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Development of Natural-Based Sunscreen Product

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Abstract

This research aimed to develop an environmentally friendly natural-based sunscreen formulation in the form of a hybrid sunscreen comprising physical sunscreen agents, natural sunscreen agents, and natural SPF boosters. The study utilized a physical sunscreen base and incorporated three natural sunscreen agents: ethyl ferulate, castor oil, and coffee oil, each at 1-2% in the formulation. The emulsifier was adjusted between 2-3% of the total formulation to select the most stable composition for subsequent *in vitro* evaluation of sun protection efficacy. The results revealed that the physical sunscreen base formulation, containing 8% ZnO dispersion and 15% TiO_2 dispersion, exhibited a sun protection factor (SPF) of 33.03 ±3.87. Upon the addition of ethyl ferulate, castor oil, and coffee oil, each at 1%, the SPF increased to 59.49±9.04, representing a 80.01% enhancement. Furthermore, when ethyl ferulate, castor oil, and coffee oil were added at 2% each, the SPF rose significantly to 107.80 ±13.19, marking a substantial 226.46% increase from the base formulation. The stability tests conducted on the formulations with centrifugal stability testing at 4,500 rpm at room temperature for 30 minutes and heating cooling cycle stability testing. The result revealed no phase separation.

Keywords: Natural-Based Hybrid Sunscreen, Ethyl Ferulate, Castor Oil, Coffee Oil, SPF Booster

Introduction

The growing consumer concern about the environmental impact of cosmetic products has led to a significant shift in product selection criteria. In addition to safety and the absence of harmful chemicals, consumers now prioritize the environmental sustainability of cosmetic products used in their daily lives (Rocca et al., 2022). This trend is particularly evident in the sunscreen product category, where consumers demand both safety and environmental responsibility. As a result, regulatory bodies worldwide have responded by banning various synthetic chemical sunscreen agents due to their detrimental environmental effects (Hollert, 2020).

Physical sunscreen agents, such as zinc oxide (ZnO) and titanium dioxide (TiO_2), are generally considered safe and eco-friendly alternatives to synthetic chemical sunscreens. However, their use in natural and sustainable sunscreen formulations is limited by the high concentrations required to achieve a high sun protection factor (SPF) value. These high concentrations often result in thick, difficult-to-spread textures and white cast residue, which are undesirable to consumers (Tortini et al., 2022). On the other hand, natural sunscreen agents have gained popularity due to their antioxidant properties and UV protection capabilities. Nevertheless, their SPF values are typically lower compared to synthetic and physical sunscreen agents (Couteau et al., 2009), which hinders their competitiveness in the market.

The challenges in developing natural-based sunscreen formulations that simultaneously address safety, environmental friendliness, and consumer expectations have prompted researchers to explore innovative solutions. A review of the literature suggests that the SPF efficacy of natural-based sunscreens can be enhanced through hybrid formulations. These formulations strategically combine physical sunscreen agents (e.g., ZnO and TiO_2 with varying particle sizes for broad-spectrum UV protection) that provide UV protection via light scattering mechanisms, with natural sunscreen agents that absorb UV radiation and exhibit antioxidant properties. Furthermore, optimization strategies, such as selecting appropriate physical sunscreen agent types and particle sizes and incorporating SPF boosters like waxes that form lipid films and enhance SPF performance, can be employed to improve the overall efficacy of natural-based sunscreens (Huynh et al., 2019; Punia et al., 2021).

The review of literature reveals the potential effectiveness of several natural substances as sunscreen agents. Ethyl ferulate, a stable compound, has garnered attention for its antioxidant and anti-inflammatory properties, effectively acting as a reactive oxygen species (ROS) scavenger. Its ability to protect against sunlight has been widely recognized, with commercial use in sunscreens being supported by multiple studies (Horbury et al., 2017). Further, research by Laszlo et al. (2015) highlights its ability to form feruloylated structured lipids when reacting with natural oils, which enhance SPF through a transesterification process. Castor oil and coffee oil are also noted for their sunscreen properties. Castor oil, containing ricinoleic acid, has a natural SPF of 5 and absorbs UV light with a maximum absorption at 270 nm, positioning it as a natural UV filter (Johnson, 2007). Moreover, castor oil's film-forming ability may contribute to increased sunscreen efficacy by creating a protective barrier. On the other hand, coffee oil contains chlorogenic acid, a cinnamate derivative that absorbs UV radiation and releases it in the form of longer-wavelength energy, reducing skin irritation. Its antioxidant properties also help to minimize UV-induced free radicals, thus potentially replacing synthetic sunscreen components (Saewan et al., 2015). Together, these natural substances provide a strong foundation for developing effective, environmentally conscious sunscreens.

