How long does beauty last?

A stability analysis can give insights into shelf-life behavior and temperature-dependent changes of cosmetic products over time

By Dr. Michaela Laupheimer

Cosmetic products, often consisting of multiple components and phases, should stay stable and uniformly mixed over long periods of time. The MultiScan MS 20 system developed by the German laboratory equipment manufacturer DataPhysics Instruments can analyze how the stability of a multi-phase liquid changes over time and at different temperatures. Researchers in the cosmetic industry can therefore ensure high product quality and longevity from the get-go.
Cosmetic products, such as creams, emulsions, lotions, or cleansers, consist of multiple ingredients and are usually multi-phase mixtures. Their formulations are elaborately composed and refined to meet numerous requirements regarding function, appearance, texture, odor, and stability. It must be ensured, for example, that the product is still homogeneous when applied at body temperature, when stored at room temperature for several months or when forgotten in the car on a hot summer day. To analyze and optimize long-term stability at different temperatures, an in-depth analysis is crucial during product development.

Precise measuring device ensures reliable stability analysis

A straightforward way to test the stability is the so-called "shelf-test". Here, development researchers put samples in test tubes on a shelf and inspect them periodically. This approach is certainly simple, but lacks reliability, quantification, and scientific rigor. When the separation process becomes visible, it is already quite advanced. The beginning of separation often comes on slowly and on a small scale. Those slight changes in appearance are hardly visible to the naked eye. Hence, a reliable and precise measuring device like the stability analysis system MultiScan MS 20 developed by DataPhysics Instruments is needed for product development and improvement (see **Figure 1**). Dr. Sebastian Schaubach, Managing Director and Head of the Application Center at DataPhysics Instruments, says: "We developed the MS 20 for a fast and objective analysis of dispersion stability. This analysis also allows valuable conclusions on possible destabilization mechanisms, such as coagulation or creaming."

The MS 20 system carries out optical stability and aging analy-

Figure 1: The stability analysis system MultiScan MS 20 from DataPhysics Instruments is equipped with six independent ScanTowers.

sis of liquid dispersions in their original concentrations, measuring their transmission and backscattering over time and at different positions. Up to six samples can be measured at the same time by linking up to six so-called ScanTowers to the base unit of the MS 20. "Each sample can be held under different experimental conditions to emulate real storage or application conditions", says Schaubach. Temperatures can be set between 4 °C and 80 °C. Temperature curves are also possible: for example, the operator can heat and cool a sample within the time period of one experiment.

"Our devices are developed to ensure uncomplicated handling. In fact, the MS 20 is as easy to operate as a standard coffee machine", says Schaubach. The sample liquid is filled into a transparent container and put into a ScanTower. Here, it is illuminated by a LED light source. On the other side of the sample container, a detector measures the transmission light intensity. Additionally, a second light source, positioned at a 45° angle from the detector, shows the backscattering intensity, which is particularly beneficial for opaque samples; there is no need to alter and dilute such samples as the analysis is possible in the original state. Both light sources and the detector are mounted on a scanning plate, which moves up and down along the sample height and inspects it in defined time intervals (see **Figure 2**). Hence, the researcher obtains profiles in the device's software, in which the transmission and backscattering intensity are plotted against the position within the sample.

According to scattering theory, the detected intensities depend on the number, size and type of the particles obstructing the irradiated light. In other words, when suspended particles agglomerate in a sample, or emulsion droplets coalesce, these changes become visible as changes in transmission and backscattering intensity. When phase separation takes place in the form of sedimentation or creaming, this manifests in a characteristic progression of the measured intensity profiles. In the following, we will look at two examples for stability and aging analysis using the MS 20: first, an analysis of a make-up remover, second a study of a cosmetic emulsion.

Make-up remover needs to separate quickly

To remove eye make-up neatly, make-up removers usually consist of both aqueous and oily ingredients – which, generally, constitute immiscible phases. Often, efforts are made the by manufacturers to emulsify both components into a uniform mixture. However, some products are sold in a separated two-phase state, which looks nice on the shelf. Those are to be shaken and mixed by the user before application. A fast phase-reseparation after usage is, in this case, the target in product development.

