Total Solutions in Particle Characterization

APPLICATION NOTE

Pigment Particle Size Using Laser Diffraction Technology

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Application Note

SL-AN-30 Revision C

Provided By:

Microtrac, Inc.

Particle Size Measuring Instrumentation

Since early times, natural pigments have played a crucial role in the development of many areas of human endeavor including art, construction, communication, food, cosmetics, plastics, textiles and pharmaceuticals. Biologically, the color of plants and their ability to sustain life is controlled by chlorophyll (green) and xanthophylls (yellow). The earliest use of pigments by humans dates back to pre-historic cave paintings providing the first recorded communications at the dawn of civilization.

Since the early 1980s Microtrac has been at the forefront of measuring pigment particle size and particle size distributions using the originally patented (1976) Small Particle Analyzer (SPA). The SPA was the first analyzer to use mulitple wavelengths of light and intensity polarization measurements. With the advancement of semiconductor lasers and fiber optics during the mid-1980s, Microtrac developed the Ultrafine Particle Analyzer (UPA). The UPA measured submicron and nanosize particles using Dynamic Light Scattering employing an intensity-frequency distribution analysis (1990) overcoming the analytical limitations of Photon Correlation Spectroscopy. The UPA and Nanotrac (2000) have since gained global recognition as the instrument of choice by major manufacturers of inks for its speed, sensitivity, repeatability, and robust design. For other pigment materials containing particles outside the normal range of the Nanotrac, the Microtrac Tri-laser Diffraction instrument (patent 1993, Bluewave 2005) is applied when particles larger than 6 microns are important to the quality and final use criteria. The advanced blue laser technology in Microtrac BLUEWAVE (0.010 – 2800 microns) has incomparable resolution and sensitivity particularly below 2 microns. This is a result of newly advanced optical design criteria including detection, analysis and mathematics.

Applications notes describing the use of Microtrac instruments for measuring dry powder coatings and inks are available upon request. This note describes the more general measurement of all pigment types using the Microtrac patented tri-laser diffraction analyzer.

A pigment may be described as a substance that modifies the color of another substance to produce a product that provides identity (pharma tablets and capsules), warning (stop signs), living enhancement (cosmetics, house paint), artful pursuits (artwork) and general utility (printer inks). Pigments generally have properties that allow for selective absorption and reflection/refraction of particular wavelengths and frequencies of light. White light contains many frequencies of light. When a wave of light encounters a colored substance, the light not absorbed, but reflected or refracted into the observer’s eyes provides the color observed. Thus color and the need for pigments affect and enhance all facets of life.

In the coatings industry particle size of the pigments can affect the “finish” of the product. Exterior and interior construction paints can have a range of finishes such as flat, egg-shell, semi-gloss and gloss.

There are many terms used by paint engineers and pigment chemists to describe the product quality parameter that particle size can affect.

Flocculation – Particles attracted to one another affect the degree of rubbing (shear durability). Hegman fineness gauges often are used for this test but are often not discriminatory enough to provide greater size distribution information to determine or test for the best size. For instance, there can be a 10X difference in size for a Hegman reading of 7 compared to a reading of 7 ¾. This is because the Hegman grind test emphasizes the presence of coarse particles. www.pfonline.com.

Tint strength – The ability to modify the color of paint or another pigment. Decrease in size is associated with increased tint strength. Narrow distribution and optimum size, the stronger the tint strength. For TiO_2, the optimum requires size smaller than 1 micron.

Transparency/hue – Size distribution must be monitored and controlled

Hiding – Larger particle size (greater than 10 microns) cause a greater pigment loading. Control is necessary to avoid color drift and optimize hiding.

Flatting, transparency, gloss – These are highly dependent on light scattering (reflection, refraction) where the smallest size promotes uniform coating and higher gloss where the incident angle of light is closer to the angle of reflection. Gloss can be increased 55 to 80 by increasing the volume percent less than 1 micron from 3% to 18%. (www.azonano.com).

Film appearance and industrial coatings – Waxes, polyethylenes and hard polymers impart special properties to films (reduced metal working, scuff and mar resistance, etc). When polymer size is too large the transparent quality of clear coatings and reflective images (colored coatings) is affected. Smaller particle size distributions reduce film imperfections while increasing additive chemical activity, for instance in auto paints coatings.

