

IFSCC 2025 full paper (IFSCC2025-1273)

“SPF BOOSTER COMBINATIONS: A STRATEGY FOR ACHIEVING HIGHLY EFFECTIVE AND SENSORY SUN CARE PRODUCTS USING MICROSCOPICAL, RHEOLOGICAL AND TEXTURE CHARACTERIZATION”

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1. Introduction

The market of suncare has been growing year by year and people's awareness of UV radiation-related issues is arising with it, leading to a high market demand and the need for appealing and sensorial formulations [1]. Nowadays UV filters represent a timely issue due to the ongoing scrutiny by the regulatory organizations [2] and their potential effects on human health and the environment [3,4]. For all these reasons, suncare formulation has become an issue in terms of achieving a high SPF protection with the filters still allowed, obtaining a pleasant sensorial profile and at the same time developing sustainable products, also not forgetting to contain costs. In this context the cosmetic sector has begun focusing on identifying alternative raw materials with the aim of boosting the activity of UV filters, maintaining a high protection factor and decreasing their concentration to address the health and environmental issues. These so called “SPF Boosters” seem to be groundbreaking ingredients for suncare as they function by enhancing the product spreadability on the skin, the scattering of sunlight and the thickness of the sunscreen layer [5,6], with no activity on solar radiation absorbance. However SPF boosters, generally polymers or solid insoluble particles, could lead to incompatibility issues with the vehicle and to the modification of the stability, viscosity and sensorial profile of the product. Another challenge lies in the booster's efficacy on the Sun Protection Factor which is often correlated to high concentrations and this could significantly impact on the formulation costs. A rheological instrumental approach has demonstrated to be effective for predicting how the SPF changes in suncare formulations through the analysis of physical properties like viscosity and flowing behaviour [7,8]. In light of this, the aim of this work is to utilize microscopical, rheological and texture analysis to characterize selected SPF boosters added to an O/W emulsion, first singularly and then combined, in order to evaluate their stability, homogeneity and application properties, thus maintaining comparable characteristics to the original emulsion. By using a systematic approach this study has investigated how to obtain booster combinations to potentially maximize the performance of sun care products with no heavy impact on the applicative properties of the product.

2. Materials and Methods

12 raw materials with commercial information supporting the SPF booster effect were tested.

Hydrophilic boosters: R (Microcrystalline Cellulose), V (Cellulose/cellulose gum, 3% fibrillated cellulose in water).

Liphophilic boosters: N (Hydrogenated castor oil, jojoba esters, tocopherol), U (Hydrogenated palm oil, saccharum officinarum extract), H (Copernicia Cerifera Wax, Oryza Sativa Wax).

Film former crosspolymers: W (Trimethylpentanediol/Adipic Acid/Glycerin Crosspolymer) and F (Polyglyceryl-3 Stearate/Sebacate Crosspolymer).

Solid boosters: four spherical particles Z (Zeolite), C (Calcium Sodium Borosilicate), S (Silica), D (Cellulose) and a lamellar particle, M (Magnesium Potassium Fluorosilicate).

Reference sunscreen emulsion: oil in water fluid emulsion containing both organic and mineral filters (BASF Sunscreen Simulator calculated SPF 40) based on an anionic primary emulsifier (Potassium cetyl phosphate) associated with a non-ionic co-emulsifier (Tribehenin PEG-20 esters) and an oily phase (18.2%w/w) formed by synthetic lipids and 7.2% w/w organic filters. The oil phase (75°C) was added to the water phase (70°C) using a Silverson L5T laboratory mixer and homogenized for 5 min at 4500 RPM. Polyacrylate Crosspolymer-6 and Hydroxyethyl Acrylate/Sodium Acryloyldimethyl Taurate Copolymer were added as thickeners at 60°C.

After initial screening of individual boosters at their technical data sheet-recommended concentrations, the boosters were combined at the percentages indicated in Table 1. The percentage of each booster was subtracted from the aqueous phase (water q.s. to 100%) to prevent significant emulsion alterations.

Table 1. Concentration of boosters associations in emulsions.

