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"Multi-benefit holistic approach tailored for Brazilian market necessities: Hybrid Sunscreen & Make-up product"

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1. Introduction

Tinted sunscreens have surged in popularity within the Brazilian market, dominating pharmacy sales in 2023. This trend reflects consumer demand for products that combine sun protection with aesthetic benefits [1]. However, achieving optimal broad-spectrum protection alongside desirable makeup attributes, especially in Brazil's hot and humid climate, remains a challenge. This paper details the development of a novel tinted sunscreen designed to address this need. Through strategic formulation, incorporating an inverse emulsion, optimized ingredients, and components derived from Brazilian biodiversity, a high-performance product meeting photoprotection regulatory requirements while delivering a superior sensory experience and long-lasting coverage was created. The following sections will outline the specific formulation strategies and testing methodologies employed.

2. Materials and Methods

Formula development and stability

Formula development were done in a bench scale using MaxiLab OLSA 5kg mixture reactor (OLSA, MascoGroup®). Prototypes stability were conducted in a 100 mL glass jars, volume filled with 80% of its capacity at a climate chamber (ICH, Memmert©) during 2 months in different temperature conditions. Microscopic analysis were done in a Optical Microscopy (Carl Zeiss, model Axio Imager M2 Zeiss).

Sun Protection Factor (SPF) and Water Resistance & UVA-PF factor (UVA-PF) determination

To determine the Sun Protection Factor (SPF) of formula and its water resistance a single-blind, controlled clinical study in accordance with international standards (ISO 24444:2019, AS/NZS 2604:2021, ISO 16217:2020, ISO 18861:2020) was conducted with eleven female subjects (18-64 years old). Additionally, following ISO 24442:2022, a single-center, open-label, randomized study measured the UVA-PF of formula on ten subjects (19-61 years old, 7 female).

Assessment of the protective effect on visible light induced pigmentation

To assess the protective effect of formula on visible light-induced skin pigmentation, a clinical study with twenty subjects (15 female), Fitzpatrick skin phototypes III-IV (22-50 years old) was peformed. Study design was monocentric, double-blind and randomized. Formula and control were applied (2 mg/cm²) to eight test zones (8 x 2.5 cm) on the back (igually divided into exposed and non-exposed areas). Skin color colorimetric parameters (Lab*) were measured using Konica Minolta Chromameter® CR 400 and the Individual Typology Angle (ITA°) and Delta E were calculated based on these measurements. This assessment was done at baseline and multiple timepoints during 5 days, following visible light exposure using an ORIEL simulator (92292-1000, 144 J/cm² - equivalent to one hour of midday sun exposure at 45° latitude). VL-PF was calculated based on the slope of the change over time in Delta ITA° from Day 1 to Day 5, using the formula: VL-PF = (Slope_untreated) / (Slope_product).

Preclinical evaluation of formula on prevention against Infrared-A (IR-A) radiation effects

Preclinical *in vitro* study to determine the formula's restorative activity on the extracellular matrix by measuring Matrix Metalloproteinase-1 (MMP-1) production in human skin explants culture subjected to IR-A radiation. Skin fragments were divided into control, IR-A only, and product + IR-A groups. Product (2 mg/cm²) was applied 48h before IR-A exposure (360 J/cm²) using a Hydrosun 750 HBM1 (Hydrosun Medizintechnik GmbH) device. Culture supernatants were collected and MMP-1 was measured via sandwich ELISA assay kit (R&D Systems).

Long lasting coverage, color durability, film resistance and coverage under extreme conditions

This non-comparative study enrolled 22 women (18-64 years old, Fitzpatrick skin phototypes II-III) with facial imperfections. A 600 mg dose was applied to the entire face and images were captured at baseline (T0), 15 minutes post-application (Timm), 6, 10, and 12 hours post-application (T6h, T10h, T12h). Coverage and color durability were analyzed within a defined region of interest (ROI) using the FrameScan® "Morphology of Spots – Blue Channel Filter" module (parameter: Total Surface). Decreases in Total Surface indicated improved coverage and product retention. Cross-polarized images were used for durability assessment.

