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Application Note

Particle Size Analysis by Laser Diffraction

Introduction

The underlying assumption in the design of laser diffraction instruments is that the scattered light pattern formed at the detector is a summation of the scattering pattern produced by each particle that is being sampled. Deconvolution of the resultant pattern generates information about the scattering pattern produced by each particle and, upon inversion, information about the size of the particle.

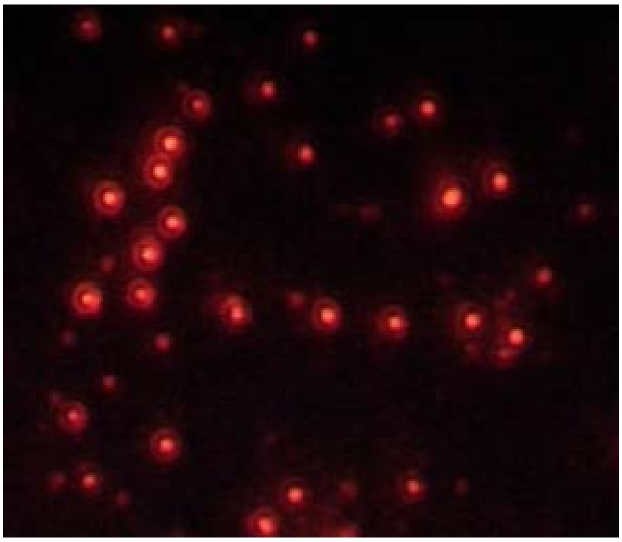

Figure 1: Light scattering patterns observed for polystyrene latex particles

In essence, each particle scatter pattern is matched to a theoretical pattern using comprehensive mathematical solution to the scattering of incident light by spherical particles; the Mie Theory. This theory indicates the necessity for a precise knowledge of the real and imaginary components of the refractive index of the material being analysed, to determine particle size and particle size distribution. When a good fit is obtained, then we know all of the relevant information in order to deconvolute the Mie pattern into meaningful particle size information. All laser diffraction instruments rely on three basic tenets:

1. The particles scattering the light are spherical in nature.
2. There is little to no interaction between the light scattered from different particles.
3. The scattering pattern at the detectors is the sum of the individual scattering patterns generated by each particle.

Experimental

Prior to analysis, the dispersion cell is filled with clean, deionised water and left to allow thermal equilibrium to take place. The instrument automatically aligns so that the incident path of the laser is aligned with the optical arrays. The cleanliness of the system is then checked, and a background is taken. By comparing the signal intensity of the system without a sample present, to the intensity with a sample, the obscuration of the laser beam may be calculated, giving some idea of the material concentration in the dispersal cell. Too high a concentration results in multiple scattering, too low and the signal strength is inadequate to register at the detectors. Figure 2 below illustrates a comparison between the signals obtained from an empty system (red), with that from a particle sample (green). The background signal should always be lower than your sample signal!

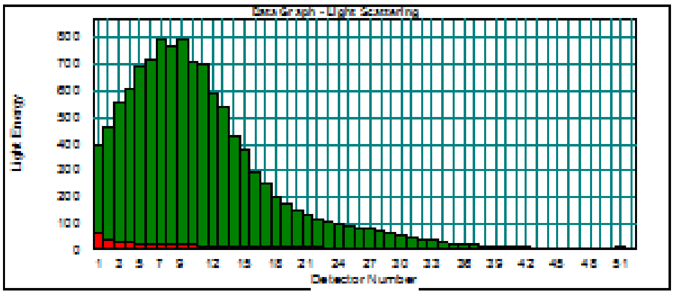

Figure 2: Background and sample scattering data

Suitable dispersion procedures should be followed to ensure that the powder is dispersed and minimum agglomeration has taken place. Care must also be taken to ensure that the dispersal cell contains no air bubbles or that particle fracture is not occurring as the instrument is not capable of distinguishing between agglomerates, air bubbles or primary particles.

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Sonification is an option to aid particle dispersion although again, care must be taken to ensure the correct intensity and duration of sonification to avoid primary particle breakage.

