Rheology and Texture Analysis to Predict Stability and Scale-Up in Particle-Stabilized Fluid Emulsions for Dermatological Applications: A Systematic Approach

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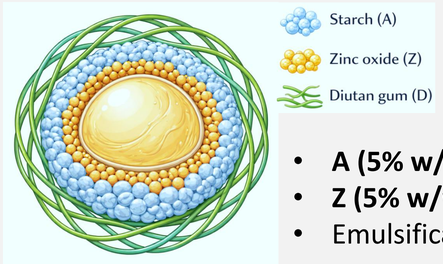
INTRODUCTION

Pickering emulsions stabilized by solid particles are gaining increasing interest in cosmetic science due to their enhanced skin and environmental compatibility. However, their use is limited by drawbacks such as instability, the need for high solid loads, and the resulting poor sensoriality[1].

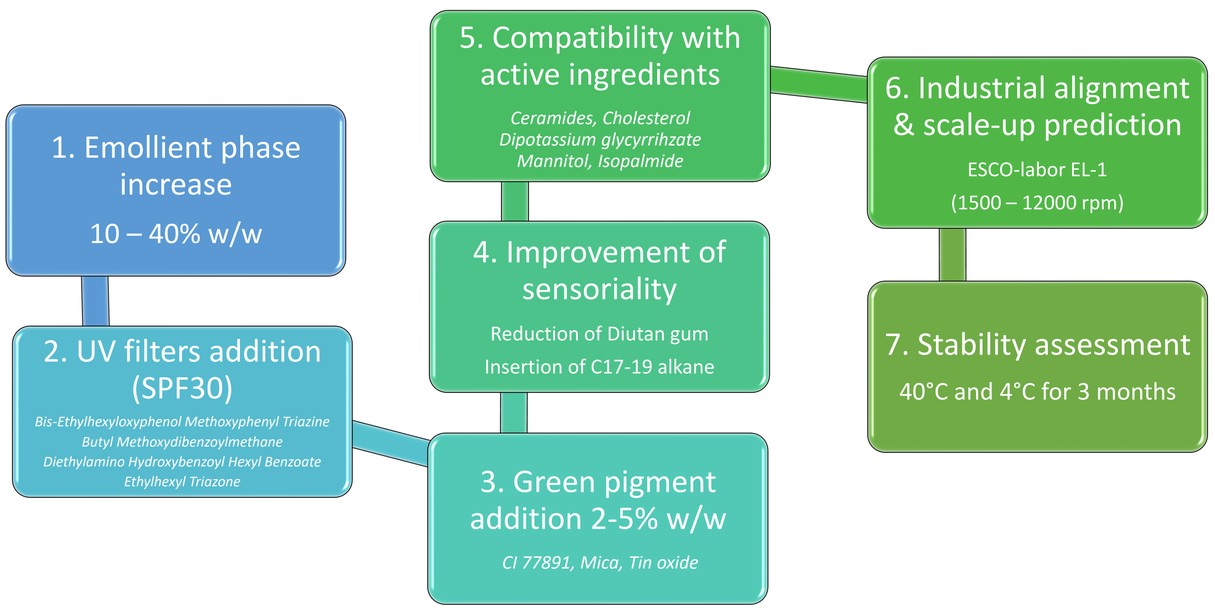
This work aims to develop a **fluid, light-texture, stable cosmetic base** suitable for incorporating **UV filters** and **dermatological active ingredients**.

FORMULATION OF THE O/W PICKERING EMULSION[2]



- **10% w/w solid phase**: A + Z **(1:1)**
- **10% w/w oil phase**: Dicaprylyl carbonate + Caprylic/capric triglyceride + Diisopropyl sebacate (1:1:1)
- **A (5% w/w)** and **D (0.1% w/w)** dispersed in water phase at 40°C
- **Z (5% w/w)** was dispersed in oil phase at 45°C
- Emulsification under Silverson L5T at 4500 rpm

EXPERIMENTAL PLAN AND METHODS



RHEOLOGY

The samples' **viscoelastic properties** (*η viscosity, G' storage, G'' loss, G* complex moduli, and loss factor tanδ*) were measured in continuous and oscillatory flow conditions using a rotational Rheometer Physica MCR-302 (Anton-Paar) at 23 ± 0.05°C, equipped with CC17 coaxial cylinders for low-viscosity systems.

TEXTURE ANALYSIS

An immersion/de-immersion test was performed with the Texture Analyzer TMS-Pro (Food Technology Corporation) equipped with a 10 N load cell and a nylon spherical probe (2 cm diameter). The probe penetrated the sample at a speed of 80 mm/min to a depth of 10 mm. From the **texture curve** the related parameters were obtained (*firmness, consistency, cohesiveness, adhesiveness, stringiness*).[3]

OPTICAL MICROSCOPY

Emulsions **morphology** was observed under LEICA DM1000 microscopy with 40X and 100X immersion oil objectives.