	F	FU CM	FH CM	FV	FH	FVH	FD	FC	FVH C	FVH CM	FVH D	FVH DM
% F (Polyglyceryl-3 Stearate/Sebacate Crosspolymer)	1	1	1	1	1	1	1	1	1	1	1	1
% U (Hydrogenated palm oil, saccharum officinarum extract)		2				2						
% H (Copernicia Cerifera Wax, Oryza Sativa Wax)			1		1	1			1	1	1	1
% V (Cellulose/cellulose gum)				2					2	2	2	2
% D (Cellulose)							1.5				1.5	1
% C (Calcium Sodium Borosilicate)	2	2						1.5	1.5	1		
% M (Magnesium Potassium Fluorosilicate)	1	1								0.5		0.5

All the emulsions were analysed through the following techniques:

Centrifuge test (4800 RPM/30minutes) to assess the mechanical stability.

Optical microscope LEICA DM1000 with 40X objective and 100X immersion oil objective to evaluate the morphology of the emulsions.

Rheological tests performed by Rheometer Physica MCR e302 (Anton Paar) at 23°C ± 0.05°C using PP50-P2 (gap 1 mm) or CP50-1 sensor (gap 0.1 mm) to detect the trend of storage (G') and loss (G'') moduli.

- Amplitude sweep tests (AS): strain (γ) 0.01% - 1000% at fixed frequency (1 Hz).
- Frequency sweep tests (FS): 10 Hz - 0.01 Hz, fixed strain (within the linear viscoelastic region).
- Combined procedure: Oscillatory test: constant stress and frequency/ 3'; Steady shear test (5000 s⁻¹/ 5'); Oscillatory test: constant stress and frequency/ 30'; AS; FS.

Texture analysis performed by a Texture Analyzer TMS-Pro (Food Technology Corporation), using a nylon, spherical probe (\varnothing 2 cm) and a load cell of 10 N to perform the immersion/de-immersion test (depth 10 mm, speed 80 mm/min) in a 50 mL container (\varnothing 5.3 cm) filled with

the emulsion. The following parameters were detected from the curve plotted as load (N) vs cumulative displacement (mm) obtained with the SoftwareTexture Lab Pro: firmness (N), the maximum value of force; consistency (N.mm), the area under the positive curve; cohesiveness (N), the negative peak; adhesiveness (N.mm), the area under the negative portion of the curve. **SPF VITRO test** (ISO 24443:2022) performed by an external laboratory.

3. Results and discussion

2.1. Characterization of single SPF boosters

The first screening phase presents the characterization of single SPF boosters belonging to different categories inserted in a light fluid O/W SPF 40 suncare emulsion at defined concentrations, according to each raw material Technical Data Sheet. The raw materials are considered acceptable for the second phase of the study if they allow the emulsion to remain stable, exhibiting a uniform morphology and a rheological and texture profile similar to the base emulsion.

As far as hydrophilic SPF boosters are concerned, “*Microcrystalline cellulose*” (R) and “*Cellulose, Cellulose gum*” (V) are compared to the base formula (B). The frequency sweep rheological analysis shows how R brings a significant increase in the value of both storage (G') and loss moduli (G'') already at low concentration of 1% (Figure 1 a). In addition, “*Microcrystalline cellulose*” has also a huge impact on the viscosity of the emulsion, which represents a formulation limit considering the aim of keeping a comparable sensorial profile to the starting emulsion. On the other hand, V shows a slight increase in the value of the moduli while keeping a comparable rheological behaviour to the base formula, also not showing significant differences between the two tested concentrations (Figure 1 b).

Due to the obtained results, “*Microcrystalline cellulose*” is not pursued further in the study in favour of “*Cellulose, Cellulose gum*”.

