Formulation of a concentrated emulsion for natural agrochemical

Introduction

Oils are used as components of agrochemicals for the treatment of beetroot, maize and wheat as they increase the foliar penetration of the pesticides. For their application, oils have to be dispersed in water to form an emulsion that has to be stable for few hours (time of the treatment). The dispersion in water has to be spontaneous. In order to achieve that, the oils are pre-formulated and can be found in two formats:

- Emulsifying emulsion (a surfactant solution in oil)
- Concentrated emulsion (a O/W emulsion at 70 to 80% oil).

The company ARD has formulated a concentrated emulsion with methyl ester of canola seed as oil and a natural surfactant based on sugar as emulsifier. The main constraints of the formulation are the stability of the concentrated emulsion in storage and its ease of use (fluidity, dilution) as well as the relative stability of the diluted emulsion for the use in the field. A homogeneous emulsion has to form spontaneously without bringing any external energy and has to be stable during application.

Reminder on the technique

Turbiscan® technology, based on Static Multiple Light Scattering, consists on sending a light source (880nm) on a sample and acquiring backscattered (BS) and transmitted (T) signal all over the sample height. By repeating this measurement over time at adapted frequency, the instrument enables to monitor physical stability.
The signal is directly linked to the particle concentration (φ) and size (d) according to the Mie theory knowing refractive index of continuous (n_f) and dispersed phase (n_p): $BS = f(\varphi, d, n_p, n_f)$

Method

The experimental plan mimics the use of the product. Samples are prepared directly in the Turbiscan™ cells and analyzed immediately. 400µL of concentrated emulsion are added to 10 mL of water. The cell is manually agitated by turning it upside down three times.
Samples are analyzed with the Turbiscan™ by scanning them every 30 seconds during 1 hour.
3 different emulsions are analysed:

- a commercial product
- An environmental friendly emulsion made by ARD
- An optimization of the ARD emulsion

Results

By scanning the 3 samples according to the method described in the previous paragraph, the following results are obtained for commercial and optimized emulsions:

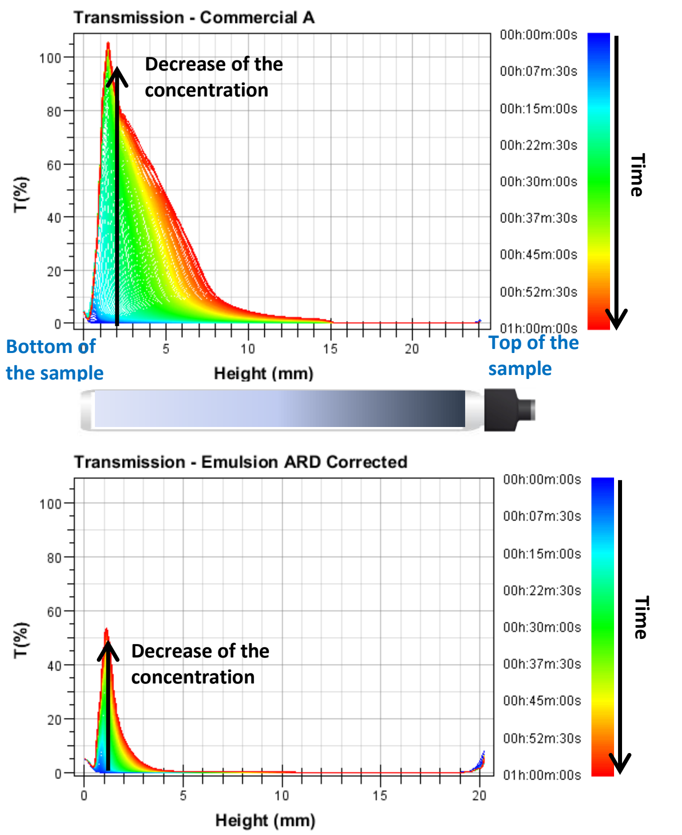

Figure 1: Variation of the transmission versus sample height for commercial (top) and ARD optimized (bottom) products

From the graphs in Figure 1, we can visualize the following destabilization:

- At the bottom of the sample, an increase of the intensity of transmission is observed, meaning the light is less scattered by the droplets and so a decrease of the concentration.

