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TiO₂ water-based dispersion for inclusive sunscreens

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1. Introduction

In the fight against the damage caused by solar radiation to human health, sunscreens have become indispensable allies in the prevention and mitigation of the effects of UVA and UVB rays on the skin. Exposure to UVB radiation triggers an inflammatory response in the skin, known as erythema or sunburn, thickens the stratum corneum, causes photoaging, and has immunosuppressive effects. UVA radiation, in addition to tanning, is also responsible for photoaging, pigment disorders, and photodermatoses [1]. However, the greatest damage related to sun exposure is skin cancer.

The majority of skin cancer cases are due to exposure to UV light without protection. Therefore, it is essential that the habit of using sunscreen before sun exposure becomes increasingly common among consumers, as its regular use has been proven to reduce the risk of melanoma, the most aggressive type of skin cancer [2]. The primary challenge lies in developing sunscreens that are not only effective but also safe for both human health and the environment. These sunscreens must also offer desirable aesthetics, a pleasant skin feel, and affordability.

As the beauty industry steers towards a more natural and safety-conscious approach to photoprotection, the spotlight is fixed on inorganic UV filters. Beyond the fact that they are generally recognized as safe and effective (GRASE) by FDA, they are also photostable, considered safe and non-irritant, provide broad-spectrum protection, and their dispersions are easy to incorporate into product bases [3].

Despite their efficacy in shielding against harmful UV rays, the challenge lies in formulating mineral sunscreens that do not leave an undesirable white film on the skin. This quest for inclusive beauty, catering to diverse phototypes, demands exploration and innovation.

Mineral sunscreens contain active ingredients like ZnO or TiO₂, which can impart a white cast on the skin. This is caused by the reflection and scattering of visible light by mineral particles. To diminish the white cast in inorganic sunscreens, an association of strategies can be considered, like working with nano-sized particles, and experimenting different types of surface treatment and particle coatings [4].

Titanium is the 9th most abundant element on earth and is found in almost all living things, as well as water, rocks and soils. TiO₂ is the most common oxide of titanium. As a UV filter, it is

obtained by transformation of titanium compounds mined from the earth, using an aqueous process with saltwater waste streams.

TiO₂ is well-known as a safe material that has been used in cosmetics for decades. In more recent times extensive safety reviews have been conducted by the US Food & Drug Administration (FDA) [5] and the EU Scientific Committee for Consumer Safety (SCCS) [6] and these reviews have concluded that whatever its size (nanomaterial or not) TiO₂ largely does not penetrate the skin. The FDA classify them as GRASE (Generally Recognised as Safe & Effective) Category 1 [7]. TiO₂ is approved by regulatory bodies globally at levels up to 25% active, which is the highest active level permitted of any UV active, along with Zinc Oxide (ZnO).

TiO₂ also has a very low potential for skin irritation making it a great choice for formulators working on products for all skin types including babies, children, and sensitive skin. In contrast, there is some evidence of allergens with chemical/organic sunscreens, specifically benzophenone, cinnamates and dibenzylmethanes. In addition, the FDA state there is evidence that they are/or may be absorbed through the skin, and the consequences of this absorption are not known [5]. Finally, the permitted use levels of organic sunscreens are limited to 5-10% in most cases, depending on the country.

This work aims to explore a titanium dioxide (TiO₂) water-based dispersion with a technology that delivers a tightly controlled particle size distribution. As a result, the undesirable whitening effect typically associated with TiO₂ is effectively eliminated, while the SPF efficacy remains uncompromised. Made without EO, PEG/PPG, phenoxyethanol, and silicone, its composition is suitable for natural products.

2. Materials and Methods

Whitening test

A draw down whitening test was carried for the simple frame formulation (Table 1) to compare the whitening of water-based dispersion to the other TiO₂ particle sizes. An assessment of whiteness on black and white contrast card was taken using LabScan XE Spectrophotometer. A small quantity of the formulation was pipetted on the top of the black and white contrast card. A 'drawn down' was conducted using a number 2 'K' bar with the application of a small amount of even pressure to get an even film. The sample was then left to dry for 15 minutes, after which photographs were taken.