Film resistance and coverage under extreme conditions

This open-label, comparative, randomized study enrolled 21 women (20-49 years old, Fitzpatrick skin phototypes I-III, high sebum) prone to facial perspiration. A 315 mg dose was applied to one randomized half-face, with the other half serving as a control. Images were taken at baseline (T0), 15 minutes post-application (Timm), after sauna (40°C ± 1°C, 75% ± 5% RH), after cold room exposure (21°C ± 1°C, 45% ± 5% RH) and after light physical activity. FrameScan®'s colorimetric module was used to quantify UV film formation and resistance on the treated half-face, with T0 and Timm as references. Coverage and its resistance under these conditions were analyzed as described in previous study, using the "Morphology of Spots" module.

Design of experiments for fillers concentration definition

A design of experiments (DoE) approach, employing internal software, was utilized to investigate the impact of Silica and Silica Silylate concentrations on product shine and rheological properties. Ten experiments were conducted, varying the concentrations of the two components across five levels each. Prototypes were formulated to have a fixed total amount of fillers, in this way Aluminum Starch Octenylsuccinate was used to balance the remaining formulation when necessary. Prototype shine was assessed by applying 50 mL of formula to a 30 cm² delimited area on a SkinFx synthetic skin substrate (Axon' Cable).

Application was standardized using 10 seconds of circular spreading moviments followed by zig-zag motions in two directions to ensure a homogenous film. After drying at 36°C in a heat plate for 15 minutes, shine was assessed using a LightCam image analyzer (Newtone Technologies) through analysis of shininess gloss (GS) parameter that represents the average shininess level of all the pixels indetified as shiny (0-100 scale).

Rheological behavior was determined using an Anton Paar MCR302 rheometer equipped with a CP150-1/S cone-plate measuring system. Strain sweep tests were performed at 25°C, measuring storage modulus (G') and loss modulus (G") as a function of deformation rate (0,01-1000).

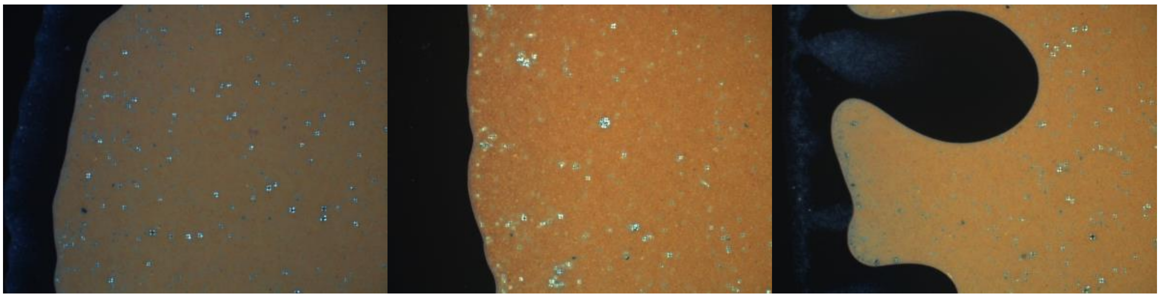
Consumer perception validation test

A sequential monadic incomplete block with 4 products (two prototypes of our investigational product and two main market benchmarks) were done to validate the performance of proposal technology by consumer point of view. Samples were tested at home by 60 volunteers during

5 days. Test design counts with women from 18 to 54 years old from Rio de Janeiro, Brazil, majority with oily skin. 24 attributes from 6 different categories were evaluated by them and student test were apllied to determine main differentiations between prototypes and bench-marks.

