APPLICATION NOTE

Printer Ink-Jet Nanosize Measurement Using Microtrac Dynamic Light Scattering Particles Size Analyzers

Instrument: Wave II / Wave II Q / Nanotrac flex

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Introduction

Microtrac dynamic light scattering (DLS) instruments are perfectly suitable for measuring the particle size of pigment suspensions used in printers. One big advantage is that measurements can be performed at high concentrations to avoid effects of dilution on dispersion.

Printing and writing have involved the use of color or black materials contained in various vehicles since the time of cavemen. The first pigment believed to be used was probably lampblack dating back as long as 4000 -5000 years. Modern printing inks contain many components each having a specific purpose in maintaining color, intensity, dispersion, viscosity, as well as acting as a milling aide. The table below shows the general type of chemical and its application. These materials impart specific properties to the ink to allow for design considerations of printers and final use.

Material	Purpose
Buffers	Control of acidity and pH
Humectants	Avoid too rapid drying
Polymers	Binding to substrate
Defoamers	Eliminates bubbles which cause interruption of ink flow from a printer –head tip
Wetting Agents	Dispersion, reduction of surface tension
Biocides	Biological stability
Thickeners	Viscosity
Dispersants	Maintenance of separated particles
Clays	Filler

Approximately 50% of the cost of inkjet ink is due to the coloring agent. The colorant can be of two types: pigment or dye. Dyes are very small molecules usually considered to be in complete solution. Molecules smaller than 1 nm are considered to be in solution while those larger than 1nm are considered to be in suspension. Pigments are used and are considered to be in suspension (not solution) and may be either organic or inorganic in chemical composition. The type of black pigment used in the past was spinel, rutile or iron. These have been substituted mostly by carbon black.

The ink is developed by mixing the components in a primary blender. Milling or grinding is performed after the more gentle blending operation and includes addition of one or more components. Addition of surfactant reduces surface tension to allow mixing of all the components in water. Along with dispersants, surfactants also assist in maintaining dispersion during the subsequent step of ball or roller mixing. Dispersants may also to be used to lower the mechanical energy required for grinding. Polymers such as polyacrylates, polyurethanes and polyesters are used to obtain the best “blocking” characteristics of adherence to a substrate. It is necessary to be careful with balancing the relative quantities of surfactants and polymers since they may interact which would reduce their effectiveness in maintaining suspension applicability and color strength.

The purpose of all the components is to finally provide a colloidal suspension of crystals or particles which scatter light according to the characteristics of the pigment and other materials present. The resulting light scattering affects light fastness, shade and intensity of color.

Modern advances in electronics and paper coatings allowed the commercial development and availability of color printers in the early 1980s. Of these printers, probably the most popular is inkjet technology. The ink for these printers has the same general composition but small particle size and low viscosity are necessary to pass through the small nozzle of the print head. There are three types of technology used for transfer of the ink: Drop on demand, Continuous Ink Jet and Piezo Ink Jet DOD. These vary depending upon printer manufacture.

Microtrac DLS Analyzers

Microtrac MRB has taken an innovative approach to dynamic light scattering (DLS) by using a proprietary probe design to deliver and collect light. By focusing the laser probe at the material interface, Microtrac combines the benefits of a short path length with reference beating and 180° backscatter, delivering the best accuracy, resolution and sensitivity. The probe as the catalyst for Microtrac MRB’s Reference Beating, an enhancement to traditional DLS, increases the optical signal back to the detector anywhere from 100 to 1,000,000 times more than systems that use “self-beating.” The enhanced signal produces superior analysis results when measuring single modes or multi-modes across the widest concentration range. Figure 1 shows the three DLS instruments available from Microtrac. The NanotracFlex has a dip-in probe for in-situ measurements so than almost any vessel can be used as a sample cell. With the Wave II Q five different cuvette types can

be used, including micro-volume, glass or stainless steel / sapphire. The Wave II has a specially developed measuring cell made of Teflon (stainless steel is available). Thanks to the integrated electrode, this device can also be used to determine the zeta potential.

Fig. 1: *Microtrac DLS analyzers. From left to right: Nanotrac Flex, Nanotrac Wave II Q, and Nanotrac Wave II*

Application of DLS to Measuring Inks

Microtrac DLS instruments like the Wave II provide an exceptional avenue to provide Dynamic Light Scattering data. Use of the Power Spectrum analysis concept provides high repeatability and advanced analysis while the probe technology permits measurement at high particle concentrations. For more information on design concepts see Microtrac Applications Note SL-AN-14 Nanoparticle Sizing - Dynamic Light Scattering in the Frequency Spectrum Mode.

Sample is placed in a beaker or other suitable container. Wave II FLEX software is initiated and a background measurement is performed using water or other clean fluid matrix. The choice of fluid is dependent upon the base fluid and truly-soluble components. The probe is transferred to the waiting sample and the measurement is started. Background measuring time is set at 15-30 seconds while measuring time is set to 30-60 seconds.

