HORIBA Scientific

Application Note

Particle Size Analysis of Inks

AN183

An ink is a liquid containing various pigments and/or dyes used for coloring a surface to produce an image, text, or design. Ink is a complex medium composed of solvents, pigments, dyes, resins, lubricants, solubilizers, surfactants, particulate matter, fluorescers, and other materials. Particle size of the pigment particles can significantly affect the color strength, surface finish, and durability of the final product. Measuring the particle size of ink pigments was typically done using dynamic light scattering (DLS). Laser diffraction can also now be used to measure these pigments due to the state of the art enhancements in the LA-960.

Introduction

The components of inks serve many purposes. The carrier, colorants, and other additives are used to control flow and thickness of the ink and its appearance when dry. The colorant can be either a dye (solution) or a pigment (particulate). Since the color strength of the pigment depends on the exposed surface area, a smaller particle size generates a stronger color. Other ink qualities affected by the pigment particle size include saturation, brightness, opacity, and viscosity. The opacity of the ink tends to maximize at a mean particle size near 0.25 - 0.50 µm. Particle size also plays an important role with ink rheology since a reduction in particle size increases the suspension viscosity. Particle size distribution is another important physical parameter requiring measurement and control. In general, a wide distribution reduces color strength and can be indicative of poor stability. Figure 1 shows a basic diagram of how particle size influences several ink physical properties.

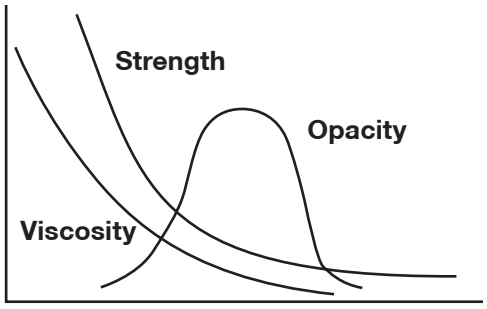


Figure 1: Generalized relationship between ink particle size and other physical properties

It is often desirable to reduce the pigment to the primary particle size in order to achieve optimum benefits. Monitoring the milling process to achieve the desired size requires frequent use of a particle size analyzer. It is therefore quite important to be able to measure the particle size of ink pigments.

Choice of Technique

Most ink pigments fall into the range of 50-200 nm. Several techniques are capable of measuring in this range including dynamic light scattering (DLS) and laser diffraction. DLS can measure particle sizes within the range of roughly 1 nm – 1 µm, so this technique has been used to measure ink pigments for many years. Using the HORIBA LB-550 DLS system for ink pigments has been documented (1,2,3) and can be found performing these measurements in labs around the world.

Due to enhancements in small particle sensitivity, laser diffraction is now also being used to measure sub-micron ink pigments. The HORIBA LA-960 has proven to be able to measure real world samples with mean sizes as small as 30 nm (4), but has the ability to measure larger particles up to the millimeter range. This technique provides more flexibility to measure larger agglomerates/flocculates and be used for other applications due to its broad dynamic range.

Figure 2: LA-960 Laser Diffraction Particle Size Analyzer

Experimental

Ink Sample 1 was analyzed on both the LB-550 and LA-960 systems. The concentrated sample was diluted in an organic solvent using both instruments. The operating conditions are shown below:

LB-550:

1. Add 1 drop of the sample in the organic solvent. Mix well.

2. Add one drop of pre-diluted sample to cell.

3. Mix with pipette in the cell.

4. Set the sample holder temperature to 25°C and wait until sample reaches this temperature.

5. Take 3 consecutive readings and record results.

LA-960:

1. Fill MiniFlow sampler with organic solvent.

2. Circulate until system reaches equilibrium.

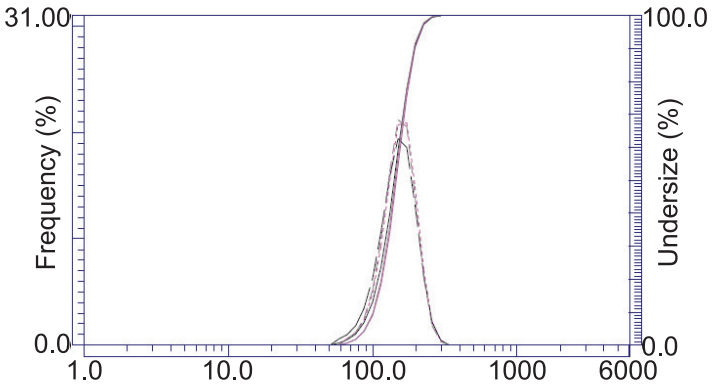
3. Align and blank optics.

