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

Fig. 2: DataPhysics MS 20 stability analysis system

Background

Protein powder is a well-known dietary supplement among athletes, which can contribute to strength and performance enhancement [1]. Typically the powder is consumed as a protein shake where it is dispersed in water or milk (see Fig. 1 left).

In addition to other supplements such as vitamins and minerals, ready-made protein shakes contain stabilisers. The stabilisers are added in order to prevent the de-mixing of the product for as long as possible thus ensuring longer shelf life e.g. at room temperature.

The shakes seem therefore “stable” and de-mixing processes such as separation of individual components are very often invisible to the naked eye for weeks or even months.

But how stable are such products in reality?

Unless they are desired, separation processes are one of the key challenges faced in product development and require thorough stability optimisations. Even the slightest changes within dispersions can be detected and evaluated by the MultiScan 20 (MS 20) (see Fig. 2) and its matching software MSC developed by DataPhysics Instruments.

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 commercial milk-based protein shake will be presented throughout this application note.

Experiment

A small vial filled with the desired dispersion was 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 moved 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.

Prior to the measurement and as advised by the producer, the protein shake was shaken for 1 minute in its original container.

20 ml were then poured in a transparent glass vial and measured at T=25 °C every 5 min during 16 days and 18 hours. The measured zone is between 0 mm (bottom of the glass) and 55 mm (fill level of the vial). Fig 1 right shows the protein shake vial at the end of the experiment.

Fig. 1 left: milk-based protein powder mixed in water
right: protein shake in glass vial after 16 days an 18 hours

Results

Fig. 3 shows the plots of the transmission and backscattering intensities against the position. The colour-coding of the curves indicates the time at which they were recorded, from red (start of the experiment) to purple (end of experiment). Every curve represents one individual measurement.

The transmission diagram, shows a constant mean intensity value of I_{tr} = 0% with no changes throughout the whole experiment. This can be explained by the opaque appearance of the protein shake which prevents the transmission of any incident light.

The backscattering diagram, on the other hand, shows a clear mean signal between 2 mm and 55 mm of I_{BS} = 22% and a time dependent change of this signal. This becomes even more obvious once the change in intensity compared to the first measurement is being plotted (see Fig. 4)

The changes in backscattering intensity indicate that the protein shake dispersion destabilises 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.

As shown in Fig. 4, the backscattering signal is being divided into 3 sections for analysis:

1st section 3 mm – 50 mm: Position independent increase of the backscattering intensity over time.

2nd section 1 mm – 4 mm: Time-dependent formation of a maximum in backscattering intensity.

3rd section 52 mm – 54mm: Decrease in intensity and signal shift towards lower positions.

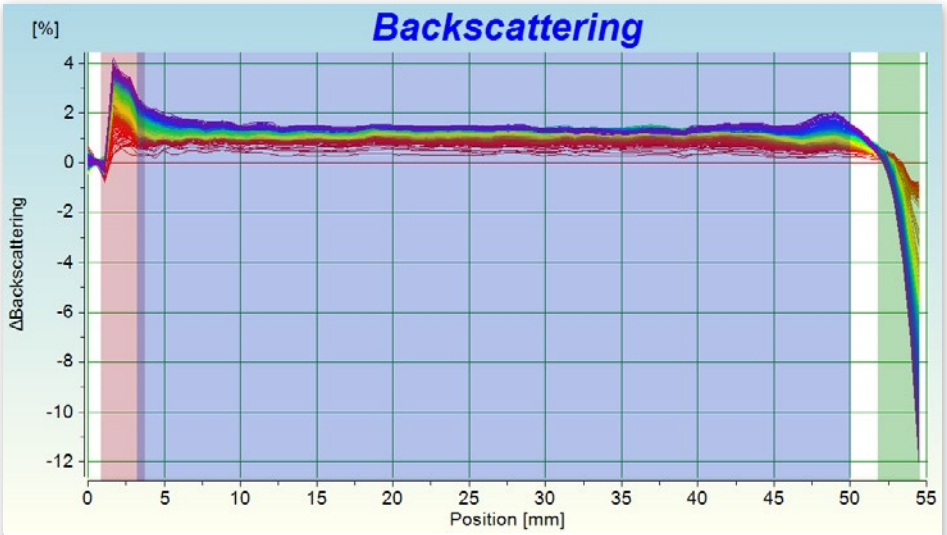

Fig. 4: backscattering intensity diagram relative to the intensity at t = 0 s

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 increase of the backscattering intensity can be observed during the first day.

