HLB EFFECT ON 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, a method to formulate and optimize a double emulsion is shown using the Turbiscan™ technology.

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:

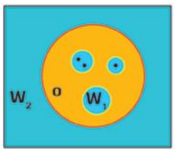
$$d = f(BS, \varphi, n_p, n_f)$$

More information

METHOD & RESULTS

The formulation of a double emulsion is quite complex and require several steps to insure a stable product. In this note, the formulation of a w/o/w (encapsuled water in droplet of oil in external water phase) is explained in three steps:

- Pre-formulation of the emulsion
- Formulation of the double emulsion
- Optimization of the double emulsion



1. Pre-formulation of the emulsions

To formulate a double emulsion (w/o/w), it is necessary to choose one oil (caprylic/capric triglyceride) and two surfactants, one with a low HLB (HLB < 5) and one with a High HLB (HLB > 10). Six different surfactants (Table 1) are tested at 3 different concentrations (0.1; 1 & 10%) given us 18 emulsions with a ratio 50/50 (v/v oil/water).

Surfactant	High HLB	Surfactant	Low 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

Table 1: Choice of the surfactant

The simple emulsion oil in water and water in oil has to be stable in order to be able to formulate a stable double emulsion. Thanks to the Turbiscan™ and its automatic computation of the **T**urbiscan **S**tability **I**ndex (TSI), the emulsion stability can be discriminated. The TSI sums all the variations detected in the sample (creaming, size variation, …). At a given ageing time, the higher is the TSI, the worse is the stability of the sample.

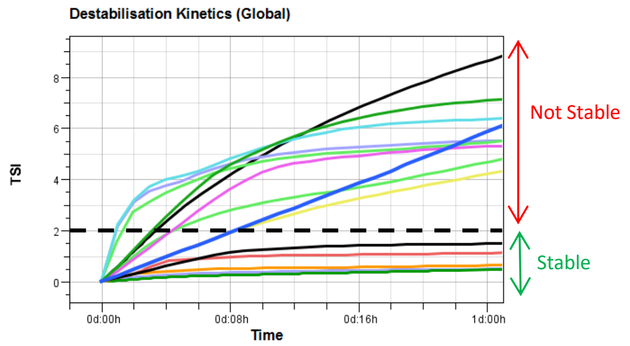


Figure 1: TSI graph for all samples

Surfactant	TSI (1day)			Surfactant	TSI (1day)		
	0.1%	1%	10%		0.1%	1%	10%
Tween 40	8.6	6.3	5.3	Span 60	7.1	5.9	5.1
Tween 60	6.8	6.7	5.4	Span 80	0.5	1.1	1.1
Tween 80	0.5	1.5	1.4	Span 85	7.1	4.2	0.6

Table 2: TSI values for all samples

For this study the following surfactant will be used:

- High HLB : Tween 80 at 0.1% & 1%
- Low HLB : Span 50 at 0.1% & 1% and Span 85 10%

No stability improvement at 10% for Tween 80 and Span 85 so in order to limit formulation cost, they will be not evaluated. A total of 6 combinations are possible.

2. Formulation of the double emulsion

The double emulsion is generated by first creating a water in oil emulsion using high shear process and then this emulsion is dispersed in water with a low shear process. The emulsions are analyzed with the Turbiscan in order to validate the formation or not of a double emulsion. The graph in Figure 2 is generated:

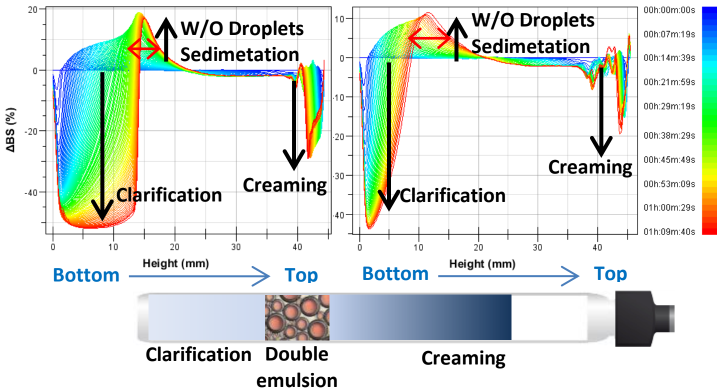


Figure 2: Backscattering profiles for various double emulsions

From the Graph above, we can conclude:

- At the bottom of the sample, a light intensity decrease is observed meaning a decrease of the concentration (clarification) due to the creaming of the oil toward the top of the sample.
- Just above the clear layer at the bottom of the sample, a light intensity increase is observed meaning an increase of the concentration. In fact the density of the double globule is higher and so sediment toward the bottom of the sample. The presence of this layer is a proof of a double emulsion. 3 combinations lead to the formation of a double emulsions:
    - **Span80 (0.1%) & Tween 80 (0.1%)**
    - **Span80 (1%) & Tween 80 (1%)**
    - **Span85 (10%) & Tween 80 (0.1 %)**
- The area of the sediment peak is higher for the graph on the right (see red arrow on Figure 2), The formulator may choose to formulate a double emulsion rich in Span 85 (about 10%) or an emulsion that is less rich in surfactant and so less expensive (1% Span 80), but also less suitable than the previous one, to form a double emulsion.

3. Optimization of the double emulsion

Once the components and the concentrations are coarsely defined by the pre-formulation steps, 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)

The thickness of the clear layer at the bottom of the sample due to the creaming of the oil is used to compare the different emulsions

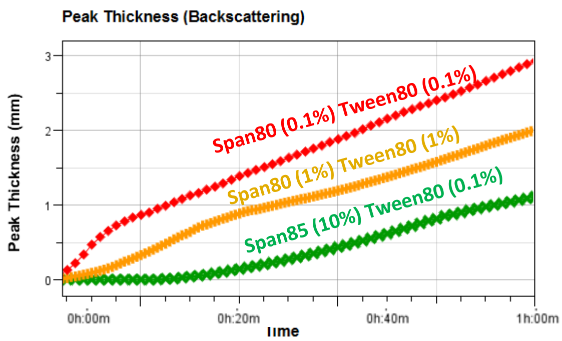


Figure 3: Thickness of the clear layer over time

Sample	Migration rate (mm/hr)
Span80 (0.1%) Tween80 (0.1%)	2.19
Span80 (1%) Tween80 (1%)	1.38
Span85 (10%) Tween80 (0.1%)	1.20

Table 3: Migration rate of the oil droplets

For this study, the best compromise between the cost of the formulation and the stability properties is:

- Span 80 (1%) & Tween 80 (1%)

SUMMARY

In this application note, we show a quick and simple method to formulate and optimize a double emulsion using the Turbiscan™ technology. The double emulsion is generated by first looking the stability of the regular emulsion and then, for the more stable emulsions, measure if the double emulsion is forming. Once this process done, some parameters can be adjust to optimize the final stability of the formulation. All measurements were done in less than an hour per measurement using the Turbiscan™.