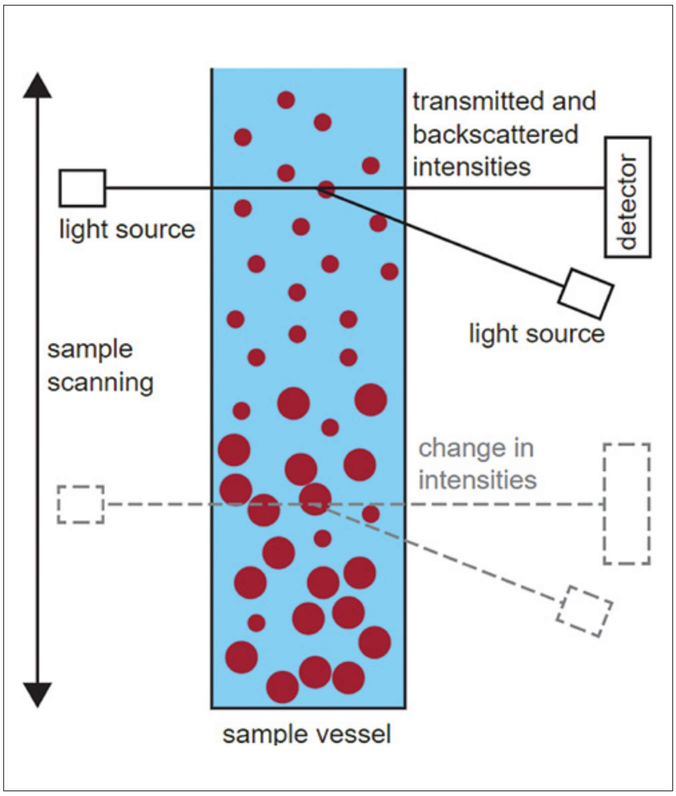

Figure 2: The scanning plate, with its two light sources and the detector, scans the sample by moving up and down, detecting transmission and backscattering.

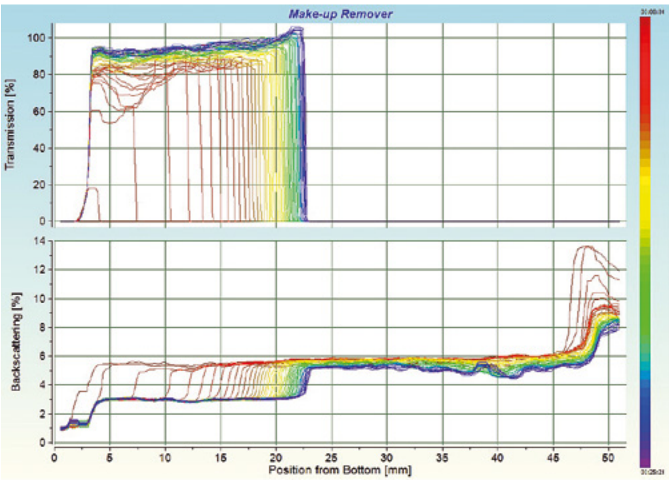

Figure 3: This figure shows the transmission (top) and backscattering (bottom) profiles of a make-up remover during the phase-separation process.

In **Figure 3**, drawn by the device's software, you can see the transmission and backscattering profiles measured with the MS 20 for a two-phase make-up remover during the phase separation process. The sample make-up remover, filled into a sample container up to a height of 52 mm, was measured every half minute over a time span of 25 minutes, starting just after shaking. The time is indicated in the figure by the color change from red to purple, following the rainbow-spectrum. This way, the phase separation process can be made visible comprehensively. As you can see, in the beginning (red color) the transmission is low over the whole sample height, because the two phases of the shaken make-up remover are mixed homogenously. Then, the aqueous phase begins to accumulate at the bottom and the sample is slowly clearing up here. Accordingly, the transmission increases at the bottom over time. Additionally, the oily emulsion droplets are creaming up. Hence, in the bottom there are less and less particles obstructing the light and so, the backscattering intensity decreases there. The migration front of the creaming oil droplets is shifting upwards; its position can be analyzed as a function of time (see **Figure 4**). In the last ten minutes of the experiment a relatively stable creaming rate of 0.14 mm/min is reached. Looking at the upper phase (position range >24 mm) you can see that the backscattering continuously decreases, i.e., the phase slowly begins to clear up, too. However, one can conclude that the phase separation process of the product was not fully completed when the experiment was stopped after 25 minutes.

Schaubach draws the following conclusion from this study: "To further improve product performance, it could be advisable for the manufacturer to adjust the composition of the make-up remover formulation to accelerate drop coalescence. To achieve this, they could, for example, change the type or concentration of the emulsifying additives and reassess the effectiveness in another stability measurement."

