APPLICATION NOTE

Metallic Effect Pigments

Introduction

Effect pigments are pigments that impart additional properties to the system in which they are embedded, such as angle-dependent changes in hue or gloss or texture. Effect pigments are divided into metal effect pigments and special effect pigments. Metal effect pigments are luster pigments made of metal that have a parallel orientation in the application and exhibit a metal-like effect produced by reflection of light from the metal platelets. Important metal effect pigments are aluminum, brass and copper flakes. Non-metallic effect pigments are, for example, mica (sheet silicates), graphite, iron oxides or molybdenum sulfide. What most effect pigments have in common is the flaky structure of the particles. In contrast to classic pigments, their effect is not primarily based on absorption of certain wavelengths, but on reflection and interference. While classical pigments such as titanium dioxide often have particle sizes of a few 100 nanometers, the platelet-shaped effect pigments usually have a diameter of 5 - 100 µm, but much smaller layer thicknesses.

In the present example, the particle size distribution of two aluminum effect pigments was investigated by laser diffraction (ISO 13320). The analyses could be carried out in aqueous dispersion. The measuring device used, SYNC analyzer from Microtrac, has a measuring range of 10 nm to 4 mm and is suitable for all types of pigments. The camera, which is available as standard, allows dynamic image analysis (ISO 13322-2) to be performed in addition to laser diffraction, thus providing information on the particle shape. Likewise, the sensitivity for small amounts of oversize particles is significantly increased by the image analysis.

Measurement Conditions

With the SYNC, Microtrac MRB extends its established Tri-Laser technology with a powerful image analysis that offers users a new, unique measurement experience. Particle size measurement by laser diffraction (LD) is the most widely used technology in research and industry and is considered the standard for quality control. The measurement is based on the interaction of laser light with particles in a dispersion. This produces a scattered light pattern that is recorded over an angular range of 0.02 - 163° using Microtrac MRB technology, allowing the size distribution to be calculated. The SYNC uses two detector arrays and three lasers. The analyzer can be equipped with red lasers or a combination of red and blue lasers (Figure 1).

Small particles scatter light to large angles, while large particles scatter light to small angles. The evaluation is performed using Microtrac MRB's innovative modified Mie scattering theory. This algorithm provides accurate particle size distributions for both round and non-spherical particles as well as for transparent, reflective, and absorbing materials.

Modern particle analyzers are often expected to do more than simple size analysis. Dynamic Image Analysis (DIA) provides important insights into particle shape and thus detailed information on physical material properties. While laser diffraction only provides an equivalent diameter based on the assumption of spherical particles, image analysis can separately evaluate length and width of the particles in addition to shape parameters. A particle stream illuminated by stroboscopic light source is photographed by a high-resolution digital camera, and a video file of the particle stream is created from the images.

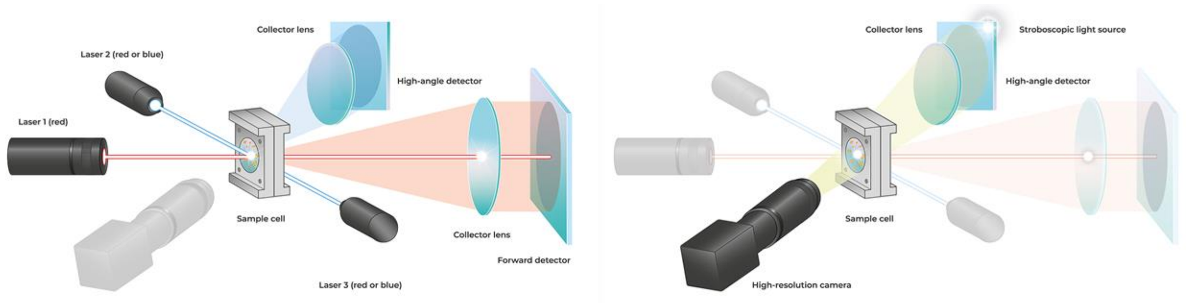


Figure 1: Optical design of the SYNC analyzer. For laser diffraction (left), three red or blue lasers and two detector arrays are used. Dynamic image analysis (right) is performed in the same measurement cell using a stroboscopic light source and a camera. The particles are detected as shadow projections.

