Nanoparticle distribution in concentrated transparent and opaque dispersions

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The Next STEP® in Dispersion Analysis and Materials Testing

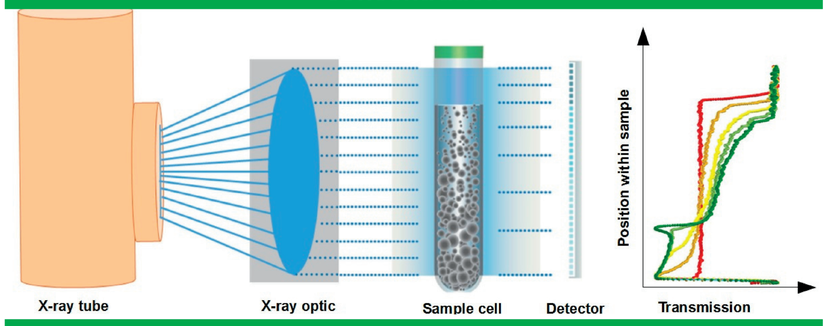
Introduction

Highly concentrated dispersion of nanoparticles are increasingly getting practical importance (e.g. energy storage media, functionalized inks and coatings etc.)
These products have to be analyzed during design, processing and in quality control.
Overmost analytical techniques need sample dilution, which change the dispersion behavior, like rheology, phase separation, flocculation etc.
A new technique is presented to analyse separation behavior, consolidation without dilution and to characterize sediment/cake structures.

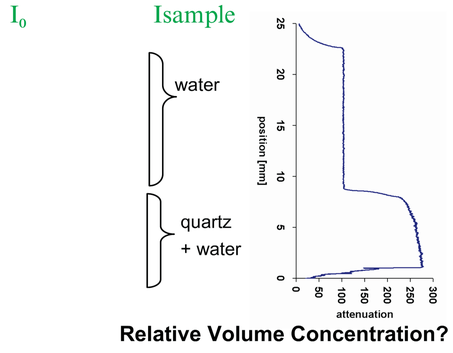
Experimental

For easy and reliable characterization of dispersions the STEP®-Technology (Space and Time resolved Extinction Profiles) was developed, which measures concentration changes inside the samples by high resolution photometric detection. The measurement setup contains a typical Molybdenum X-ray tube. A HOPG (highly ordered pyrolytic graphite) is used to monochromize the X-rays. The attenuation through the sample is measured as function of the local position (see figure below). Knowing the attenuation coefficient of the dispersed and continuous phase the concentration of the dispersed phase can be calculated. These calculations in contrast to optical techniques do not depend on particle size.

Space and time resolved determination of particle concentration



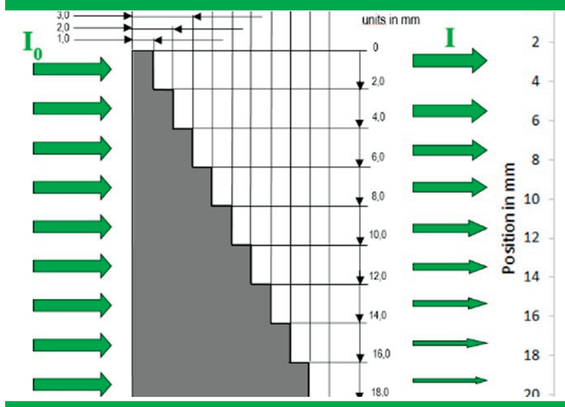
X-ray attenuation measurement

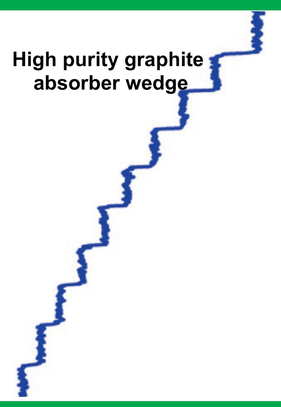


Attenuation:

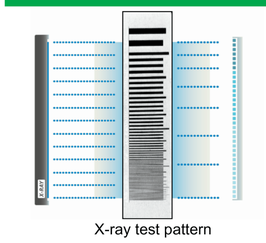
Relative Volume Concentration?