Dynamic Light scattering is governed by certain basic concepts as described in the Stokes-Einstein Theory. Accordingly, the viscosity of the clean, suspending fluid is required. Contrary to some promoters of DLS instruments the viscosity of the suspension is not needed and a viscometer is not required to make viscosity measurements of individual samples. Data are shown below for a variety of inkjet and flexographic inks.

Example Results

Figure 2 shows a typical printout for blue colored flexographic ink where Wave II is used as a quality control measure. The image is that shown on the monitor following measurement of the ink. Note the presence of the bimodal distribution. The mode at the larger sizes was verified by optical (light) microscopy as was the well-dispersed state. The sample was measured using a 3:1 dilution. The second mode may be indicative of agglomerated particles or individual coarse particles. It may also be characteristic of the ink.

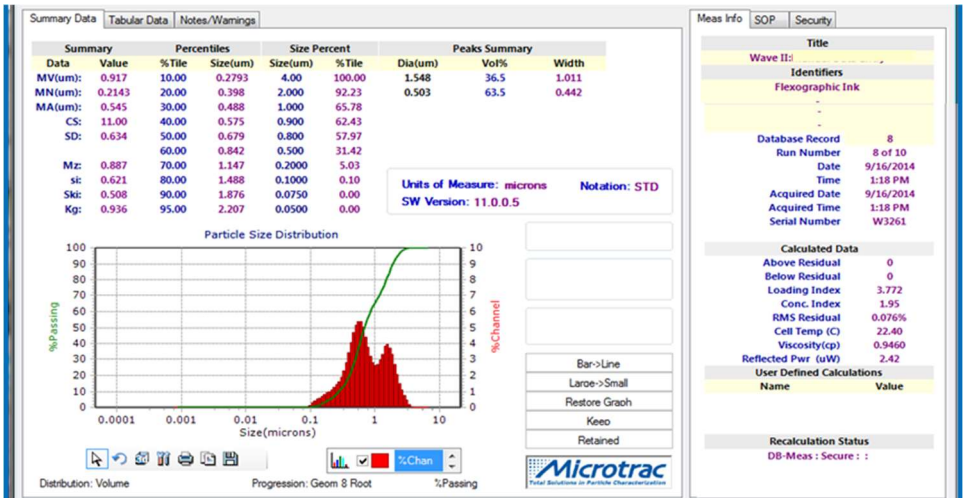

Fig. 2: *Measurement result of ink (Nanotrac Wave II Q). Some features of the data display: (1)Distribution is bimodal, easily discerned due to the Frequency Spectrum Analysis used by Microtrac; (2) D50 and amount of each peak is shown (Peaks Summary); (3) Data are protected according to FDA 21 CRF Part 11 (lower right corner); (4) High calculation assurance shown with RMS residual 0.076%.*

In Figure 3, FLEXOGRAPHIC blue ink distribution is compared with that of magenta. The comparative graph was developed using Microtrac FLEX software which allows direct copying into PowerPoint or other office programs. The magenta sample does not show the presence of a second distribution mode by either Wave II or as confirmed by microscopy.

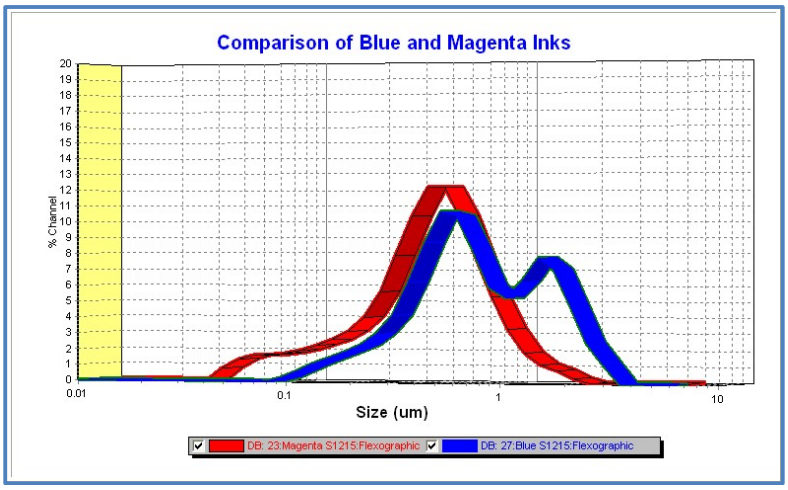

Fig. 3: *DLS result for magenta ink and blue ink. In contrast to the magenta sample, the blue ink is bimodal.*

The following figures show measurement result for ink-jet inks of various colors. Interestingly the yellow ink shows a second mode near 20nm. This feature is present in yellow ink from some manufacturers.

