Particle Size of Inkjet Inks

AccuSizer®

Figure 1. The AccuSizer A7000 AD

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 Entegris AccuSizer® single particle optical sizing (SPOS) system is ideal for determining if the final inkjet ink contains any oversized particles that pose a risk for clogging jets and causing other reductions in performance. This application note shows several examples where the Entegris AccuSizer solves inkjet ink manufacturing challenges.

INTRODUCTION

Biotherapeutics have been shown to be susceptible to inducing the elicitation of antidrug antibodies (ADA). Evidence suggests that protein aggregations have the ability to enhance immunogenicity and therefore enhanced immune responses to the monomeric form of the protein.

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.

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. Depending on the sensors incorporated in the system, the AccuSizer can cover a dynamic range of 0.15 – 400 µm. The system shown in Figure 1 includes the standard LE400 light and scattering sensor that measures from 0.5 – 400 µm mounted in the AD sampler that provides automated dilution of the sample to the optimum concentration for the measurement.

APPLICATION EXAMPLE 1: EFFECT OF STIRRING

There are many factors that can impact pigment dispersion, one of which is stirring time. It is necessary to determine an optimal stirring time to reduce the number of oversized particles within a dispersion. It is also important to monitor stirring time since over homogenization can lead to increased particle size.

Two pigment dispersions, magenta and cyan, were analyzed to monitor the effects of stirring time on oversized particles. Figures 2 and 3 show the results for the magenta sample that was stirred for 50, 70, and 90 minutes. The tail particles decreased from 4×10^6 particles/mL to 2×10^5 particles/mL.

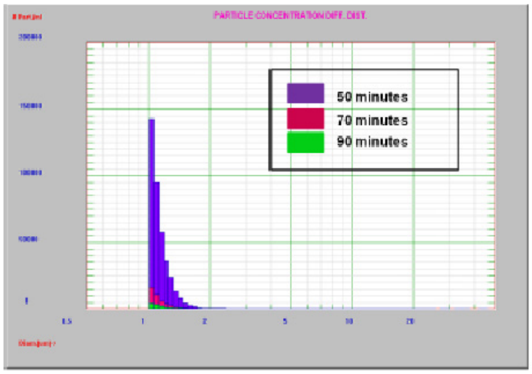

Figure 2. Effect of stirring on magenta sample

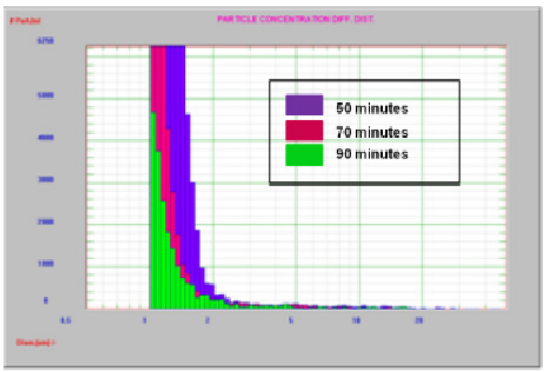

Figure 3. Expanded view of magenta sample

Concentration of large particles >1 µm:

50 min: 4×10^6 particles/mL

70 min: 5×10^5 particles/mL

90 min: 2×10^5 particles/mL

The cyan sample shown in Figure 4 also undergoes a decrease in the number of oversized particles with stirring time. The concentration of particles decreases from 9 million particles/mL to approximately 3 million particles/mL with just 10 additional minutes of stirring.

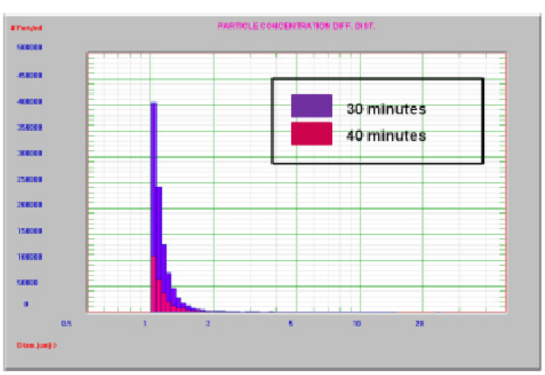

Figure 4. Effect of stirring on cyan sample

APPLICATION EXAMPLE 2: EFFECT OF FILTRATION

This example shows the results of a filtration test in which an inkjet ink is filtered using a 2 micron and 5 micron filter, see Figure 5. Both filters show an improvement in the concentration of particles >1 micron. The unfiltered sample contained >100,000 particles/mL greater than 1 micron while the 5 micron and 2 micron contained ~70,000 and ~20,000 particles/mL respectively.

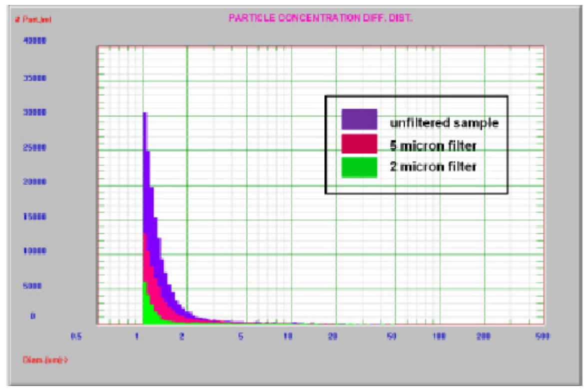

Figure 5. Effect of filtration on inkjet ink

CONCLUSIONS

The AccuSizer 780 is an ideal system for quantifying the size and concentration of large particle tails in inkjet ink. In this document the effect of stirring and filtration are 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. A variety of sensors and configurations are available depending on the application requirements.

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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