SPES Classification of Inks, Watercolours, and Pigments

INTRODUCTION

Inks, watercolours, and oil pigments are mixture of microscopic and sub-microscopic grains, typically suspended in a liquid that allows the pigment to be applied. The liquid itself is a mixture of components and additives having physical and chemical role in defining the paint before and after use. Shelf life, manufacturing costs, handling of paint, and final appearance are examples of issues traded off by optimizing the liquid. Pigments are milled so that a given mass is divided into smaller and smaller particles, thus increasing the surface area. This increases the quality of the paint but also requires more liquid to wet the pigment. Depending on the material the pigments are made of, dispersants can be added to reduce clustering or clumping. Ink, watercolour, and oil pigments are usually manufactured to a well-defined particle size, or with a given particle size distribution, depending on the material and application. Particle size affects the paint features like transparency, saturation, but also staining and even shelf life and effectiveness of use. From a physical point of view, refractive index plays a major role in determining the optical properties of the particles.

Measuring size and refractive index of pigments is of utmost importance for characterizing them and for improving product formulation. The further presence of secondary populations in the formulation, as oil emulsions in suspension, limits and/or precludes the traditional particle sizer to perform an effective and reliable measure.

In this application note, the classification, the quantification, and the characterization via EOS Classizer™ ONE of commercial products and colour/oil emulsion mix are presented.

PARTICLE ANALYSIS METHOD

Among the several methods currently adopted, optical ones have unique advantages, and therefore, have brought light scattering into the forefront of analytical methods in many scientific and industrial applications. Unfortunately, the number of parameters typically affecting the scattering properties of a given particle is such that the basic measure of the scattering power (or even the power removal from a light beam -extinction- from one particle) is far from being enough to recover something more than a rough estimate of its size. Things change appreciably when considering a collection of many scatterers, with the immediate drawback of introducing the need for mathematical inversion and ill-posed problems to interpret experimental real data.

EOS Classizer™ ONE particle analyser is based on patented Single Particle Extinction and Scattering (SPES) method. It introduces a step forward in the way light scattering is exploited for single particle characterization.

EOS Classizer™ ONE – front view

EOS Classizer™ ONE provides data that go beyond the traditionally optical approaches. EOS Classizer™ ONE discriminates, counts, and analyses single particles through their optical properties. It retrieves to the user several pieces of information such as: particle size distribution of the single observed populations, absolute and relative numerical concentrations, particle stability, information on optical particle structure and oversize. Classizer™ ONE works offline and online/real-time, enabling to verify consistency of intermediate and final formulations with target QbD, SbD, and Quality Control target expectations.

For a general introduction to SPES data please refer to the Application Note AN001/2021, available online along with other application notes and example of applications at EOS website: www.eosinstruments.com/publications/

APPLICATION EXAMPLES

EOS Classizer™ ONE is exploited to characterize pigment suspensions in pure water. Cases discussed in this document are reported below:

1. Analysis of inks
2. Analysis of a mix of inks
3. Analysis of watercolours
4. Analysis of a mix of a pigment and an oil emulsion in water

1) Analysis of Inks

Ink is a gel, sol, or solution that contains at least one colourant, such as a dye or pigment, and is used to colour a surface to produce an image, text, or design. Ink is used for drawing or writing with a pen, brush, reed pen, quill, or an inkjet printer. Thicker inks, in paste form, are used extensively in letterpress and lithographic printing. Ink can be a complex medium, composed of solvents, pigments, dyes, resins, lubricants, solubilizers, surfactants, particulate matter, fluorescents, and other materials. The components of inks serve many purposes; the ink's carrier, colorants, and other additives affect the flow and thickness of the ink and its dry appearance. In Figure 1 three data sets obtained with commercial calligraphy ink are presented.

