How to best formulate aesthetically pleasing mineral sunscreens

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Introduction

The North American sunscreen's market has been changing drastically over the past few years. With the growing use of mineral filters (Titanium Dioxide and Zinc Oxide), there is a need to understand and learn how to best formulate in order to optimize SPF protection, product performance and formulation aesthetics considering skin diversity and customers lifestyle. Formulating with mineral filters is very different from developing conventional sunscreens and formulators are looking for advice and best practices to develop the next generation of sun care products. The purpose of this study was to understand the key parameters when developing sunscreen formulations in order to maintain aesthetics while optimizing SPF values: the influence of using surface treatments, the importance of choosing the right emollients and the best manufacturing process in order to develop optimized mineral sun care formulations. The best sunscreen is ultimately the one people use so it is key to take in consideration not only the performance but also the final aesthetics on all skin types.

Methodology

We selected different Titanium Dioxide and Zinc Oxide mineral filters, U.S.P. grades: untreated, surface treated with a synthetic hydrophobic surface treatment (INCI: Triethoxycaprylylsilane) and treated with a natural hydrophobic surface treatment (INCI: Persea Gratissima [Avocado] Oil, Hydrogenated Vegetable Oil, Tocopherol).

❖ Surface Treatment Added Value

The first part of the study was to evaluate the performance benefit of using a surface treatment on mineral sunscreens (Sun Protection Factor). Three individual sunscreen formulations were made using the same structure and process with the exception of the mineral filters. The mineral filters used were either untreated or treated with the synthetic or natural hydrophobic surface treatments. The formulations were then sent externally for in-vivo SPF testing (FDA Monograph, 5 valid subjects).

❖ Influence of Manufacturing Process

The second part of the study was to evaluate the influence of using different processes when manufacturing the same formulation with different lab equipment and manufacturing procedures. The formulation was made five different ways:

- Process 1: High shear mixing (Silverson, L5M-A mixer, square hole high shear screen stator).
- Process 2: Three roll mill (EXAKT 50I) to create mineral filter pasty dispersion and then emulsion created under propeller mixing (IKA Eurostar 60 Control, 3-bladed propeller stirrer).
- Process 3: Three roll mill (EXAKT 50I) to create mineral filter pasty dispersion and then emulsion created under propeller mixing (IKA Eurostar 60 Control, 3-bladed propeller stirrer).

Batch was homogenized at the end with high shear mixing (Silverson, L5M-A mixer, square hole high shear screen stator) for 5min.

- Process 4: Dispersions used were commercially available and emulsion created under propeller mixing (IKA Eurostar 60 Control, 3-bladed propeller stirrer).
- Process 5: Dispersions used were commercially available and emulsion created under propeller mixing (IKA Eurostar 60 Control, 3-bladed propeller stirrer). Batch was homogenized at the end with high shear mixing (Silverson, L5M-A mixer, square hole high shear screen stator) for 5min.

The formulations were then sent externally for in-vivo SPF testing (FDA Monograph, 5 valid subjects).

❖ Influence of Key Ingredients

The last part of the study was to evaluate the influence of two key ingredient categories used to formulate sunscreen products: mineral filters and emollients.

We chose to evaluate the mineral filters in combination with 13 commercially available emollients ranging from different polarities and chemical structures. 78 mineral filters dispersions were made using a three roll mill (EXAKT 50I) in order to obtain homogeneous particle size dispersions with regular and fine paste consistency. The concentration of the pasty dispersions were then adjusted to 25% solid content under propeller mixing (IKA Eurostar 60 Control, 3-bladed propeller stirrer). The dispersions were evaluated and compared under two criteria: fluidity and color.

Fluidity: The viscosity of each dispersion was measured with a viscometer (Brookfield, DVE, spindle: RV-04, speed: 100 rpm, time: 30 sec) in order to evaluate the pigment load capacity of each combination.

Color:

- CIELAB Color Space was used to quantify the color of each dispersion by measuring L*a*b* values (HunterLab, UltraScan Vis).
- In-vivo evaluation on different skin types were conducted to validate predicting method through CIELAB Color Space measurement.

Results

❖ Surface Treatment Added Value

The results of the in-vivo SPF testing showed higher SPF values for sunscreen formulations made with hydrophobic treated mineral filters (synthetic treatment: SPF 34.0; natural treatment: SPF 34.8) compared to untreated filters (SPF 12.8).