Evaluations were also conducted *in-vivo* on skin using the same formulations at 7.5% TiO₂ (Table 1). 0.05g of sample was applied to the subjects. After application the subjects were instructed to rub in the sample 40 times, the skin was left to dry for 5 minutes before images were taken.

Table 1: Frame formulation used in efficacy evaluations

Ingredients (INCI Name)	Functionality	% w/w
Phase A		
PEG-30 Dipolyhydroxystearate	W/O Emulsifier	3.00
Candelilla Wax (Euphobia Cerifera)	Structuring Agent	1.00
Sorbitan Isostearate	Dispersing Agent	3.00
Caprylic Capric Triglyceride	Emollient	37.50
Cyclopentasiloxane	Emollient	5.00
Phase B		
TiO ₂ powder content	UV Filter	7.50
Phase C		
Water		37.50
Glycerine	Humectant	4.00
Magnesium Sulphate Heptahydrate	Stabiliser	0.70
Phase D		
Phenoxyethanol (and) Ethylhexylglycerine	Preservative	0.80

SPF and UVA performance

A O/W emulsion was produced using different emulsifier systems (Table 2) and processing methods when used at 7.5% active, adding TiO₂ water-based dispersion before or after emulsification, and an in vivo test was conducted to evaluate the impact of process and emulsifier system on SPF performance.

Table 2: Formulation used in efficacy evaluations of SPF

Ingredients/ INCI Name	Functionality	A (%)	B (%)	C (%)
Deionized Water		To 100	To 100	To 100
Glycerin	Humectant	3.00	3.00	3.00
Magnesium Aluminum Silicate	Rheology modifier	0.70	0.70	0.70
Xanthan Gum	Rheology modifier	0.30	0.30	0.30
TiO ₂ water-based dispersion	Inorganic UV Filter	23.44*	23.44*	23.44*
Propylene Glycol Dicaprylate/Dicaprate	Emollient	7.50	7.50	7.50
Caprylic/Capric Triglyceride	Emollient	5.50	5.50	5.50
Glyceryl Stearate (and) PEG-100 Stearate	Emulsifier	3.00	-	-
Sorbitan Stearate	Emulsifier	3.00	-	-
Polysorbate 60	Emulsifier	1.50	-	-
Stearyl Alcohol	Emulsion stabilizer	1.00	1.00	1.00
Cetearyl Alcohol (and) Dicetyl Phosphate (and) Ceteth-10 Phosphate	Emulsifier	-	3.00	-

Cetearyl Alcohol (and) Ceteth-20 Phosphate (and) Dicetyl Phosphate	Emulsifier	-	-	3.00
Glyceryl Caprylate (and) Glyceryl Undecylenate	Preservative	1.00	1.00	1.00
*7,5% TiO ₂ active				

Synergy with organic UV filters

O/W emulsions were produced including only TiO₂ water-based dispersion, organic UV filters or combining both systems (Table 3) to evaluate a synergistic effect on SPF result through *in vivo* test.

Table 3: Formulation used for evaluation of synergistic effect between TiO₂ water-based dispersion and organic UV filters

Ingredients/ INCI Name	Functionality	A (%)	B (%)	C (%)
Phase A				
Deionized Water		To 100	To 100	To 100
Glycerin	Humectant	3.00	3.00	3.00
Potassium Cetyl Phosphate	Emulsifier	3.00	3.00	3.00
Magnesium Aluminum Silicate	Rheology modifier	0.40	0.40	0.40
Xanthan Gum	Rheology modifier	0.10	0.10	0.10
Phase B				
Ethylhexyl Cocoate	Emollient	1.00	1.00	1.00
C12-15 Alkyl Benzoate	Emollient	6.00	6.00	6.00
Diethylhexyl Adipate	Emollient	5.00	5.00	5.00
Sorbitan Stearate	Emulsifier	1.00	1.00	1.00
Glyceryl Stearate (and) PEG-100 Stearate	Emulsifier	1.50	1.50	1.50
Octocrylene	Organic UV filter	6.00	-	6.00
Bis-Ethylhexyloxyphenol Methoxyphenyl Triazine	Organic UV filter	4.00	-	4.00
Phase C				
TiO ₂ water-based dispersion	Inorganic UV Filter	-	9.50	9.50
Phase D				
Phenoxyethanol (and) Ethylhexylglycerin	Preservative	1.00	1.00	1.00
Cyclopentasiloxane	Skin sensory modifier	1.00	1.00	1.00

3. Results

Whitening test

Due to its tightly controlled particle size distribution the TiO₂ water-based dispersion delivers clarity when applied to skin. The photographs taken are shown in Figure 1 and Figure 2.

