ANALYSIS OF ABRASIVES VIA SPES TECHNOLOGY

INTRODUCTION

Abrasives are natural or artificial substances of great hardness used in mechanical processing. The characteristics that distinguish an abrasive are high hardness, very low brittleness, and crystalline nature. They have countless uses for countless materials, depending on which support changes, some applications are sharpening, cutting, abrasive soaps, abrasive pastes. The best-known natural abrasives are quartz, corundum, silica, pumice, sandstone, diamond, emery, diatomaceous earth, garnet. Among the artificial ones there are the oxides of aluminum, chromium, iron, silicon carbide, glass, boron carbide. The use of abrasives can be done in the form of powder; applied to sheets of paper or canvas or sintered to form abrasive wheels or stones.

Modern industries and research centers rely on scientific instrumentation as particle analyzers to characterize and improve abrasive formulations and perform product quality control. Two factors must be considered when looking at the particle size distribution: larger particles provide high impact energy, while smaller ones have more impact per unit area. So, it is important to calibrate the size of the particles to suit the purpose for which they will be used. The more data you retrieve, the better the quality of formulation of the abrasive is. EOS Classizer™ ONE and SPES technology support the task in several different ways.

In this application note the novel classification and characterization of oxide particles in liquids via patented SPES technology and EOS Classizer™ ONE is presented for key abrasive materials and commercial products.

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

Examples of abrasives covered in this document are:

1. SiO_2 particles
2. SiO_2 powder
3. CeO_2 slurry
4. High quality diamond paste
5. Commercial polishing Al_2O_3 and clay pastes

1) SiO_2 particles

Commercial silica particles are commonly available on the market. In Figure 1 a 2D SPES histogram (EOS CLOUDS) of a mix of monodisperse 0.3 µm and 2.0 µm spheres is presented. Two separate clouds appear and may be analysed independently. The one in the lower left corner is the 0.3 µm sample, the one on the right is the 2 µm sample. The measured numerical concentrations are 1.4×10^6 ptc/mL for the 0.3 µm sample and 1.3×10^5 ptc/mL for the 2 µm sample.

The EOS Classizer™ software estimates for the 0.3 µm sample estimates a RI of 1.47 and an average diameter of 0.35 µm, while for the 2 µm sample a RI of 1.45 and an average diameter of 1.9 µm. Both results agree within experimental errors with values expected.

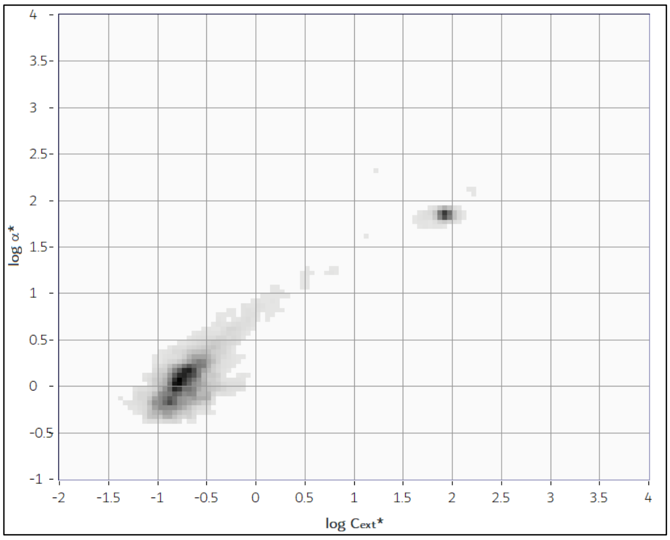

Figure 1 EOS CLOUDS of a mixture of silica spherical particles.

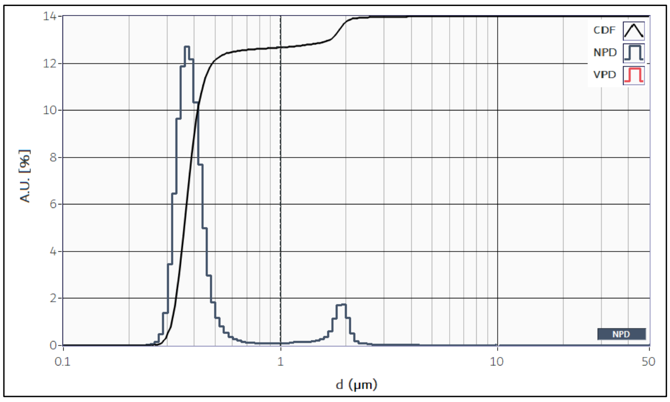

Figure 2 PSD of the silica samples in Figure 1.