From the second day onwards, a slight increase of the intensity can be detected, which is constant during the rest of the experiment with a rate of 0.04% per day.

The strong increase observed during the first day of measurement is due to the decrease in air bubbles initially dispersed in the sample. These bubbles appeared during the shaking of the vial prior to the beginning of the measurement and then migrated towards the surface of the sample.

The slight but constant increase of the backscattering intensity observed in the 1st section affects almost the entire height of the sample. It can be explained by a change of particle size of one or more components, for instance through a process of agglomeration. The scattering capacity of particles is dependent, among other things, on their particle size. [2].

In the 2nd section the Peak-Area Analysis method was used to evaluate the bottom part of the sample. The resulting diagram of peak area vs. time is given in figure 6 and shows similar trends to the ones observed in the 1st section (Fig. 5).

Likewise one can observe a significant increase of the signal (area below the backscattering curves in the 2nd section) during the first day. The same assumption can be made in regards to the migration of air bubbles towards the top of the vial.

Throughout the rest of the experiment, the backscattering peak and its related area continue to increase. The same goes for the width of the peak, as seen in figure 4.

This can be explained by the accumulation of sedimented particles at the bottom of the vial. This phenomenon increases the overall light scattering.

The analysis of the migration front obtained from the 3rd section the upper part of the sample confirms the assumption of a sedimentation process of single components (Fig. 7).

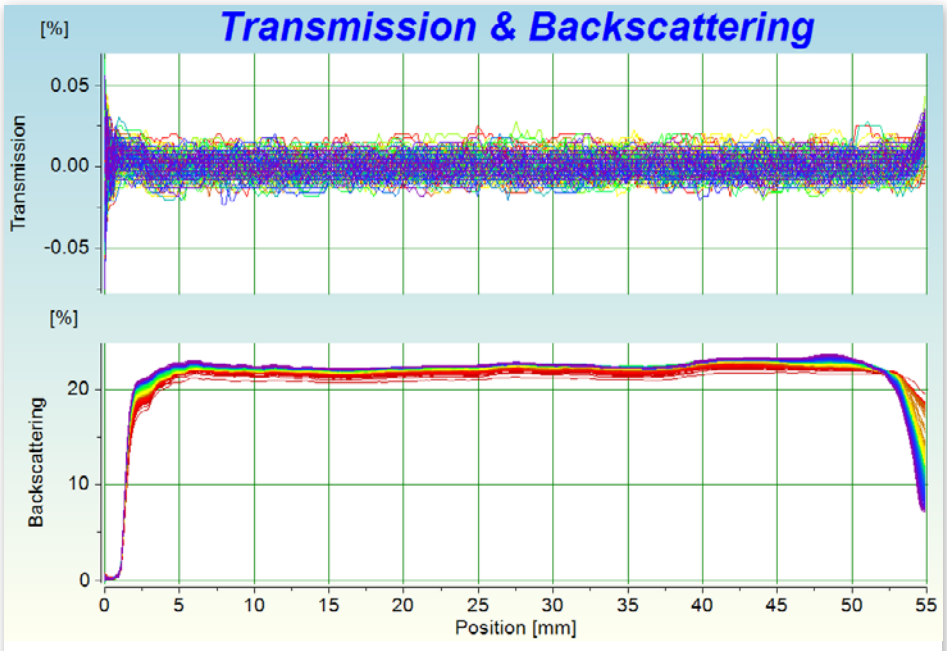

Fig. 3: intensity vs. position diagrams of transmission (top) and backscattering (bottom)

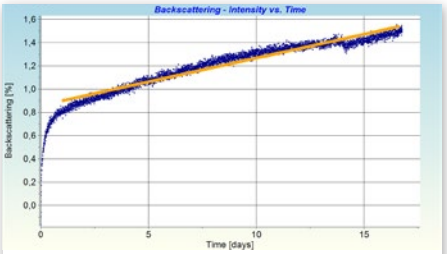

Fig. 5: mean relative backscattering intensity of the 1st section vs. time (increase rate: 0.04 %/d)