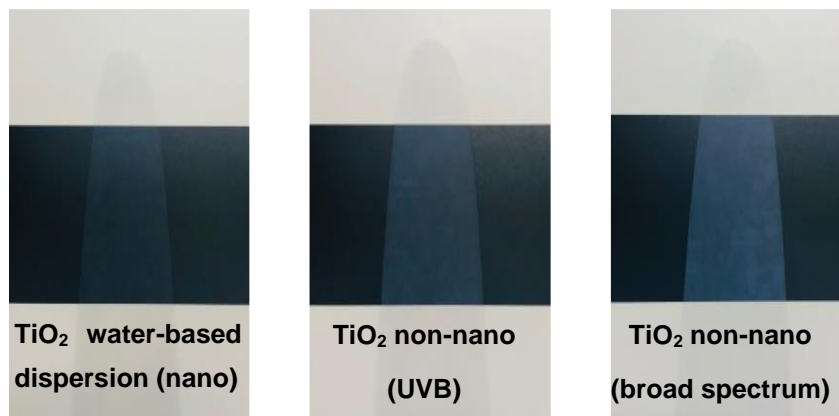


Figure 1: Visual whitening comparisons of TiO₂ with different particle sizes

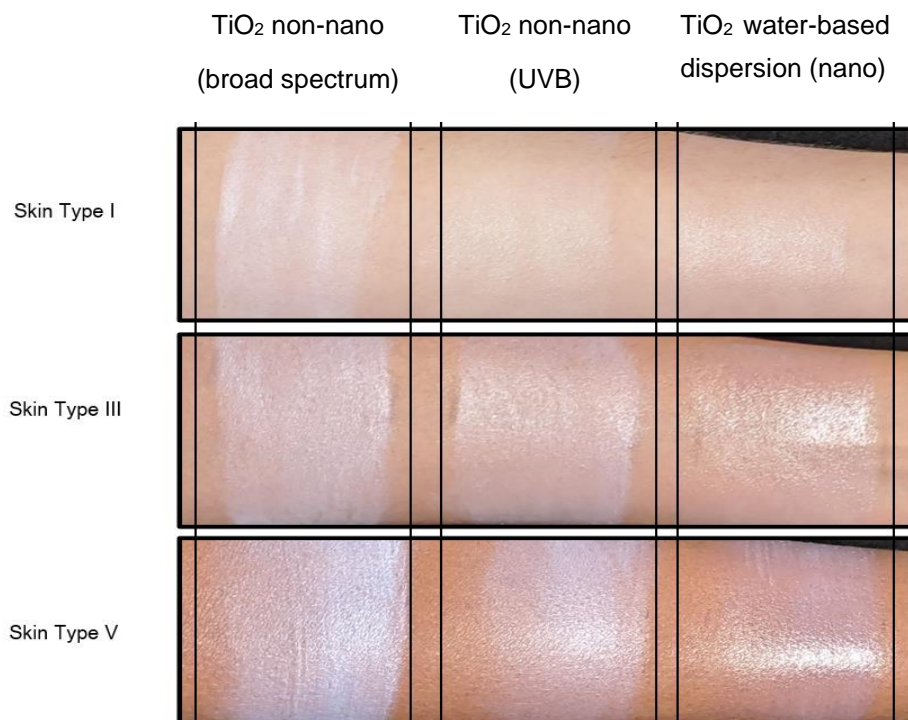


Figure 2: Visual whitening comparisons in vivo of TiO₂ with different particle sizes on various skin phototypes

The results demonstrate that TiO₂ water-based dispersion shows the lowest level of whitening compared to non-nano TiO₂ in both contrast cards and *in vivo* tests.

