*The stability of a liquid pesticide formulation sample was tested using the MultiScan 20 stability analysis system. By analysing the time-dependent transmission and backscattering behaviour of the dispersion, unstable components were detected and various destabilisation mechanisms could be distinguished within a short period of time.*


Fig. 2: DataPhysics Instruments MS 20 stability analysis system

## Background

Pesticides are an integral part in the worldwide food production, playing an important role in securing the worldwide supply of food and prosperity [1]. Adverse effects such as environmental risks and health issues are often accepted or ignored. In order to develop safer and more environmentally friendly pesticides, new selective pesticide compounds and formulations need to be found that can guarantee for an efficient and sustainable use.

Pesticides are commonly applied as dispersions which need to be stable to guarantee for optimal distribution and to minimize the effective pesticide dose that needs to be applied. Thus, for each pesticide formulation it is essential to study the stability in order to make sure it can be used well. The separation of individual components is very often invisible to the naked eye for weeks or even months making technical help a must have for an up-to-date product development process.

Separation processes are one of the key challenges faced in formulation and product development and require thorough stability optimisations. For stability optimisation the MultiScan 20 (MS 20, see Fig. 2) from DataPhysics Instruments with its matching software MSC is an ideal partner since even the slightest changes within dispersions can be detected and evaluated. The MS 20 enables a fast and objective analysis of the dispersion stability as well as conclusions on possible destabilisation mechanisms. The study of a pesticide formulation will be presented throughout this application note.

## Experiment

A small vial filled with the desired dispersion is placed in one of the “Scan Towers” of the MS 20. The scanning system is composed of a transmission and backscattering LED along with a detector. This system moves along the vertical side of the vial (z-axis).

The obtained transmission and backscattering intensity is represented in an intensity-position diagram. The sample was scanned at regular time intervals. Changes in the detected measuring signal can provide explanations on the stability properties of the sample. Fig. 2 shows the MS 20 and its six independent Scan Towers.

20 ml of the pesticide formulation were poured in a transparent glass vial and measured at T = 25 °C every minute during 2 hours and 12 minutes.

The measured zone is between 0 mm (bottom of the glass) and 57 mm (fill level of the vial). Figure 1 shows the sample vial filled with the pesticide formulation at the end of the experiment.

Fig. 1 left: tractor distributing pesticide on a field
right: pesticide formulation in sample vial after 2 hours and 12 minutes