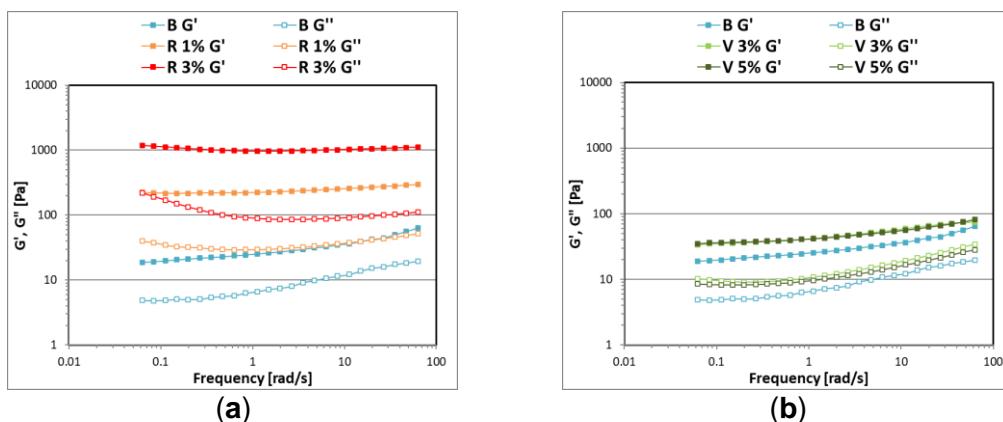


Figure 1. Trend of elastic G' and viscous G'' moduli in function of the oscillation frequency for samples containing (a) R and (b) V at different concentrations compared to B, the base formula.

Moving forward to the following category, three different lipophilic SPF boosters have been selected for characterization. “*Hydrogenated castor oil, jojoba esters, tocopherol*” (N) shows mechanical stability (centrifuge test 4800RPM /30minutes) but morphological issues, highlighting a inhomogeneous droplet distribution compared to the base formula, as it can be observed in Figure 2 (b). For this reason, the material is not pursued further within the study.

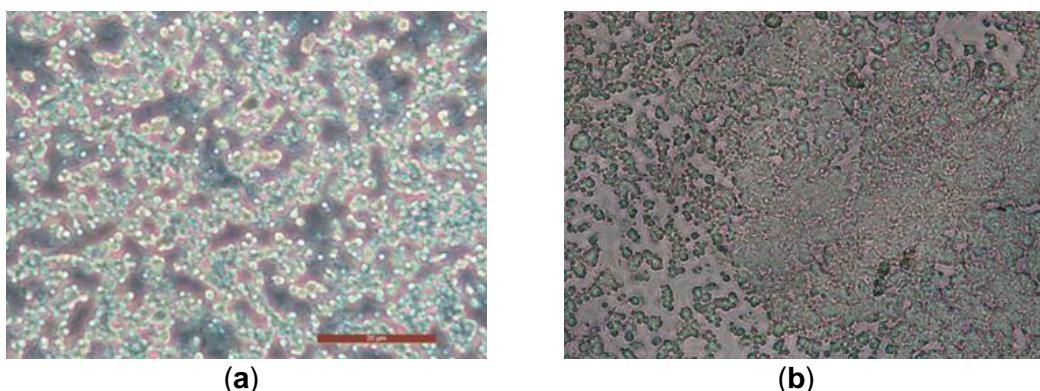


Figure 2. Microscopy image with objective 100X of (a) the base emulsion B and (b) the emulsion containing N.

Two lipophilic materials are analysed and compared to base formula. “*Hydrogenated palm oil, saccharum officinarum extract*” (U) shows an increase in the viscoelastic properties consistently with its concentration, but at 3%w/w the elastic moduli remains within the same decade of the one of the base emulsion. (Figure 3 a). “*Copernicia Cerifera Wax, Oryza Sativa Wax*” (H) presents a similar increase in the value of moduli G' and G'' at 3% w/w, while a decrease of the viscoelastic properties is observed at higher concentration, suggesting a compatibility limit over 3%w/w in the emulsion (Figure 3 b).

According to the results discussed above, both U and H are selected to move to the second phase of the study.

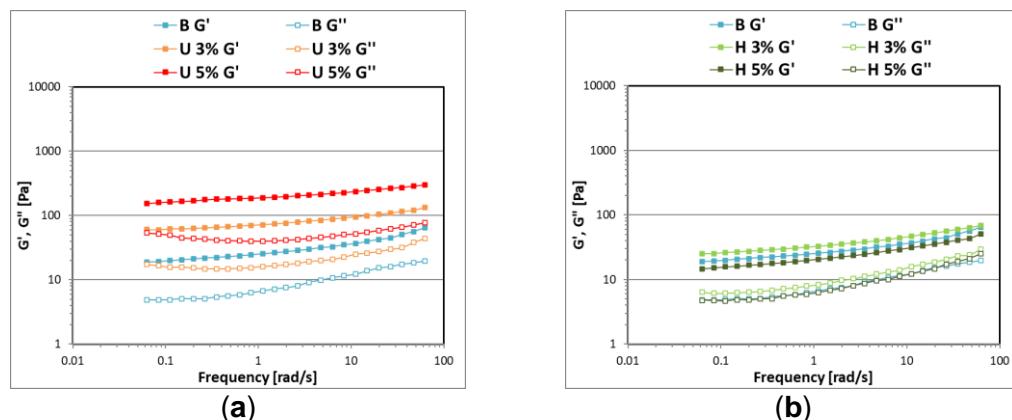


Figure 3. Trend of elastic G' and viscous G'' moduli in function of the oscillation frequency for samples containing (a) U and (b) H at different concentrations compared to B, the base formula.