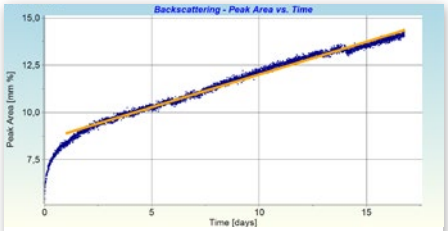

Fig. 6: peak area of backscattering intensity of the 2nd section vs. time (increase rate: 0.35 mm % / d)

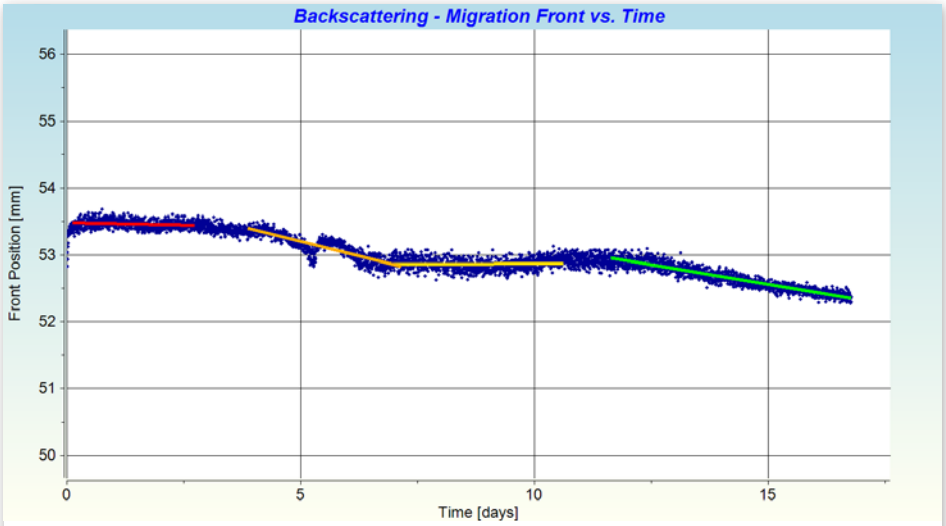

Fig. 7: changes in migration front position throughout the experiment

According to Fig 7, the migration front shifts only very slightly during the first three days. Between day 4 and day 7 it moves towards the bottom of the vial at a speed of 0.17 mm per day and across a distance of approximately 0.5 mm. This initial migration is explained by the separation of the components and the resulting sedimentation of particles.

A stable position of the migration front can be seen from day 7 to day 12 at a height of 53 mm. From day 12 onwards, the migration front moves once more towards the bottom of the vial at a speed of 0.12 mm per day.

From the previously described processes seen in Fig 7, it is evident that there are no changes in intensity due to sedimentation during the first 3 days of measurement. The sedimentation process can be confirmed from day 4 onwards.

Along with the observations from the 1st section, it can be concluded that a sedimentation process is indeed occurring from day 4 onwards, once a critical particle size is reached, due to e.g. agglomeration, thus resulting in a shift of the migration front towards the bottom of the vial.

After day 7 the sedimenting particles have already sedimented below the 3rd section. Between day 7 and 12 only components of the mixture which are sedimentation resistant remain in the 3rd section.

But as seen in the analysis of the 1st section a global change in particle size still occurs. Hence, after day 12 another component has reached a critical particle size and another sedimentation process can be observed in the 3rd 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 commercial protein shake.

By recording transmission and backscattering intensity diagrams for a period of 16 days and 18 hours, and with the help of various analysis options provided by the software, it was observed that a certain amount of components were not stable in the liquid dispersion which led to the conclusion that multiple destabilisation processes had occurred.

The opportunity to observe even the smallest changes within a very short period of time enables the producer of such protein shakes (as well as producers of all kinds of dispersions or emulsions) to obtain fast and objective experimental results. With the use of the MultiScan 20 (MS 20) stability analysis system, destabilisation processes may be observed after only a few days which enable the producer to anticipate long term stability and thus guarantee time and cost optimal product development.

References

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