

Particle Size of Inkjet Inks

*Nicomp® DLS system and
AccuSizer® SPOS system*

OVERVIEW

Inkjet inks are colloidal dispersions of pigments in solution. Proper dispersion of the pigments is necessary to avoid sedimentation, instability, or inkjet nozzle failure due to agglomeration. Assuring optimum formulation and manufacturing requires a reliable method to determine the particle size distribution of the final product. The Nicomp® dynamic light scattering (DLS) system can be used to determine the mean size and zeta potential of ink dispersions. The AccuSizer® single particle optical sizing (SPOS) system is ideal for determining if the final inkjet ink contains any oversized particles (tails) that pose a risk for clogging jets and causing other reductions in performance. This application note shows several examples where the Nicomp system and AccuSizer system solve inkjet ink manufacturing challenges.

INTRODUCTION

The pigments formulated into inkjet inks are typically dispersed to small particle sizes (between about 50 and 200 nm, depending on the application) and need to be made colloidally stable. The colloidal stability can be achieved either by surface modification to form an adequate surface charge (zeta potential), or by adsorption of certain compounds on the surface of pigment particles (steric stabilization).

The size of the pigment particles is critical because large particles may plug the jets and channels, causing damage to the print head. Controlling the large particle content (>0.5 – 1.0 μm) requires having a technique that is sensitive to a small number of large particles, the tail of the distribution.



The AccuSizer A7000 APS system.

PARTICLE SIZING AND COUNTING TECHNIQUES

There are several methods for determining the mean particle size of ink dispersions, such as dynamic light scattering (DLS), but most are not capable of determining small amounts of oversized material. Methods that are based on sizing and counting individual particles are particularly well-suited for this type of analysis, where even small amounts of outliers are sized and counted in the process.

The AccuSizer SPOS system is ideal for quantifying the size and concentration of large particle tails present in inkjet inks. The AccuSizer system includes the LE400 sensor that measures from 0.5 – 400 μm mounted in the APS dilution fluidics and autosampler that provide automated dilution of the sample to the optimum concentration for the measurement.

APPLICATION EXAMPLE 1: BLACK INK MEAN SIZE AND TAILS

A commercial black inkjet ink sample was analyzed using DLS to determine the mean size and zeta potential. The sample was diluted 1000:1 in deionized (DI) water and a quick study was performed to assure size did not change with additional dilution. The results for the mean size and zeta potential as measured by the Nicomp DLS system are shown in Figures 1 and 2.

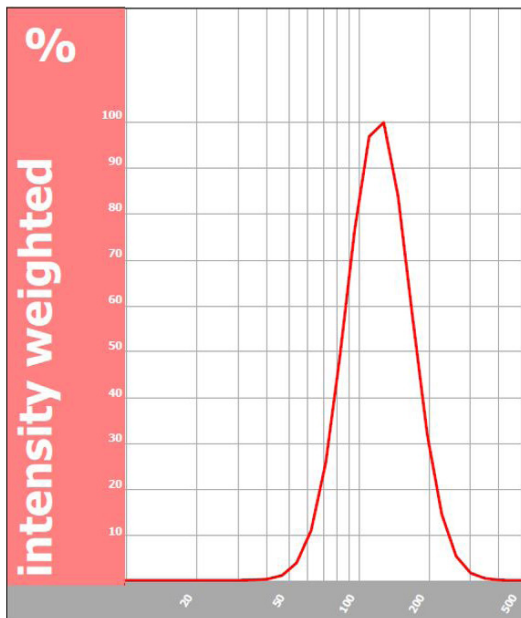


Figure 1. Black ink intensity mean size = 127.49 nm by DLS.



Figure 2. Black ink zeta potential = -32.1 mV.

The AccuSizer APS system was then used to quantify the large particle tail greater than 0.5 μm . The sample was measured by directly injecting 100 μL of prediluted 10:1 (D10) and 100:1 (D100) sample directly into the dilution vessel and using single-stage exponential dilution. This approach is useful when first studying unknown samples. Once the proper dilution ratio is determined the AccuSizer APS system can also operate using two-stage dilution to keep the count rate in an optimum range during the measurement. The results for the D10 and D100 samples are shown in Figure 3.

Notice the close agreement between the two dilutions, validating the ability of the AccuSizer instrument to accurately calculate back to the actual sample concentration.

