Multiparametric Analysis of Particles in Toothpastes

INTRODUCTION

Toothpaste is used to clean and maintain the aesthetics and health of teeth. Toothpastes are formulated with a variety of components. The three main ones are abrasives, fluoride, and detergents. Abrasives constitute 10-20% of a typical toothpaste. These insoluble particles are designed to help remove plaque from the teeth. Abrasives include, e.g., particles of aluminum hydroxide (Al(OH)3), calcium carbonate (CaCO3), magnesium carbonate (MgCO3), sodium bicarbonate, calcium hydrogen phosphates, silicas and zeolites, and hydroxyapatite (Ca5(PO4)3OH). The typical particle size range is between 0.25µm and 10µm. Further small particles may be added for the remineralization and capillary sealing of teeth. In this document, the optical classification and analysis of particles in popular toothpastes using Classizer™ ONE and LMS01™ sample manager is presented.

PARTICLE ANALYSIS METHOD

Traditional light scattering methods are widely used in scientific and industrial applications. However, the number of parameters affecting the scattering properties of a given particle is such that the basic measure of the scattering power (or even the extinction) is far from being enough to recover something more than a rough estimate of its size. Traditional approaches become even less reliable when mixtures of particles with different properties are analysed.

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

EOS Classizer™ ONE particle analyzer + LMS01™ - front view

EOS Classizer™ ONE retrieves the particle size distribution, numerical concentration, oversize, optical structure, other insights for each particle population detected. Classizer™ ONE works offline and online/real-time, enabling to verify the consistency of the intermediate and final formulations with target QbD, SbD expectations.

For a general introduction to SPES and EOS Classizer™ ONE, please refer to the Application Note AN001/2021, available online along with other application notes and examples at: www.eosinstruments.com/publications/

APPLICATION EXAMPLES

EOS Classizer™ ONE is exploited to classify 2 types of commercial popular toothpastes of different formulation:

1. Toothpaste with titanium dioxide
2. Toothpaste based on zinc hydroxyapatite

EOS Classizer™ ONE retrieves key indicator of particle populations in toothpastes delivering unique quantitative information on particle population quality as numerical concentration and particle size distribution.

EOS Classizer™ ONE Operative Procedure is:

Refractive Index (RI) – **Automatic SPES classification of effective RI for each single population detected in each sample** – Accurate Particle Size Distributions and other information retrieved accordingly to measured RI.
An aliquot of toothpaste is dispersed in 20mL milliQ H_2O
Sample flow: measured at 4ccm / Stirrer speed: 10%
Sample run time: 10min (internal checks comprised)
Background concentration: <10^4 ptc/mL
Target sample concentration: 10^6 ptc/mL
During each run, thousands of particles are measured, yielding an appropriate statistic for the data interpretations discussed below. Each sample was analysed in triplicate.

1) Toothpaste with titanium dioxide and hydrated silica

In Figure 1 the EOS CLOUDS of the first toothpaste is shown. Classizer™ ONE classifies two main populations.

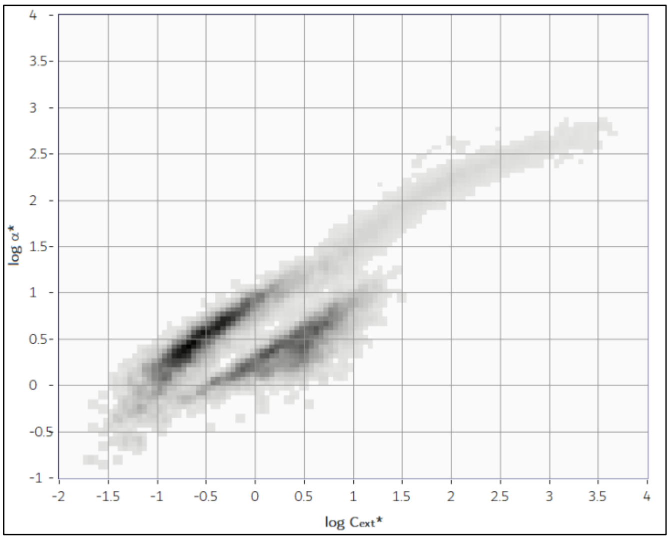

Figure 1 Two clouds in the EOS CLOUDS indicate a formulation based on a mixture of two particle population.

