Pre-formulation of a double emulsion

Done in collaboration with ademtech

Application
Cosmetics

Objective
Choose the nature and amount of surfactants to formulate a double emulsion

Device
TURBISCAN® LAB

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 characterisation is also quite limited with classical techniques that, for most of them require dilution (particle size), or are not quantitative (microscopy).
The study of the kinetics of release of the active encapsulated in the internal emulsion is also very interesting for the formulator. However, here again, very few techniques are available apart from the Turbiscan LAB (see application note “Study of the release of an encapsulated active in a double emulsion”).

METHOD

To formulate a double emulsion it is necessary to choose, at least, an oil and two surfactants, one low in HLB and one high in HLB. In the example mentioned here, we have been working with Span surfactants (HLB<5) and Tween surfactants (HLB>10) and with a vegetable oil (caprylic/capric triglyceride).
The first stage involves making "state diagrams" that provide the means for pre-selecting formulas. Figure 1 a picture of some samples is presented. Each tube contains 50% aqueous phase and 50% oil. The concentrations of surfactant studied are 0.1, 1 and 10%. Each tube is agitated for 10 seconds using a vortex, then examined.
All of the samples that lead to an emulsion where drops are discernible to the naked eye or that give rise to a rapid separation of phases are rejected due to their instability. The emulsions that sediment are also eliminated (inverse emulsions).
Those selected for in-depth study are the very white emulsions that cream slowly and/or moderately. These pre-formulations are then analyzed with the Turbiscan LAB.

Surfactant	HLB	Surfactant	HLB
Tween 40	15.6	Span 60	4.7
Tween 60	14.9	Span 80	4.3
Tween 80	15.0	Span 85	1.8

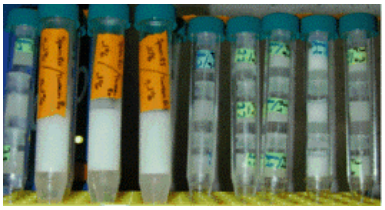

Figure 1. Preselection of the most stable formulations.

RESULTS

1. Analysis of Backscattering

The backscattering spike that appears in the lower part of the cream is the mark of the double globules whose density is higher than simple drops of oil (Figure 2). This is why, during the creaming of oil drops, they remain in the bottom of the cream. This is confirmed by microscopic observation of samples (Figure 3).

Backscattering profiles made with the Turbiscan LAB highlight noticeable differences between the three formulas.

- The average backscattering intensity in the core of the sample gets higher from left to right (40 to 50%). This translates a decrease of the average drop size in the emulsion hence, an increase in stability.
- The area of the backscattering spike is at its maximum for the formula presented above on the right. The formulator may choose to formulate a double emulsion rich

in Span 85 (about 10%) or an emulsion that is less rich in surfactant (1% Span 80), but also less suitable than the previous one, *a priori*, to form a double emulsion.

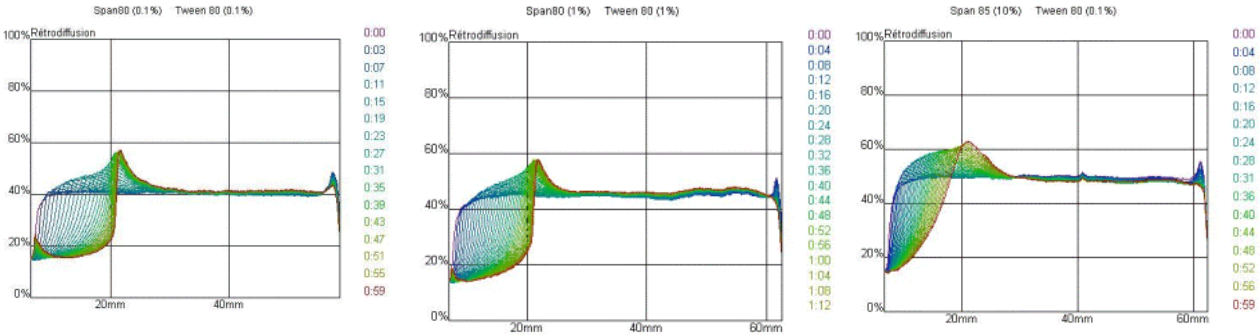

Figure 2. Backscattering profiles for various double emulsions

2. Optimisation of the formulation

Once the components and the concentrations are coarsely defined by the pre-formulation stage, what remains for the formulator is to implement the process of his choice. It is during this last phase that he can optimize the formula regarding :

- the concentration of the high HLB surfactant, a potential source of destabilization of the double structure
- the concentration of the low HLB surfactant, linked to instabilities between globules
- the osmotic pressure of the internal and external aqueous phases
- the creaming (addition of a thickener in the external aqueous phase)

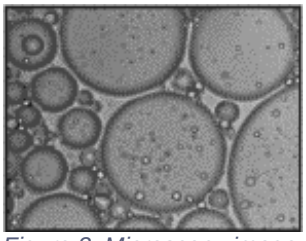

Figure 3. Microscopy image of Span 80 1%, Tween 80 1%

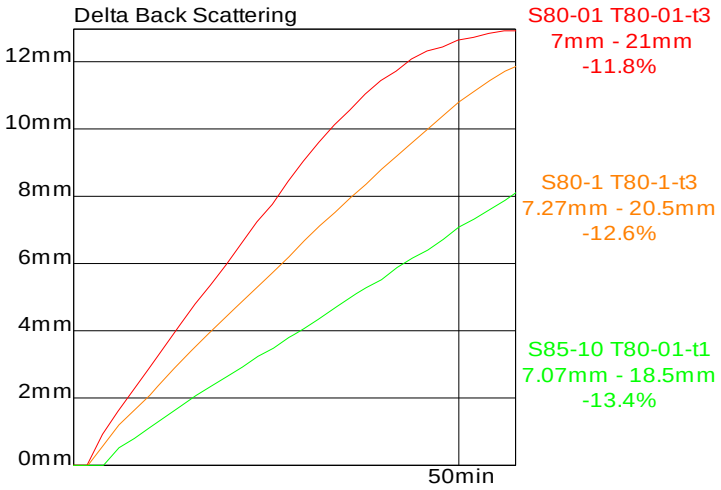

Figure 4. Creaming velocity

SUMMARY

We have presented a possible method to formulate a double emulsion. Once the components chosen, one has to study different mixtures and pre-select some formulations according to the macroscopic appearance. The formulations selected in this way are analyzed with the Turbiscan LAB. Those that present a backscattering spike in the lower part of the cream (the mark of a double emulsion when formulating emulsions) are kept. By comparing the latter formulations (backscattering intensity in the core of the sample, area of the backscattering spike, speed of creaming of double globules), the best one is selected. The formulator will then have to find the right process by adjusting the different parameters.