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“The ultimate O/W sunscreen by means of “fiber emulsification”~Visualization of its unsurpassed performance through advanced OCT technologies ~”

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1. Introduction

The global sunscreen market continues to grow, driven by rising awareness of UV protection and climate-related factors like heat and perspiration [1]. These conditions expedite sunscreen film breakdown, increasing the need for more durable formulations.

Oil-in-water (O/W) emulsions are favored for their lightweight feel but are easily disrupted by water and sweat, making durability a key challenge [2]. Traditional evaluation methods, such as plate or replica testing, often fail to reflect actual skin behavior. Discrepancies between *in vitro* and *in vivo* results further hinder optimization [3][4].

To address this, we used Optical Coherence Tomography (OCT), a non-invasive tool enabling real-time observation of film breakdown. Our analysis showed that uniform oil layers, typical of water-in-oil (W/O) structures, provide enhanced water resistance due to continuous oil-phase networks [5].

To blend the comfort of O/W types with W/O-like durability, we introduced bamboo-derived cellulose fiber, a material from outside the cosmetics field. Used in areas such as construction and sports, cellulose fiber offers both strength and amphiphilic properties that stabilize emulsions without surfactants [6].

This interdisciplinary strategy led to a novel formulation combining OCT-based insights with materials science, achieving both pleasant usability and high durability.

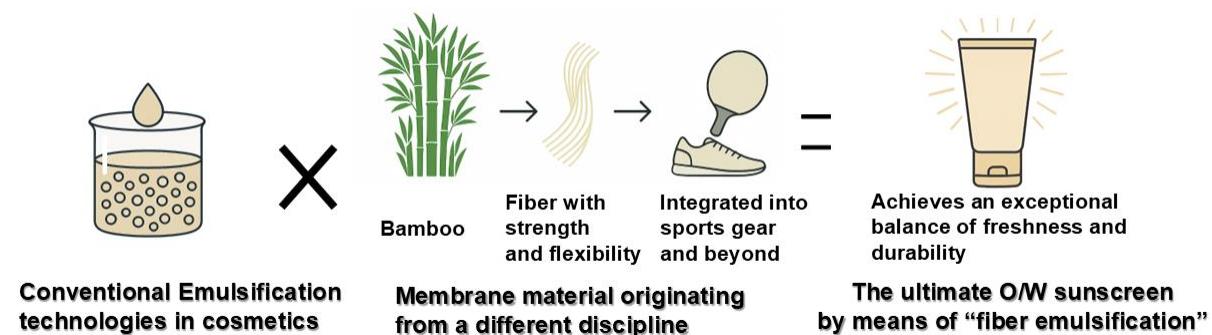


Figure 1. Integration of cross-disciplinary innovation into sunscreen development.

2. Materials and Methods

2.1. Evaluation of Sunscreen Film Degradation Using OCT and Artificial Sweat

To investigate how sunscreen films degrade on the skin, non-invasive imaging capable of micrometer-level depth resolution is required. In this study, Optical Coherence Tomography (OCT) was used to fulfill this need [8]. OCT employs near-infrared light and optical interference to generate high-resolution cross-sectional images and is widely applied in medical diagnostics, including skin and retinal imaging. Its moderate tissue penetration makes it particularly suitable for observing surface structures such as sunscreen films.

As illustrated in Figure 1, OCT enables real-time, non-invasive observation of sunscreen film detachment triggered by artificial sweat.

Measurements were conducted using a swept-source OCT system (SS-OCT, IVS-2000-HR; Santec Corp., Japan) with a $5\text{ mm} \times 5\text{ mm}$ field of view in a temperature-controlled room at 20°C . Sunscreen was applied to a $3\text{ cm} \times 3\text{ cm}$ area on the outer upper arm at 2.0 mg/cm^2 . After drying for 10 minutes, OCT was used to image the film before and after applying artificial sweat (0.5 mg/cm^2), capturing degradation at 3, 5, and 10 minutes post-application.

Ten commercial sunscreens (O/W and W/O types) were evaluated and classified based on their time-lapse OCT profiles following sweat exposure.