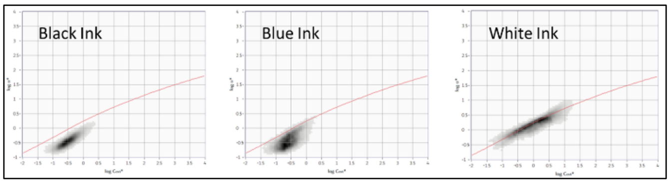

Figure 1 EOS CLOUDS of black, blue, and white calligraphy inks. The red line is just a guide for the eye and represents the expected position for TiO_2 particles (white ink).

For the measurement, small aliquots of each ink are diluted in milliQ water. The three populations are classified and located in different position in the EOS CLOUDS accordingly to the measured optical properties of the pigments at the wavelength of the laser light used for the study (λ640nm). The high extinction cross section at fixed polarizability of the black and blue inks is compatible with absorption from these scattering particles. Semi-automatic evaluations of the optical properties via EOS software of these parameters allow a better evaluation of the numerical PSDs, the cumulative PSDs and of the oversize of the data in Figure 1 as represented in Figure 2, Figure 3, Figure 4.

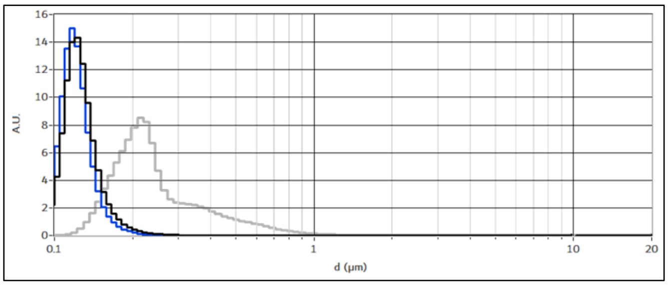

Figure 2 Numerical PSD of the calligraphy inks in Figure 1.

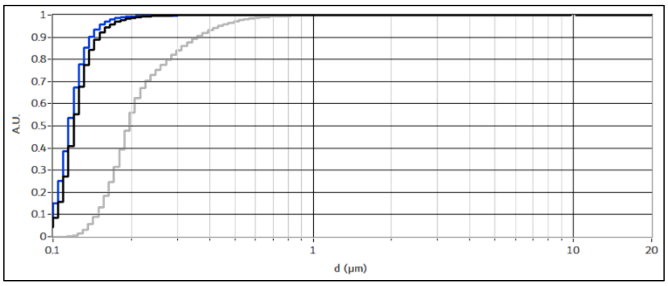

Figure 3 Cumulative PSD of the calligraphy inks in Figure 1.

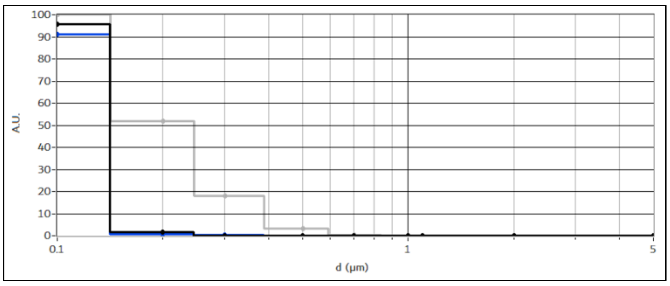

Figure 4 Oversize study of calligraphy inks in Figure 1.

2) Analysis of a mix of inks

Through the study of the properties of the individual particles dispersed in a liquid, the SPES technique classifies the presence of particles having different characteristics in a liquid. In Figure 5 we report the analysis of a 50:50 mixture of white ink and black calligraphic ink.