Another category of SPF boosters is then evaluated, that is the one of mineral solid particles for a scattering effect. “*Zeolite*” (Z) shows mechanical instability, inducing the surfacing of oil at the centrifuge test. Moreover, it also does not provide pH stability within the range needed. For these reasons the material is dropped from the study.

On the other side “*Calcium Sodium Borosilicate*” (C), “*Cellulose*” (D), “*Magnesium Potassium Fluorosilicate*” (M) and “*Silica*” (S) have all passed preliminary stability test and are therefore analysed and compared to base formula. The frequency sweep rheological analysis of the materials reports that the addition of solid particles induces a slight increase in the viscoelastic properties of the formula with the exception of “*Silica*” (S), which provides a more structured and elastic rheological profile, as reported in Figure 4.

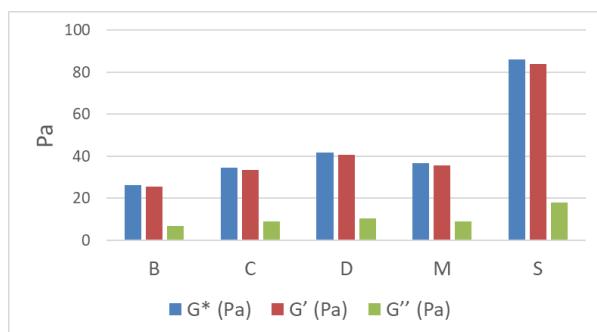


Figure 4. G^* , G' and G'' values obtained by Frequency sweep test at 1 rad/s of the base emulsion (B) compared to samples containing solid particles C, D, M and S.

For the reasons described above C, D and M are selected for the second phase of the study.

Lastly one more SPF booster category is tested in order to find the optimal film forming agent. A semi-synthetic crosspolymer “*Trimethylpentanediol/Adipic Acid/Glycerin Crosspolymer*” (W) is compared to a 100% natural origin (NOI 1) one “*Polyglyceryl-3 Stearate/Sebacate Crosspolymer*” (F). As expected, the performed analysis (rheological and texture studies) confirms that the addition of film forming agents does not alter the stability and the morphology of the studied emulsion (**Figure 5**) and has no impact on the viscoelastic and textural properties of the formula.

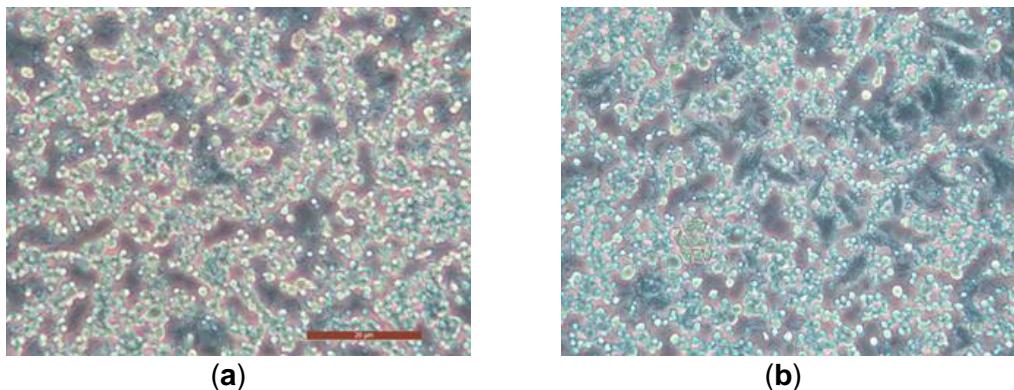


Figure 5. Optical microscopy at 100X of (a) the base emulsion B and (b) the emulsion containing F.