Of all the ingredients present in this toothpaste, the main insoluble ones are hydrated silica and titanium dioxide. Selecting the lower population as represented in Figure 2, the user retrieves the effective refractive index of these particles. EOS Classizer™ software automatically estimates an experimental RI of 2.4, in agreement with the expected value for titanium dioxide (RI of 2.4-2.6).

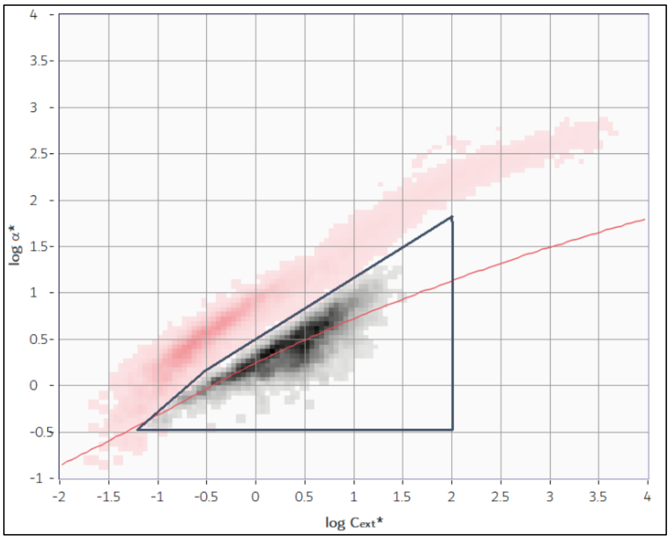

Figure 2 The selection of the titanium dioxide population.

PSD of the selected particles has a peak at 0.22µm and AVG of 0.32µm. This selection corresponds to the 40% of the numerical concentration of whole data measured.

On the other hand, the upper population is estimated to have the lower effective refractive index (Figure 3) of 1.38. Hydrated silica has an expected bulk refractive index of about 1.45 when compact spherical particles are considered. Based on rough preliminary analysis, the experimental value suggests that these particles are non-compact (mesoporous), non-spherical, and/or aggregated.

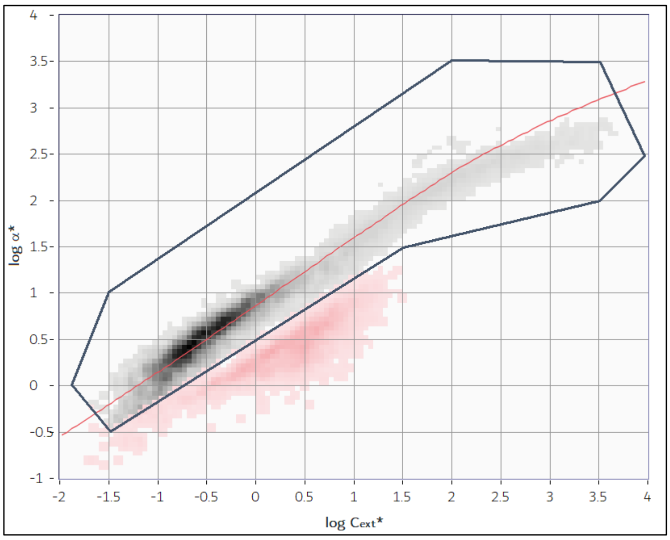

Figure 3 The selection of the hydrated silica population.

Experimentally determined the refractive index that best describes this population, the particle size distribution from the analyses of the single selected particles is retrieved. The numerical PSD shows a peak at 0.65µm, the AVG value of 1.1µm, and the D90 at 2.0µm. Considering the volumetric PSD, the D[4,3] is 8.2µm.

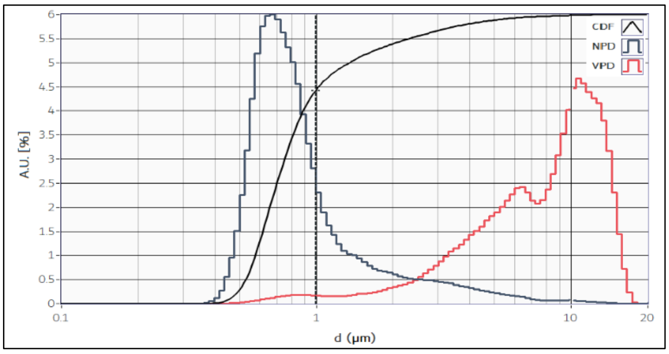

Figure 4 Numerical (blue) and volumetric (red) PSDs of Silica particles; (black) cumulative distribution of the numerical PSD.