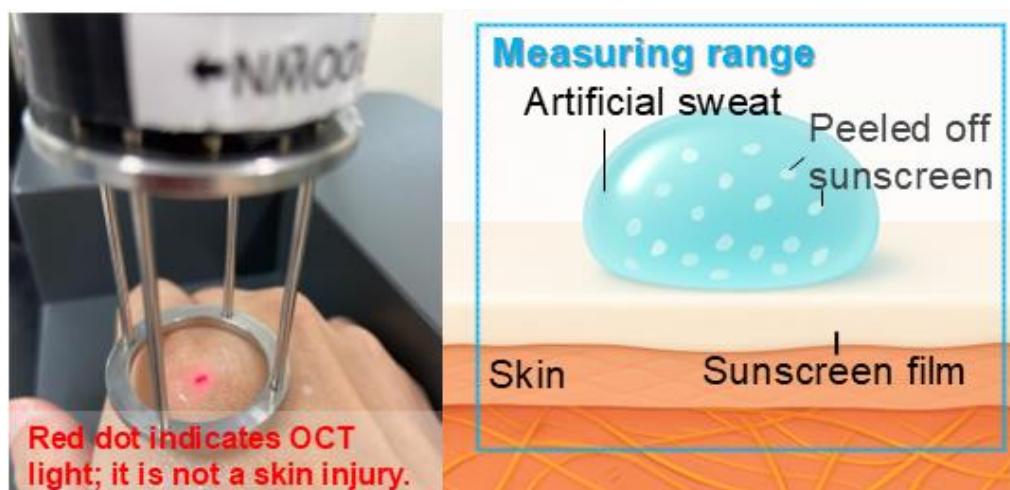


Figure 2. OCT measurement setup (left) and schematic of durability evaluation under artificial sweat (right).

2.2. Preparation and Evaluation of Samples for Uniform Oil Film Formation

To develop sunscreen formulations capable of forming a uniform oil film even after exposure to shear stress and evaporation, samples were prepared and evaluated as follows.

2.2.1. Preparation of Emulsion Samples Using Cellulose Fibers

Cellulose fibers derived from bamboo were employed as stabilizing agents for the emulsions. These fibers possess amphiphilic properties, which make them suitable for stabilizing oil-in-water systems. An oil phase containing 30 wt% UV absorbers was emulsified into an aqueous phase using the fibers.

2.2.2. Evaluation of Emulsion State

The dispersion quality of oil droplets and the distribution of cellulose fibers within the emulsions were evaluated using freeze-fracture scanning electron microscopy (SEM). The samples were rapidly frozen in liquid nitrogen and physically fractured to expose the internal structure.

SEM images were acquired using a Helios 5 Plasma PFIB system (Thermo Fisher Scientific, MA, USA) at an accelerating voltage of 1 kV, and magnifications of 2,000 \times , 5,000 \times , and 10,000 \times were used to examine the microstructure of the emulsions.

2.2.3. Analysis of Film Formation Behavior

The spatial distribution of the oil phase within the formed films was analyzed using Raman microscopy (inVia Raman microscope, RENISHAW, Tokyo, Japan). Two types of samples were prepared: one stabilized with fibers and the other with surfactants, and their states before and after application were evaluated.(Table 2.)

Applied film samples: A film with a thickness of 10 μm was applied to a substrate using a single-sided applicator and allowed to stand for 10 minutes at room temperature.

Additionally, the morphology of fibers remaining in the oil phase was observed using a laser microscope (LEXT OLS5100, Olympus, Tokyo, Japan).

Table 1. Formulation of Fiber-Stabilized Emulsion Sample.

	Ingredient (w%)	Fiber-stabilized emulsion	Surfactant-based emulsion (Reference)
Water phase	WATER (AQUA)	64.49	61.99
	BUTYLENE GLYCOL	4.80	4.80
	XANTHAN GUM	0.10	0.10
	CARBOMER	0.15	0.15
	POTASSIUM HYDROXIDE	0.06	0.06
Emulsifier	POLYSORBATE 60		3.00
	MICROCRYSTALLINE CELLULOSE (Derived from Bamboo)	0.50	
Oil phase	ETHYLHEXYL SALICYLATE	9.00	9.00
	DIPHENYLSILOXY PHENYL TRIMETHICONE	7.00	7.00
	CAPRYLIC/CAPRIC TRIGLYCERIDE	3.00	3.00
	POLYSILICONE-15	2.50	2.50
	TRIETHYLHEXANOIN	2.00	2.00
	DIETHYLAMINO HYDROXYBENZOYL HEXYL BENZOATE	3.00	3.00
	BIS-ETHYLHEXYLOXYPHENOL METHOXYPHENYL TRIAZINE	3.00	3.00
	PHENOXYETHANOL	0.40	0.40
Total		100.00	100.00

2.2.4. Durability Evaluation

The durability of the applied films was assessed using a simplified evaluation method involving the optical coherence tomography (OCT) system described in Section 2.1, in combination with artificial sweat. Time-lapse observations were performed to evaluate film degradation behavior.