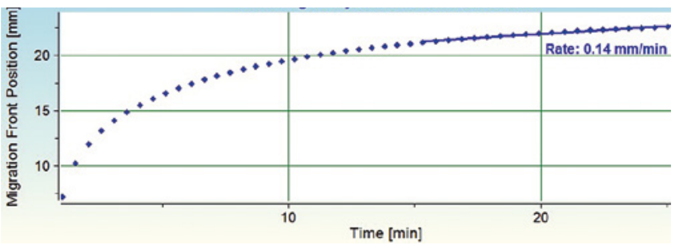

Figure 4: Plot of the migration front position vs. time for the analysis of the creaming process

Latex emulsions less stable at higher temperatures

Many cosmetic products contain natural rubber latex particles which aid good film-formation and high elasticity. The products are shipped, stored, bought, and used at varying temperatures. Just think of the sun lotion you took on your last holiday, which sat on the dashboard of a car in the blazing sun. Even under such extreme circumstances, those emulsions should keep their intended "look and feel", or, in another word, their stability. Schaubach explains: "It is therefore advisable to study the stability of cosmetic products at different temperatures to draw conclusions for optimal processability and performance of the formulation."

In this example, a stability study was carried out with two different latex emulsions (Latex A, Latex B) at 25 °C and 40 °C, respectively. They were measured for six days, being scanned every hour. Schaubach says: "With the MultiScan MS 20, all four measurements could be carried out simultaneously, as up to six independent ScanTowers can be used at once with one device. For long-term measurements, this yields significant time savings for researchers." After shaking the mixtures to create homogeneous samples, they were poured into transparent glass vials, which were then put in the ScanTowers and scanned from the bottom to the top fill level.

Since the latex emulsions appear completely turbid, no transmission was measured. However, the backscattering intensities can be used for analysis (see **Figure 5**). Again, the color-coding of the curves indicates the passage of time, from red (start of the experiment, t = 0 s) to purple (end of the experiment, t = 6 d), with every curve representing one individual measurement. In this study, the relative backscattering intensities are plotted, i.e., the intensity change with respect to the first measurement. "This approach reveals even slightest variations in intensity", explains Schaubach.

In **Figure 5**, you can see that the studied emulsions show differently pronounced signal changes over time when measured at 25 °C or 40 °C, respectively. Looking at the different position zones, it is also possible to deduce the occurring destabilization mechanisms. At 25 °C both latex emulsions show hardly any changes in both the middle and the bottom zone. The top zone, however, looks very much different with time. "The seen reduction of the backscattering signal in the top zone is typical for a sedimentation process", knows Schaubach.

At the elevated temperature of 40 °C, you can see that the emulsion stability is decreased, especially for sample Latex B. The sedimentation in the top zone is considerably more pronounced in this case, while at the same time, a backscattering increase in the bottom zone shows the buildup of sediment. Sample Latex A interestingly shows a completely different behavior at 40 °C. Here, a drop of backscattering intensity is visible in the bottom zone, indicating clarification. The top zone, at the same time, shows a backscattering increase which suggests that a creaming