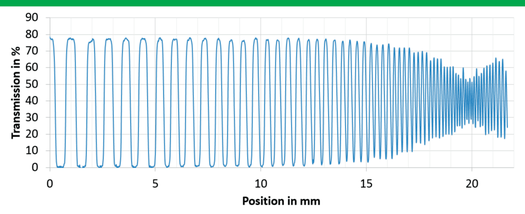
Contrast resolution

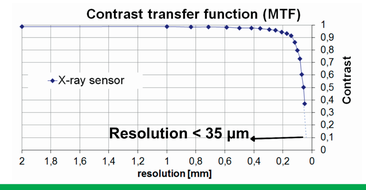




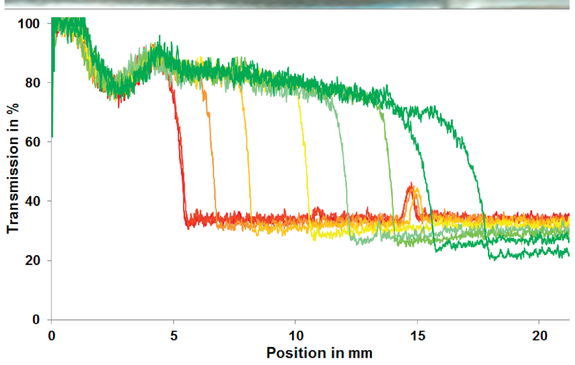
Space resolution

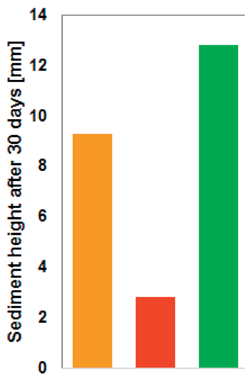






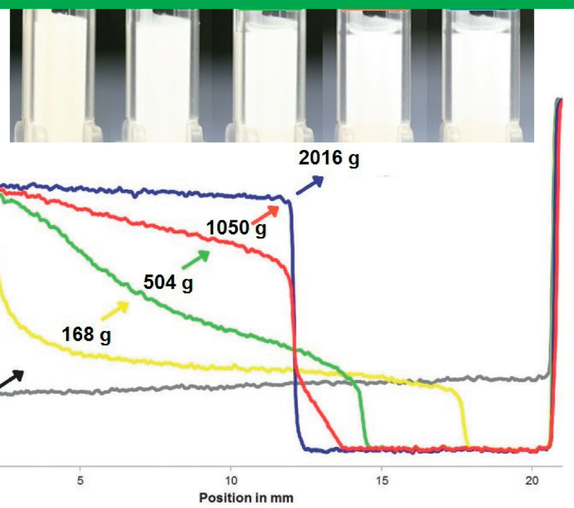
Phase separation of adhesives with nano fillers



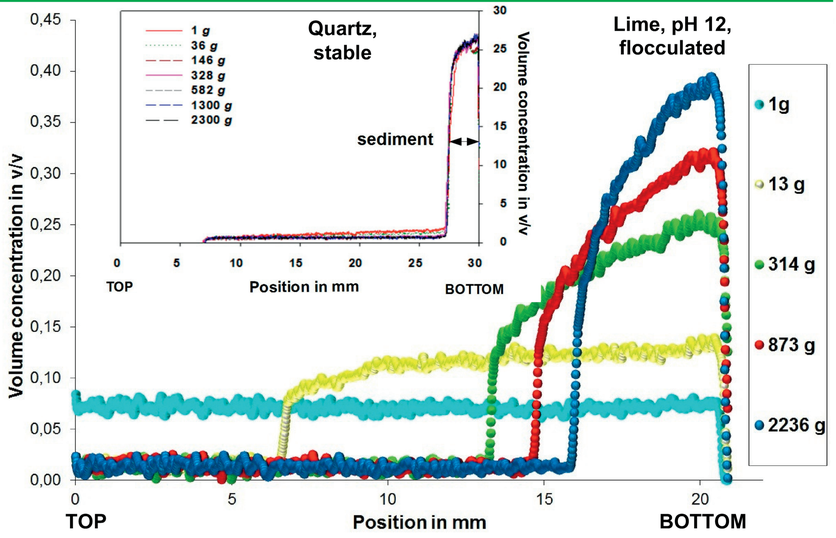


X-ray profiles allow to trace in real-time kinetics of invisible processes. Separation of a filler inside an adhesive matrix (earth gravity). Right comparison of 3 adhesive formulations.