❖ Influence of Manufacturing Process

The highest in-vivo SPF value (SPF 43.5) was obtained with Manufacturing Process 5, when formulating with commercially available mineral filter dispersions, emulsifying with propeller mixing and then homogenizing the batch with high shear mixing as shown in Figure 1. The aesthetics such as the transparency of the formulation was also improved by using a commercially available dispersion versus straight mineral filters (Figure 2).

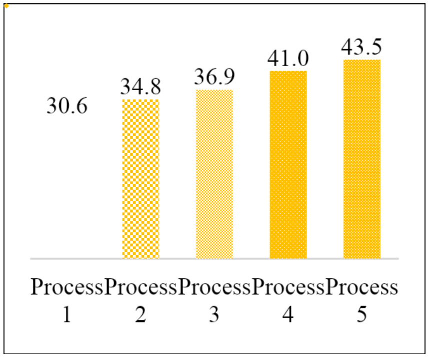

*Figure SEQ Figure * ARABIC 1: Influence of Manufacturing Process SPF in-vivo results.*

*Figure SEQ Figure * ARABIC 2: 1mg/cm^2 sunscreen applied on skin type III.*
Left: sunscreen made with Process 1
Right: sunscreen made with Process 5.

❖ Influence of Key Ingredients

<u>Fluidity:</u> The choice of surface treatment and emollients are key to achieve low viscosity mineral sunscreen dispersions.

Untreated mineral filters dispersions were always thicker than the ones made with the hydrophobic surface treated ones. The natural surface treated mineral filters offer the most fluid dispersions.

Each combination (mineral filter and emollient) provided a different viscosity (Figure 3).

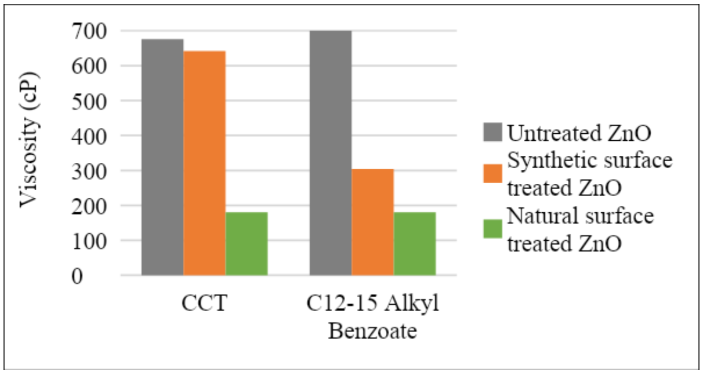

Figure 3: Viscosity results of Zinc Oxide dispersions.

<u>Color:</u> In order to quantify the whitening and blueish effects of each combination (mineral filter and emollient), we compared L* and b* values obtained from the CIELAB Color Space.

The natural hydrophobic surface treatment offers the least white and more yellow application with low L* and b* values in every emollients, allowing to formulate more natural-looking sunscreens (Figure 4). Evaluating the color of each dispersion prepared allows the formulator to choose wisely the best combination of mineral filters and emollients to develop more transparent and natural-looking sun care products for all skin types.

Conclusion

The choice of using a surface treatment, emollients and process are key parameters to consider when developing sun care products. They have a direct impact on formulation aesthetics including fluidity, spreadability, skin feel, visual appearance as well as consumer's perception and more importantly efficacy. With viscosity and LAB measurements, we were able to demonstrate the importance of choosing the right ingredients to develop more transparent mineral sunscreens with optimized SPF value and minimum whitening effect.

About the speaker

Stellie Balthazard is the North America Technical Services Manager for Sensient Beauty and is responsible for leading its applications development team in developing skin care, sun care, hair and color cosmetic prototypes. She began her career in the personal care industry as an intern with Firmenich, followed by an R&D apprenticeship at Chanel. This assignment drove her passion for formulating innovative cosmetic products. In addition, she quickly discovered the impact ingredient suppliers can have during the product formulation phase. Stellie received her Master's degrees in both Chemistry and Chemical Engineering from Ecole Supérieure de Chimie Organique et Minérale (ESCOM) in Compiègne, France.