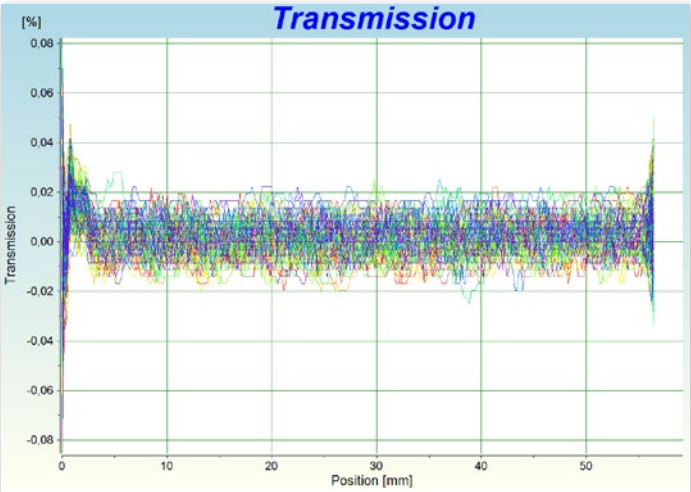


Fig. 3: transmission intensity diagram relative to the intensity at t = 0 s

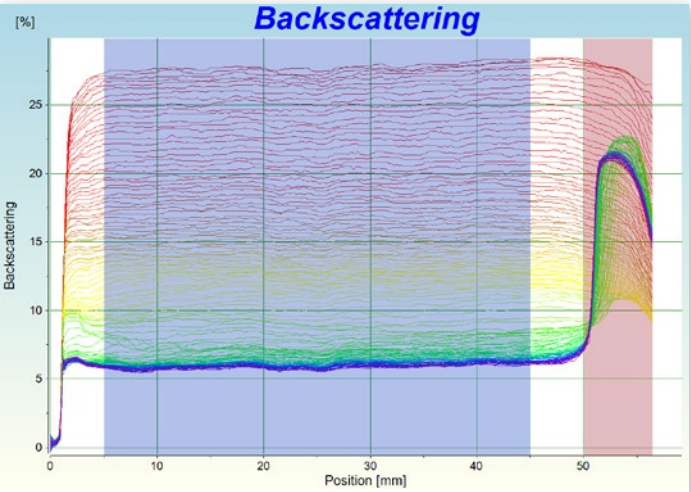


Fig. 4: backscattering intensity diagram

## Results

Figure 3 shows the plot of the transmission intensities against the position. The colour-coding of the curves indicates the time at which they were recorded, from red (start of the experiment, t = 0 s) to purple (end of experiment, t = 2 h 12 min). Every curve represents one individual measurement.

The transmission diagram (Fig. 3) shows a constant mean intensity value of $I_{tr}$ = 0 % which does no change throughout the whole experiment. This can be explained by the turbidity of the pesticide formulation which prevents the transmission of any incident light.

The backscattering diagram (Fig. 4), on the other hand, shows a clear time-dependent change of the signal between 2 mm and 57 mm, as well as a position-dependent change of this signal between 50 mm and 57 mm.

The changes in backscattering intensity indicate that the pesticide formulation destabilizes over the period of time it is measured. Thanks to the MSC software, it is possible to determine which mechanisms led to the destabilisation during this experiment.

Looking at Fig. 4, the backscattering signal can be divided into 2 sections for analysis:

1st section: 5 mm – 45 mm: Position-independent decrease of the backscattering intensity over time.

2nd section: 50 mm – 57 mm: First decrease then after 1 h increase of the backscattering intensity, i.e. formation of an intensity peak.

The 1st section is evaluated using the Value Analysis Method of the MSC software. The obtained diagram (Fig. 5) plots the mean intensity of this section against time. A strong decrease of the backscattering intensity can be observed.

The decrease of backscattering intensity in the 1st section, which almost comprises the whole sample height, indicates a global change in particle size of one or more components of the dispersion through, e.g., agglomeration. The scattering capacity of particles is dependent, among other things, on their particle size [2].

The 2nd section is evaluated by the Peak Width Analysis Method and a Migration Front Analysis.

The obtained diagram for the Peak Width Analysis (Fig. 6) plots the Peak width at a defined Intensity level against time. In the focus is the timespan from 1 h to the end of the experiment at 2 h 12 min where the backscattering intensity peak apparently forms in this section. The analysis shows that over time a creaming layer is formed with a final thickness of 6 mm.

Through a Migration Front Analysis (Fig. 7) it is furthermore possible to quantify the speed of the creaming process. At the beginning (1 h to 1 h 10 min) the creaming rate was determined with -25.03 mm/h, slowing down to -0.50 mm/h after about 1.5 h of the measurement.