2.3. Investigation of Adhesion Enhancement Using Fatty Acids

2.3.1. Incorporation of Fatty Acid Soaps and Emulsion Observation

Based on the findings in Section 2.1, it was suggested that the addition of solid or semi-solid lipids may improve adhesion and reduce film detachment. Therefore, this study examined the use of naturally derived fatty acid soaps, which are also highly compatible with cellulose fibers.

To determine the optimal blending range, a total of 200 emulsion samples were prepared by varying the cellulose fiber concentration (0–1.0 wt%) and fatty acid soap concentration (0–2.0 wt%) in 0.1% increments. All samples were based on a standard formulation (STD), with water content adjusted to maintain constant overall concentration. The emulsions were visually inspected and observed using an optical microscope (BZ-X800L, Keyence, Osaka, Japan).

2.3.2. Evaluation of Adhesion and Durability

To confirm whether the developed formulations offered enhanced adhesion and sufficient durability, a simplified durability test was conducted using the OCT system and artificial sweat as described in Section 2.1. Comparative evaluations under simulated real-use conditions were also conducted against reference samples containing conventional surfactants as emulsifiers.

After cleansing the outer upper arm ($3\text{ cm} \times 3\text{ cm}$) of participants using a designated cleanser and facial wash, the area was acclimated for 10 minutes. Each sample was applied at 0.2 mg/cm^2 . Following a 10-minute drying period, the initial film state was observed using OCT. Participants then performed a 10-minute treadmill run at 35°C and 65% relative humidity to induce perspiration. The film condition was recorded using OCT at 3, 5, and 10 minutes after running to evaluate time-dependent degradation. All evaluations were conducted in a climate-controlled room maintained at 20°C .

2.3.3. Sensory Evaluation

To ensure that the improved adhesion did not compromise the formulation's fresh skin feel, a paired comparison test was conducted with 50 expert evaluators. Participants applied both the test and a standard surfactant-based O/W-type sunscreen and were asked to choose which felt more refreshing.

2.3.4. Comparison of UV Protection Efficacy

The UV protection performance was evaluated by an external laboratory through in vivo testing, in accordance with ISO 24444:2019/AMD.1:2022. SPF values were measured before and after 80 minutes of water immersion to assess the persistence of protective efficacy.

2.3.5. Evaluation of Washability

Washability of the developed formulation was tested on the forearms ($5\text{ cm} \times 5\text{ cm}$) of healthy adult participants. A sample (0.05 g) was applied and left to air-dry for 10 minutes. The film was then washed with a commercially available soap.

Post-wash, UV light (blacklight) was used to visually confirm whether any residue remained on the skin. Residual presence under UV illumination was determined by detecting fluorescent components included in the formulation.

3. Results

3.1. Classification of Sunscreen Film Degradation Mechanisms

Through this evaluation, the mechanism of film disruption in sunscreen formulations was clearly visualized.

Ten representative formulations were evaluated and categorized into four distinct types based on the mode of film degradation: dissolving type, peeling type, splitting type, and retention type, in order of increasing durability. Each type exhibited characteristic film degradation behavior, as illustrated in Figure 2.

The dissolving type (Figure.3a) was observed in formulations containing a high ratio of high-HLB surfactants. These films readily dissolved and dispersed upon exposure to artificial sweat.

The peeling type (Figure.3b) appeared in O/W formulations that incorporated a high amount of fibers or film-forming agents, but lacked viscous emollients. This resulted in insufficient skin adhesion, leading to the film peeling away from the surface.

The splitting type (Figure.3c) was frequently seen in O/W formulations with moderate-HLB surfactants, where the film fractured and partially detached.

In contrast, the retention type (Figure 3d), exhibiting the highest durability, was only observed in W/O formulations with a heavier and more occlusive feel. Such behavior was not seen in lighter-feeling O/W types.

Notably, the presence or absence of UV-protective agents did not significantly influence the durability classification.

Furthermore, all O/W formulations were observed to swell in contact with artificial sweat and formed visibly white and thickened films. This strong affinity to sweat is considered a key factor contributing to the reduced durability of these formulations.