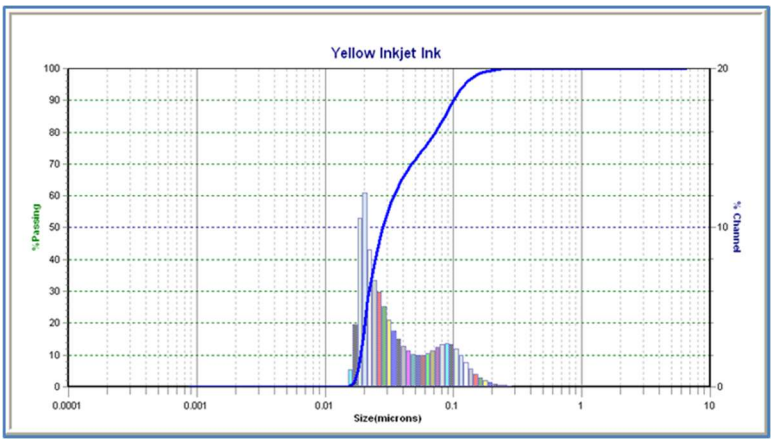

Fig. 4: *DLS result for yellow and blue ink.*

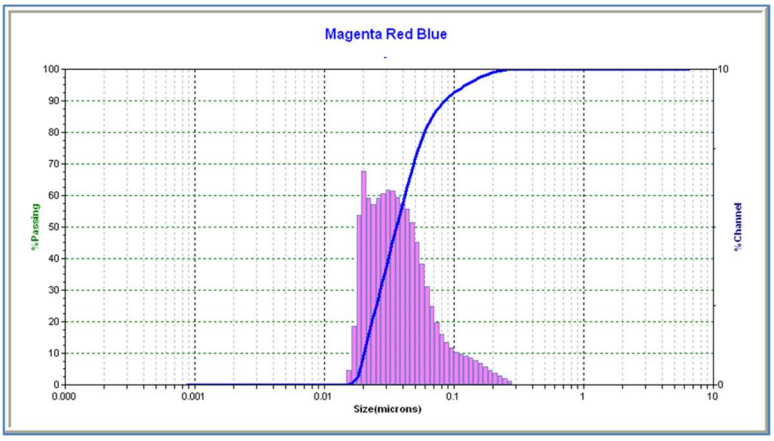

Fig. 5: *DLS result for magenta ink.*

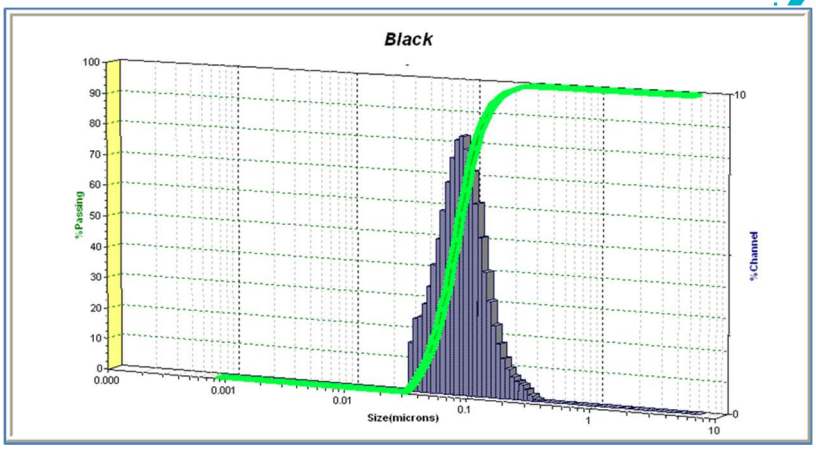

Fig. 6: *DLS result for black ink.*

Conclusion

Wave II Q and other Microtrac DLS analyzers have the capability to measure inks of all colors including black, magenta, yellow and blue. The measurement can be conducted using high concentrations and can reveal special distribution features such as bi-modal distributions and changes in particle size.

NANOTRAC series capabilities

- Patented "Controlled Reference Method" for high signal and true particle size distribution by frequency spectrum analysis.
- Measures multimodal distributions without assumptions or "fitting"
- True background measurement eliminates need for high purity diluent or filtration operations.
- Withstands plant environment
- Peltier temperature control 5°C to 100°C (Wave II & Wave II Q)
- Full database management capability exportable using HTML, ASCII, pdf formats and to all popular spreadsheets and database managers
- High concentration measurement up to 40% solids (sample dependent)
- Simplicity of operation
- NO extensive sample preparation
- NO selection of special distribution models where operator decides on cumulants, NNLS, CONTIN or other complex choices.
- Compatible with most common organic solvents and aqueous solutions.
- Many models to choose from:
- Internal cell (200 - 2000µl) available in Teflon or stainless steel.
- Cuvette cell (Wave Q) uses both disposable plastic and reusable glass cells
- Polystyrene - (Min vol. /Max. Vol.): 50/1000µl, 300/2000µl and 1000/3000µl.
- Glass – 1000/ 3000µl
- External probe an excellent choice for:
- Dip 'N' Run method (like making a pH measurement), or
- Any volume greater than 500 µl covering probe tip.
- Zeta Potential capability (optional) – 150/2000µl

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For further information please contact us at:

www.microtrac.com