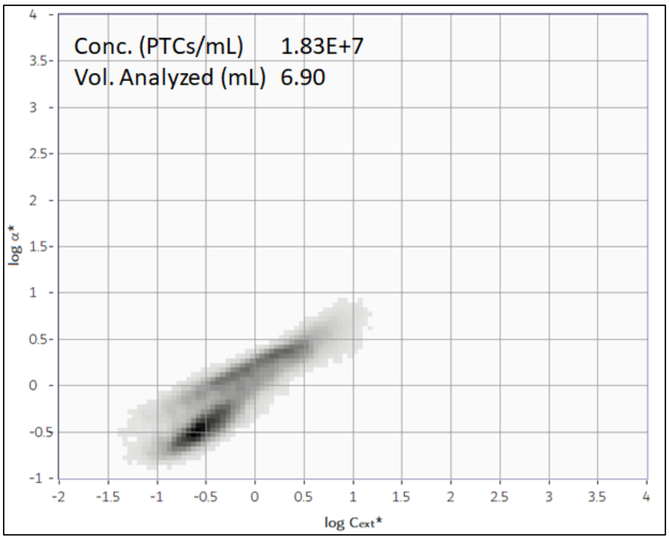

Figure 5 A 50:50 mixture of white ink and black calligraphic inks.

As described in the application notes AN001/2021 and AN006/2021, one of the added values of Classizer™ ONE and of the SPES technology is the possibility of using the multiparametric measurement and following two-dimensional representation to select and analyse the individual particle populations in a mixture. It is possible to experimentally evaluate the ratios between the numerical concentrations of the components of a mixture in a formulation, as shown in Figure 6 for the case considered.

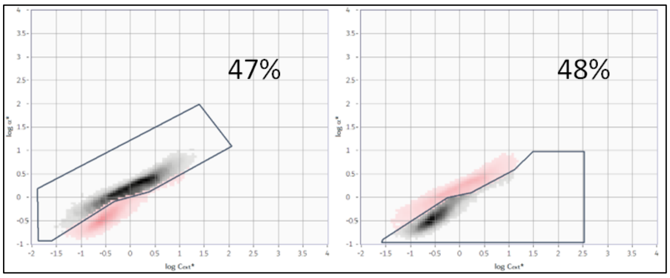

Figure 6 Experimental estimation of the relative number of particles attributable to the individual components of the mixture.

The dimensional analysis of the particles in the selected area is naturally carried out allowing to monitor their characteristics and stability in complex systems.

2) Analysis of Watercolours

Watercolour, also aquarelle (French, from Italian diminutive of Latin aqua "water"), is a painting method in which the paints are made of pigments suspended in a water-based solution. In Figure 7 three data sets obtained with commercial watercolours are presented.

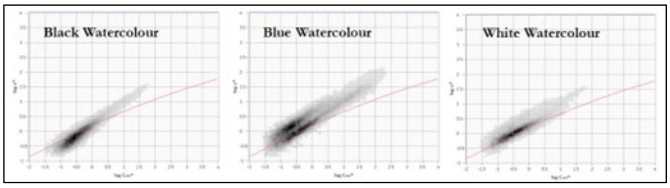

Figure 7 EOS CLOUDS of black, blue, and watercolours. The red line is just a guide for the eye and represents the expected position for TiO2 particles (white ink).

It is observed that the populations and are more polydisperse and larger particles respect inks are observed. Furthermore, the blue watercolour has a double population indicating that its formulation has material with differentiated optical properties in suspension. Automatic evaluation via EOS Software of the optical properties of these samples provides the numerical PSDs, the cumulative PSDs, and of the oversize of the data in Figure 7 as represented in Figure 8, Figure 9, and Figure 10.

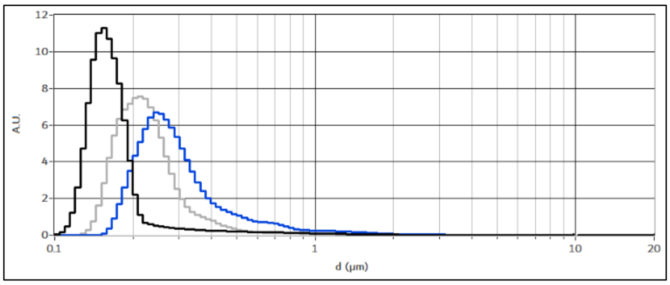

Figure 8 Numerical PSD of the watercolours in Figure 7.