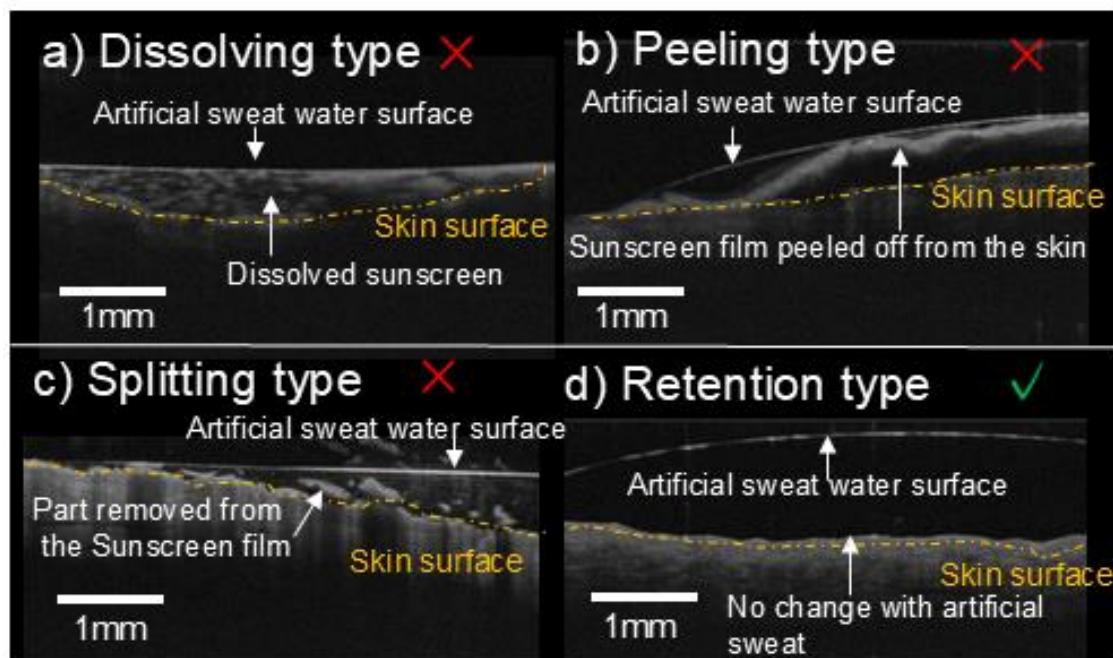


Figure 3. Representative OCT images of four types of sunscreen film degradation behaviors upon artificial sweat exposure.

3.2. Preparation and Evaluation of Samples Aimed at Uniform Oil Film Formation

3.2.1. Appearance of Emulsified Samples Using Fibers

Both the fiber-stabilized emulsions and the surfactant-based reference samples exhibited a white, cream-like appearance. No visible aggregation or phase separation was observed upon visual inspection, indicating successful and stable preparation of emulsions in all samples.

3.2.2. Evaluation of Emulsion State

The emulsification state of the fiber-based emulsions was evaluated using freeze-fracture scanning electron microscopy (SEM). As shown in Figure 4, oil droplets were uniformly dispersed with an average diameter of approximately 5 µm. No signs of coalescence or phase separation were observed. Cellulose fibers were found to be concentrated near the droplet interfaces, suggesting their role in interfacial stabilization (Figure 4).