The paste-like, highly concentrated pigment dispersions were mixed with demineralized water and shaken well (Figure 2). 1-2 drops of this were then added to the SYNC analyzer until a suitable concentration for analysis was achieved. The signal strength is detected by the evaluation software and is displayed to the user as a "Loading Factor". If this is too low, more sample must be added. If the loading factor is too high, dilution can be performed automatically.

The following measurement conditions have been used:

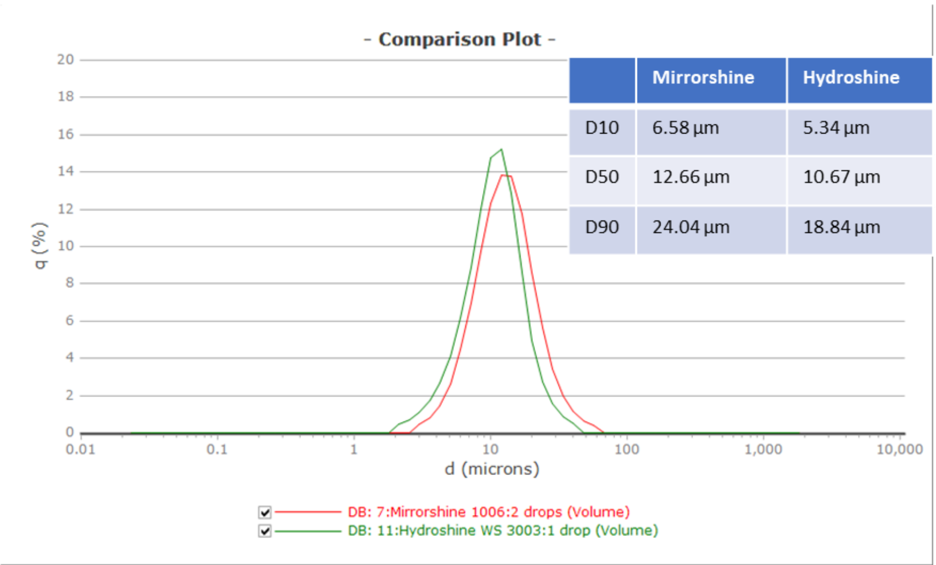
- SYNC analyzer configuration 3R L (3 red lasers, “low-range” camera)
- Wet measurement with “FlowSync”
- Flow Rate: 50%
- Measurement time: 30 seconds
- Evaluation: optical model for reflecting particles

Figure 2: Already diluted Pigment dispersions „Hydroshine“ and „Mirrorshine“. Only 1-2 drops of these are required for analysis.

Results

Laser Diffraction

Figure 3 shows the result of the laser diffraction analysis for both pigment samples "Hydroshine" and "Mirrorshine". The size distribution ranges from about 2 µm to about 50 µm, with Mirrorshine having slightly larger particles. The table shows the percentile values at 10%, 50% (median) and 90%.



	Mirrorshine	Hydroshine
D10	6.58 µm	5.34 µm
D50	12.66 µm	10.67 µm
D90	24.04 µm	18.84 µm

Figure 3. Size distributions of Mirrorshine (red) and Hydroshine (green), measured with laser diffraction.

Both samples were analyzed three times, Figure 4 shows the results of these replicate measurements, which compare very well.

