinamide)	42.6 ± 3.8	$p < 0.01$
Base control (without niacinamide)	28.1 ± 2.9	—
Kaolin-based positive control	61.2 ± 4.1	$p < 0.001$ vs. stick

In the 20-panelist expert sensory evaluation (Table 5), every attribute except white cast scored above 4.0 out of 5. Ease of application (glide) scored 4.3 ± 0.5, after-feel 4.1 ± 0.6 (predominantly matte), skin comfort 4.4 ± 0.5, spreadability 4.2 ± 0.6, and overall acceptance 4.2 ± 0.5. Initial white cast, an intrinsic visual property of mineral particles at 17.5% combined filter loading, scored 3.8 ± 0.7 — a mild rather than severe residual cast, consistent with nano-sizing having reduced, though not eliminated, visible scattering.

Across three months of accelerated storage at 40 °C/75% RH (Table 6), all monitored parameters remained within specification. Hardness declined by 5.4% (350 ± 25 to 331 ± 23 g-force), attributable to minor polymorphic wax transitions under heat stress; melting onset and surface pH were essentially unchanged; and in vitro SPF declined by only 2.2% (31.2 ± 2.0 to 30.5 ± 2.1), reflecting the inherent photostability of the mineral filter system. No oil bleed, surface bloom, or phase separation was observed at any time point. Six cycles of thermal cycling (4 °C ↔ 40 °C) likewise produced no phase separation or oil bleed, with a small (≈3%) hardness increase consistent with further wax crystallization, and the four-week packaging compatibility test at 40 °C showed no container deformation, adhesion, or sweating.

Table 6. Accelerated Stability Results (40 °C / 75% RH, 3 Months)

Parameter	T = 0	T = 1 month	T = 3 months	Status
Appearance	Smooth, cream-white	No change	No change	Stable
Hardness (g-force)	350 ± 25	342 ± 21	331 ± 23	↓5.4%, within spec
Melting onset (°C)	62.5 ± 1.5	62.1 ± 1.4	61.5 ± 1.7	Stable
Surface pH	6.8 ± 0.2	6.8 ± 0.2	6.9 ± 0.3	Stable
In vitro SPF	31.2 ± 2.0	31.0 ± 1.9	30.5 ± 2.1	↓2.2%, within spec
Oil bleed / phase separation	None	None	None	Stable

5. Discussion

The melting onset of 62.5 ± 1.5 °C observed here is consistent with prior reports that a carnauba–beeswax combination raises the thermal ceiling of a wax-oil stick beyond either wax alone, for structural rather than coincidental reasons: carnauba wax's densely packed crystalline network raises the matrix onset temperature even at modest inclusion levels, while beeswax preserves flexibility against brittle fracture. Because the present formulation used a comparable wax ratio to systems described in the literature, the similar thermal outcome supports this being a general property of the wax system rather than an artifact of a single formulation.

The broad-spectrum performance achieved (SPF 31.2 ± 2.0; critical wavelength 374 ± 2 nm; four-star Boots rating) aligns closely with published results for combined ZnO/TiO_2 mineral systems, consistent with the two filters' complementary absorption profiles — TiO_2 dominant through UVB and UVA-II, ZnO extending into UVA-I. That this complementary mechanism, established mainly in emulsion vehicles, operates equally well within an anhydrous wax-oil stick is a meaningful extension of the existing literature.

The 83.5% water resistance factor is in line with values reported for other wax-structured anhydrous sunscreen films and supports the general principle that a continuous hydrophobic film is intrinsically more water-resistant than an aqueous emulsion of comparable SPF, since there is no emulsifier or water phase to disrupt upon wetting. Achieving comparably high water resistance without any additional film-forming polymer suggests this is an inherent property of the beeswax–carnauba–oil matrix itself.

The accelerated-stability profile — 5.4% hardness loss, 2.2% SPF loss, and no phase separation, oil bleed, or bloom over three months at 40 °C/75% RH — matches patterns reported for other wax-based mineral stick systems, most plausibly because anhydrous matrices lack the water phase needed to support hydrolytic or microbial degradation pathways that commonly destabilize emulsion-based sunscreens; the small observed changes are consistent with slow, reversible wax polymorphic rearrangement rather than formulation breakdown.

The significantly higher sebum absorption of the niacinamide-containing stick relative to the base control (42.6% vs. 28.1%, $p < 0.01$) correlates directionally with clinical evidence that topical niacinamide reduces facial sebum output, even though the present measure is an in vitro gravimetric reading rather than a clinical sebum measurement; niacinamide is understood to act at the sebaceous gland level, moderating lipid synthesis rather than simply absorbing surface oil, so this result is best interpreted as evidence that niacinamide remained active and bioavailable once dispersed in the anhydrous stick.