This study aims to evaluate the *in vitro* photoprotection properties of natural-based sunscreen formulations that combine physical sunscreen agents (ZnO and TiO_2 in varying particle sizes) with natural sunscreen agents such as ethyl ferulate, castor oil, and coffee oil. By comparing different formulations, the research seeks to develop a high-performing hybrid sunscreen with SPF 50+ and PA+++ ratings. Additionally, the study aims to align with sustainability and Green Chemistry principles, ensuring consumer appeal by reducing white casting and enhancing suitability for daily use. The goal is to develop a prototype natural-based sunscreen that addresses both consumer demands and environmental concerns, contributing to the advancement of eco-friendly sunscreen formulations.

Methodology

1. Preparation of Sunscreen Formulation

This research focused on developing sunscreen formulations using exclusively ingredients derived from natural sources. The formulations were categorized into two distinct systems.

Table 1 Ingredients of all sunscreen creams.

Part	INCI Name	(%) w/w		Function
		Base	Vary formulas	
A	Squalane	2	1	Emollient
	Caprylic/Capric Triglyceride	0.5 - 2	0.5 - 2	Emollient
	C12-20 Alkyl Glucoside, C14-22 Alcohols	2 - 3	2 - 3	Emulsifier
	Zinc Oxide (And) Simmondsia Chinensis (Jojoba) Seed Oil (And) Jojoba Esters (And) Polyhydroxystearic Acid	8	8	Physical sunscreen agent
	Caprylic/Capric Triglyceride (And) Titanium Dioxide (And) Polyhydroxystearic Acid (And) Aluminum Stearate (And) Alumina	15	15	
	Castor Oil	-	1 - 2	Natural sunscreen agent
	Coffee Oil	-	1 – 2	
	Ethyl Ferulate	-	1 - 2	
	Rice Bran Wax	-	0 - 1	Wax/ SPF Booster

Table 1 (Continued)

Part	INCI Name	(%) w/w		Function
		Base	Vary formulas	
B	C15-19 Alkane	6.5	6.5	Texturing
	Silica	1.5	1.5	Texturing
C	Capryloyl Glycerin/Sebasic Acid Copolymer	1.5	1.5	Natural film former
D	GLYCERYL CAPRYLATE (AND) GLYCERYL UNDECYLENATE		1.5	Preservative
E	DI Water	q.s. to 100	q.s. to 100	Solvent
	Sodium Phytate	0.1	0.1	Chelating agent
	Sodium Cholride	1.5	1.5	Stabilizer
F	Glycerin 99.5	2	2	Humectant
	Xanthan Gum	0.5	0.5	Thickener
G	Pentylene Glycol	2	2	Preservative
	Total	**100**	**100**	

To prepare the sunscreen cream, the components A, B, C, D, E, F, and G were weighed. The ingredients of part B were mixed to form a silica gel and set aside at room temperature. Parts E and F (aqueous phase) were blended into a gel. Parts A and EF were heated to 80-90°C to induce the phase inversion temperature. Part EF was gradually added to part A while homogenizing at approximately 10,000 rpm for 1-2 minutes. Subsequently, parts B, C, and D were sequentially added to AEF while maintaining homogenization. The mixture was further homogenized for 3 minutes. The homogenization process was then switched to stirring at 500-700 rpm, and the mixture was cooled using a water bath for 5-10 minutes until it reached room temperature. Finally, part G was incorporated, and the cream was allowed to set for 24 hours.