2.2. SPF boosters association and characterization

Based on the screening results, different associations of selected boosters are presented with the aim of exploring their compatibility and potential synergistic effect on the overall SPF through the application of morphological, rheological and texture analysis.

In order to combine boosters rationally, associations are made considering the category of each raw material. As a first step the base emulsion is enriched with a 1% w/w of the film forming agent “*Polyglyceryl-3 Stearate/Sebacate Crosspolymer*” to provide water resistance to the formula. This ingredient is going to be in all the emulsions from this point of the work to the end, substituting base formula (F).

In the first systematic a **lipophilic booster alongside spherical and plane mineral particles** are combined to provide a thicker oily layer that could extend the optical path length of the solar radiation on the skin, while also inducing a scattering effect due to the presence of solid particles. Solid particles are combined together in order to obtain a synergistic scattering effect

between spherical and plane patterns, which is the reason behind the use of “*Magnesium Potassium Fluorosilicate*” (M) in combination.

“*Hydrogenated palm oil, saccharum officinarum extract*” (U) at 2% w/w is added to the emulsion together with “*Calcium Sodium Borosilicate*” (C) at 3% w/w and “*Magnesium Potassium Fluorosilicate*” (M) at 0,5% w/w. Simultaneously the emulsion is compared to an equivalent system containing “*Copernicia Cerifera Wax, Oryza Sativa Wax*” (H) at 1% w/w associated with the same solid particles.

On a preliminary mechanical test, the emulsions are stable but within days FUCM starts to change its organoleptic properties and evolve into a thick, not fluid and dry compound. The texture analysis comparing base formula (F), the emulsion containing “*Hydrogenated palm oil, saccharum officinarum extract*” (FUCM) and the emulsion with “*Copernicia Cerifera Wax, Oryza Sativa Wax*” (FHCM) shows a significant increase in consistency and cohesiveness of FUCM (Figure 6), testifying an incompatibility between “*Hydrogenated palm oil, saccharum officinarum extract*” and the solid particles, suggesting an adsorption process taking place within time.



Figure 6. Histograms of texture parameters (a) consistency and (b) cohesiveness measured by means of an immersion de-immersion test comparing samples F, FUCM, FHCM.

The second systematic has the aim of associating **lipophilic booster** “*Copernicia Cerifera Wax, Oryza Sativa Wax*” (H) at 1% w/w with the **hydrophilic** one “*Cellulose, Cellulose gum*” (V) at 2% w/w, in order to provide a thicker and more efficient layer for UV filters along with a better stabilization of the water phase. In addition to that, the use of both can mitigate the greasy feeling of waxes with a light watery texture. Both raw materials are trialed first alone in the base emulsion and then associated to evaluate their contribution to the formula, as it can be observed in Figure 7. The frequency sweep rheological analysis (Figure 7 a) reports a general increase in the viscoelastic properties of the emulsion once boosters are added, but the most difference is observed when boosters are combined together (FVH). Accordingly, $\tan\delta$ (Figure 7 b), which represents the ratio between loss modulus G'' and storage modulus G' , reports a decrease regarding the combinations tested, indicating a strengthening of the emulsion's elastic behaviour.

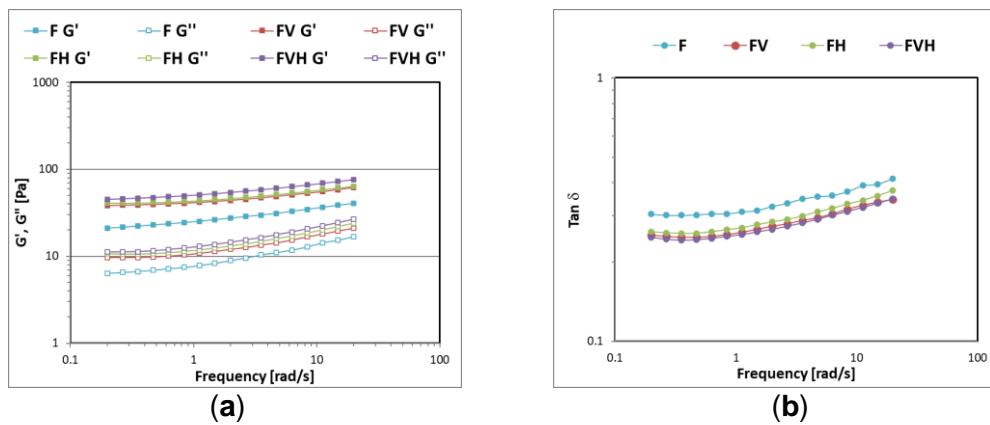


Figure 7: (a) Trend of storage modulus G' and loss modulus G'' in function of oscillation frequency and (b) dumping factor $\tan\delta$ for samples F, FV, FH, FVH.