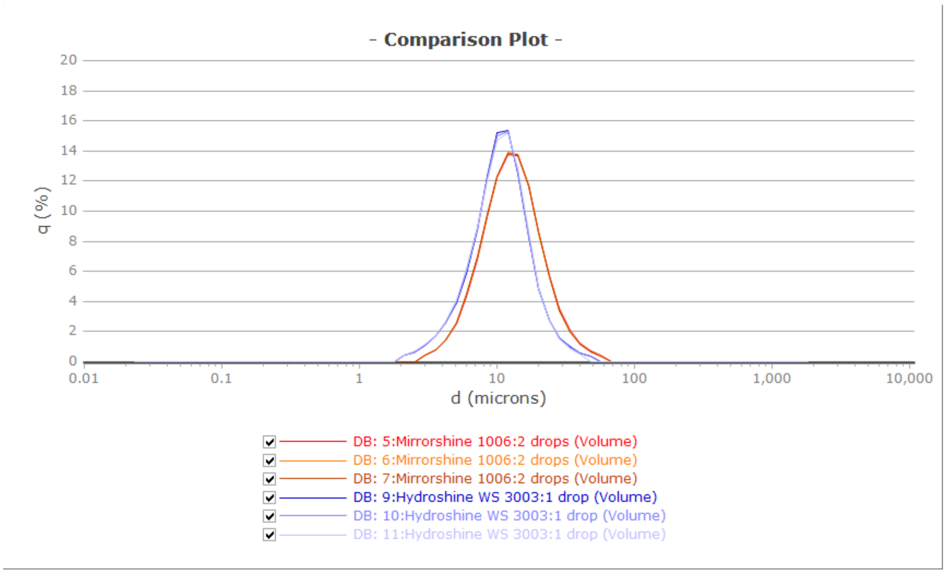


Figure 4: Results of three consecutive measurements of Mirrorshine (red) and Hydroshine (blue)

An important setting in the evaluation of laser diffraction data, especially for small particles, is the so-called optical model. This includes the optical properties of the particles and the surrounding medium. Essentially, these are the refractive indices and the absorption. For large particles, simplified evaluations such as the Fraunhofer approximation can be used in some cases.

When analyzing diffraction patterns with Microtrac analyzers, it must first be specified whether the particles are absorbing, transparent or reflecting. Figure 5 shows a comparison of the analysis with the "reflecting" and "absorbing" affects the result for the "Hydroshine" sample. This is the same raw data set, which was only evaluated with different models. The "absorbing" model shows a fine fraction at 100 - 200 nm. Since the particles reflect light to high angles, this is misinterpreted as a signal from small particles that characteristically scatter light to high angles. With the "reflective" setting, a correct distribution is output.

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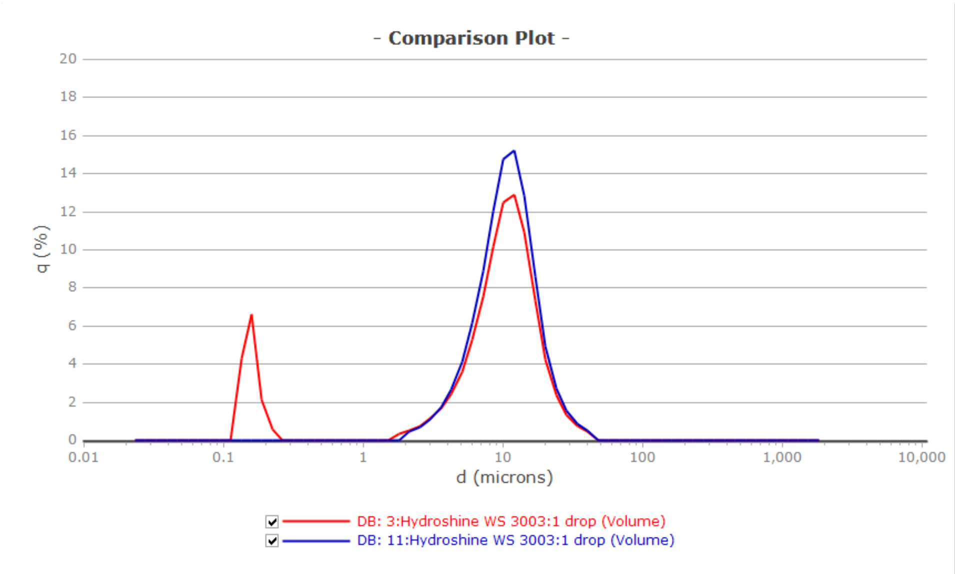

Figure 5: Comparison of the evaluation for "absorbing particles" (red) with unrealistic fine particles and the evaluation for "reflecting particles" (blue) with the correct result

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Dynamic Image Analysis

In addition to the actual laser diffraction measurement, the Microtrac SYNC offers the possibility of simultaneously performing an image analysis. Especially for large particles, this method offers higher accuracy and sensitivity. The results can be combined with those of laser diffraction to form a size distribution via a BLEND algorithm (Figure 6). While there is no difference between the laser diffraction result and the combined evaluation for the sample "Hydroshine", a coarse fraction shows up for "Mirrorshine" when image analysis data are considered. Due to the small percentage of this oversize, laser diffraction cannot detect it.