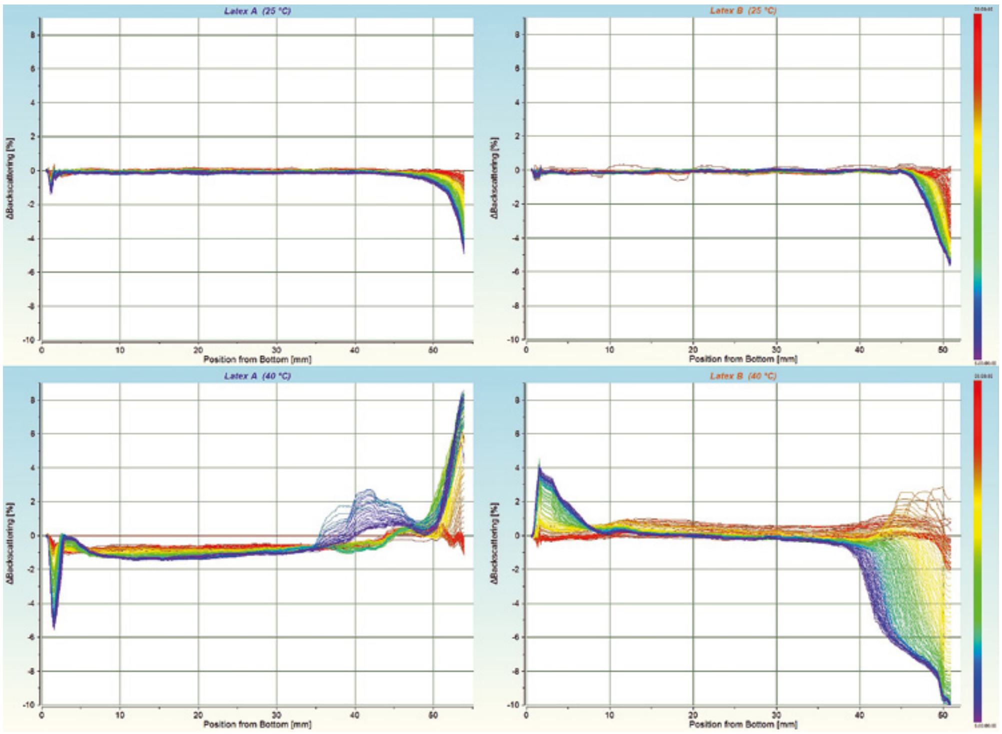

Figure 5: Backscattering Profiles for Latex A (left) and Latex B (right) at 25 °C (top) and 40 °C (bottom)

process is taking place. Schaubach additionally points out the uniform backscattering intensity decreases in the middle zone. "This could mean that the emulsion particles not only cream, but also change size over time due to agglomeration or coalescence", he explains.

As the destabilization mechanisms of the two samples at the two studied temperatures are quite different from another, it is not necessarily opportune to look at parameters like the sedimentation rate or creaming layer thickness, when trying to compare their stability. Fortunately, the MultiScan software MSC from DataPhysics Instruments can automatically calculate the Stability Index (SI), which assesses the overall stability of samples. **Figure 6** depicts the SI functions for all four experiments. All Stability Index values lie in a range between 0 and 0.35, which is, notably, very low and shows that the MS 20 can detect even the smallest sample changes. The SI functions for the two experiments at 25 °C illustrate, in line with the measured backscattering profiles, that both latex emulsions possess a similar stability at room temperature. "The higher SI at 40 °C indicates stronger destabilization processes", explains Schaubach, which means that both samples are less stable at the elevated temperature. The SI plot furthermore allows to distinguish between the two samples: you can see that, in particular at 40 °C, sample Latex A is more stable than sample Latex B, which was not immediately obvious in the backscattering profiles.

Summing up, the two studies exemplified how the stability and aging behavior of cosmetic products (and, of course, any other liquid dispersions) can be reliably and revealingly studied using the stability analysis system MultiScan MS 20 from DataPhysics Instruments. With the MS 20, it is possible to measure up to six samples at the same time, each at a different temperature if desired. Moreover, the samples can remain unaltered, in their original concentration, while they are studied under real storage or application conditions. With the insights gathered from analyses like this, product developers can refine and optimize product formulations regarding their stability. Hence, customers can depend on their cosmetics longevity, even when re-discovering them on the bathroom shelf during the spring clean, or after forgetting them in the car during a hot summer day at the beach.

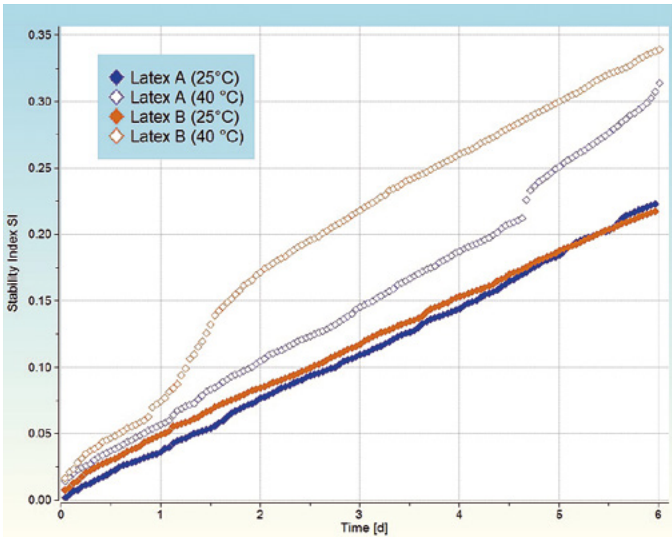

Figure 6: This figure shows the Stability Index (SI), plotted for Latex A in blue and Latex B in brown at 25 °C (filled diamonds) and 40 °C (open diamonds).

Dr. Michaela Laupheimer studied chemistry at the University of Stuttgart, Germany. She specialized in physical chemistry and received her PhD in 2013 for her distinguished thesis about gelled bicontinuous microemulsions. Being keen in sharing her knowledge and expertise in surface and interfacial science, she today works for DataPhysics Instruments, where she is responsible for public relations and knowledge transfer.

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