2. Stability Assesment

2.1 Centrifugal Stability Testing: The stability of the developed sunscreen formulations was assessed using centrifugal stability testing. The products were subject to centrifugation at 4,500 rpm at room temperature for 30 minutes to observe any potential phase separation.

2.2 Heating Cooling Cycle Stability Testing: In addition to centrifugal stability testing, the sunscreen formulations underwent accelerated stability testing using heating-cooling cycles. The products were alternately exposed to low temperature (5°C in a refrigerator for 24 hours) and high temperature (45-50°C in an oven for 24 hours), constituting one complete cycle. Three cycles were performed, and key parameters such as pH, viscosity, and texture were monitored throughout the testing process.

3. *In Vitro* Study of Photoprotective Properties

3.1 UVB Protection (SPF) and UVA Protection Factors (UVAPF) Assessment and comparative evaluation of SPF values and UVA Protection Factors (UVAPF)

3.2 The overall UVA protection efficacy of the sunscreen formulations was evaluated by comparing UVAPF, PA, and BOOTS STAR ratings. These assessments were conducted in accordance with the guidelines accepted by the Thai Food and Drug Administration for product notification.

Results and Discussion.

1. Preparation of Sunscreen Formulas.

The emulsifier concentration was varied at 2 and 3 percent across the different systems, resulting in a total of 7 formulas as specify in Table 2. This was done to select the most stable formulation. The research study formulations contained equal amounts of physical sunscreen agents: 8% ZnO (KOBO-JOP80MZCJ, 75% micronized ZnO, 250 nm) and 15% TiO_2 (SOLAVEIL CT300, 33% nanoparticles, 74-100 nm). According to manufacturer data, 16% JOP80MZCJ is suggested for SPF 50, and 20-25% SOLAVEIL CT300 is recommended for SPF 50. To avoid high concentrations that could affect stability and texture, the formulations in this study used 8% JOP80MZCJ and 15% SOLAVEIL CT300, which were estimated to provide an initial SPF of at least 30.

Regarding natural sunscreen agents, a study by Laszlo et al. (2015) demonstrated that reacting ethyl ferulate with any natural oil through a transesterification process can form a lipid film structure called feruloylated structured lipids. Transesterification involves the reaction of plant or animal oils containing fatty acids with an alcohol, catalyzed by an acid or base, yielding ester products (Charoensukhon, 2021). The study found that transesterifying castor oil and ethyl ferulate in ionic liquids at 90°C formed feruloylated structured lipids, which exhibit properties that can enhance the SPF value (acting as SPF boosters). Although this research did not experimentally conduct the transesterification reaction of castor oil and ethyl ferulate, based on the literature review, the researchers hypothesized that combining the three natural sunscreen agents (ethyl ferulate, castor oil, and coffee oil) possessing UV protection properties, along with ingredients exhibiting film-forming and SPF-boosting abilities, such as castor oil, ethyl ferulate, and rice bran wax, would synergistically optimize the efficacy of the natural-based sunscreen formulation. Using high percentages of these agents to achieve a high target SPF value could destabilize the emulsion. Therefore, the researcher employed the lowest feasible concentrations, starting at 1% (1-2% w/w) for each natural sunscreen agent. Additionally, all formulations contained a film-former (Capryloyl Glycerin/Sebasic Acid Copolymer) to enhance SPF performance and silica to improve texture.

Table 2 Details of sunscreen cream formulations divided into 5 formulas

Formulation	(%)	(%) w/w		
No.	Emulsifier	Ethyl Ferulate	Castor Oil	Coffee Oil
No.1	2	-	-	-
No.2	3	-	-	-
No.3	2	1	1	1
No.4	3	1	1	1
No.5	3	2	2	2

Form the result, formulations with a 2% emulsifier concentration showed a lighter, more fluid texture than formulations with a 3%. This allowed for easier application and faster absorption on the skin, providing a fresh, lightweight feel. However, Formulas containing 3% emulsifier created a thicker, more emollient texture.