2) SiO2 powder

One of the most common uses of the silica powders is as abrasive, due to its round and angular varieties. As an example, mineral silica is commonly used for industrial blasting where reclaiming is not feasible, such as in unconfined abrasive blasting operations. In A commercial powder of silica in suspended in a water-based solution of 0.01% SDS, then sonicated for five minutes and measured.

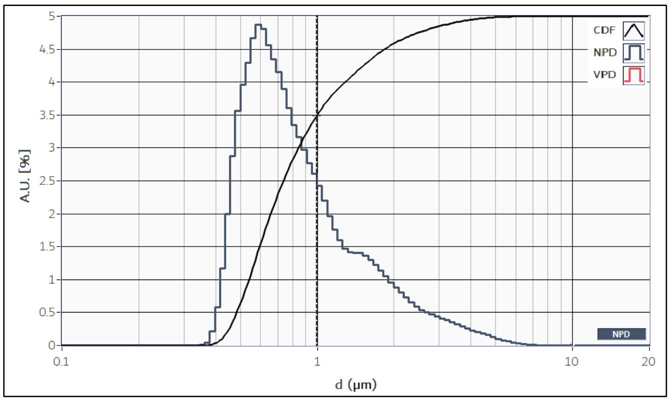

Figure 3 PSD of polydisperse silica powder suspended in water.

The EOS Classizer™ software estimates an AVG diameter of 1.0 µm, a D50 of 0.74 µm, and a D[4,3] of 3.4 µm.

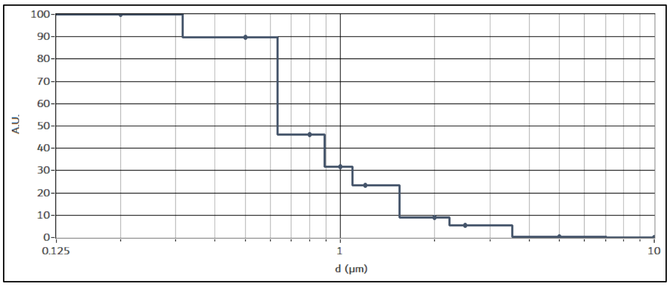

Figure 4 Oversize analysis of polydisperse silica powder suspended in water.

The oversize detection finds that 46% of the numerical concentration is above 0.8 µm, while the 9% is more than 2 µm and less than 0.5% is more than 5 µm.

3) CeO2 slurry

Cerium oxide (CeO2), called also ceria, is an abrasive with a 8.5 MOHS and largely employed for the chemical-mechanical polishing (CMP) of silicon surfaces. Premium cerium oxide required by most demanding nanoelectronics applications needs a precise submicron size distribution. It is demanding to monitor the oversize particles to prevent scratches and failures in the quality of the production.

An aliquot of few microliters of a commercial CeO2 slurry for silicon surface CMP is properly dispersed in filtered water. SPES experimental data are presented in Figure 5.

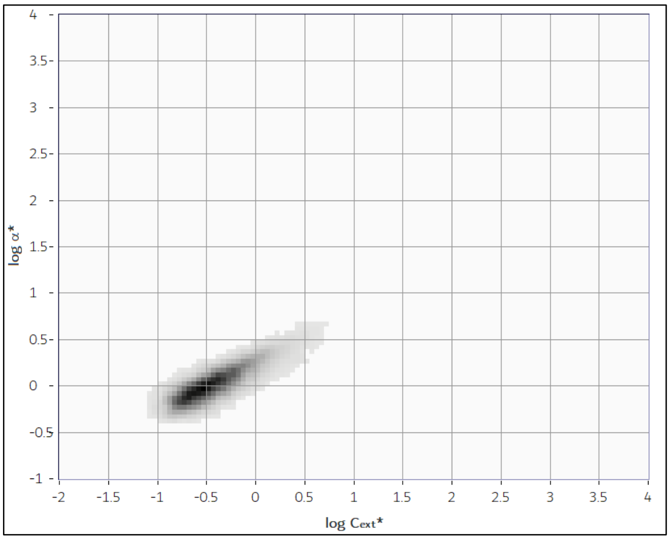

Figure 5 EOS CLOUDS of a CeO2 suspensions.

The EOS Classizer™ software retrieves a refractive index of 2.0±0.4, in agreement with the theoretical value of 2.2@λ640nm. In Figure 6 and in Figure 7 the Particle Size Distribution and the Oversize analysis are presented.