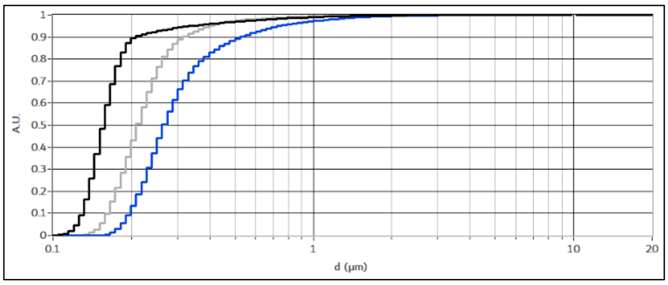

Figure 9 Cumulative PSD of the watercolours in Figure 7.

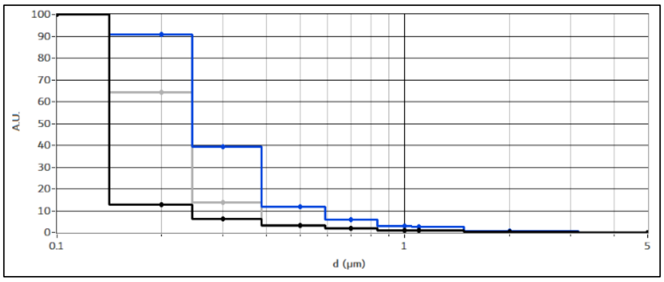

Figure 10 Oversize study of watercolours in Figure 7.

2) Analysis of a mix of a pigment and an oil emulsion in water

Formulations of pigments in cosmetics, paints, and multiple sectors require the dispersion of different materials such as oil emulsions and ground pigments in a liquid. The analysis of the pigment particle populations and the identification of oil droplets individually suspended in the liquid require the ability to identify populations according to e.g., their respective optical properties. The SPES patented technology and Classizer™ ONE provides the adequate information as needed and suitable for the purpose to distinguish the different types and relative concentrations of the suspended materials as shown in the figure for an uncalibrated mixture of blue ultramarine and an oil emulsion.

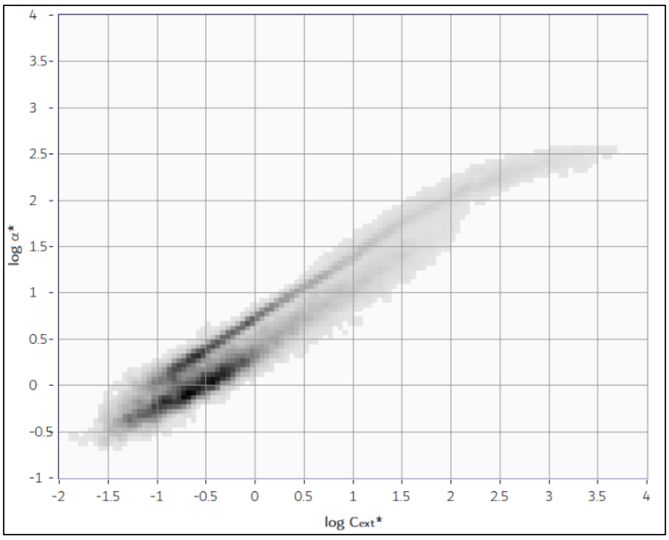

Figure 11 An uncalibrated mixture of blue ultramarine and oil emulsion. Two populations are visible in the EOS CLOUDS.

It is possible to experimentally evaluate the ratios between the numerical concentrations of the components of a mixture in a formulation, as shown in Figure 12.

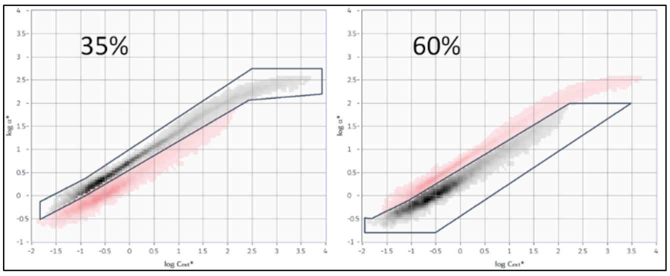

Figure 12 Experimental estimation of the relative number of particles attributable to the individual components of the mixture of ultramarine pigment and the oil emulsions.

