MEASURING ORGANIC PIGMENTS WITH LASER DIFFRACTION PARTICLE SIZE ANALYSIS

Introduction

A general definition of a pigment could mean any substance that alters the color of a material through selective color absorption. Pigments are used to color a broad range of products including ink, paint, cosmetics, fabric and food.

Many pigments work by selectively absorbing and reflecting specific wavelengths of light. When the multiple wavelengths of white light encounter a pigment some wavelengths are absorbed by the chemical bonds of the pigment and others are reflected. The newly created spectrum creates the appearance of a color (see Figure 1).
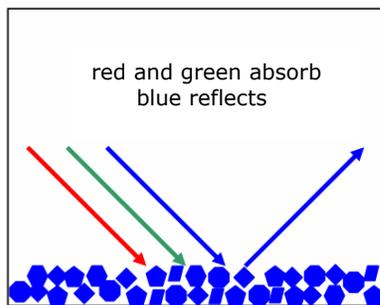

red and green absorb
blue reflects

Figure 1

Particle size is a critical physical characteristic of pigments, affecting many performance factors. Hiding power of some pigment dispersions can be determined by measuring the fraction of material less than 0.3 micron and gloss strength can be characterized by measuring the fraction of material above one micron (see Figure 2). Tinctorial strength - the ability to absorb light - increases with decreasing particle size.
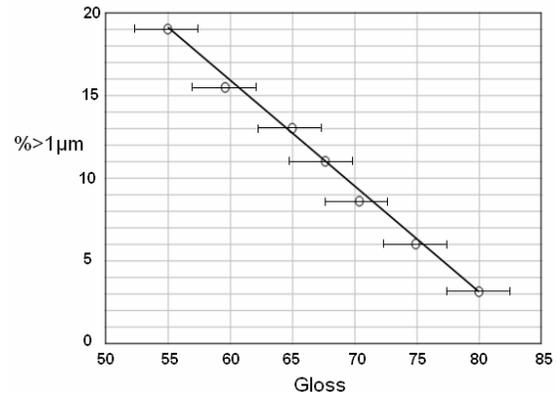

Figure 2

Final Product Functionality

Pigments are typically mixed into various systems such as paint or ink dispersions. Organic pigments are intensely colored particulate organic solids (powders) that do not dissolve in the medium they are mixed with. These powders are added to a vehicle (or matrix) that acts as a binder.

Measurement of pigment particle size serves as an excellent predictor of final product performance. Particle size of pigment particles in paint is a critical parameter that affects surface finish. The difference between flat, eggshell, semi-gloss, and gloss finishes is determined by the PSD of the pigment particles. Accurate monitoring of the particle size is an important parameter to providing good quality paints.

Rheological characteristics such as flow rate, viscosity, adhesion or thixotropic behavior are also affected by the size distribution. Flocculation or agglomeration is avoided with the use of additional surfactants to stabilize the dispersion.

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Particle Size Analysis of Organic Pigments

Pigment particles are milled to ensure they are below the size required by the desired type of finish. The final quality check has often been the Hegman gauge (see Figure 3). This cheap and easy to use gauge provides an indication of the largest particle size, which is often the most critical parameter for surface finish.

Figure 3

While this traditional technique is inexpensive, easy to understand, and can provide a reasonable measure of the final behavior of the paint, the full size distribution allows characterization of a host of other final product performance criteria.

Laser Diffraction Benefits

A complete measurement of the particle size distribution down to much finer sizes can allow optimization of the paint formulation and of the manufacturing process.

The capabilities of current laser diffraction instruments extend into the nanometer sizes, allowing characterization of even the finest size components in a product. The same analyzer used for this study is also perfectly suitable for sub-micron pigments such as TiO_2 and carbon black.

Advantages of this technique include speed, ease of use, a wide dynamic range, and the ability to measure both powders and dispersions. The data collected for this study was analyzed on the HORIBA LA-950 Partica laser diffraction particle size analyzer, capable of measuring particle sizes from 0.01 – 3000 microns.

Particle size analysis by laser diffraction can be performed in the dry state while the milling is taking place. The pigment does not need to be mixed into the final vehicle as required by the Hegman gauge test. This allows tighter control of the milling operation, assuring achievement of the proper specification without over grinding.

Experimental

Four different organic pigments were analyzed using the LA-950 Partica with the PowderJet dry feeder (see Figure 4):

- Diarylide Yellow 83
- Hansa Yellow 74
- Phthalo Blue 153
- Opaque Yellow 83

All samples were properly mixed and sampled prior to analysis. The test method for all samples is shown below.

1. Select the small, high-dispersion nozzle for the fine particle sizes expected.
2. Set the automatic measurement conditions to acquire data in the proper testing range (96-94%T for the laser).
3. Set dispersion air pressure to Low.
4. To prevent nozzle clogging, remove large agglomerates from sample using 1mm sieve (U.S. No. 18).
5. Load enough filtered sample onto feeder chute to perform multiple measurements.

6. Take 3 consecutive measurements using the Auto sequence function.

Figure 4

Results

All samples were measured multiple times to test reproducibility. Table 1 below shows the Dv50, mean and Coefficient of Variation (COV %) for each of the results.

Sample ID	Results	Mean	COV %
Diarylide Yellow 83	18.386		
	18.082		
	18.032	18.17	1.05
Hansa Yellow 74	50.125		
	50.096		
	50.442	50.22	0.38
Phthalo Blue 153	36.49		
	36.618		
	36.603	36.57	0.19
Opaque Yellow 83	25.114		
	25.546		
	25.531	25.4	0.97

Note that all COV % values are well below the 3% level ISO 13320 "Particle size analysis - Laser diffraction methods - Part 1: General principles" suggests to be the maximum acceptable value for this technique. A major reason these values are so low is the automatic control of sample feed rate in the PowderJet Dry Feeder, assuring constant mass flow rate and reproducible results. This also eliminates the creation of false peaks sometimes created by other systems when sampling dry powders.

Overlay plots of the three measurements for each sample are shown in Figures 5 – 8, along with photographs of the samples taken using a digital camera and microscope.

Conclusions

The LA-950 Partica proved to be an excellent choice of instruments to measure the particle size distribution of organic pigments. The PowderJet Dry Feeder system could easily disperse the powder and provided a constant mass flow rate during the measurement. Sample analysis time was under 2 minutes per sample, including change over time from sample to sample. Results include a complete picture of the distribution including calculated moments of the distribution and percent below any chosen size range.

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Diarylide Yellow 83
Figure 5

Hansa Yellow 74
Figure 6

Phthalo Blue 153
Figure 7

Opaque Yellow 83
Figure 8

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