Opaque dispersions and sediments



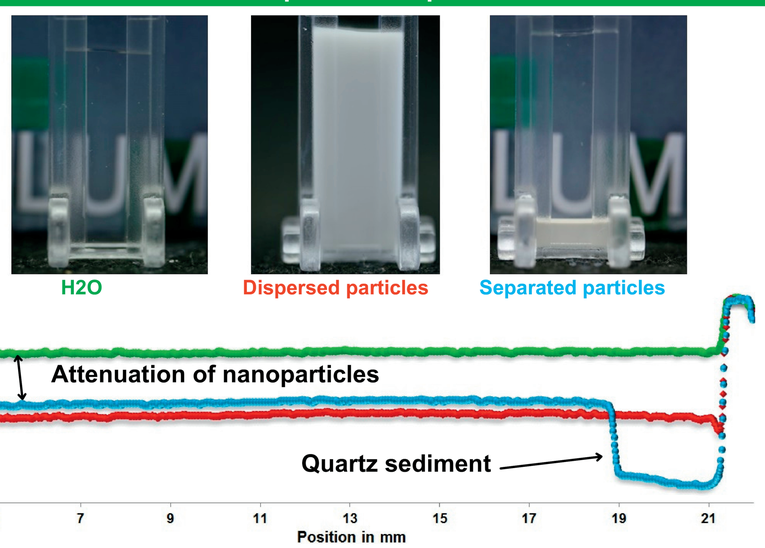
Concentration profiling of filler dispersed in polymer (61% m/m) with LUMiReader X-Ray after accelerated separation for 60 min at 25°C, RCA = 168 *g* to 2016 *g*



Sediment packing profiling by X-ray after consolidation (different centrifugal forces and particle interaction)

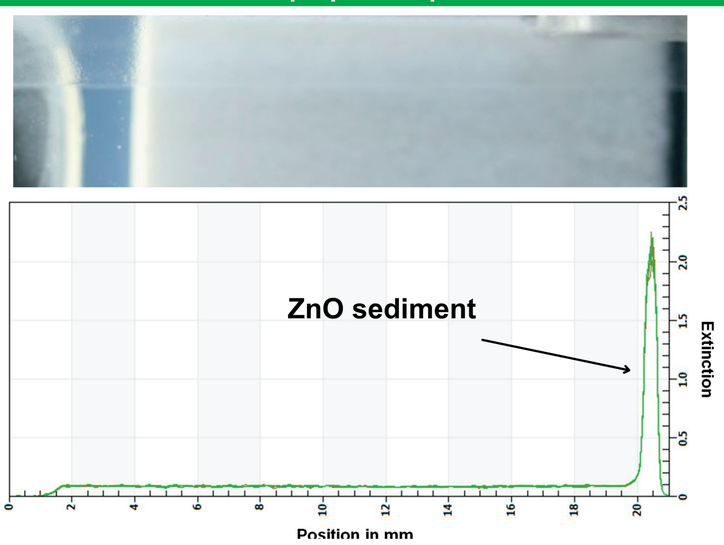
Nanoparticle detection

Transparent samples



Detection of nanoparticles in supernatant of suspension consisting of 10 % Quartz + 30 % w/w Koestrosol® (15 nm) in water after accelerated separation (10 min at 25°C, RCA = 2300 *g*)

Opaque sample



Pigment (ZnO) segregation within opaque sun protection cream: Accelerated separation for 120 min at RCA = 2300 *g* (LUMiSizer) and attenuation profiling (LUMiReader X-Ray)

Conclusion

- In-situ analysis from transparent to very opaque dispersions
- No dilution of emulsions or suspensions
- Signal does not depend on particle size and shape
- High resolution of phase separation of multicomponent systems
- Determination of concentration gradients within dispersions and sediments, mean and space resolved packing densities
- Applicable to stability, separation, segregation and consolidation phenomena
- Real-time, non-invasive and non destructive
- Consolidation analysis in combination with LUMiFuge/ LUMiSizer

Acknowledgement *We thank Bundesministerium für Wirtschaft und Technologie for financial support (INNOWATT IW082075)*