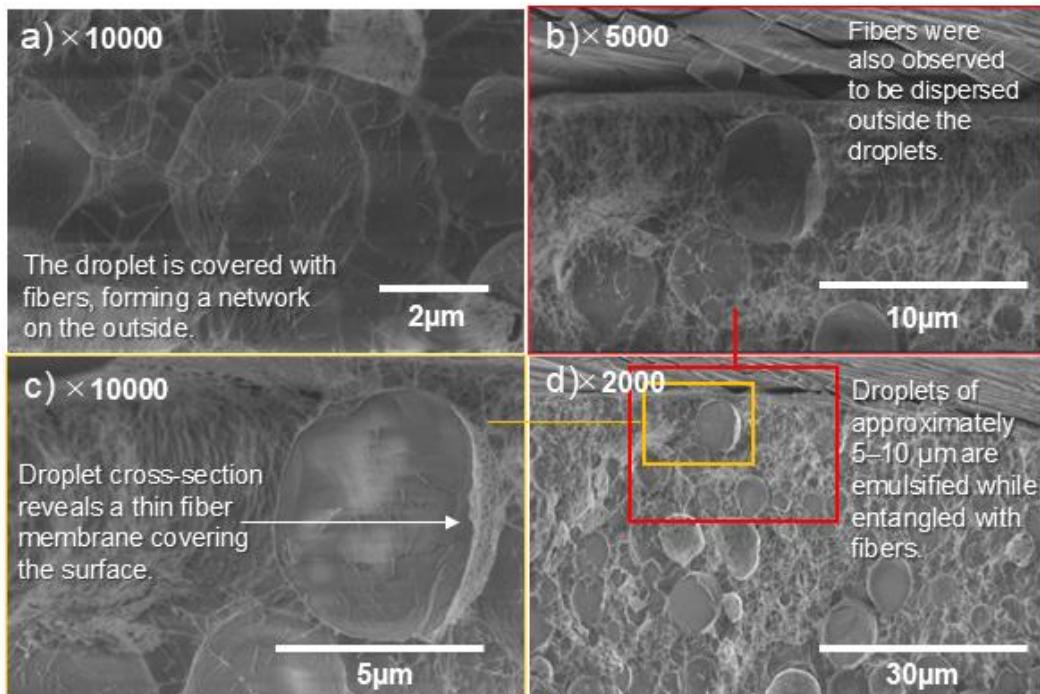


Figure 4. SEM observations and formulation of fiber-stabilized emulsions.

3.2.3. Observation of Film Formation Behavior

Continuous observation using an optical microscope revealed that, immediately after application, emulsified particles (5–10 μm) were uniformly distributed within the film. Within a few minutes, the emulsion droplets rapidly collapsed, and by 10 minutes post-application, the emulsion structure had disappeared, leaving behind a uniform oil film.(Figure 5)

In contrast, in the surfactant-based emulsion used as a reference, finer emulsion droplets were dispersed throughout the film and showed little structural change after 10 minutes. The characteristic structural collapse behavior observed in the fiber emulsions was not seen.

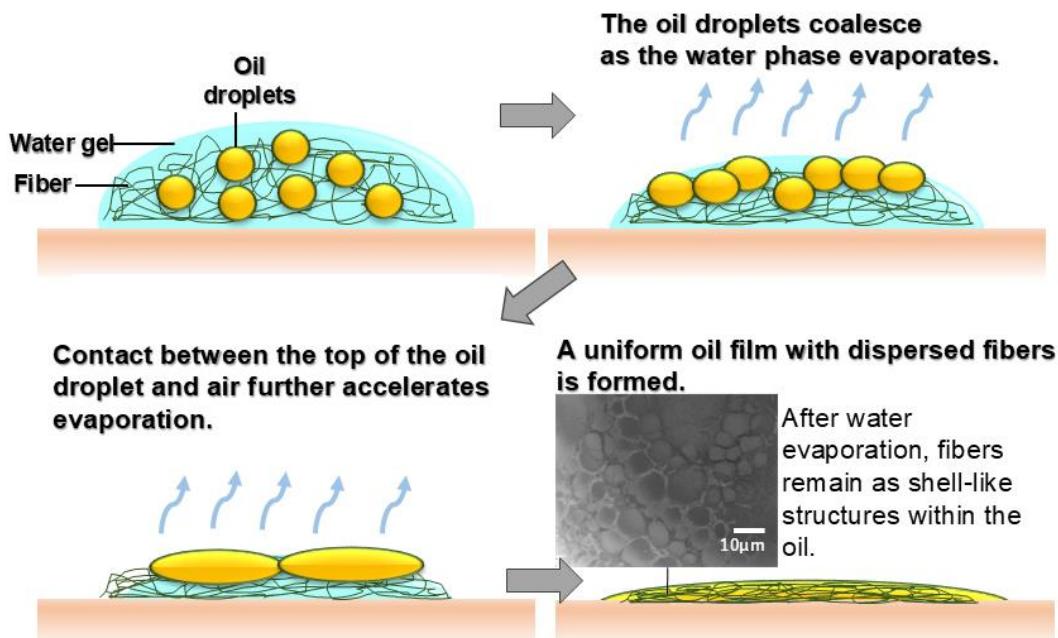


Figure 5. Uniform oil film formed during the application process of fiber emulsion.