The different emulsions are characterized by measuring:

- The global stability (TSI)
- Migration rate of the oil droplets

The global stability (TSI)

It is possible to monitor the destabilization kinetics in the samples versus ageing time, thanks to the Turbiscan Stability Index (TSI). It sums all the variations detected in the sample (creaming, clarification, size variation, …). At a given ageing time, the higher is the TSI, the worse is the stability of the sample.

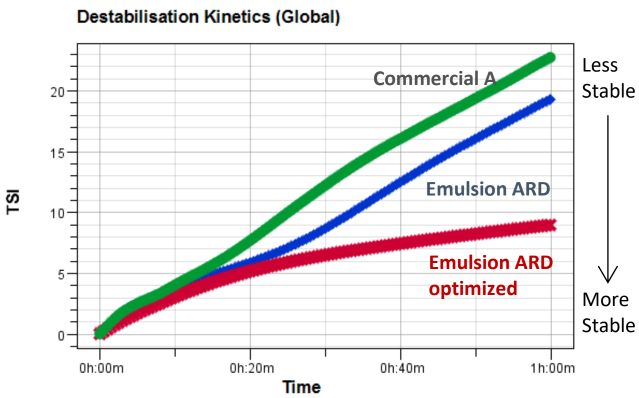

Figure 2: Turbiscan Stability Index for all samples

Sample	TSI (1 hour)
Commercial A	22.4
Emulsion ARD	19.0
Emulsion ARD corrected	8.9

Table 1: TSI values after 1 hour of measurement

Thanks to Figure 2 & Table 1, we can identify that the emulsion formulated by ARD is equivalent in term of stability to the commercial product. After optimization of the emulsions, the stability properties are significantly improved.

Migration rate

In Figure 3, the thickness of the bottom clear layer of the sample is measured over the time of the analysis. By computing the slope of the curves Thickness = f(time), we obtained the migration rate of the droplets in these emulsions.

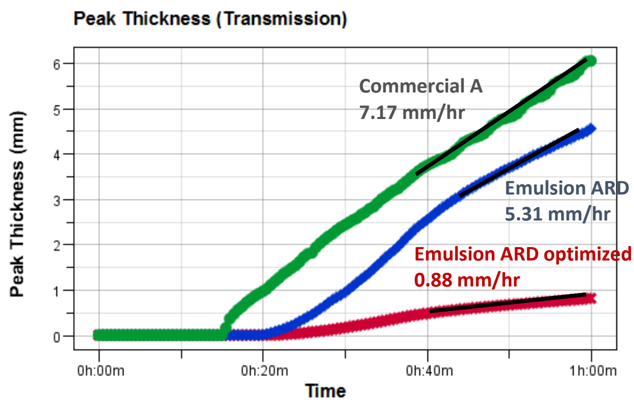

Figure 3: Peak thickness of the clear layer over time

Sample	Migration rate (mm/hr)
Commercial A	7.17
Emulsion ARD	5.31
Emulsion ARD optimized	0.88

Table 2: Values compute from graph in figure 3

The same conclusion than with the TSI study can be done. The environmental friendly product formulated by ARD is more stable than the commercial product and after optimization, the stability of the emulsion is significantly improved.

CONCLUSION

This application note shows a quick and simple method to validate the stability of concentrated emulsion used in agrochemical area in only one hour of measurement. Two parameters, the global stability and the migration rate, were studied for 3 different emulsions. The graph below resumes the computation done during the study.

Both parameters lead to the same conclusion, the ARD emulsion is similar to the commercial product and the optimized formulation provides better results.