STUDY OF THE RELEASE OF ON ENCAPSULATED ACTIVE IN DOUBLE EMULSION (DONE IN COLLABORATION WITH ADEMTECH)

INTRODUCTION

Double emulsions are colloidal systems of great interest for the cosmetics industry as they enable to encapsulate both hydrophilic and lipophilic molecules. However, their formulation is very delicate and requires an important know-how. Their characterization is also quite limited with classical techniques that, for most of them require dilution (particle size), or are not quantitative (microscopy).

In this application note, the release of an encapsulated active in the internal emulsion is studied using the Turbiscan technology. To release the active, the double emulsion has to be destabilized into a simple emulsion were the active will results in the external phase. This principle is usually used for topical emulsions were some constituents on the surface of the skin will trigger the destabilization of the double emulsions and so activate the release of the active. By measuring the emulsion breakdown of the formulation using the Turbiscan™, the release of the active is evaluated

PRINCIPLE

Measurement with Turbiscan®

Turbiscan™ instrument, based on Static Multiple Light Scattering, consists in sending a light source (880 nm) on a sample and acquiring backscattered and transmitted signal. Combining both detectors (BS & T) enables to reach wider concentration range. The backward reflected light comes from multiple scattering as the photons scatter several times on different particles (or drop).

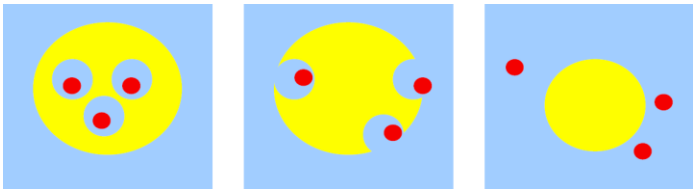
This signal intensity is directly linked to the diameter (d), according to the Mie theory:

$$d = f(BS, \varphi, n_p, n_f)$$

More information

METHOD

The coalescence of the internal droplets of a double emulsion on the wall of the globules leads to the release of the encapsulated active ingredient. This coalescence is trigger by the addition of a surfactant of high HLB in the external phase. The emulsion that was initially double is transformed into a simple emulsion. This instability is critical, both for the emulsion storage and for the release of the active ingredient during application (Figure 1).



Close-up of a droplet in proximity to the wall of the globule. The active ingredient is represented by a red dot.

Close-up of the oil film that separates the droplet and the external aqueous phase. The high HLB surfactant helps the formation of a hole in the film.

Following the formation of a hole in the film: the active ingredient is released into the external aqueous phase.

Figure 1: Coalescence of the internal droplets

In this application, a double emulsion of water/hexadecane/water is formulated with encapsulated salt in the internal phase.

First the stability of the double emulsions is studied to validate the stability of the double emulsion during storage.

Then the effect of an excess of surfactant (SDS) is evaluated on the double emulsions stability. The aim is to simulate the action of the skin playing a role of hydrophilic surfactant and so trigger the destabilization. The double emulsion is analyzed with the Turbiscan at 33°C (skin temperature) during 30 minutes. The SDS is added at the concentration of:

- 0.5 CMC
- 1.5 CMC
- 3.0 CMC

RESULTS

By scanning the formulation according to the method, the graphs in Figure 2 are obtained