Following the results described above, the two **solid spherical materials** selected for the study, “*Calcium Sodium Borosilicate*” (C) and “*Cellulose*” (D), appear to exhibit very similar properties, thus a comparative analysis between them is conducted. Both “*Calcium Sodium Borosilicate*” (C) and “*Cellulose*” (D) are added to the base emulsion containing the film forming agent at a concentration of 1.5% w/w and then compared. Figure 8 reports the rheological analysis of the formulas, where D is shown to provide a greater elastic contribution compared to C. This is evidenced by both the frequency sweep analysis and the $\tan\delta$ values, which tend toward zero.

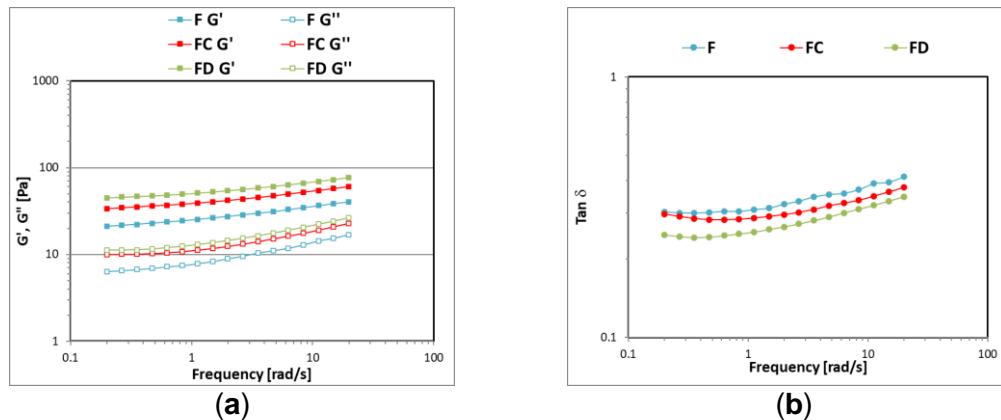


Figure 8: (a) Trend of storage modulus G' and loss modulus G'' in function of oscillation frequency and (b) dumping factor $\tan\delta$ for samples F, FC and FD.

The research proceeded by introducing spherical and plane solid particles to the association FVH.

The former spherical particle introduced in formula is “*Calcium Sodium Borosilicate*” (C) which is subsequently combined with plane particles of “*Magnesium Potassium Fluorosilicate*” (M) for a total particle concentration of 1,5% w/w.

The microscopy reports an increase in the thickness of the internal phase when powders are introduced in formula, with close and homogeneous oil droplets and geometrical spherical particles dispersed around the emulsion. Also no apparent differences are observed in the presence of M (Figure 9 b).

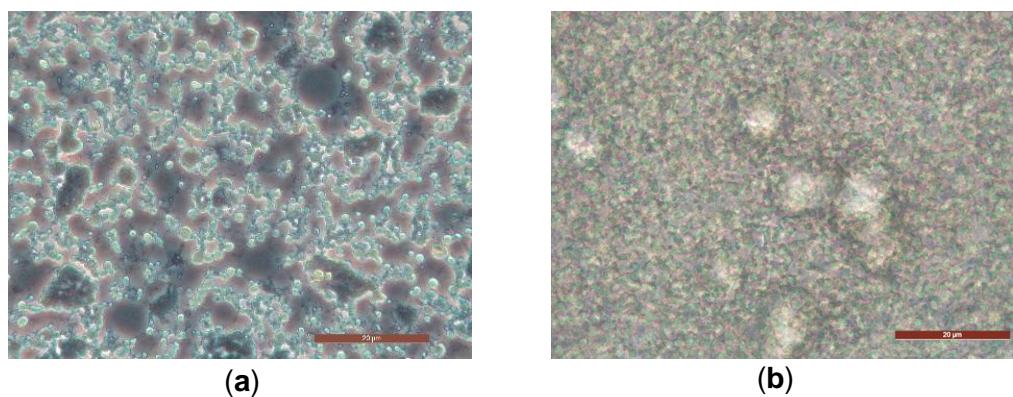


Figure 9: Optical microscopy at 100X of (a) sample FVH and (b) FVHCM.