3. Results and Discussion

A stable water-in-oil (w/o) emulsion was developed to suspend high pigment and filler loads, utilizing PEG-30 Dipolyhydroxystearate and Octyldodecyl Xyloside as surfactants, and Di-asterdimonium Hectorite as a stabilizer. Prototypes exhibited excellent stability over two months under three distinct temperature conditions, with no discernible changes in macrscopic appearance, odor, color or viscosity. The formulation readily adapted to various shades, in-corporating different iron oxide/titanium dioxide ratios without impacting stability. A blend of five emollients (Diisopropyl Sebacate, Dicaprylyl Carbonate, Propylene Glycol Dicaprylate/Di-caprate, Dicaprylyl Ether, and C15-19 Alkane) was carefully selected based on their physical-chemistry characteristics – such as polarity and average molecular weight – to provide a non-greasy, lightweight texture while maintaining UV filter solubility, prioritizing biodegradability and bio-based origins [2]. Optical microscopy confirmed UV filter solubility, with no crystalliza-tion observed, even at low temperatures (Figure 1), ensuring consistent sunscreen perfor-mance and preventing inaccuracies in advertised SPF values [3].



(a) (b) (c)

Figure 1. Optical microscopy magnified 10x, polarized image for 2 months (a) low temperature, (b) room temperature and (c) high temperature

SPF, UVA and other light spectrum protections

A minimalist, yet effective, sunscreen system comprising three organic filters – Diethylamino Hydroxybenzoyl Hexyl Benzoate (DHHB), Drometrizole Trisiloxane (DTS), and Ethylhexyl Tri-azone (EHT) (10.75% total formulation weight) – was developed to provide broad-spectrum protection minimizing undesirable sensory effects. Precise filter concentrations were deter-mined via iterative testing to achieve spectral homeostasis [4]. This approach, avoiding inor-ganic filters due to their potential for an undesirable white cast on darker skin tones [5], re-sulted in an average SPF of 72.4, a water resistance of 54.2%, and a UVAPF of 24.2. While

UVB and UVA protection are essential, they are not sufficient to provide comprehensive protection against sun damage, particularly considering the diversity of skin tones. Passeron et al. (2021) highlight that while darker skin may offer inherent protection against UVB, it is more susceptible to hyperpigmentation induced by visible light (VL), especially high-energy visible light (HEV) and UVA. Therefore, protection against VL, particularly HEV, is crucial for individuals with darker skin tones. Tinted sunscreens containing iron oxides and/or pigmentary titanium dioxide offer this protection, and color-matching these products to the user's skin tone is essential for maximizing both efficacy and user acceptance [6].
Our formulation demonstrated significant visible light (VL) protection, with statistically significant differences in Delta Individual Typology Angle (ITA°) between the untreated and product-protected zones observed from day 3 onwards, as illustrated in Figure 2. Furthermore, calculating the parameter (1 - 1/(VL-PF)) * 100, which represents the protection factor as a percentage ranging from 0% (untreated skin) to 100% (a completely opaque screen), yielded a VL protection factor of 46%, the double when compared to regular sunscreens results from internal research.

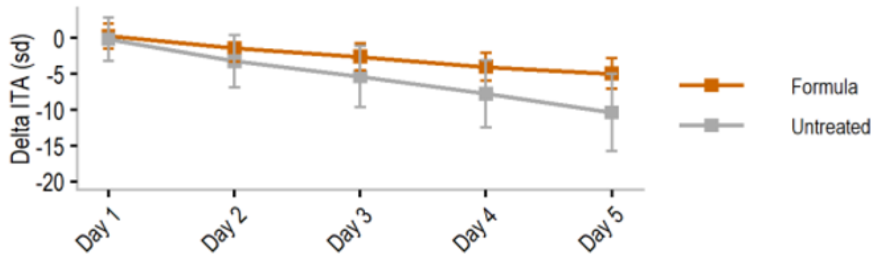


Figure 2. Mean values of Delta ITA° over time of untreated and treated zones

Infrared (IR) radiation can induce thermal effect, which can be beneficial or harmful depending on the dose, and oxidative damage, primarily associated with infrared-A (IR-A). IR-A radiation (760-1500 nm) penetrates deeply into the skin, with 35% absorbed in the epidermis, 48% in the dermis, and 17% in the subcutaneous tissue [7]. To address this, the formulation incorporates two antioxidants, Tocopherol and Dilauryl Thiodipropionate, to combat oxidative stress. As shown in Figure 3 and consistent with existing literature, IR-A radiation significantly increased matrix metalloproteinase-1 (MMP-1) production compared to the non-irradiated control (135.64%; P<0.01), contributing to photodamage. Conversely, our formulation demonstrated a protective effect, significantly reducing MMP-1 production compared to the IR-A group (64.63%; P<0.01).