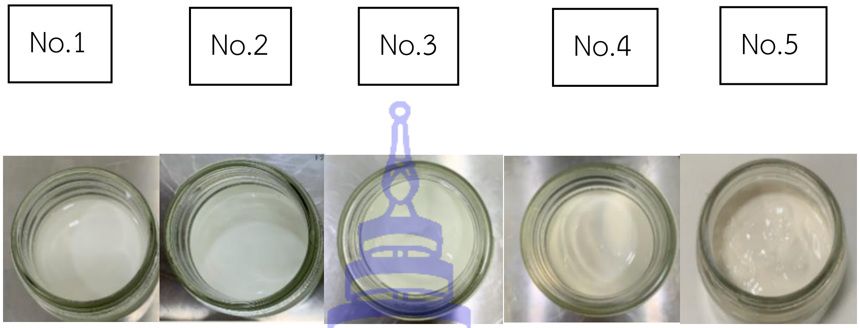

Figure 1 Physical Characteristics of All Sunscreen Cream Formulations

From Figure 1, Formula No.1 is the most lightweight and fluid, featuring a thin consistency that spreads easily on the skin. It contains 2% emulsifier and does not include any natural sunscreen agents, resulting in a simple and light texture. In comparison, Formula No.2 is slightly creamier than Formula No.1 due to its 3% emulsifier content, while still maintaining a lightweight feel. Like Formula No.1, it does not contain natural sunscreen agents, which contributes to its basic yet stable texture. Formula No.3 has a soft, creamy texture with a moderate thickness. It contains 2% emulsifier and includes 1% of each natural sunscreen agent (ethyl ferulate, castor oil, coffee oil). The addition of natural sunscreen agents (ethyl ferulate, castor oil, and coffee oil) slightly enriches the texture of the sunscreen, providing a light yet protective feel. These agents also contribute to a slight decrease in the pH value. However, despite this adjustment, all formulations maintain a similar range of pH values, as shown in Table 3. Formula No.4 has creamier and slightly thicker than Formula No.3, with 3% emulsifier and 1% of each natural sunscreen agent. The texture is stable and provides a more substantial layer on the skin. Formula No.5 has the thickest and richest texture among all formulas. It contains 3% emulsifier and 2% of each natural sunscreen agent, creating a dense, protective barrier. This formulation feels more substantial and offers enhanced skin conditioning and protection, making it suitable for extended sun exposure.

2. Stability Assessment

2.1 Centrifugal Stability Testing

The accelerated stability of all sunscreen formulas was assessed via centrifugation at 4,500 rpm for 30 minutes. Initially, all formulations displayed good homogeneity. Post-centrifugation, none of the formulations exhibited phase separation, indicating robust stability under accelerated conditions.

2.2 Heating-Cooling Cycle Stability

Stability testing under accelerated conditions employing a heating-cooling cycle was conducted by subjecting the sunscreen cream formulations to alternating low and high temperatures. The formulations were exposed to temperatures ranging from 5°C in a refrigerator for 24 hours, followed by 45-50°C in an oven for 24 hours (considered as one cycle). Three cycles were performed in total.

Table 3 pH value and viscosity of all formulas before/after heating-cooling

Formulation	pH ±SD		vicosity (CPs) ±SD	
	Before	After	Before	After
No.1	7.84 ±0.01	7.88 ±0.15	2067 ±5	2764 ±3
No.2	7.84 ±0.01	7.82 ±0.15	3514 ±6	4773 ±6
No.3	7.75 ±0.01	7.74 ±0.01	5120 ±5	5710 ±3
No.4	7.81 ±0.01	7.80 ±0.15	6113 ±3	6385 ±5
No.5	7.75 ±0.01	7.71 ±0.15	6224 ±4	6445 ±4

*Needle #0.4, RPM = 30, 10 seconds

As shown in Table 3, after undergoing three heating-cooling cycles, all sunscreen products exhibited slight changes in pH values. All formula showed a slight increase in pH after heating-cooling. However, the post-heating-cooling pH remained within the acceptable range for human skin and the suitable pH range for physical sunscreens (pH 6-8) (Smaoui, Slim et al., 2012). For viscosity, formula No.1 and No.2 showed a lightweight, fluid texture due to a lower emulsifier concentration and no natural agents resulted the lowest viscosity. Similar to No.3 and No.4, Formula No.4

has 3% emulsifier and 1% of each natural agent, with viscosity at 6113 ±3 CPs, giving it a thicker, stable feel sunscreen. Formula No.5 has the highest viscosity at 6224 ±4 CPs, with 3% emulsifier and 2% of each natural agent. This formula has the richest, highest viscosity value.