SPF and UVA performance

Generally TiO_2 with nano particle size delivers high UVB protection, with a typical SPF efficacy between 2-3 SPF units per 1% solids. The water-based dispersion has achieved higher SPF efficiency than this in an O/W emulsion based on Glyceryl Stearate (and) PEG-100 Stearate, the efficacy ratio was 4 SPF units for 1% active. The graph in Figure 3 below shows the *in vivo* SPF of TiO_2 water-based dispersion in different emulsifier systems when used at 7.5% active. It also shows the differences that can be expect with different processing methods. The efficacy ratios range between 2.7 SPF units/1% active and 4.1 SPF units/1% active.

TiO_2 nano is optimised to give clarity on skin and as a result it has low UVA protection. Therefore, it must be combined with a UVA absorber to achieve a broadspectrum protection formula. Typically, the following UVA performance can be expected from TiO_2 water-based dispersion. It has a UVA PF per 1% solids of around 0.5. Used alone it does not pass the EU requirements for UVA (that is SPF/UVA PF ratio of less than 3) its ratio is around 4. It also does not meet the US requirements for broad spectrum claims when used alone (that is a critical wavelength greater than 370nm) its critical wavelength is around 360nm. In the UK the UVA/UVB ratio would be 0.35-0.40 which is equivalent to 1 star.

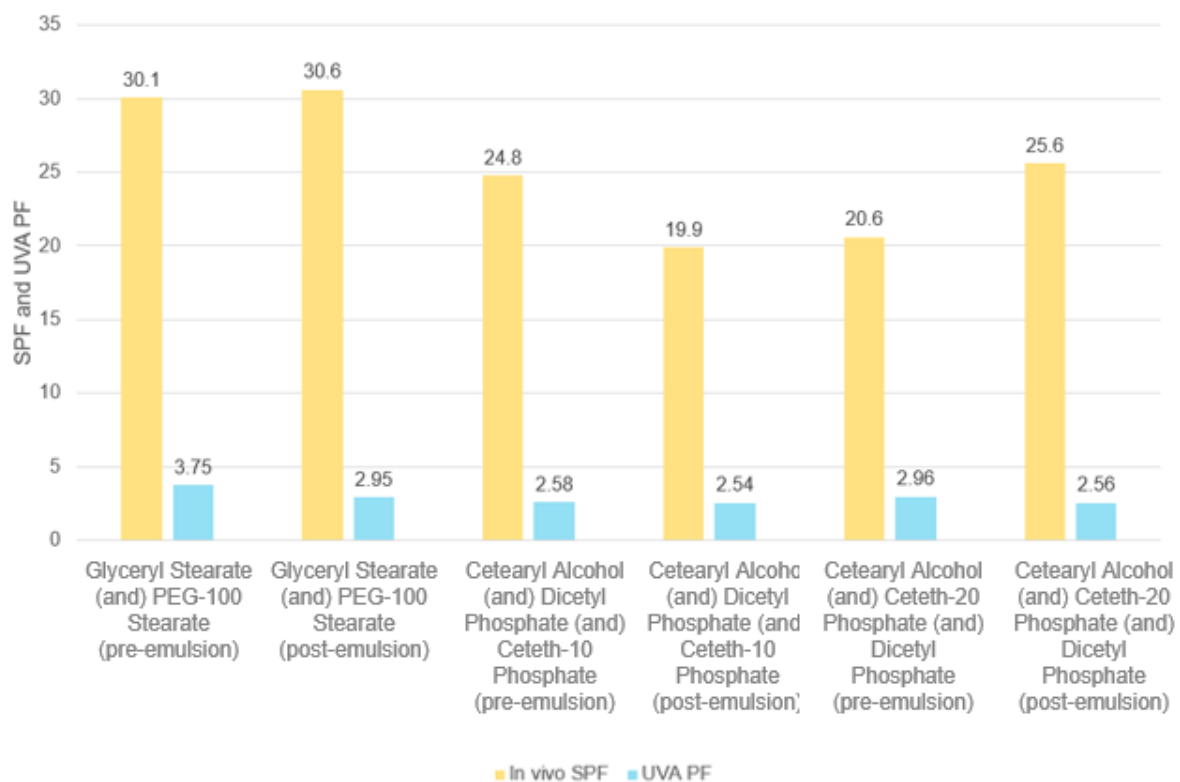


Figure 3: Efficacy of various formulations, *in vivo* SPF, *in vitro* UVA PF

Synergy with organic UV filters

The formulation containing only organic UV filters showed SPF 9.2, the one containing TiO_2 water-based dispersion showed SPF 5.0, and combining both UV filters the formulation achieved SPF 40.4, confirming the synergistic effect.