The particle size distribution will depend on the optical model used to calculate it! The real and imaginary components of the refractive index are a vital part of the particle characterisation equation. In the case of an unknown particle refractive index (if a literature value is unavailable), the optical properties may be derived by varying the input values and comparing the resultant scatter pattern with the measured data until a good fit is obtained.

In figure 3 below, we see the difference the imaginary part of the refractive index makes to the resulting size distribution. The question is which value gives the correct distribution?

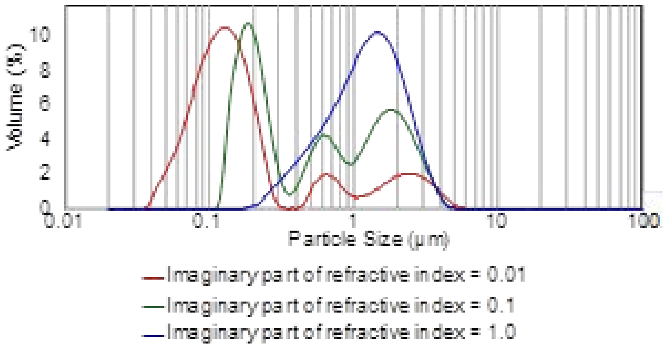

Figure 3: Size distributions using varying imaginary RI values

If we look at the theoretical scatter pattern compared with the measured data, we get an instant idea of the goodness of fit. Figures 4, 5 and 6 illustrate the comparison when using an imaginary refractive index of 0.01. 0.1 and 1.0 respectively.

We clearly see the evolution in scatter pattern as the actual data starts to approach the theoretical, until at an imaginary RI component of 1, the patterns coincide. At this point, we can say that this scatter pattern is the most correct, and in this case, the true size distribution will be the blue line in figure 3.

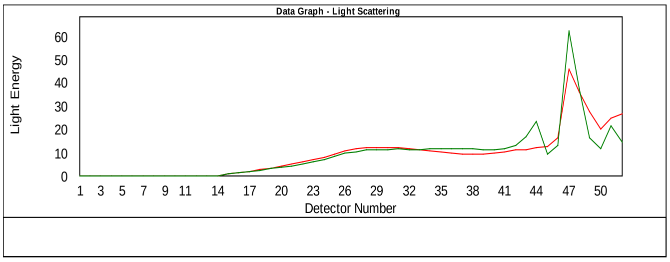

Figure 4: Imaginary RI component = 0.01

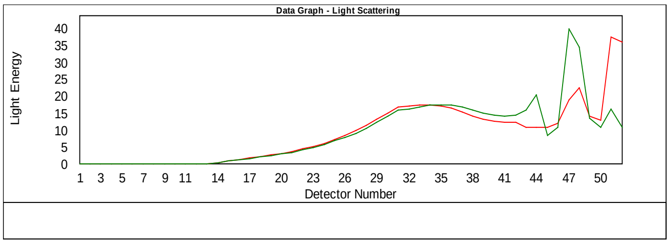

Figure 5: Imaginary RI component = 0.1

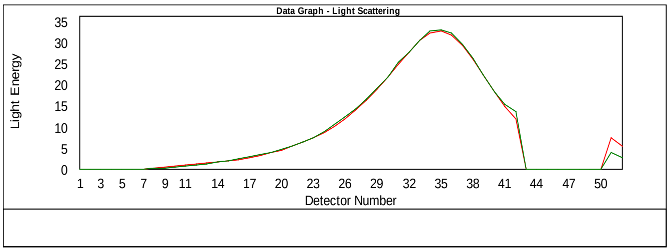

Figure 6: Imaginary RI component = 1.0

Conclusions

1. Sample preparation is important; the instrument must be aligned and clean.
2. Laser diffraction particle sizing requires both the real and imaginary components of the particle under analysis
3. By adjusting RI values manually, the scatter pattern may be manipulated until actual and theoretical are congruent. At this point, we may say our input RI is correct, and the resultant size distribution is representative of the system under analysis.

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