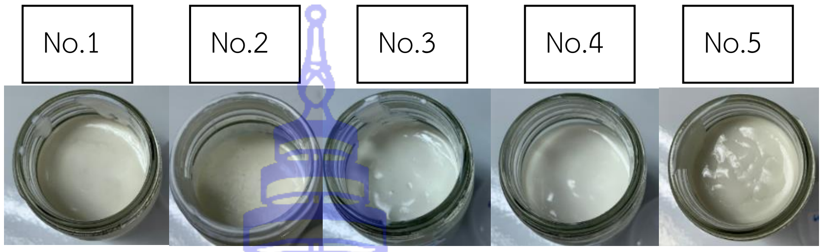

Figure 2 Physical characteristics of all sunscreen cream formulations after heating-cooling

After heating-cooling condition from figure 2, formulas No.1-3 observed slightly phase separation with oil floating on top. Thus, for the base formula, No.2 with 3% emulsifier was selected. Other formulations with 3% emulsifier (No. 4 and No.5) which had the most viscosity value and the creamier texture were also selected for further SPF and UVA-PF testing.

3. *In Vitro* Study of Photoprotective Properties

3.1 Evaluation of Sunscreen Protection Efficacy (SPF-UVB)

Table 4 The effectiveness values of UVA-PF and SPF sunscreen protection value.

Formula	SPF ± SD	SPF differ from No.2 (%)	UVA-PF ±SD	UVA-PF differ from No.2 (%)
No.2	33.03±3.87	-	5.45 ±0.34	
No.4	59.49±9.04	+80.01	8.29 ±0.76	+52.11
No.5	107.80±13.19	+226.46	9.89 ±0.72	+81.47

From Table 4, the SPF value of the base formulation (No.2) was at 33.03 ± 3.87. Meanwhile, formulation No.4, incorporating natural sunscreen agents (ethyl ferulate, castor oil, and coffee oil) each at 1%, exhibited an SPF of 59.49 ± 9.04, representing an 80.01% improvement over the base formula. The results also revealed that formulation No.5 exhibited an SPF of 107.80 ± 13.19, representing a 226.46% increase over the base formulation (No.2). When comparing formula No.4 to formulation No.5, where the concentration of natural sunscreen agents (ethyl ferulate, castor oil, and coffee oil) was increased from 1% to 2%, the SPF improved by 81.15%, rising from 59.49 ± 9.04 to 107.80 ± 13.19. By increasing 1% of all natural sunscreen agents, can potentially increase SPF value up to almost 50%.

Table 4 also shows that the UVA-PF values are consistent with the SPF values across all formulations. The natural-based hybrid sunscreen formulas (No.4 and No.5), which included natural sunscreen agents and an SPF booster, demonstrated the highest UVA-PF values. Formula No.5, which contained 2% of each natural sunscreen agent, exhibited the highest UVA-PF value of 9.89 ± 0.72, an 81.47% increase over the base formula (No.2). This significant increase in both SPF and UVA-PF values attributed to the interaction between ethyl ferulate and natural oils, which may potentially lead to a transesterification reaction that enhances UVA/UVB absorption (Laszlo et al., 2003). The increase in SPF and UVA-PF may also be due to the direct effect of increasing the concentration of each natural sunscreen agent, along with the improved cream structure, which enhances both reflection and absorption mechanisms of the physical and natural sunscreen agents.

3.2 Comparison of UVA Protection Efficacy including UVAPF, PA, and Boots Star Rating.

Table 5 The effectiveness values of UVA ratio of sunscreen.