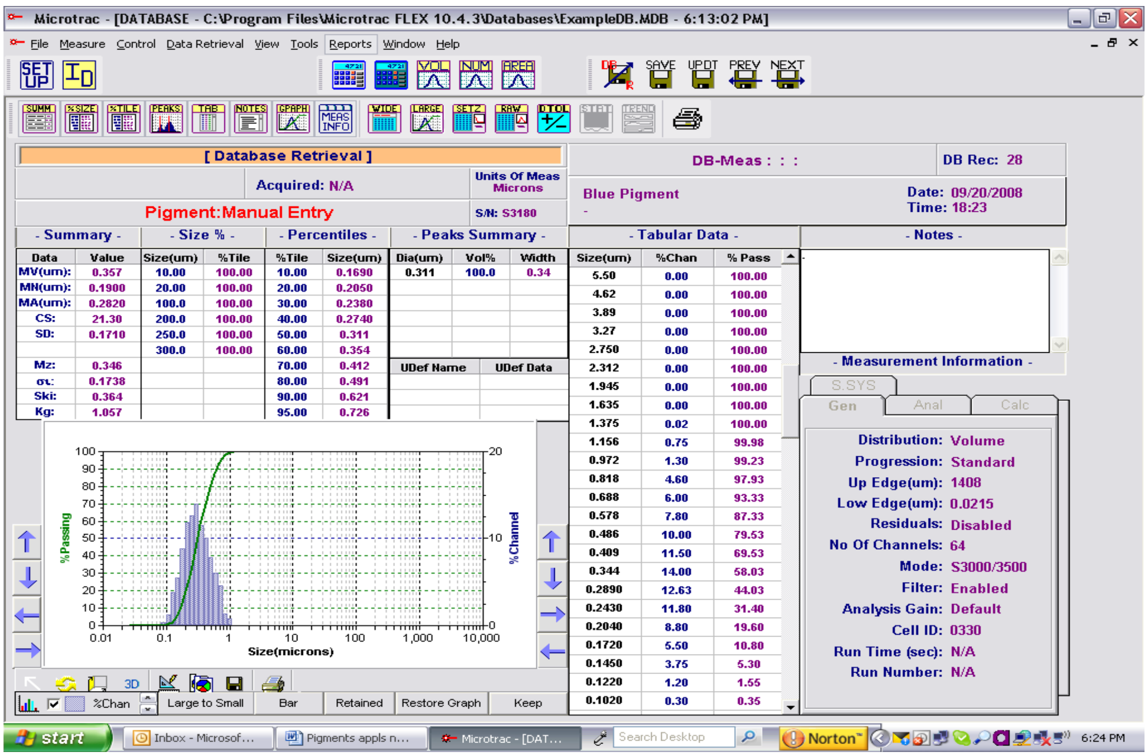
Rheological properties and stability – Viscosity is increased by smaller size particles which prevents settling and re-flocculation. These changes can result in loss of color intensity. Consistency of size for all non-soluble ingredients promotes suspension stability.

Chalking, weather resistance – Chalking refers to removal of paint from a surface for instance by rubbing a finger across the surface and seeing a residue. Small size increases undesirable chalking effects while small particles provide better weathering. Thus monitoring of size distribution is important to maintaining the balance of these properties.

Pigment size varies over a wide range (www.handprint.com.). Modern luster and iridescent paints are probably the largest with particles as large as 100 microns similar to historical mineral pigments. For reference, note that a human hair is typically 100u in diameter. Pigments used for printing inks may have sizes smaller than 50 nanometers. Other paint pigments such as carbon black in its smallest physical form, iron (Prussian) blue and phthalocyanines have sizes as small as 50 nanometers but most demonstrate a size range of 0.1 to 10 microns depending upon type and use. The most desirable size range is 0.1 to 1 microns because of the optical, rheological and coating characteristics imparted at this size. The Microtrac S3500 and Bluewave advanced tri-laser diffraction instruments are uniquely suited for measuring these materials.

