DLS vs. Diffraction of Flavor Emulsions

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The utility of many industrially produced materials such as emulsions is often determined by the particle size. Dynamic light scattering provides fast, accurate and repeatable nanoparticle size information and is, therefore, an important tool for the nanoparticle technologist. Laser diffraction can also be used to characterize these important materials. Even though each method probes the sample in a different manner, similar trends are observed in measurement results.

Figure 1: SZ-100 Nanoparticle Size Analyzer.

Introduction

The particle size distribution of many flavor (food or beverage) emulsions has a substantial effect on their performance. Particle size distribution can affect mouth feel, appearance, and emulsion stability (shelf life). Therefore, it is important to monitor and control size distribution to continuously provide optimal product.

There are two optical techniques for characterizing these emulsions-laser diffraction and dynamic light scattering. These techniques are both fast and reliable. However, since each technique performs the size measurement in a different way, different results are obtained for most real samples. This application note compares the differences to help the reader understand why the results of analysis of the same material may not be the same.

Laser diffraction (LD) is arguably the most popular modern particle sizing technique (1). Modern instruments can be used to characterize particles ranging in size from tens of nanometers to millimeters in just a few minutes. The results are extremely repeatable and the instruments can be used to analyze samples that are both dry powders and particles or droplets in suspension.

Dynamic light scattering (DLS) is the technique of choice for analyzing the size of submicron particles including nanoparticles (2). DLS can be used to characterize particles with sizes ranging from less than a nanometer to several microns. As in laser diffraction, the measurement is fast and repeatable, but DLS can only be used with liquid suspensions. On the other hand, the volume of liquid required can be as little as 10 microliters. In addition, particle charge, that is, zeta potential can be determined with an appropriately modified DLS instrument.

Figure 2: LA-950 Particle Size Analyzer.

For this application note, a series of emulsions used for food and beverages were analyzed with each technique. It is well established that all particle analysis techniques yield the same results for spherical particles with a narrow size distribution. See TN156 Colloidal Silica as a

Particle Size and Charge Reference Material, (3). In fact, both techniques are often checked with the same standards. What is less clear is what happens when real samples with unknown geometry and broader size distributions are analyzed by these techniques. While the results here are unique to this process, the results illustrate real manufacturing issues.

Materials and Methods

Four different proprietary edible emulsions were obtained from a manufacturer. These emulsions had an aqueous continuous phase and each emulsion had different compositions (raw ingredients) and processing histories.

Samples were analyzed with a HORIBA LA-950 laser diffraction instrument. The droplet refractive index estimated from the scattering data with the Method Expert (4) closely matched the expected value for the proprietary material under test. The samples were then immediately transferred to an SZ-100 DLS nanoparticle size analyzer for measurement by DLS.

Figure 3: Lemons provide excellent flavor for cookies and other food and drinks. Image courtesy of André Karwath and Wikimedia Commons.

It should also be noted that the laser diffraction measurement required a few milliliters of suspension while the DLS measurement required only a few drops of suspension. The volume of sample required for both techniques can be reduced by choosing different sample cells. Nevertheless, the general trend is that DLS requires less sample.

Results and Discussion

The median size values obtained from the volume based size distribution obtained with the LA-950 and SZ-100 are given in Table 1 below. In addition, the Z-average diameters obtained with the SZ-100 are listed in the same table.

	D_{50} (vol. basis) LA-950	D_{50} (vol. basis) SZ-100	Z-avg. Diam. SZ-100
E-1	129.8	146.6	118.3
E-2	149.8	170.5	138.7
E-3	110.0	100.2	112.7
E-4	49.4	45.5	32.4

Table 1: Size results from industrial food emulsions by two different techniques, laser diffraction and dynamic light scattering. D_{50} results along with the more reliable Z-average diameter are shown for the dynamic light scattering results.

From this data, it is clear that the laser diffraction and dynamic light scattering data follow each other quite uniformly. This is highlighted by plotting the values obtained by DLS as a function of the value obtained by laser diffraction in Figure 4.

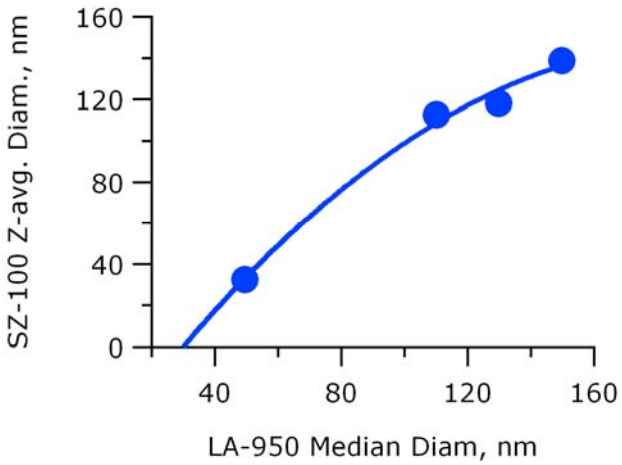

Figure 4: Measured Z-average diameter by DLS as a function of measured median diameter by laser diffraction. Note that the results follow each other quite closely.

The observed differences in the results are readily ascribed to the radically different measurement technique used by each instrument and the corresponding effects on the obtained results. Laser diffraction uses the measured change in light scattering intensity with angle to extract particle size. On the other hand, DLS derives particle size from particle motion and, therefore, the size obtained by DLS is a measure of hydrodynamic size. In both cases, the particles are treated as if they are spherical, but deviations from this assumption will appear differently in each technique. In addition, the relative effect of large particles compared to small particles is quite different in the two techniques. Therefore, as the size distribution deviates from a perfectly narrow unimodal distribution, the obtained size results are expected to differ.

For these industrial materials, the expectation of uniform spheres and extremely narrow size distributions is inappropriate. In the case at hand (emulsions) the particles are expected to be spherical. Thus, while the shape may not cause the results obtained by the two techniques to differ, the breadth of the size distribution will. Even so, both techniques show the same trends.

Conclusions

The results of these measurements show that both the SZ-100 and the LA-950 can be used to characterize submicron size emulsion droplets. The choice of instrument rests on other considerations. In this case, the primary outside consideration was that particles in many of the customer samples were too large to be measured with the SZ-100.

In selecting analysis equipment, it is critical to work with vendors who are able to evaluate multiple techniques in order to make the optimum choice.

References

(1) ISO 13320 Particle Size Analysis – Laser Diffraction Methods – Part 1: General Principles

(2) ISO 22412:2008 Particle Size Analysis – Dynamic Light Scattering

(3) TN156 Colloidal Silica as a Particle Size and Charge Reference Material, http://www.horiba.com/fileadmin/uploads/Scientific/Documents/PSA/TN158.pdf

(4) See http://www.horiba.com/scientific/products/particle-characterization/technology/laser-diffraction/simplify-choosing-a-refractive-index-with-the-method-expert/

Figure 5: Ice cream is a tempting food emulsion often prepared with ingredients such as the ones under study. Image courtesy of morguefile.com

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