RESULTS AND DISCUSSION

1. EMOLLIENT PHASE INCREASE (from 10 to 40%) to assess the ability of the system to stabilize higher oil fractions and enable UV filters incorporation. All formulations were stable under centrifugation (3000 rpm, 30 min).

Amplitude sweep analysis showed a progressive increase in G' and G'' with increasing oil phase (Fig. 1a).
Texture analysis revealed increased adhesiveness and stringiness, suggesting a potential rise in greasiness and stickiness (Fig. 1b).

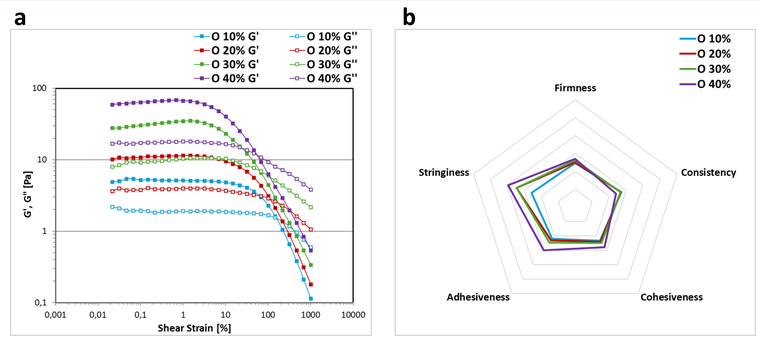


Figure 1: *viscoelastic moduli in function of shear strain (a) and texture parameters (b) for emulsions with increasing oil phase "O" amount.*

2. UV FILTERS ADDITION to achieve SPF 30 (estimated with BASF Sunscreen Simulator).
The total emollient phase was increased to 22.5%, with an oil-to-filter ratio of 3:2 to ensure proper solubilization and formulation stability. Rheological parameters increased as reported in Table 1.

Table 1: *Rheological parameters measured for emulsion without and with UV filters.*

Parameters	NO SPF	SPF 30
η (0,01 s^{-1})	140 Pa.s	405 Pa.s
G* (1 Hz)	14 Pa	42 Pa
tanδ (1 Hz)	0,3	0,3

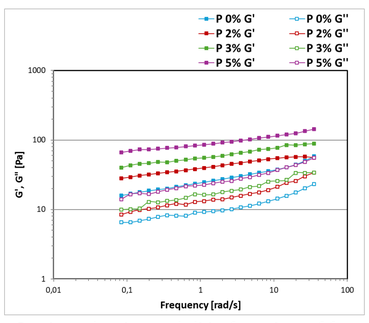


Figure 2: *viscoelastic moduli in function of frequency for emulsions with increasing amount of pigment "P".*

3. GREEN PIGMENT ADDITION to counteract discoloration associated with couperose. Frequency sweep analysis showed a progressive increase in viscoelastic structure with increasing pigment concentration (Fig. 2). A 2% pigment level was selected to avoid excessive structuring while maintaining a light and fluid texture. However, this sample showed poor spreadability and skin pilling effect during application.

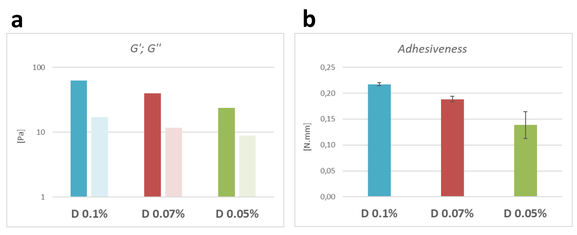


Figure 3: *G' and G'' (1Hz) (a) and adhesiveness (b) measured for emulsions with decreasing amount of "D".*

4. IMPROVEMENT OF SENSORIALITY of the pigment-containing sample. *Caprylic/capric triglyceride* was reduced to 3% to limit greasiness, while 3.5% *C15–19 alkane* was introduced to enhance spreadability. *Diutan gum* was reduced to minimize skin pilling, leading to a significant decrease in viscoelastic moduli (Fig. 3a) and adhesiveness (Fig. 3b). The lowest concentration resulted in phase separation under centrifugation, thus 0.075% was selected as the optimal compromise.