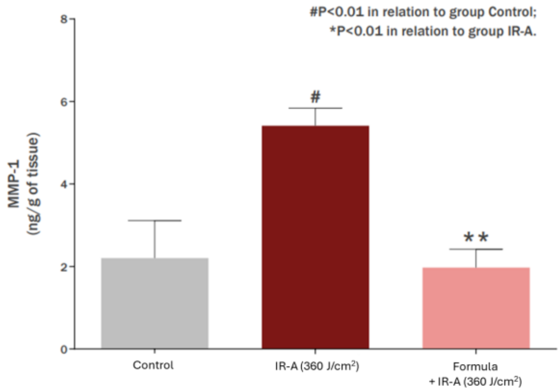


Figure 3. Effects of formula on the production of MMP-1 in human skin culture subjected to IR-A radiation. Data represents the average ± standard error of mean

Coverage, resistance, sensoriality and consumer perception validation

Beyond sun protection, aesthetic characteristics such as coverage and its durability are also crucial, given the product's dual function as both sun care and makeup. Therefore, coverage, long-lasting wear, and the resistance to heat and humidity of both color and coverage were evaluated.

Coverage assessment, conducted using software-based image analysis, revealed a statistically significant reduction in total surface area of imperfections at time points Timm, T6h, T10h, and T12h compared to the initial time point T0. This indicates effective coverage of skin imperfections (heterogeneous areas). Moreover, the lack of significant differences between Timm and the subsequent time points demonstrates that coverage was maintained for a full 12 hours. This confirms the long-lasting coverage claim – as illustrated in Figure 4.

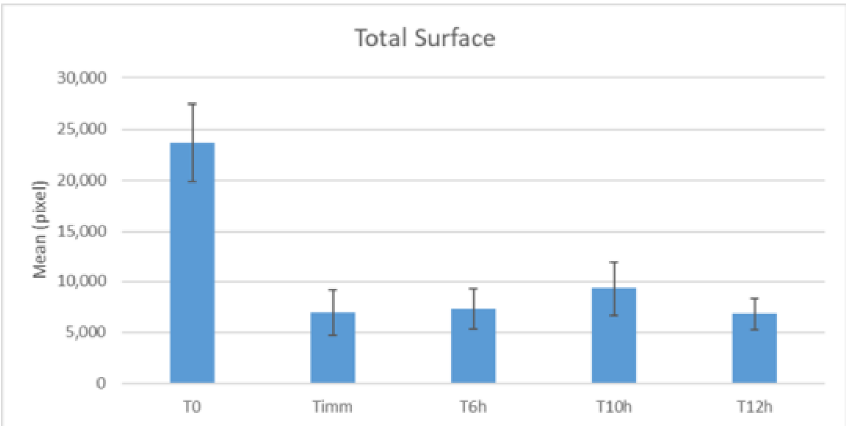


Figure 4. Average of total surface and its standard error per time point of analysis

The product demonstrated excellent durability and color resistance, with a minimal reduction in color intensity over time. Image analysis (Figure 10) revealed a maximum reduction of only 1.5% after 10 hours, confirming the long-lasting wear properties of the formulation.