Formulation	Critical Wavelength (CW) ±SD	PA	UVA/UVB RATIO ±SD	BOOTS STAR
No.2	372.78 ±0.44	++	0.448 ±0.01	★★
No.4	373.00 ±0.00	+++	0.503 ±0.00	★★
No.5	370.00 ±0.00	+++	0.499 ±0.00	★★

The UVA/UVB ratio of formula No.2 was 0.448 ± 0.01, corresponding to a 2-star level according to the BOOTS star rating system (Table 5). When 1% of each natural sunscreen agent (ethyl ferulate, castor oil, and coffee oil) was added, as seen in Formula No.4, the UVA/UVB ratio increased to 0.503 ± 0.00, representing an 11.83% improvement over the base formula. This enhancement brought formula No.4 to a 3-star level, indicating a better balance between UVA and UVB protection. Formula No.5, which increased the concentration of natural sunscreen agents to 2%, had a UVA/UVB ratio of 0.499 ± 0.00, which is slightly lower than that of Formula No.4 but still comparable. Despite its lower ratio, Formula No.5 maintains a 3-star rating. This suggests that while formula No.5 achieved the highest SPF and UVA-PF values, it had a slightly less balanced UVA/UVB proportion, compared to Formulas No.2 and No.4, which showed slightly better UVA/UVB balance. However, the differences in the UVA/UVB ratios across all formulas are relatively small, ranging from 0.448 to 0.503. Although these values fall short of the ideal range (0.7- 1.2) suggested by Diffey (2009), they still lie within the acceptable range (0.3-1.0) according to Wang & Lim (2011). Furthermore, all formulas provide reasonably good UVA protection efficacy, with high UVA-PF values.

From Table 5, the critical wavelength (CW) values of all formulas confirm that as the natural sunscreen agent increase, the formulas not only achieve richer textures but also exhibit enhanced broad-spectrum protection. The consistency in critical wavelength values close to or above 370 across all formulations indicates strong UVA protection, with the thickest formula (No.5) providing optimal coverage. Formula No.5 has a critical wavelength of 370.00 ±0.00, also with a PA rating of "+++" and a UVA/UVB ratio of 0.499 ±0.00, with 2 BOOTS stars. This formulation maintains high UVA protection and broad-spectrum coverage. The richest texture, due to the highest concentration of both emulsifier (3%) and natural agents (2% each), helps form a substantial protective layer, suitable for extended sun exposure. Therefore, Formula No.5 remains a better option overall due to its significantly higher SPF and UVA-PF values, despite having a slightly lower UVA/UVB ratio compared to the other formulas.

Conclusion and Suggestion

This research delineates that formulation No.5 is the preferred choice. It can be concluded that utilizing a natural-based hybrid sunscreen system, particularly employing the combination of ethyl ferulate, castor oil, and coffee oil following the blending approach of ethyl ferulate with castor oil by Laszlo et al. (2015), can yield high SPF values. This allows for the development of natural-based hybrid sunscreen cream formulations with SPF 50+ efficacy achievable at low concentrations, typically 1-2%. Moreover, it enhances SPF, UVAPF, UVAPF/SPF ratio, and PA value while aiding in reducing white cast residue from physical sunscreens like ZnO and TiO_2. This reduction in the quantity of physical sunscreens used in sunscreen cream formulations, by employing a combination of physical and natural-based sunscreens, mitigates potential formulation destabilization issues that may arise from high levels of single-type physical sunscreen usage (Tortini et al., 2022).

However, there should be more study on the following subject:

1. Analysis of physical and biological effects of the product: In addition to SPF values and product stability, other potential long-term effects on the skin after use should be considered. This includes examining other properties due to the special characteristics of ethyl ferulate, castor oil, and coffee oil in various aspects.

2. Analysis of stability in industrial production: Developing high-quality sunscreen formulas may face challenges in large-scale production or under standard manufacturing conditions. This analysis should ensure that the product remains stable and maintains its specified sun protection efficiency.

3. Analysis of market expansion feasibility: Consider opportunities and possibilities for expanding this product to other markets, both domestic and international. For instance, the product could be developed to achieve a higher UVA/UVB ratio. Adjusting the UVA efficiency proportion could help increase the Boots star rating to meet the demands of markets where the sunscreen will be sold, such as in the UK and European countries.

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