A subsequent association containing spherical particles of “*Cellulose*” (D) combined with “*Magnesium Potassium Fluorosilicate*” (M) is also trialed, maintaining a total concentration of particles of 1,5% w/w. Both materials are evaluated morphologically in the formula FVH in order to observe their dispersion in the emulsion, as shown in Figure 10 b. As depicted, the spherical solid particles are clearly visible and dispersed around the emulsion, which appears thicker than the one not containing solid boosters.

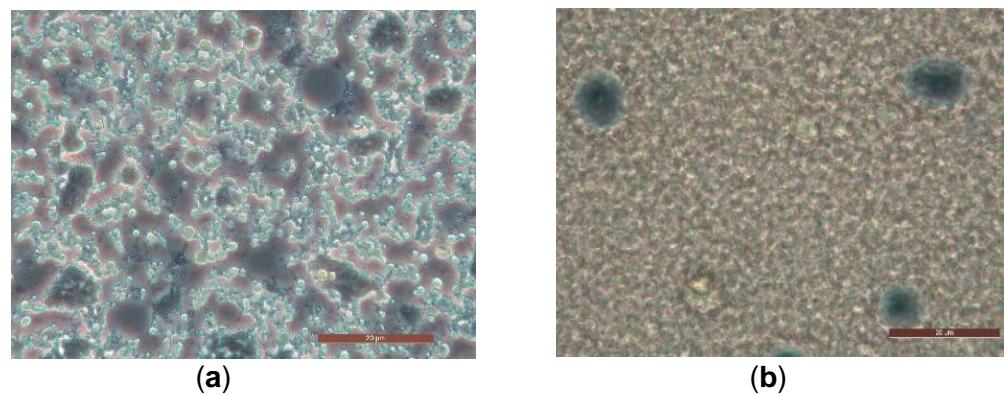


Figure 10: Optical microscopy at 100X of (a) sample FVH and (b) FVHDM.

In order to evaluate the boosters contribution to the internal structure of the formula, rheological and texture analysis of both combinations are performed and thereby presented. The frequency sweep rheological analysis reports an increase in the value of moduli G' and G'' coherently with the addition of solid particles, showing that the combination of C alongside M might be better than C alone. On the contrary, a difference between D alone and its association with M is observed, suggesting M incompatibility with D in the formula. The increase in the viscoelastic properties is also supported by the tanδ graph which indicates a general strengthening of the elastic properties without underlying any difference between the single particles tested.

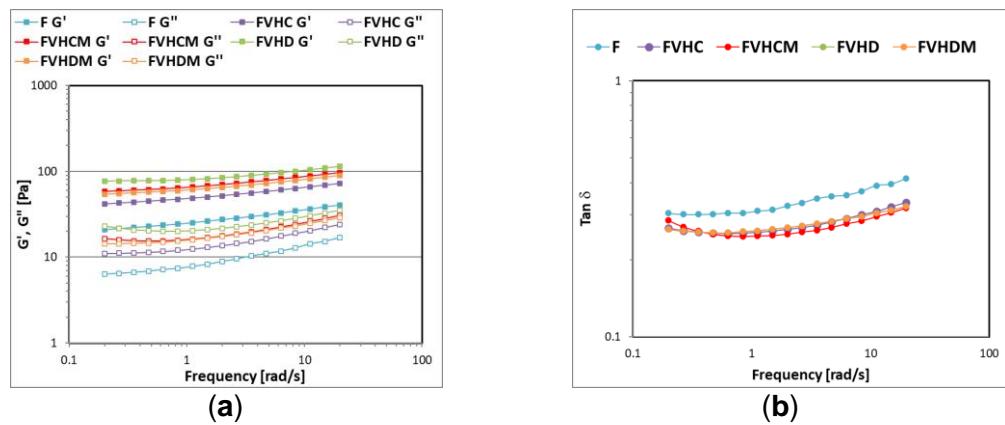


Figure 11: (a) Trend of storage modulus G' and loss modulus G'' in function of oscillation frequency and (b) damping factor $\tan\delta$ for samples F, FVHC, FVHCM, FVHD and FVHDM.