2) Toothpaste based on zinc hydroxyapatite

In Figure 1 the EOS CLOUD of the second toothpaste shows a single principal population. In this case the toothpaste is based on a formulation comprising only zinc hydroxyapatite as suspended particles. In **Errore. L'origine riferimento non è stata trovata.** the numerical PSD and its cumulative distribution.

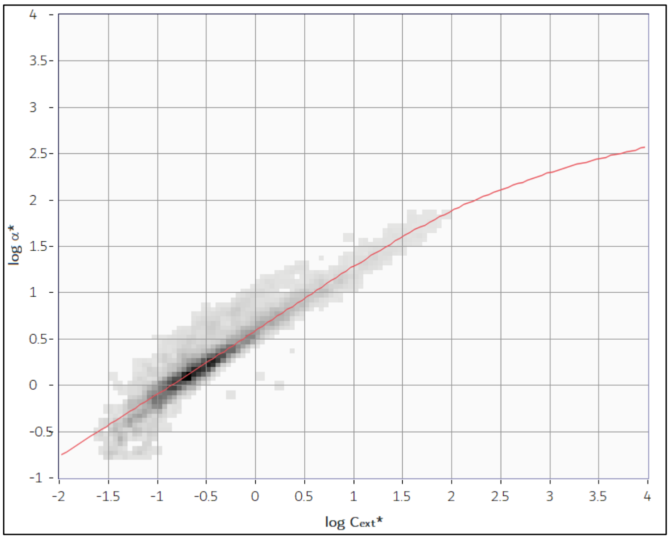

Figure 5 EOS CLOUDS of the second toothpaste.

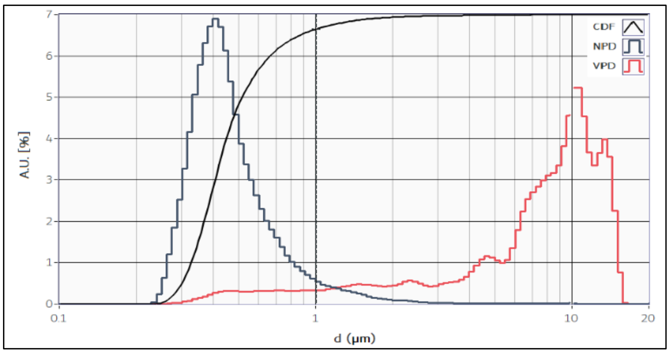

Figure 6 Numerical (blue) and volumetric (red) PSDs of particles; (black) cumulative distribution of the numerical PSD.

CONCLUSIONS

Classizer™ ONE fits the characterization needs as required for oral care and consumer products. Valuable added value is present when the formulates are based on mixtures of particles. In these cases, Classizer™ ONE provides a unique quantitative information on each single population that is not available to traditional technologies for particle sizing.

The numerical PSD shows a peak at 0.65µm, the AVG value of 1.1µm, and the D90 at 2.0µm. Considering the volumetric PSD, the D[4,3] is 8.2µm.

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 Batch-To-Batch Consistency Via Multiparametric SPES Principal Component Analysis PCA

Classizer™ ONE + Sample Managers & Autosampler
AN008-2022 Automatic Liquid Sample Management and System Cleaning with EOS LMS01™ and LMA01™

AN009-2022 Standardize SPES Operative Procedure and improve throughput of Liquid Samples via EOS LAS01™

Example of SPES application to aggregates
AN003-2021 Addressing the Issue of 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 Particles by Mean of the 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 food and 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

AN021-2023 SPES Classification of Mayonnaise

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

AN016-2021 Multiparametric Determination of Yeast Cell Viability via SPES Technology

Sanvito T et al., Nanomedicine 13, Issue 8, 2597-2603 (2017)

Examples of SPES application to inks and pigments
AN018-2022 Classification of Inks and Pigments via SPES

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

AN010-2023 Multiparametric Optical Characterization of Airborne Particles via Patented SPES/SPES² Technologies

Visit EOS website for further applications
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