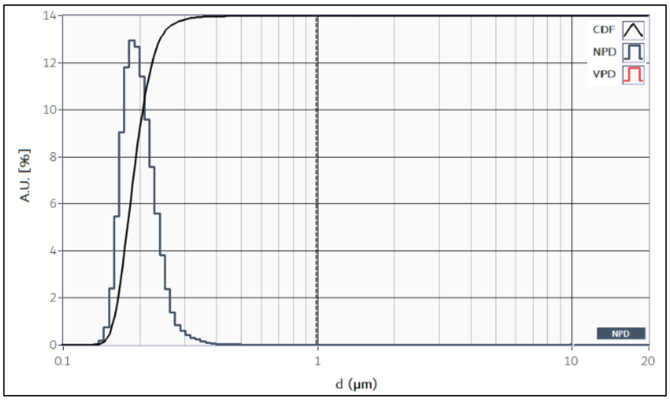

Figure 6 PSD of the powder presented in Figure 5.

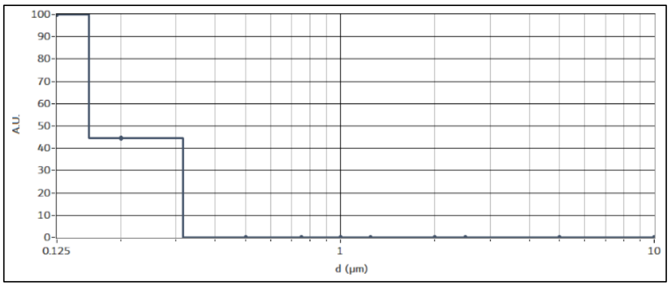

Figure 7 Oversize analysis of PSD plotted in Figure 3 is presented. In this case about the 45% of the numerical concentration correspond to particle larger than 0.2µm and less than 0.1% larger than 0.5µm.

4) High-quality Diamond paste

A diamond is forever. Indeed, with a MOHS value of 10, diamond is one of the hardest minerals known to man. Due to this peculiarity has been widely used in jewelry, because in can be scratched only by other diamonds and a few other materials. Synthetic diamonds cover high-quality cutters, chainsaws, drillers, but also polishing and lapping machines. The main advantage over traditional lapping films based on conventional abrasives is that diamond has a higher performance in cutting and durability.

In Figure 8 the result of a synthetic diamond powder is presented. The sample is dispersed in milliQ-grade water and sonicated for three minutes. The SPES measure via EOS Classizer™ ONE lasted just one minute at 5ccm.

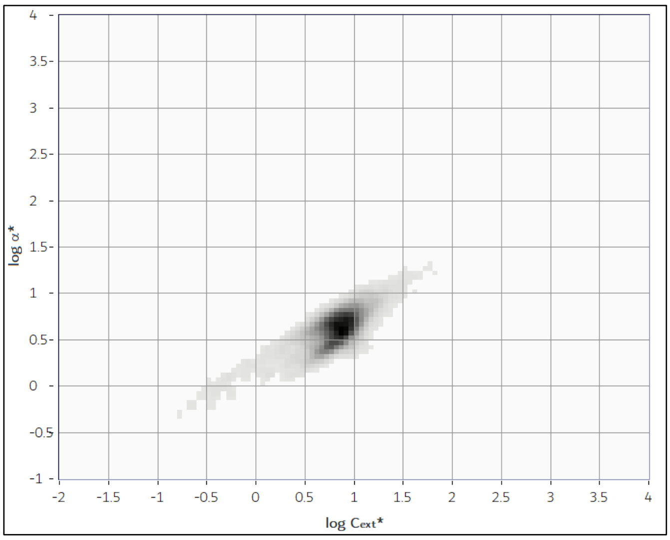

Figure 8 EOS CLOUDS of a commercial high quality diamond lapping powder dispersed in water.

The EOS software determines a refractive index (RI) of 2.4±0.4, in agreement with theoretical data of 2.41@λ640nm. From particle size distribution presented in Figure *9*, the sample is interpreted as almost monodisperse with at about half of a micron.