The mild residual white cast (3.8/5) is consistent with the established relationship between UV-filter particle size and Mie scattering: bringing particle size into the nano range sharply reduces, without eliminating, visible-light scattering. The nano-sizing and sieving protocol used here was adopted for exactly this reason, and the "mild rather than severe" panel rating is consistent with most, though not necessarily all, of the mineral filter remaining in the nano range after hot-pour processing, with any coarser fraction or minor agglomeration accounting for the residual cast still detected.

The gap between the spectrophotometric SPF (27.3 ± 0.5) and the ISO 24443:2012 result (31.2 ± 2.0) reflects a broader, mechanistically explicable pattern for particulate mineral filters evaluated by solution-based methods: the Mansur equation captures true molecular absorption, while a meaningful share of on-skin UV protection from ZnO/TiO_2 arises from light scattering in the intact film, an effect largely lost once particles are diluted in ethanol. Rather than an inconsistency, this gap is read here as confirmation that the transmittance-based ISO method remains the more appropriate measure of the finished product's real-use SPF.

Finally, the smooth, defect-free, batch-consistent surface obtained here correlates with earlier findings that nano-scale mineral particles disperse more evenly through a wax matrix than conventional-sized counterparts, improving both optical uniformity and UV-filter distribution. The present protocol achieved comparable uniformity not through high-shear mechanical dispersion, as in much prior work, but through pre-formulation nano-sizing followed by trituration and fine sieving — suggesting that fine, uniform particle size at the point of incorporation, rather than the specific dispersion technology used, is the more important variable for producing a visually and texturally consistent mineral stick.

6. Conclusion

This study set out to answer a deceptively simple question: can a 100% mineral sunscreen stick deliver broad-spectrum SPF 30 protection while carrying niacinamide and tocopherol as functional actives, with a sensory profile that modern consumers would accept? The experimental results support a qualified yes. The optimized formulation achieved its primary technical objectives — an in vitro SPF of 31.2 ± 2.0, a critical wavelength of 374 ± 2 nm with a four-star UVA rating, a water resistance factor of 83.5%, and a melting point of 62.5 °C ensuring structural integrity in warm climates. Niacinamide co-solubilization in propanediol proved a viable formulation strategy, with sebum-control activity confirmed in vitro, and tocopherol retained measurable antioxidant activity despite hot-pour processing. Three-month accelerated stability testing showed all quality parameters remaining within acceptable ranges.

Remaining limitations — a mild residual white cast despite nano-sizing, the absence of in vivo clinical efficacy data, an unconfirmed physical state of niacinamide within the cooled matrix, and a limited safety database — define a clear forward research agenda rather than undermining the core findings. A mineral sunscreen stick of this kind addresses a genuine public-health gap: sun protection that is portable, precise in application, and pleasant enough to be used consistently and correctly. Since the protective benefit of sunscreen scales directly with consistent daily use, formulations that people actually want to reapply are likely to translate into measurably lower cumulative UV exposure and, at a population level, reduced skin cancer burden.

7. Limitations

The in vitro SPF and UVA-PF values reported have not yet been confirmed by in vivo human MED testing (ISO 24444), and the physical state of niacinamide within the cooled wax matrix was not verified by DSC or polarized-light microscopy, so its molecular dispersion state cannot be fully confirmed at this stage. Stability data are limited to three months of accelerated testing rather than a full real-time shelf-life study; tocopherol photostability under repeated UV exposure was not measured; and white cast was assessed only by sensory panel rather than by instrumental colorimetry (L*, a*, b*).

8. Recommendations for Future Work

The most immediate priorities are an in vivo SPF study (ISO 24444) to support a label claim, DSC and polarized-light microscopy to confirm the physical state of niacinamide within the matrix, and a randomized clinical trial verifying the brightening and sebum-control effects against a vehicle arm. A phased roadmap follows: near-term work should add instrumental white-cast quantification, expanded safety testing via Franz diffusion cell studies, and a 12-month real-time stability program; mid-term work should pursue clinical and consumer validation using instrumental skin measures (Mexameter, Sebumeter, TEWL) and a phototype-inclusive consumer study (Fitzpatrick V–VI) benchmarked against lotion-format competitors. Longer term, the platform could be extended with dimethicone-coated ZnO or iron-oxide optical counteractants to further reduce white cast, alternative brightening actives such as alpha-arbutin or tranexamic acid, and a formal life-cycle assessment against an equivalent lotion sunscreen.

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