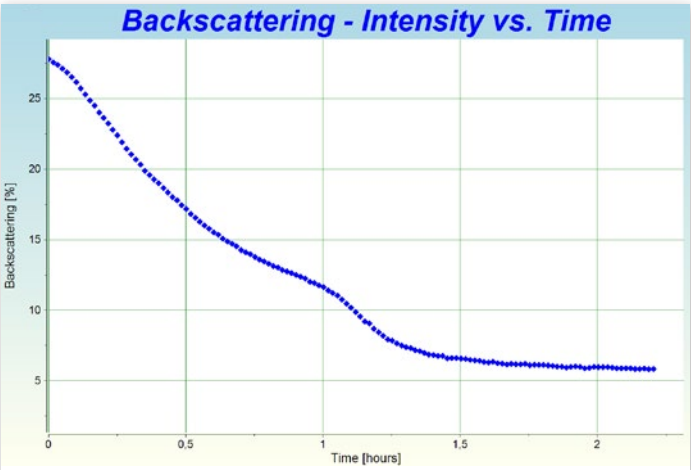


Fig. 5: mean relative backscattering intensity of the 1st section vs. time

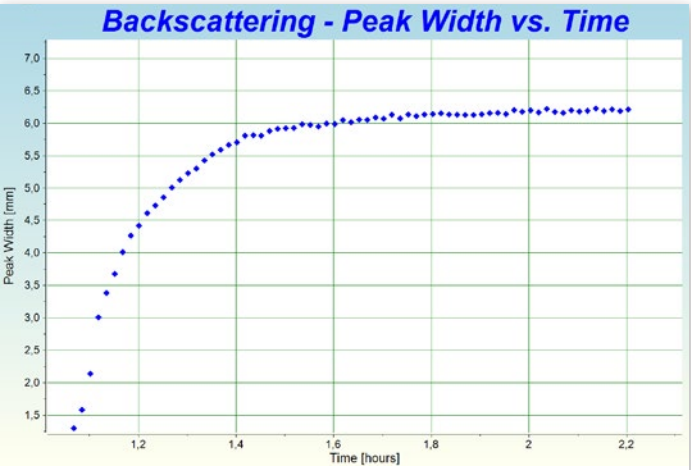


Fig. 6: peak width analysis of the 2nd section vs. time

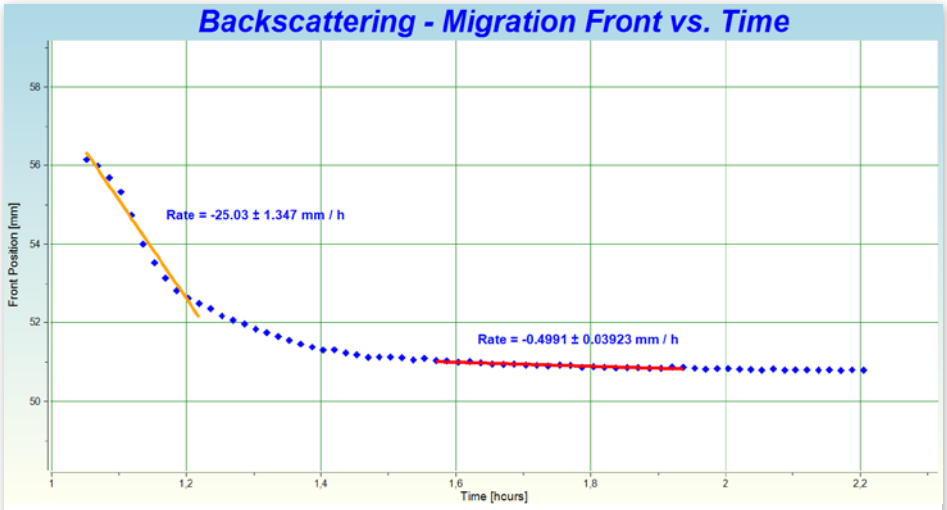


Fig. 7: migration front analysis of the creaming process in the 2nd section

## Summary

Using the MS 20 stability analysis system and its corresponding MSC software it was possible to study the mixing stability of components contained in a pesticide formulation.

By recording transmission and backscattering intensity diagrams for a period of 2 hours and 12 minutes it was observed that a certain amount of components were not stable in the liquid dispersion. The application of various analysis options provided by the software led to the conclusion that multiple destabilisation processes had occurred. The observed results indicate an agglomeration followed by a creaming process.

The opportunity to observe even smallest changes in dispersions within a very short period of time enables the producer of such pesticide formulations (as well as producers of all kinds of dispersions or emulsions) to obtain fast and objective experimental results. This enables the producer to anticipate long term stability and thus guarantee time and cost optimal product development.

## References

[1] International Code of Conduct on the Distribution and Use of Pesticides, 2005, FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS, Rome.

[2] G. Mie, Annalen der Physik 4 (25), 1908, p. 377–445.

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