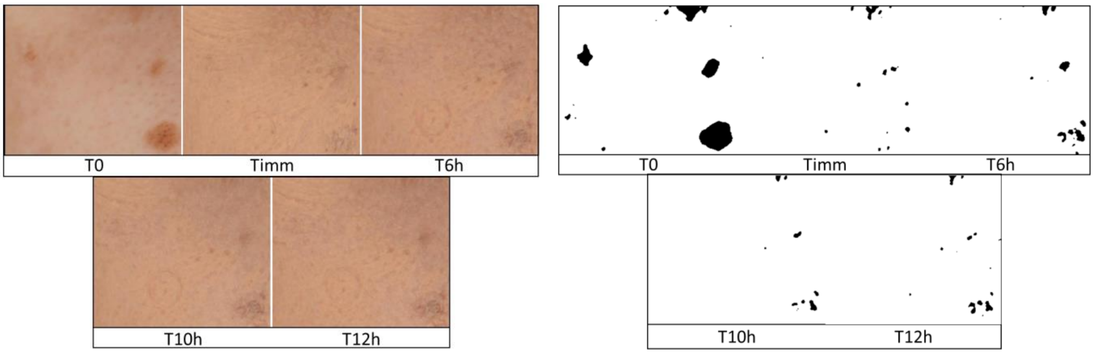


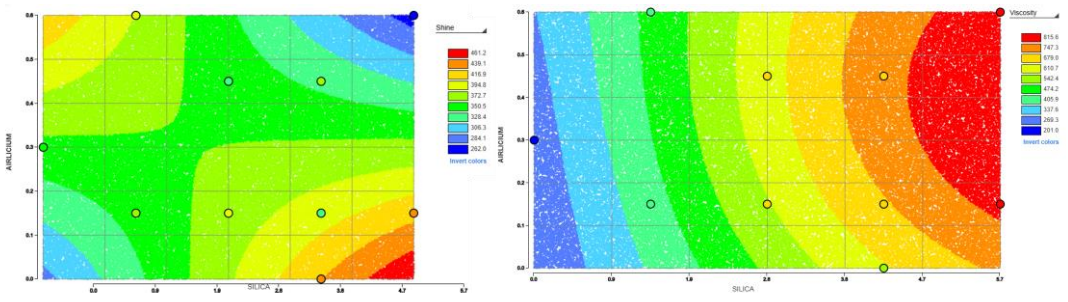
Figure 5. Image analysis of coverage of product and its durability over investigational time points

Furthermore, the product demonstrated remarkable resistance to extreme environmental conditions. Subjects exposed to a heat sauna and cold room showed no statistically significant difference in the total surface area parameter at time points Thamman (post-sauna) and Tcold-room (post-cold room) compared to Timm (immediately post-application). This indicates the film's resilience to both heat and humidity, crucial attributes given Brazil's climate.

This impressive performance can be attributed to the carefully selected film-forming polymers: Copernicia Cerifera (Carnauba) Wax and Polyamide-8. A synergic combination that creates a flexible and adherent film that can be related to their differing molecular weights, melting and gelling points, polarities, and capacity to form three-dimensional structures.

Design of Experiments (DoE) study revelead the dependence of shine and texture in terms of Silica Silylate and Silica concentration. The Gs parameter directly correlates with the product's residual shine on the skin; higher Gs values indicate a shinier appearance. Texture was assessed by analyzing the storage modulus (G') of each experimental formulation in its quasi-stationary state (at a shear deformation of 0.01%), providing a measure of viscosity/thickness before any textural changes occur.

The shine data were best fitted using a linear model, while a neural network model provided the optimal fit for the rheological data. Surface graphs illustrating these two outputs are presented in Figure 6.



(a) (b)

Figure 6. DoE Surface of response for shine (a) and surface of response for viscosity (b)

The analysis shows that both Silica and Silica Silylate contribute significantly to shine control, with both influencing the observed response. Conversely, Silica appears to play a more dominant role in determining viscosity. Analysis of the level curves on the response surface demonstrates a more pronounced increase in viscosity from left to right than vertically, indicating a stronger dependence on the Silica concentration. This detailed analysis allowed for precise tailoring of the formulation to achieve the desired balance of mattifying and rheological properties.

Assay with proportion 91% Silica and 9% of Silica Silylate exhibited the best shine control, therefore it was selected for consumer validation and designated as prototype A. However, its high viscosity prompted the creation of a modified prototype, named as prototype B, with a reduced Silica concentration (38,5% Silica, 3,8% Silica Silylate, and 57,7% Aluminum Starch Octenylsuccinate). This adjustment aimed to assess whether the thickness of the texture would directly impact sensory parameters.