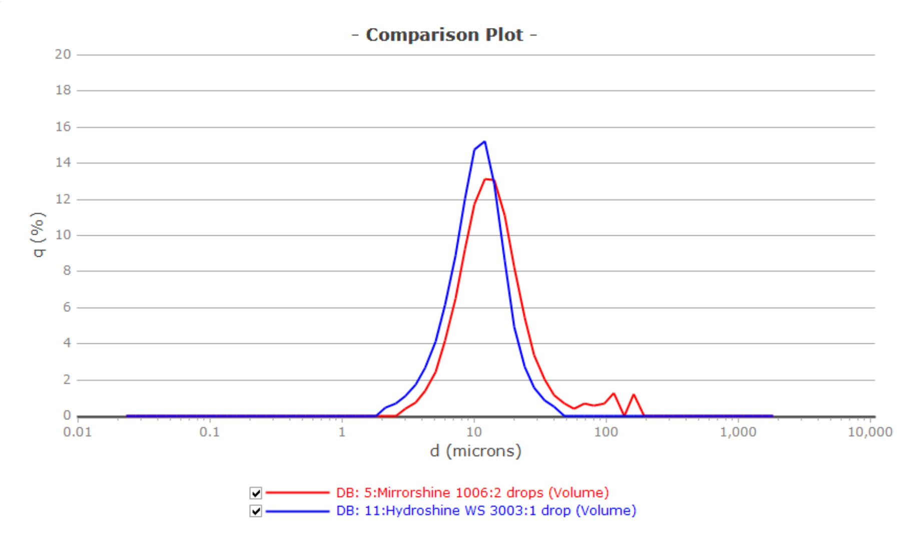

Figure 6: Evaluation with BLEND algorithm (combination of image analysis and laser diffraction). With Hydroshine, the result remains unchanged compared to pure laser diffraction, with Mirrorshine a coarse fraction shows up.

To prove that the coarse fraction is indeed particles, the images can be displayed. Figure 7 shows the largest particles in the Mirrorshine sample. The numbers under the shadow projections are the sizes in µm. Particles in this size range are missing in the Hydroshine sample.

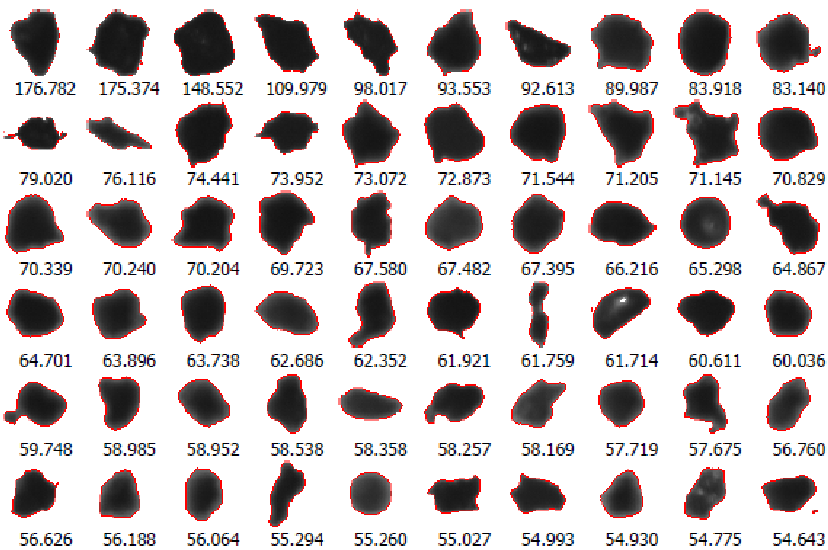

Figure7: Particle images including size in µm from sample Mirrorshine.

Summary

Laser diffraction is suitable for fast and easy characterization of effect pigments with a measurement time of only a few seconds. Since the measuring device rinses and cleans itself automatically, sample run times of 1-2 minutes are realistic. Excellent reproducibility and flexible application possibilities characterize the method. Reliable evaluation algorithms for reflective particles provide reliable results.

The simultaneous use of image analysis significantly increases the probability of detecting small amounts of oversize particles.