4. Discussion

As the TiO_2 dispersion studied is water-based, in O/W emulsions the TiO_2 goes into the external water phase. Upon application to the skin the water evaporates in a process known as “dry down” as shown in the first image in Figure 4. If hydrophilic TiO_2 is used then the active would be excluded from the remaining oil film on the skin, which could lead to a patchy coverage or a tendency to be removed by water, as shown in the second image in Figure 4. In the case of this TiO_2 water-based dispersion, the TiO_2 active is hydrophobic, so it is incorporated into the oil film on dry down which leads to better skin coverage, as shown in the third image in Figure 4.

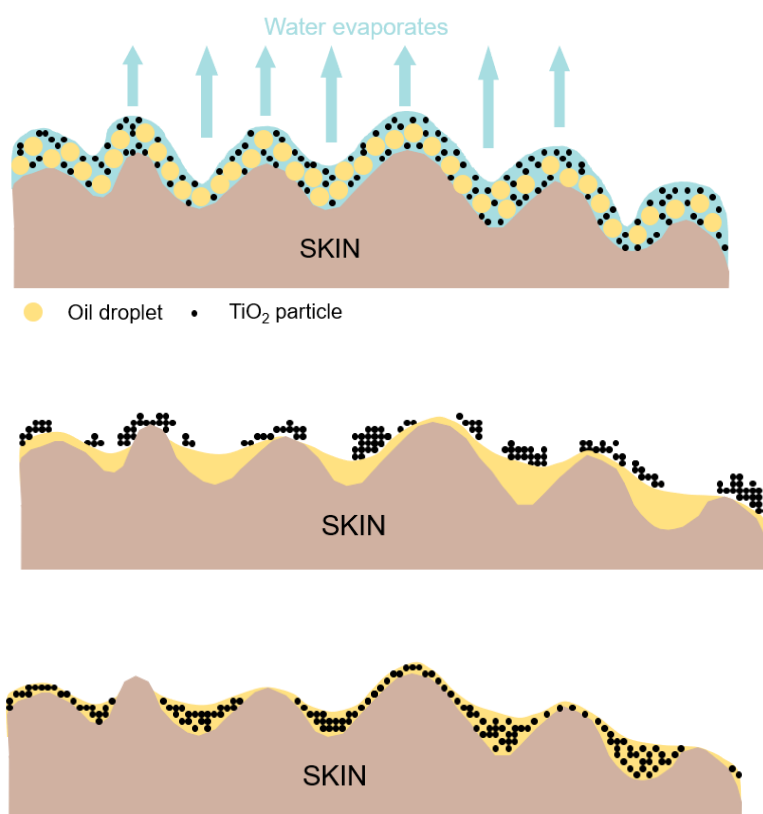


Figure 4: Diagram showing the unique behaviours of TiO_2 water-based dispersion on application

The use of TiO_2 water-based dispersion combined to organic UV filters also provide lighter sensory, since the addition is made in the aqueous phase, we will need a lower concentration of filters and emollients with solubilizing efficacy in the oily phase, in addition to increasing the FPS of the formulation *in vivo*, since the film formed on the skin is generally not completely uniform and through the combination of aqueous and oily phase filters we guarantee the delivery of active ingredients in both phases, which explained the synergistic effect observed. Nano TiO_2 water-based dispersion presented improved performance in whitening effect compared to other particle sizes due to less reflection and dispersion of light, as it has nanometric particle size.

5. Conclusion

The titanium dioxide (TiO₂) water-based dispersion has a technology that delivers a tightly controlled particle size distribution. It enables formulations high SPF claims minimizing the undesirable whitening effect typically associated with TiO₂. Made without EO, PEG/PPG, phenoxyethanol, and silicone, its composition allows “natural” formulations, appealing to those who prioritise nature identical minerals. Additionally, its mildness on skin makes it a safer choice for babies, children and sensitive skin.

6. References

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