3.2.4. Oil Phase Distribution via Raman Mapping

Raman mapping analysis of the fiber-stabilized emulsion after application revealed that peaks corresponding to the oil phase were uniformly distributed across the entire film (Figure 6). These results were consistent with the optical microscopy observations and supported the conclusion that a uniform oil film was formed following emulsion collapse. Conversely, in the surfactant-based reference sample, signals from the water and oil phases were unevenly distributed, indicating non-uniform oil dispersion within the film.

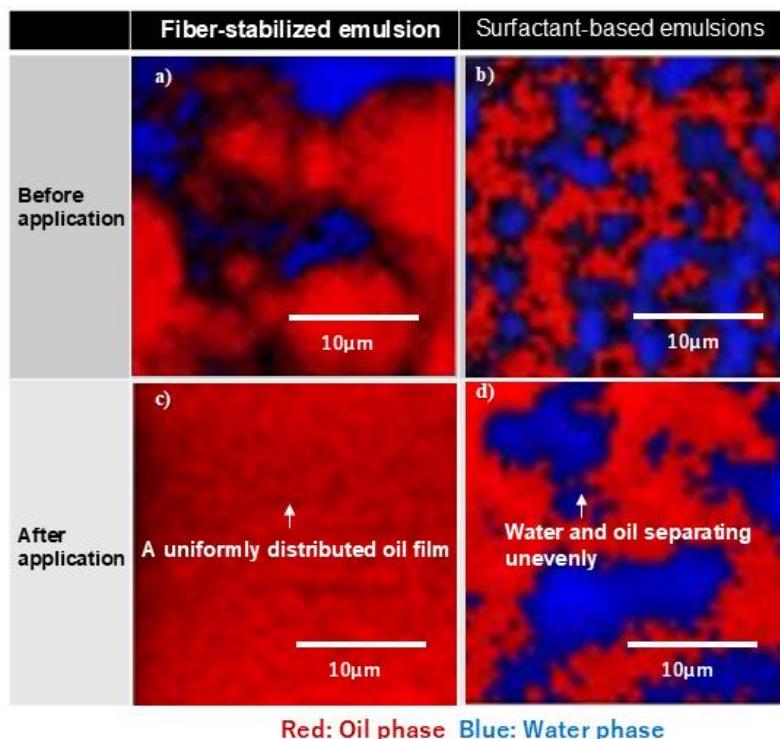


Figure 6. Raman mapping images of oil and water phase distribution before and after application.

3.2.5. Durability Evaluation

Observation of film behavior after artificial sweat exposure using OCT showed that the fiber-based film did not dissolve, but instead detached from the skin surface in a lifting manner, thus categorized as "peeling type". In contrast, the surfactant-based reference film exhibited rapid disintegration and spreading upon contact with artificial sweat and was categorized as "dissolving type".

These results suggest that fiber-stabilized emulsions are effective at forming mechanically robust oil films, although further improvement may be needed to enhance skin adhesion.

3.3. Evaluation Results of the Optimized Formulation with Fatty Acids

3.3.1. Emulsion Behavior with Fatty Acids

A total of 200 samples were prepared by varying the concentrations of fatty acids and cellulose fibers, and their emulsification states were evaluated via optical microscopy. As shown in Figure 7, emulsions were broadly classified into four categories. At low concentrations of both components, oil and water separated (O + W state), indicating a failure to emulsify.

At high fatty acid concentrations, the fatty acids acted as surfactants, producing fine droplets and O/W-like emulsions (red dotted area). However, these failed to form uniform oil films and lacked resistance to external factors like sweat.

When both fatty acids and cellulose fibers were highly concentrated, their interaction formed a viscous gel that hindered oil retention. This caused oil to separate and rise (O/W + O state), giving the appearance of emulsification but with poor stability.

Under conditions where the fatty acid content was not excessive and an appropriate amount of cellulose fiber was included (indicated in figure highlighted in blue), a stable O/W-type fiber-stabilized emulsion was formed. In this region, time-lapse observations after application confirmed a transition from the emulsion structure to a uniform oil film. The formulation developed in this study (indicated by ● in the figure) demonstrated excellent performance in terms of both emulsification stability and water resistance.