NUM D_avg (µm)	0.49
NUM D10 (µm)	0.32
NUM D50 (µm)	0.47
NUM D90 (µm)	0.64

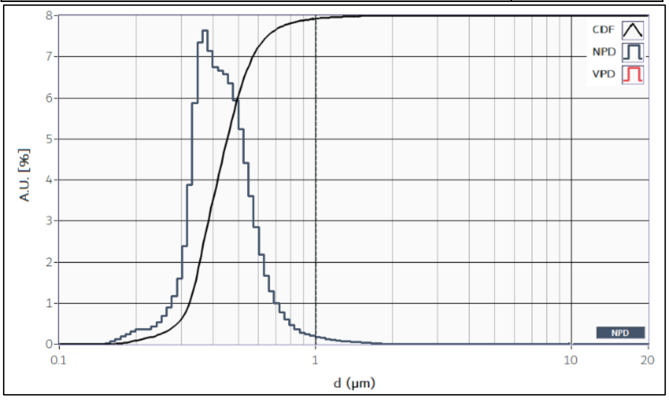

Figure 9 PSD of the powder presented in Figure 8.

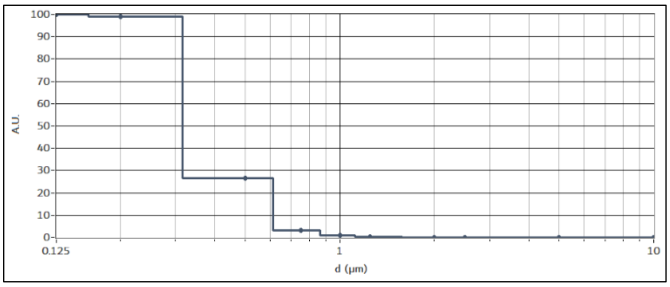

Figure 10 Oversize analysis of a diamond abrasive powder.

In Figure *10* the oversize detection of PSD in Figure 9 is presented. About the 1.2% of the numerical concentration corresponds to particle larger than 1µm. Particle larger than 2 µm are statistically negligible (< 0.01%).

5) Commercial polishing pastes

Analysis results of a couple of commercial polishing paste for metals, including stainless steel and aluminum, are presented. Apart from chemicals in the formulation, the solid part is aluminum oxide, a good quality abrasive with a value of 9 in MOHS scale. Commonly called alumina, it has a refractive index of 1.77 and a non-spherical shape in the Classizer™ size range.

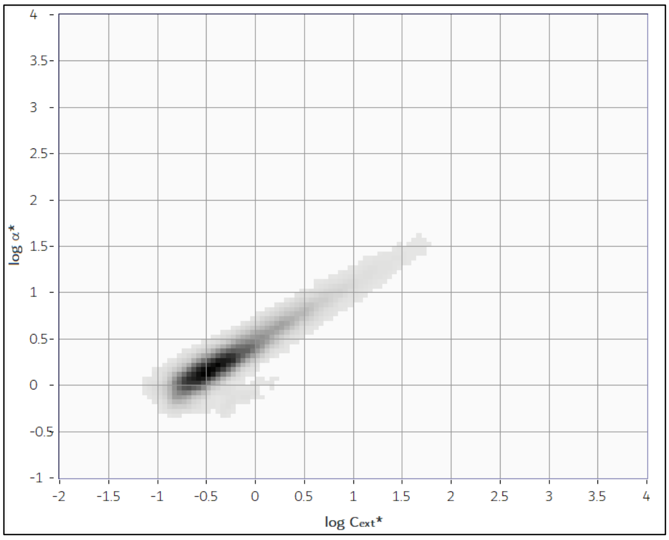

Figure 11 EOS CLOUDS of a commercial polishing paste based on Al_2O_3.

EOS CLOUDS of this alumina-based product indicate a main population with a refractive index of 1.59±0.12 (Figure 11). The discrepancy between the expected value and the experimental effective one can be due to the non-spherical form factor or to the aggregation state of particles. Carefully analysis of the formulation is needed to determine if the engineering of the particles, eg coating, affects the particles structure and particle optical properties. PSD of this material is presented in Figure 12.

NUM D_avg (μm)	0.39
NUM D10 (μm)	0.28
NUM D50 (μm)	0.34
NUM D90 (μm)	0.51

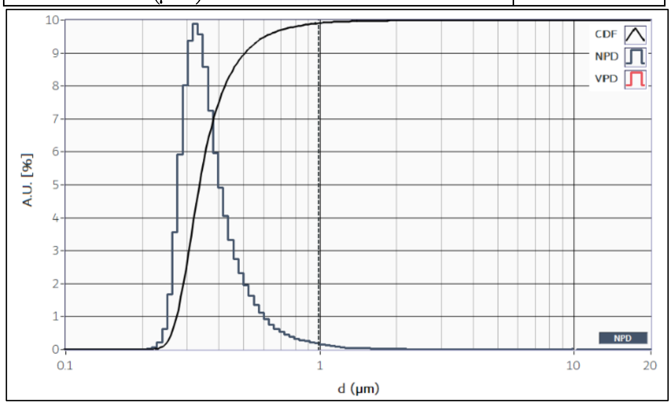

Figure 12 PSD of the powder presented in Figure 11.