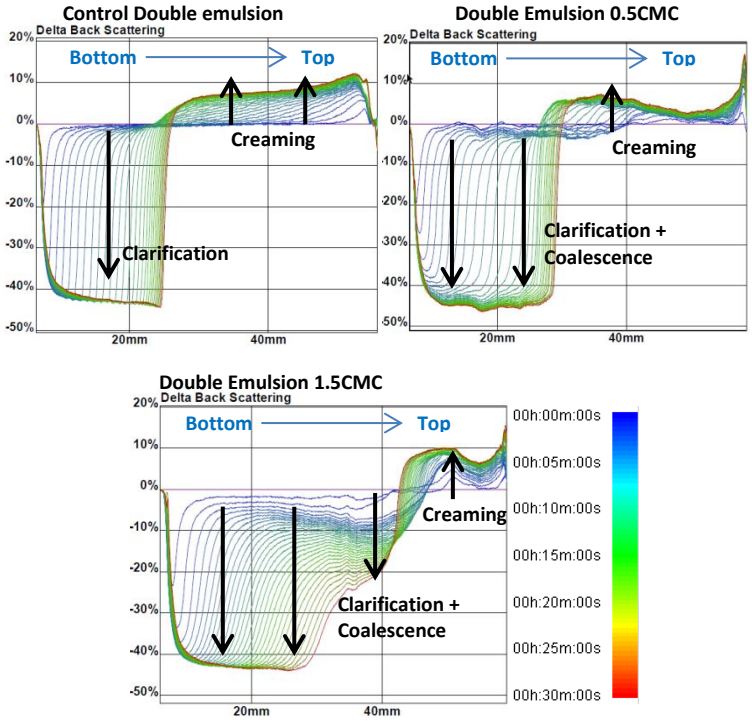


Figure 2: Backscattering variation of the samples

Thanks to the previous graphs we can observed:

- The creaming of the double globule when no surfactant is added. There is no destabilization regarding the coalescence of the internal droplets with the external phase. This can be concluded by looking the fingerprint curve of backscattering over time.
- When SDS is added, the Backscattering intensity profile is modified showing the coalescence and so the release of an active

To evaluate the effect of the concentration of SDS on the coalescence of the double emulsions, the global stability (TSI) of all samples is measured for the clarification layer at the bottom of the sample.

The **T**urbiscan **S**tability **I**ndex (TSI) is an automatic computation who sums all the variations detected in the sample (creaming, coalescence, size variation …). At a given ageing time, the higher is the TSI, the worse is the stability of the sample.

(TSI: more information)

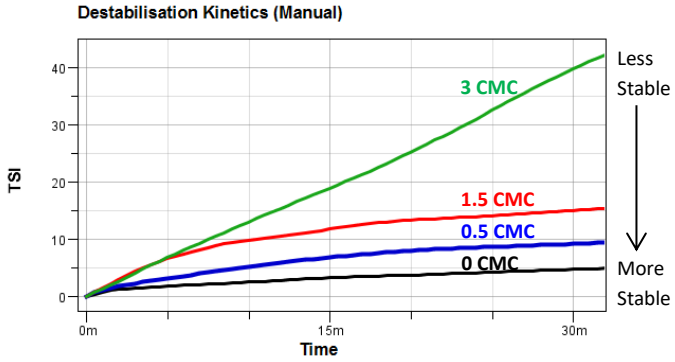


Figure 3: TSI for all samples

SDS concentration	TSI (30 minutes)
3 CMC	40.7
1.5 CMC	15
0.5 CMC	9.1
0 CMC	4.7

Less Stable ↓ More Stable

Table 1: TSI values for all samples

It is not possible to differentiate from the overall decrease of the backscattering the variation induce by the creaming and the variation induce by the coalescence but, by computing the global change for all this area, the effect of the concentration of SDS is evaluated.

A significant acceleration of destabilization kinetics is measured when the concentration of surfactant with high HLB is added. Consequently the release of the active (salt for this case) is more important.

These experiments simulate what would happen *in vivo*, for a topical formulation, as some constituents of the skin play the role of hydrophilic surfactant and trigger the release of the active ingredient initially encapsulated in the droplets.

SUMMARY

In this application, the release of an active in a double emulsion according to the concentration of surfactant in the external phase was studied. The addition of surfactant trigger the coalescence of the internal aqueous phase with the external aqueous phase and so allow the release of the active. By increasing the concentration of surfactant, the kinetic of coalescence is accelerated. It was observed that with no surfactant, the double emulsion was stable regarding the coalescence. This study was done using the Turbiscan™ technology in only 30 minute of measurement with the aim to simulate topical applications.