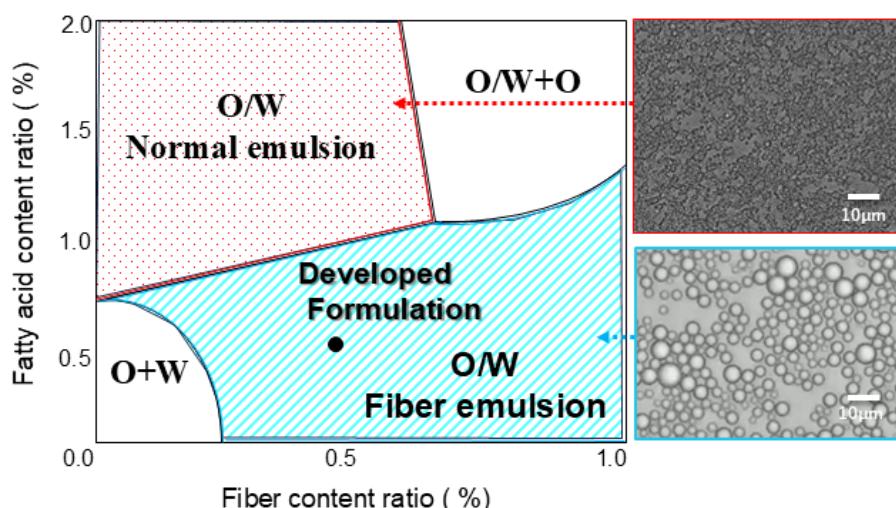


Figure 7. Phase diagram of emulsion types based on varying fatty acid and fiber content.

3.3.2. Adhesion and Durability Evaluation

OCT observations classified the developed formulation as “long-lasting,” showing no significant change even 10 minutes after the application of artificial sweat (Figure 8). In a real-use scenario simulating natural perspiration during running, conventional O/W sunscreens showed water droplet formation and film cracking due to partial dissolution. In contrast, the developed formulation maintained its structure even after 10 minutes, indicating superior durability.

3.3.3. Sensory Evaluation

In a paired comparison test conducted with 50 evaluators, 95% selected the developed formulation as having a more refreshing skin feel. This result indicates that the new technology achieves long-lasting performance without compromising sensory comfort. The refreshing sensation is believed to result from the breakdown of large oil droplets on the skin during application.

3.3.4. Comparison of UV Protection Efficacy

In SPF and PA-based comparative tests, the developed formulation exhibited UV protection equivalent to that of W/O-type sunscreens containing the same UV absorbers. It also showed significantly higher durability than O/W-type formulations, with strong resistance to degradation from sweat and other external factors. Additionally, *in vivo* testing conducted by an external agency confirmed high performance with SPF 54.0 and PA++++, and over 80% of the SPF value retained after 80 minutes of water immersion ($N = 5$).

3.3.5. Washability

No visible dark areas remained after washing, indicating complete removal of sunscreen that had darkened due to UV absorption. This suggests that the product can be effectively washed off with soap. The mechanism is likely due to a pH shift that converts the fatty acids into soap, increasing their water solubility.

