# Identification of the instability phenomenon of an emulsion

## Done in collaboration with ademtech

**Application**
Cosmetics

**Objective**
Identify the phenomenon at the origin of the flocculation of an emulsion

**Device**
TURBISCAN® LAB

## INTRODUCTION

Emulsions are unstable colloidal systems that undergo many destabilisation phenomena (coalescence, flocculation, creaming, *etc.*) that can be due to various causes (lack of surfactant to cover the interface, attractive forces, *etc.*). Therefore, it is very important for the formulator to know the origins of these processes to be able to overcome them to get a stable product.
One of the factor responsible for flocculation is the depletion due to the coexistence of small entities (micelles, microemulsions) and big droplets (emulsion). The latters undergo an osmotic pressure from the small ones that lead to a compression of the big drops. This depletion force is attractive and of low energy, hence it is reversible upon dilution.

## METHOD

Experiments were carried out on an emulsion of hexadecane (10%) in water stabilized by various amounts of SDS (from 2 to 10 times the CMC).

## RESULTS

1. Determination of depletion flocculation by various techniques

**Macroscopic observation**
Depletion flocculation gives rise to phase separation. This can be more or less easy to detect. Figure 1, we can observe in the right-hand tube, the coexistence of a cream and a less turbid phase. This phase separation is sometimes invisible to the naked eye.

*Figure 1. SDS concentrations increase from lelft to right*

**Particle size analysis**
Laser diffraction requires important dilution of the emulsion but enables to get the distribution size of the emulsion. On the other hand, it does not show flocculation in the emulsion if it is of low energy as for depletion flocculation (Figure 2).

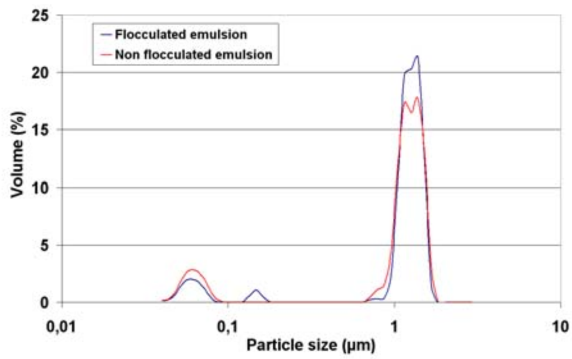


*Figure 2.Particle size analysis of flocculated and non flocculated emulsion.*

 

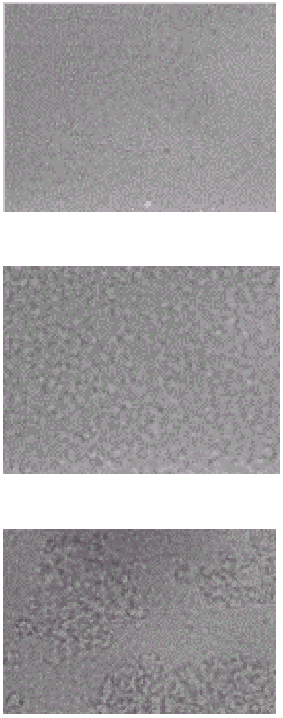

*Figure 3. From top to bottom, a perfectly disperse emulsion (Brownian drops), an emulsion with little flocculation (the flocs are very small and are barely visible), coexistence of flocculated drops and disperse drops. (lens ×100)*

**Optical Microscopy**

Using microscopy (Figure 3), we can see the presence of flocs more or less spread out or connected in equilibrium or not, together with small and Brownian drops (non flocculated).

2. <u>Determination of depeletion flocculation by the Turbiscan LAB</u>

Using the Turbiscan LAB an emulsion with high level of surfactant (10 CMC of SDS) is analysed (Figure 4). A phase separation can be rapidly highlighted in the emulsion, although it is not macroscopically noticeable. The intensity profiles backscattered over time show the formation of a phase that is rich in drops (supernatant cream) and the appearance of a more dilute emulsion in the lower part of the sample.

The Turbiscan LAB$^{expert}$ enables to calculate the drop size on the first scan. We get a mean diameter of 6.65µm when the laser diffraction techniques gives 2µm as for a non flocculated emulsion.

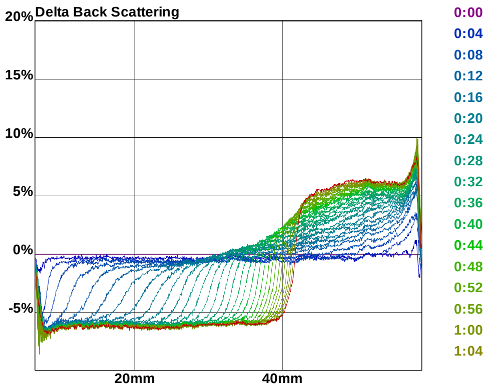


*Figure 4. Emulsion 10 CMC of SDS*

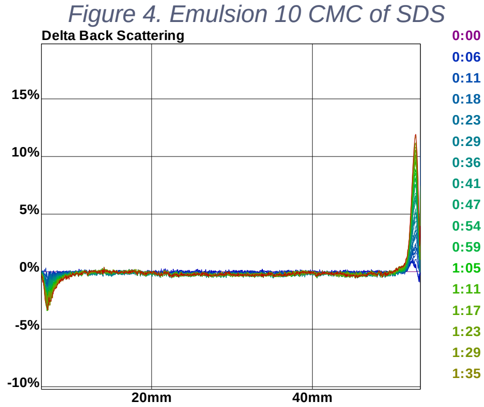


*Figure 5. Emulsion diluted 10 times*

Then the emulsion is diluted in water about 10 times, homogenized by shaking gently, and analysed with the Turbiscan LAB (Figure 5).

The backscattered intensity is now lower, as the volume fraction decreases due to dilution. In addition, the emulsion has become stable on the same time scale. We no longer observe a phase separation. The phenomenon is therefore reversible when the emulsion is diluted in water.

Knowing the reason causing the flocculation, various solutions can be used:

- Reformulation of the emulsion using less surfactant in the continuous phase.
- Simple dilution if the application allows it.
- Washing out the continuous phase by centrifugation.

## SUMMARY

The previous experiments, done with the Turbiscan LAB, show that the flocculation is due to a depletion interaction. Indeed, the dilution decreases the concentration of surfactant in the continuous phase hence the attractive interactions between the drops.