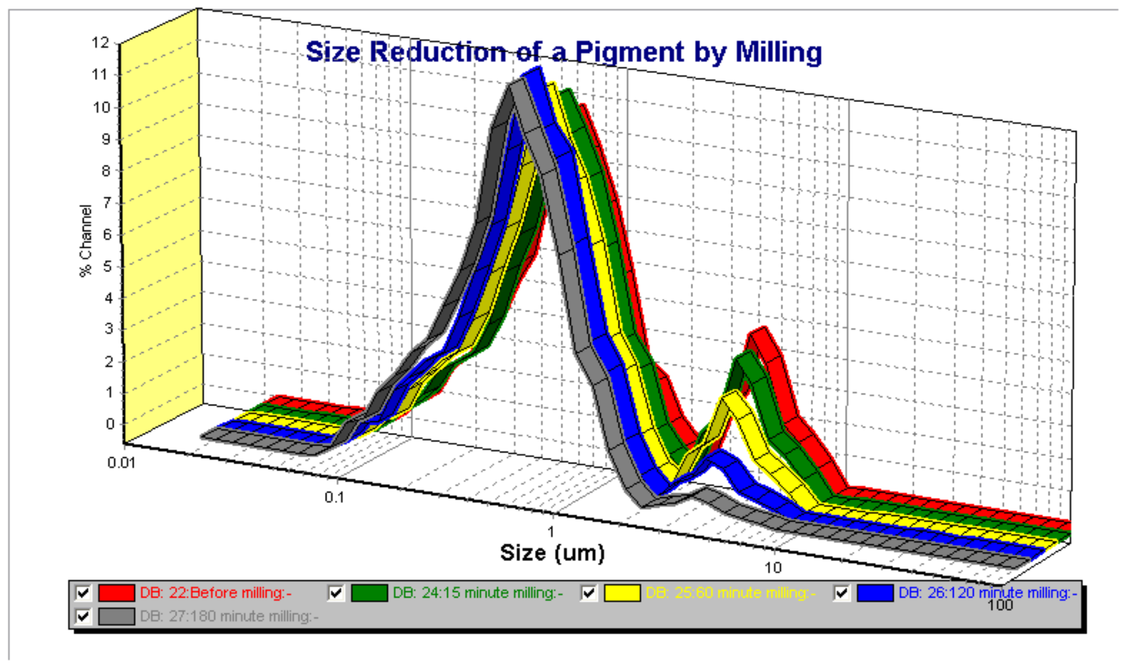
Particle size and distribution of pigments suspended in a coatings vehicle can be measured using the Microtrac laser diffraction instruments by transferring the sample to an automated fluid circulation system that contains a controllable ultrasonic probe. Any organic solvent or aqueous based solution can used to convey the particles to sample cell where particles interact with the laser light. Also, dry powder pigments can be measured using the automated Turbotrac dry powder measuring system. Either measurement requires less than one minute for measurement and presentation of data. **Microtrac Applications Note SL-AN-13** and SL-AN 47 describe the use of the S3500 and Bluewave for dry powders. For very high concentration pigment suspension measurements, the Nanotrac and Zetatrac instruments **(Applications Note SL-AN-22)** may be used for distributions having all particles smaller than approximately 6 microns.

Below is a typical printout containing particle size data for a blue pigment suspended in water containing a non-ionic surfactant such as Triton X100. Other surfactants and/or dispersants can be used, but depending upon the final composition, must be tested to assure proper wetting and dispersion conditions. It is most desirable that the particle dispersion is not disturbed to obtain an accurate view of the current condition of the coating. The Microtrac Applications Laboratory is available for free consultation and assistance to Microtrac customers and clients.



A milling experiment was conducted on a yellow pigment to exemplify the usefulness and sensitivity of the Microtrac laser diffraction instruments in maintaining control and monitoring size distribution. The particle size and distribution were measured as a function of mill time. Measurements were performed using the Triton X100 surfactant solution described above. Sample was removed from the mill and directly added to the Sample Delivery Controller (Microtrac SDC). The expectation was to achieve the smallest size possible to achieve certain properties of weathering and gloss while maintaining viscosity and other rheological properties. Milling was performed for up to 3 hours to achieve the desired quality profile. Often the most efficient surfactant to use for particle size measurement is the surfactant or dispersant used in the production of final coating.

The comparative graph was produced using Microtrac Advance FLEX software.



As expected shorter mill times produced an immediate response while longer mill times were required to reduce the size of pigment particles and agglomerates. Agglomerates are smaller particles that have conjoined into a larger particle due to Van der Waals forces or electrostatic conditions. The presence of surfactants or dispersants during final milling prevents future agglomerate formation. The Microtrac Analyzer easily follows the milling process and because of the speed of the measurement by factory personnel, can allow real time monitoring and control.

As coating science progresses to produce increasingly better surface properties while maintaining “Green”, low emission technology, Microtrac Particle Size Instruments will be in forefront of providing user-friendly yet powerful particle size measurement interface.

For information, please visit the Microtrac website (www.Microtrac.com)