A further texture analysis is carried on for the same formulations (Figure 12). Coherently with the results described above, an increase in the texture parameters is observed in the emulsions containing boosters when compared to base formula, while no significant differences are observed between the presence of C, D or M, thus maintaining a light fluid texture profile.

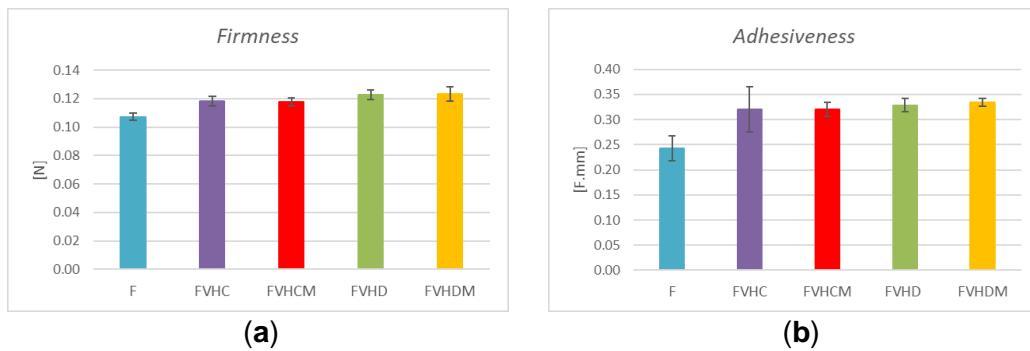


Figure 12: (a) Firmness and (b) adhesiveness texture parameter for samples F, FVHC, FVHCM, FVHD and FVHDM.

The results of the introduction of solid particles in the emulsion suggest that “*Calcium Sodium Borosilicate*” (C) could be efficiently combined with plane particles of “*Magnesium Potassium Fluorosilicate*” (M). These materials are compatible both with hydrophilic and lipophilic boosters resulting in a stable and homogeneous emulsion.

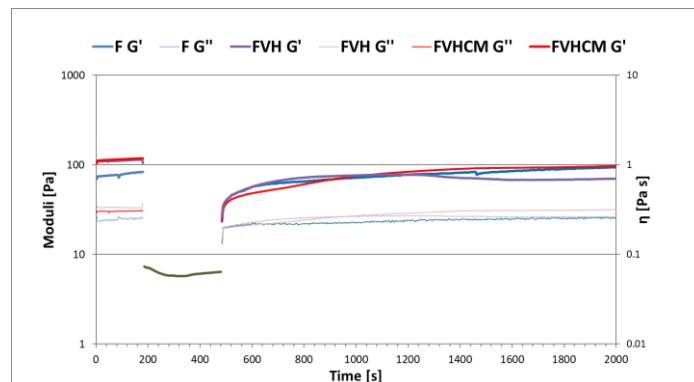


Figure 13: Trend of G' and G'' of F, FVH, and FVHCM before and after a constant shear rate of 5000 s^{-1} for 5 minutes.

The final part of the study has been carried out to evaluate the elastic recovery of selected booster-combined emulsions and the subsequent in-vitro SPF test [9], in order to detect any improvement correlating the presence of boosters to the SPF measurement. All the emulsions have a good elastic recovery of the structure without any significant differences of the emulsions with SPF boosters compared to the base (Figure 13). Also the in-vitro SPF test reveals that no significant increase in the overall SPF occurs between the analysed formulations.

4. Conclusion

This study aims to characterize and investigate SPF booster raw materials for incorporation into sun care formulations employing morphological, rheological and texture-based analysis. The systematic approach adopted for the evaluation of raw materials enables the detection of instability issues and incompatibility with the emulsion. The work demonstrates the potential of rationally combining raw materials with different functional roles, in most cases identifying stable and improved formulations compared to the original one. The instrumental protocol employed in this study serves as a valuable tool for the rational selection and combination of SPF boosters. The outcomes obtained through this method have also been verified through an in-vitro SPF test that shows no significant improvements in the overall SPF.. Nonetheless in-vivo SPF tests are required in order to make a comprehensive evaluation of the efficacy of boosters and therefore address the concerns related to UV filters on both human health and the environment.

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