The dimensional analysis of the particles in the selected area is naturally carried out allowing to monitor their characteristics and stability in complex systems.

CONCLUSIONS

The capability of EOS Classizer™ ONE and SPES patented method fits the need of a value-added application in the characterization of Inks, watercolours, and oil pigments. SPES data provide physical and statistical information, as PSD and oversize. Each characteristic can be crucial to improve the knowledge and the quality of an formulate even with secondary populations and mixes.

RELEVANT PUBLICATIONS AND REFERENCES

Presentation of Single Particle Extinction and Scattering (SPES) method for particle analysis
AN001-2021 Analysis of Polymeric Particle Mixes via SPES Technology – an introduction to SPES method

AN006-2021 Multiparametric Classification of Particles as a Pathway to Oversize Analysis in Complex Fluids via SPES Technology

Potenza MAC *et al.*, «Measuring the complex field scattered by single submicron particles », AIP Advances 5 (2015)

Example of CFA application of SPES technology
AN002-2021 Continuous SPES Flow Analysis CFA-SPES

Example of PCA application of SPES technology
AN005-2022 Multiparametric Principal Component Analysis of Heterogeneous Samples via SPES Technology

Classizer™ ONE with Sample Manager Autosampler
AN008-2022 Automatic Liquid Sample Management, Dilution, and System Cleaning with EOS Sample Manager

AN009-2022 Standardize SPES Operative Procedure of Liquid Samples Analysis via EOS Autosampler

Example of SPES application to aggregates
AN003-2021 Addressing the Issue of Particle Wetting and Clustering by means of SPES Technology

Potenza MAC *et al.*, «Single-Particle Extinction and Scattering Method …», ACS Earth Space Chem 15 (2017)

SPES application to non-spherical particles
AN004-2021 Addressing the Classification of Non-Spherical Particle by means of SPES Technology

Simonsen MF *et al.*, «Particle shape accounts for instrumental discrepancy in ice …», Clim. Past 14 (2018)

Example of SPES application to emulsions w/, w/o payload in environmental waters
AN012-2021 Monitoring the Fate of a Lipid/ZnO Emulsion in Environmental Waters

AN015-2022 Classification of Oil and Oil Mixes Emulsions via SPES Technology

Examples of SPES application to particle analysis and behaviour characterization in biotech applications
AN011-2021 Quantitative Classification of Particles in Biological Liquids via SPES Technology

Sanvito T *et al.*, «Single particle extinction and scattering optical method unveils in real…", Nanomedicine 13 (2017)

Potenza MAC *et al.*, «Single particle optical extinction and scattering allows real time quantitative…», Sci Rep (2015)

Example of SPES application to oxide particles, abrasives, and industrial slurries w/o impurities
Potenza MAC *et al.*, KONA Powder and Particle 33 (2016)

AN013-2022 Analysis of Abrasives via SPES Technology

Example of SPES application to inks and, pigments
AN018-2022 SPES Classification of Inks, Watercolours, And Pigments

Example of SPES application to ecotoxicity analysis
Maiorana S *et al.*, «Phytotoxicity of wear debris from traditional and innovative brake pads», Env Int., 123 (2019)

Example of SPES application to aerosol analysis
Mariani F *et al.*, «Single Particle Extinction and Scattering allows novel optical …», J Nanopart Res 19 (2017)

Cremonesi L *et al.*, «Multiparametric optical characterization of airborne dust …. », Env Int 123 (2019)

Visit EOS website for further applications

www.eosinstruments.com

EOS S.r.l. – viale Ortles 22/4, 20139 Milano (Mi) – Italy
email: info@eosinstruments.com Phone: +39 02 56660179

Distributed by