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High Internal Phase (HIP) Gel-in-Oil Emulsions: optimization of composition and preparation factors

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Introduction of research

The concept of HIP Gel-in-Oil to produce sustainable cold processed emulsions with original aesthetics was highlighted some years ago. One of its strengths is to achieve similar emollience perception to classical cream gel, O/W and W/O emulsions with half of the oil concentration and a fresh skin contact (IFSCC, 2014). However, despite general formulation guidelines, it has not gained popularity. Formulators are skeptical and opt for a more traditional well-known approach.

Objective: Using rheology experiments, we aimed to study key composition and manufacturing factors to precise the limitations and find a way to check quickly after manufacturing that the Gel-in-Oil formulation is optimized.

Materials & methods: Gel-in-Oil emulsions are obtained by the addition of a small quantity of lipophilic phase to a large quantity of hydrophilic phase. The performance of the surfactant system included in the lipophilic phase combined with the specific rheological profile of the polymer-gelled phase leads to phase inversion from the very first minutes, i.e. internalization of the gel phase (Figure 1).

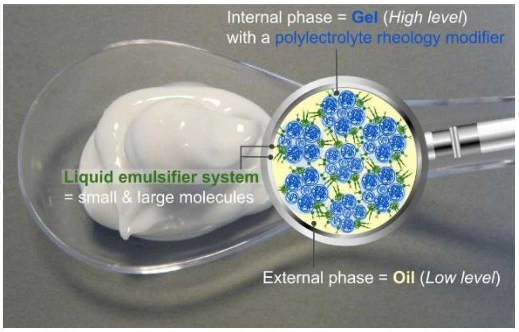

Figure 1. Main components of HIP Gel-in-Oil emulsion

Formulation experiments were carried out by changing the parameters one at a time to see how the different factors affect the emulsion structure, stability and behavior under stress: (1) gel viscosity by increasing the doses of a suitable rheology modifier, (2) ratio between the internal aqueous phase and external oily phase, (3) oil nature, (4) emulsifier level, (5) influence of preparation mode: direct or indirect, mixing or homogenization. Conventional characteristics, conductivity and stability were followed.

Rheology experiments were conducted using a rotational Controlled stress/ strain (DHR2; TA Instruments; cone 40mm/2°; 20°C). The Global flowing profile and yield stress (steady state flow protocol; Onset mode) were monitored. Viscoelasticity was analyzed using frequency sweep to evaluate the level of structure of the emulsion. G' (storage/elastic modulus) and G'/G" ratio were followed as indicators of the formula structure.

Results & Discussion: Composition factors to get an optimized HIP Gel-in-Oil emulsion were determined. The aqueous gel needs to reach a viscosity of at least 18,000 mPa.s (0.8% of tested rheology modifier). For more fluid gels, defective cream gels are obtained as signaled by higher conductivity value. Rheology experiments highlighted a stronger shear thinning character, high yield stress and a high elastic structure of Gel-in-Oil emulsions compared to cream gels, reflecting the HIP organization (Figure 1). This strong elastic structure was consistent with formula stability at room temperature and 45°C. A range of concentrations of emulsifier and internal aqueous phase could also be specified (1.5 to 3% and 80 to 90% respectively). A manufacturing process with medium shear using an anchor provided the best results while the mode of addition of the 2 phases had no impact. Flowing experiments proved to be a useful tool to precise how to reach an optimized Gel-in-Oil composition. Indeed, slope failure in the flowing profile indicated some weakness of the structure under stress (Figure 2), associated with a sudden drop in viscosity compared to optimized Gel-in-Oils that reacted with a standard shear thinning profile (Figure 2). Common parameters such as viscosity, conductivity or microscopy monitoring were not able to indicate that risk.

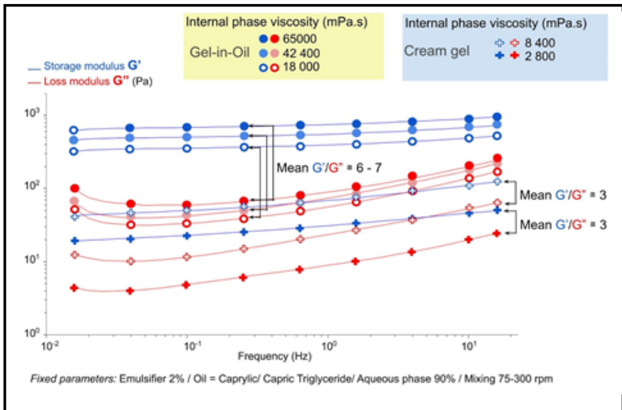

Figure 1. Impact of gel viscosity

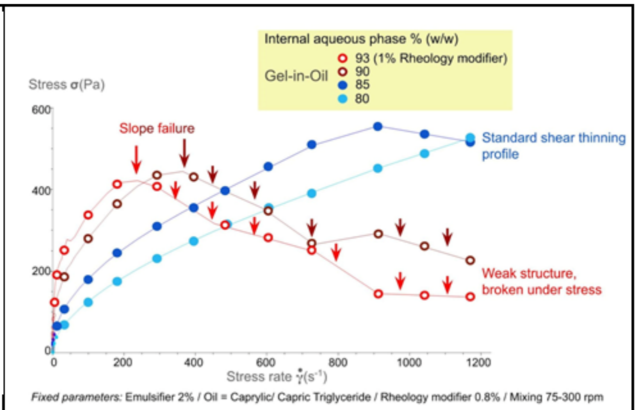

Figure 2. Impact aqueous phase concentration

Conclusion

The results of this study enable precise recommendations to get stable HIP Gel-in-Oil emulsions, both on the composition (minimum viscosity of the internal gel phase; range of concentrations of emulsifier; ratio between the two phases) and on mixing process. Weaknesses in the structure that are not visible in the short term with the parameters usually followed and can support the formula development can now be detected by rheology. In addition, Relationships between conductivity, rheology profiles and formulation stability were observed. This study removes the legitimate uncertainty of formulators in the face of an original and sustainable formulation concept.

About the speaker

Alicia Roso joined Seppic in 1986 and holds a position in the Research and Innovation team as the scientific communication manager. She has worked as a Chemical engineer for twenty years in the cosmetic R&D team. She joined the marketing team in 2006 as product manager and gained a marketing MBA from ESSEC business school in 2012. She was named as Air Liquide International Expert for health care formulation and emulsions in 2010. She is co-author of 23 patents on new ingredients or formulation technologies dedicated to cosmetology and dermopharmacy applications.