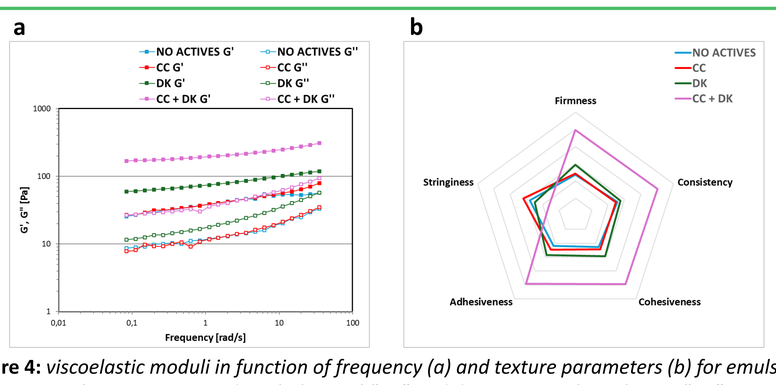


Figure 4: *viscoelastic moduli in function of frequency (a) and texture parameters (b) for emulsions with actives: ceramides, cholesterol "CC" and dipotassium glycyrrhizate "DK".*

5. COMPATIBILITY WITH ACTIVE INGREDIENTS with soothing, anti-inflammatory and barrier-repair properties.

The incorporation of Ceramides and Cholesterol in the oil phase did not significantly affect rheological properties. In contrast, Dipotassium glycyrrhizate led to excessive structuring (Fig. 4 a,b) and separation under aging test.

The combination of Mannose and Isopalmide resulted in a slight increase in moduli (Fig. 5a), while maintaining a fluid texture (Fig. 5b).

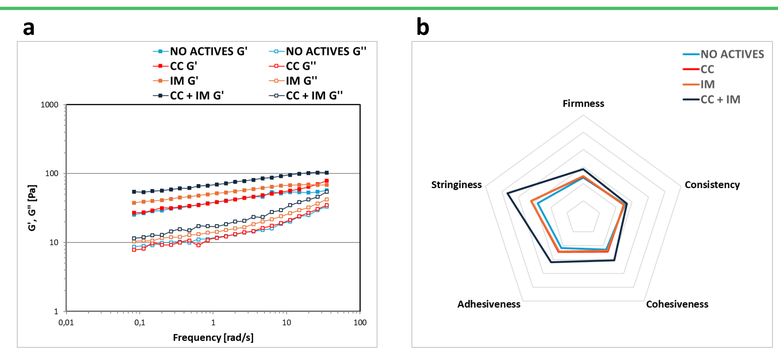


Figure 5: *viscoelastic moduli in function of frequency (a) and texture parameters (b) for emulsions with actives: ceramides, cholesterol "CC", isopalmide, and mannitol "IM".*

6. INDUSTRIAL ALIGNEMENT AND SCALE-UP PREDICTION: Formulations were prepared using an ESCO-Labor pilot EL-1 unit to mimic industrial scale-up conditions (turbine geometry and vacuum).

High shear (12,000 rpm) during emulsification at 80°C was required to ensure dispersion of zinc oxide preventing precipitation and separation (Fig. 6).

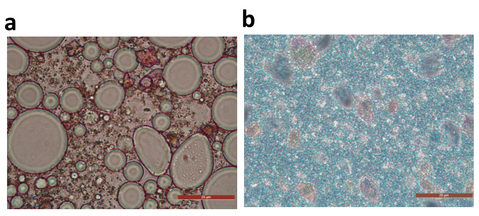


Figure 6: *Optical microscopy at 100X of emulsions formulated with ESCO-labor at 1500 (a) and 12000 (b) rpm.*

ESCO-processed samples showed slightly higher viscoelastic moduli compared to lab-prepared systems. The inclusion of active ingredients led to increased elasticity (lower tan δ) (Fig. 7).

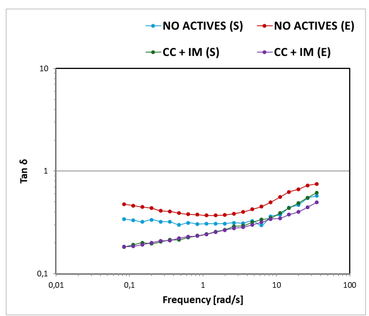


Figure 7: *tanδ values for emulsions with and without actives formulated with Silverson "S" and Esco labor "E".*

7. STABILITY ASSESSMENT: All samples were stable after 3 months at 23°C , 40°C, and 4 °C.

However, the formulation with actives showed a significant increase in viscoelastic structure and elasticity at 4 °C (Fig. 8).

This behavior can be attributed to temperature-induced structuring of the oil phase and enhanced interactions between actives and the polymer network.

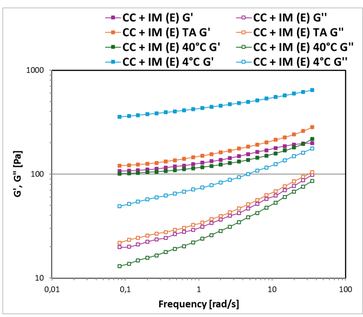


Figure 8: *Moduli in function of frequency for emulsions after 3 months at room temperature, 40°C and 4°C.*

CONCLUSIONS

The systematic approach through **Rheology and Texture analysis** allowed:

- **Rational design** of particle-stabilized emulsions,
- **Development of an innovative system** containing dermatological actives,
- Predictive **assessment of stability** and **scale-up** issues,
- Optimization of **sensorial and application properties.**

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