A second commercial polishing paste of the same brand is made for gold and silver polishing. Because of their value and softness respect to aluminum, the main ingredient of this product is kaolinite, which has a very low MOHS value of 2. Kaolinite is a clay mineral, often used in cosmetics, paper coating, fertilizers as well as in gentle polishing products. Kaolinite has a refractive index of 1.56, and the particles formed by kaolinite mineral are hexagonal in shape, which typically changes the optical scattering properties. They usually aggregate as well. In Figure *13* the result of the analysis of a polishing paste based on kaolinite is presented. The effective refractive index determined 1.44 compatible with a non-spherical shape and / or an aggregation state of these particles. Based on Mean-field method approximation, considering the latter case, aggregates with filling factor of 40-50% are estimate.

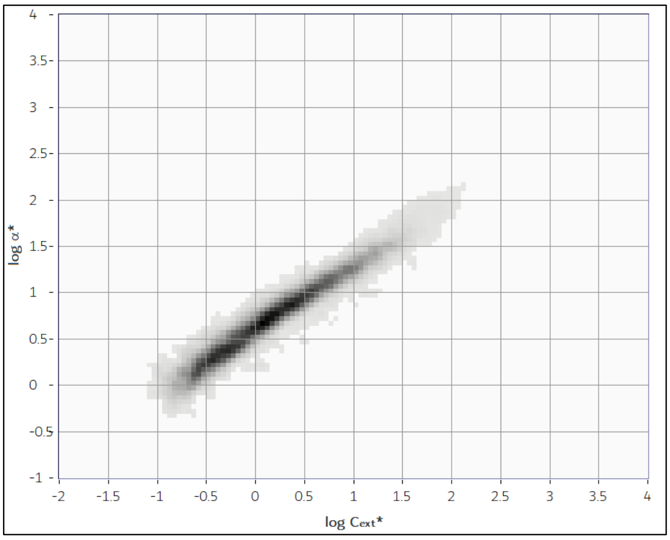

Figure 13 EOS CLOUDS of a commercial polishing paste based on kaolinite.

The EOS CLOUD is different respect to the previous ones. It is elongated on the diagonal meaning a wider particle size distribution, as presented in Figure 14.

NUM D_avg (μm)	0.76
NUM D10 (μm)	0.45
NUM D50 (μm)	0.66
NUM D90 (μm)	1.1

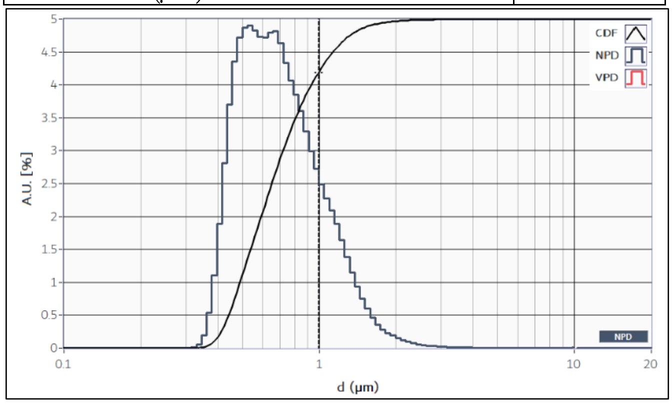

Figure 14 PSD of the powder presented in Figure 13.

CONCLUSIONS

The capability of EOS Classizer™ ONE and SPES patented method in discriminating single particle basing on their optical properties is of capital importance with abrasives and slurries. SPES data provide physical and statistical information, as particle size distribution, effective refractive index, an estimate on the compactness, the state of aggregation of particles as well as the oversized or the average filling factor. Each of these characteristics it can be crucial to improve the knowledge and the quality of a commercial product.

RELEVANT PUBLICATIONS AND REFERENCES

Presentation of Single Particle Extinction and Scattering (SPES) method for particle analysis
AN001-2021 Analysis of Polymeric Particles via SPES Technology – a general 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/o payload in environmental waters
AN012-2021 Monitoring the Fate of a Lipid/ZnO Emulsion in Environmental Waters

Examples of SPES application to particle analysis and behavior 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.*, «Optical characterization of particles for industries», KONA Powder and Particle 33 (2016)

AN013-2022 Analysis of Abrasives via SPES Technology

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)

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