4. Pre-disperse 2 drops of sample into 15 mL organic solvent in beaker.

5. Transfer sample from beaker into MiniFlow sampler using disposable pipette.

6. Measure sample 3 times, record results.

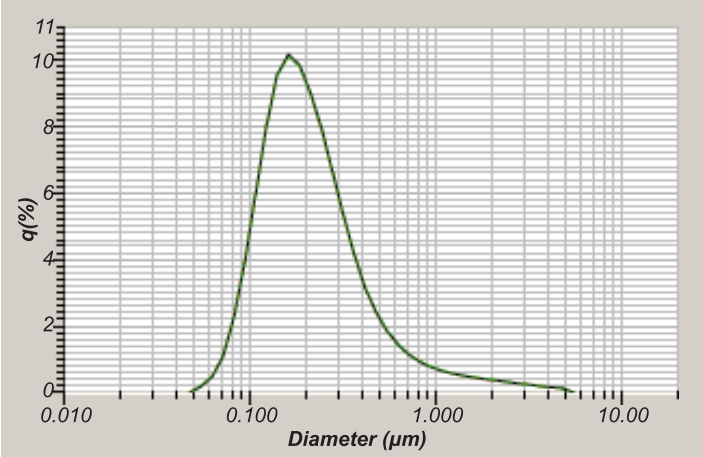
The results from the LB-550 measurements are shown in Figure 3. Note that the particle size information is reported as a volume distribution in order to compare with those results from the LA-960.



Median:	145.9 (nm)	Diameter on %:
Mean:	148.1 (nm)	(2) 10.00 (%)-101.4(nm)
Mode:	157.8 (nm)	(9) 90.00 (%)-196.5(nm)

Figure 3: DLS results for Ink Sample 1

The results from the LA-960 measurements are shown in Figure 4.



Median Size	: 0.19342 (µm)
Mean Size	: 0.30765 (µm)
Mode Size	: 0.1611 (µm)
Diameter on Cumulative %	: (1)10.00 (%)- 0.1049 (µm)
	: (2)50.00 (%)- 0.1934 (µm)
	: (3)90.00 (%)- 0.5174 (µm)
Std. Dev.	: 0.4290 (µm)
CV	: 139.4322 (%)
Span	: 2.1324

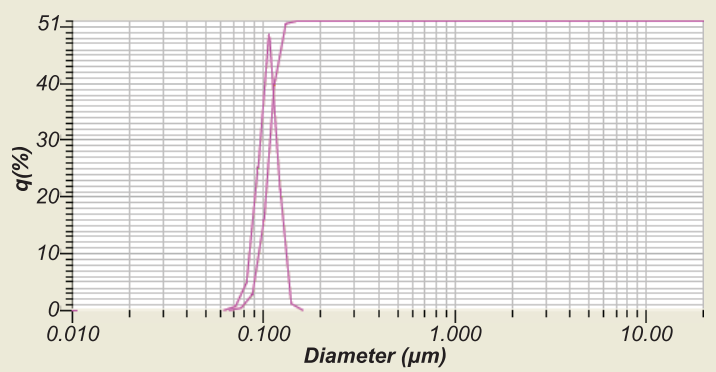
Figure 4: Diffraction results for Ink Sample 1

Although these results are not identical, the similarity confirms the ability of the LA-960 to operate in this size range. The modes of the distributions are reported as 157.8 nm using DLS and 161.1 nm using laser diffraction, indicating essentially the same peak to the frequency distribution. The differences in the other reported results are not surprising since the two techniques measure different physical properties. It is quite rare that different particle size techniques generate identical results.

Ink Sample 2 is a water-based ink called Color Wonder. This sample was measured on the LA-960 using the procedure described below:

1. Fill LA-960 sample handler with DI water.
2. Begin circulation, align and blank optics.
3. Add drops of the sample directly into the sample handler reservoir until the desired concentration (%T) is achieved.
4. Measure 3 times and report results.

The results from the LA-960 measurements are shown in Figure 5.



Median Size	: 0.10583 (µm)	Diameter on Cumulative %:
Mean Size	: 0.10585 (µm)	(2) 10.00 (%)- 0.0896(µm)
Mode Size	: 0.1065 (µm)	(9) 90.00 (%)- 0.1237(µm)
Std.Dev.	: 0.0120 (µm)	

Figure 5: Diffraction results for Ink Sample 2

Conclusions

Both DLS and laser diffraction are acceptable techniques for measuring the particle size of ink pigments in the 50 – 200 nm size range. The results shown in Figures 4 and 5 are actually overlays of three repeated measurements. It is impossible to see differences in the results due to the extreme precision of these measurements. The LA-960 is the most sensitive laser diffraction analyzer - capable of measuring below 30 nm - and the most repeatable and reproducible. These features combined with the broad dynamic range offered by laser diffraction make the LA-960 a uniquely capable instrument for ink pigments and other sub-micron dispersions.

References

1. Miyashita et. al., A new model system for studying excited states of dye aggregates of photographic color paper, Journal of Photochemistry and Photobiology A: Chemistry, Volume 194, Issues 2-3, 20 February 2008, Pages 129-135

2. Miyashita et. al., Excited-states and lightfastness of linear transquinacridone pigment nanoparticles, Journal of Photochemistry and Photobiology A: Chemistry, Volume 201, Issues 2-3, 25 January 2009, Pages 208-213

3. HORIBA AN143, Measurement of High Concentration Ink and Pigments Using Dynamic Light Scattering, available at: www.horibalab.com

4. HORIBA TN156, Colloidal Silica as a Particle Size and Charge Reference Material, available at: www.horibalab.com

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