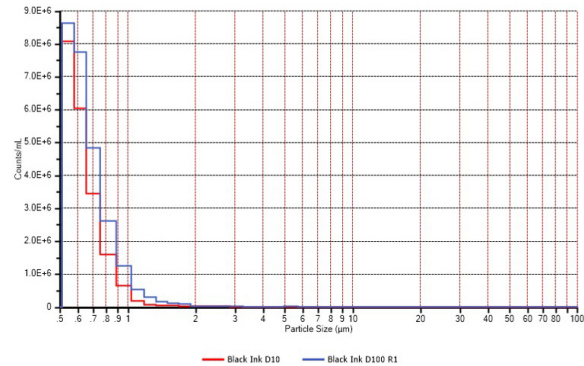


Figure 3. Black ink tail counts/mL vs. size for D10 and D100.

APPLICATION EXAMPLE 2: INK FILTRATION STUDY

Filtering out the large particles can assure proper performance of the ink in the printer. Testing the ink using the AccuSizer SPOS system is the best method for determining the optimum filtration method. Commercially available magenta and yellow inkjet ink samples were tested before and after syringe filtration using the AccuSizer APS system. The results shown in Figure 4 show the particle counts/mL vs. size of the magenta ink sample before filtration, after two passes through a 0.8/0.2 μm two-stage syringe filter, and after one pass through a 0.02 μm filter.

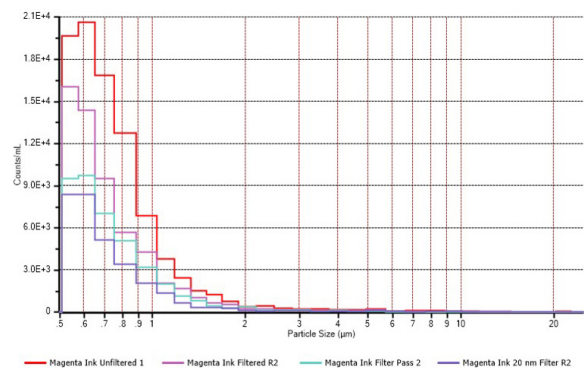


Figure 4. Magenta ink before and after filtration.

Figure 5 shows the yellow ink sample before filtration and after one and two passes through a 0.8/0.2 two-stage syringe filter.

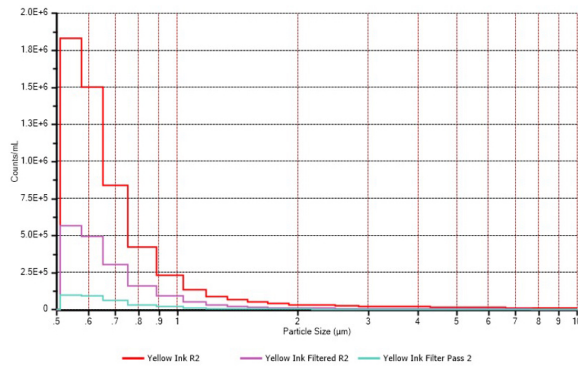


Figure 5. Yellow ink before and after filtration.

AUTOMATION

Both the Nicomp DLS system and the AccuSizer SPOS system can be interfaced with an autosampler for high sample throughput requirements as shown in Figure 6. Different concentrations, run times, and flow rates can be analyzed in the same sample tray by using different measurement protocols for each sample. Automated reporting and exporting results to Excel keeps data management easy.

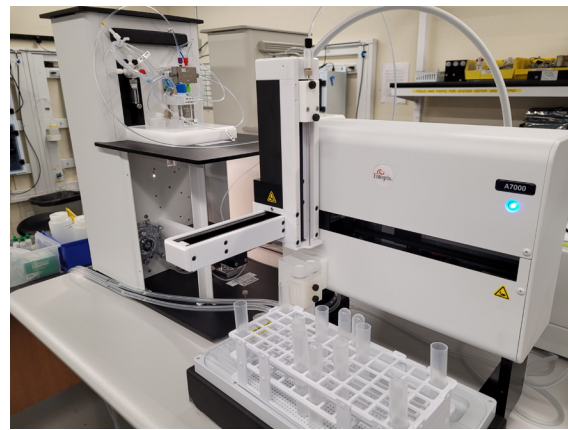


Figure 6. AccuSizer APS system (left) and autosampler (right).

CONCLUSIONS

Entegris provides multiple solutions for particle characterization of inks used in inkjet printers. Mean particle size and zeta potential can be analyzed using the Nicomp DLS system. The AccuSizer SPOS system is an ideal instrument for quantifying the size and concentration of large particle tails in inkjet ink. In this document the effect of filtration was investigated, but the system can be used both as a general quality assurance tool or as a process development tool to investigate optimum process conditions. Coupling either system with the autosampler facilitates high sample throughput requirements. Entegris field application specialists provide installation and calibration support worldwide.

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Corporate Headquarters

129 Concord Road
Billerica, MA 01821
USA

Customer Service

Tel +1 952 556 4181
Fax +1 952 556 8022
Toll Free 800 394 4083

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