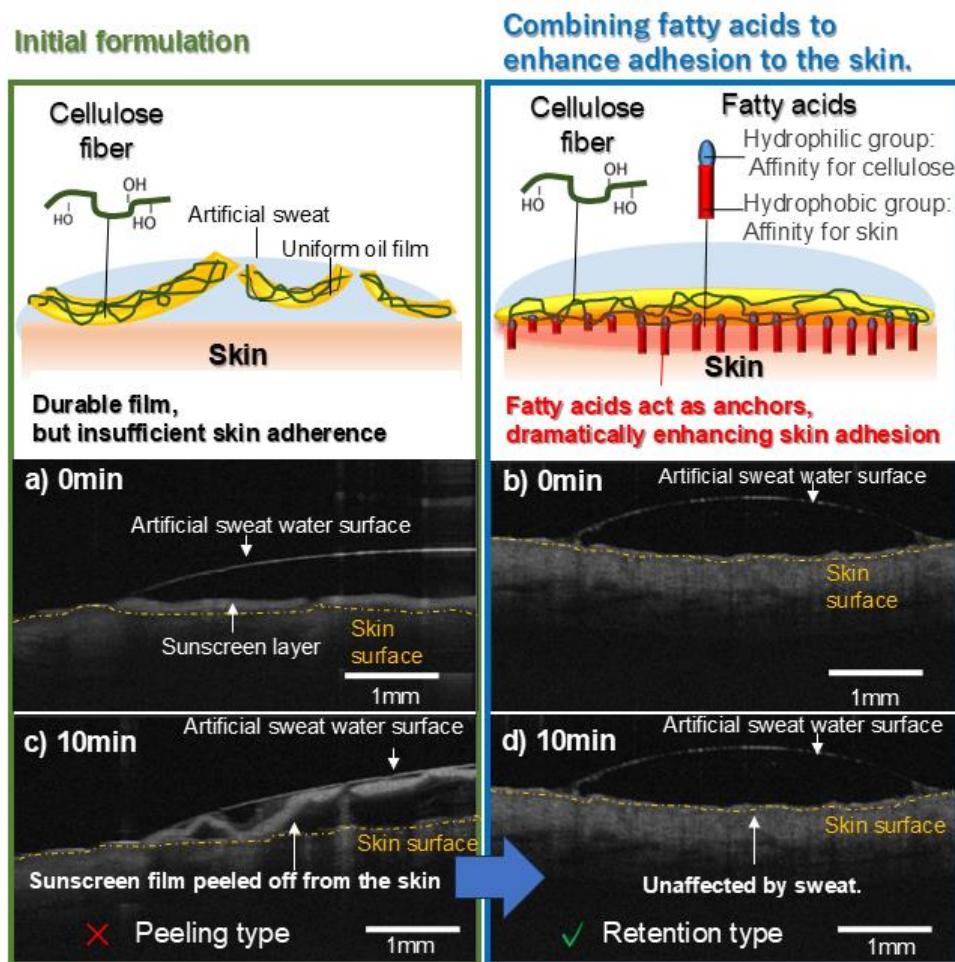


Figure 8. OCT images showing film behavior before and after artificial sweat exposure. Fatty acid addition enhances adhesion, preventing film detachment.

4. Discussion

OCT-based observations of film structure revealed that, unlike conventional O/W-type sunscreens which undergo film degradation due to perspiration, the developed formulation maintained its film structure after application and formed a uniform oil film on the skin. This property resembles the durability of W/O-type sunscreens while retaining a refreshing skin feel, making it a particularly favorable characteristic.

Of particular note is the O/W-type fiber-stabilized emulsion designed with a combination of fatty acids and cellulose nanofibers. Upon drying, this emulsion spontaneously transitions into an oil-type structure. This structural transformation is triggered by the collapse of emulsion droplets, resulting in the formation of a uniform oil film on the skin surface.

Since the resulting oil film does not contain hydrophilic surfactants, it does not swell or dissolve in water or sweat, demonstrating excellent water repellency. This contributes to sustained UV protection performance. Moreover, the addition of fatty acids enhances skin adhesion, preventing film detachment. At the same time, from a usability perspective, the film

while robust during practical use, can be easily removed with ordinary soap, offering excellent user convenience.

In terms of texture, the formulation achieves a fresh and soft feel that was not possible with Pickering emulsions. This is attributed to the smoothness during emulsion breakdown, the absence of stickiness due to the lack of surfactants, and the inherent flexibility of the fibers, which prevents a rough or squeaky sensation. Together, these features represent a highly promising approach to next-generation sensorial design in sunscreen formulations.

5. Conclusion

This study demonstrated the potential of designing an innovative sunscreen formulation by utilizing OCT (Optical Coherence Tomography) to observe film structure and combining the strengths of conventional O/W and W/O types. In particular, through the optimized combination of cellulose fibers and fatty acids, the developed formulation achieved a high-level integration of multiple performance aspects: emulsification stability, uniform oil film formation after application, UV protection, pleasant texture, and easy washability.

This brand new technology, namely "Fiber emulsification" enables the development of user-friendly yet high-performance sunscreens, potentially lowering psychological barriers toward daily sunscreen use. Moreover, this new emulsification method opens new avenues for applications beyond sun protection—such as highly moisturizing skincare, fiber-based hair care products, or allergen-blocking formulations. This offers new possibilities to engage consumer groups who have traditionally been hesitant about using skincare products.

Additionally, the formulation incorporates environmental considerations. The cellulose fibers used are upcycled materials derived from bamboo, specifically sourced from Kagoshima Prefecture, where overgrowth has become an ecological concern. Thus, the technology is not only promising in terms of product innovation but also contributes to regional sustainability and responsible material sourcing.

In summary, this study presents a formulation technology that goes beyond conventional sunscreen development. The availability of a new means of emulsification is a significant step toward the next generation of personal care products and a meaningful contribution to the future of cosmetics.

6. References

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