The final consumer validation stage involved evaluating the perceived performance of two mentioned prototypes (A and B) against internal and external market benchmarks of tinted sunscreens. These prototypes differed only in their filler composition, as previously detailed.

Figure 7 presents the overall consumer perception results, demonstrating that both prototypes achieved at least parity performance against the benchmarks. However, Prototype B notably outperformed Prototype A, particularly against the internal benchmark. Consumers perceived superiorities in attributes such as quick absorption, low stickiness, minimal oiliness, long-lasting coverage, and other key characteristics.

Although Prototype A theoretically offered better shine control, the textural impact of its higher silica content may have negatively impacted the overall user experience, leading to lower consumer preference compared to Prototype B when both were evaluated against the benchmarks. This highlights the importance of balancing performance characteristics with sensory attributes for optimal consumer acceptance.

Product Performance

		Versus Internal benchmark		Versus external benchmark	
	Fla. Number	Prototype B	Prototype A	Prototype B	Prototype A
	Base: total respondents	60	60	60	60
KPI (Key performance indicator)					
KPIs	Overall liking (T2B)	≈	≈	≈	≈
	Comparison to Usual (T2B - B2B)	+	≈	≈	≈
Detailed attributes					
BEFORE APPLICATION	Texture liking (T2B)	≈	≈	≈	≈
DURING APPLICATION	Spreads evenly on my skin (T2B)	≈	≈	≈	≈
AFTER APPLICATION	Texture (thin / thick) - <% JAR>	≈	≈	≈	≈
	Quick absorption (slow / quick) - <% JAR>	+	≈	≈	≈
	Stickiness (T2B)	+	+	≈	≈
	Scent liking (T2B)	≈	+	≈	≈
	Matches with my skin tone (T2B)	≈	≈	≈	-
	Compatible with make-up (T2B)	+	≈	≈	≈
SHORT TERM EFFECT	Skin oiliness right after application (little / much) - <% JAR>	+	≈	+	≈
	Hydration of skin right after application (T2B)	≈	≈	≈	≈
	Instant feeling of comfort on the skin (T2B)	≈	≈	≈	≈
	Conceals imperfections well (T2B)	≈	≈	+	≈
	Does not mark pores/expression lines (T2B)	+	≈	+	≈
EFFECT	Makes skin softer (T2B)	≈	≈	≈	≈
	Let skin breathe (T2B)	+	≈	≈	≈
	Liking overall make-up result (T2B)	≈	≈	≈	≈
	Shininess (shiny / opaque) - <% JAR>	+	≈	≈	≈
	No transferring (T2B)	+	≈	≈	≈
	Matches with make up routine (T2B)	+	≈	≈	≈
	Keeps the skin's natural radiance (T2B	≈	≈	≈	≈
DAY-LONG EFFECT	Makes skin feel comfortable all day long (T2B)	≈	≈	≈	≈
	Long-lasting coverage (T2B)	+	+	≈	≈
	No oiliness along the day (T2B)	≈	≈	≈	≈
	No melting along the day (T2B)	≈	≈	≈	≈

Figure 7. Overall perceived results by consumer evaluation in quantitative test (80 total responses)

4. Conclusion

This research successfully developed a high-performance, multi-benefit tinted sunscreen tailored for the Brazilian market. The optimized water-in-oil emulsion exhibited excellent stability under various temperature conditions. The sunscreen provided robust broad-spectrum protection, with average SPF 72.4, UVAPF 24.2, and visible light protection of 46%, along with significant IR-A protection (64.63%). The carnauba wax/polyamide-8 film ensured long-lasting coverage and color durability (12 hours, 98.7% product remaining). Furthermore, a DoE approach optimized shine control and texture, resulting in superior consumer-perceived performance. This holistic strategy, combining robust sun protection, desirable makeup attributes, and a focus on sustainability through bio-based ingredients, effectively addresses the increasing demand for multifunctional cosmetic products.

Conflict of interest

All employees work at L'Oréal.

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