ESRF	**Experiment title:** Investigation on structure and size of nanoscale agglomerates in the process of dispersion in liquids	**Experiment number:** ME-1179
Beamline: ID 02	**Date of experiment:** from: 14/09/05 16:00 to: 17/09/05 16:00	**Date of report:** 01/02/06
Shifts: 9	**Local contact(s):** Peter Boesecke	*Received at ESRF:*

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Report:

Introduction

Many nanomaterials rely on a homogenous dispersion of nanoscale particles in a polymeric matrix, aqueous solution or solvent [1]. Most of the anorganic nanoscale particles in industrial applications are synthesized by pyrogenic or other gas phase processes. Such particles tend to agglomerate during synthesis or during the dispersion process [2]. The agglomeration can lead to soft and hard agglomerates. Hard agglomerates can be characterized by sinter necks between the primary particles. In soft agglomerates no sintering occurs [3]. For functionalized or smart materials even the agglomerate structure plays a major role. Yet the microprocess of dispersing nanoscale particles has not been thoroughly studied and understood.

Previous studies include the development of a high pressure homogenizer which is capable of processing highly abrasive suspensions [4,5]. Using this apparatus which ensures good reproducibility, we studied the dispersion of industrial grade fumed silica particles [5]. These agglomerates consist of partially sintered primary particles. Therefore the analysis concentrated on the dispersibility leading to smaller agglomerate sizes as well as stabilization of the nanoscale suspension. It has been shown that dispersion is possible by high specific energy input down to agglomerate sizes of 150nm in the case of Aerosil 200 from Degussa AG. The hard agglomerates can be stabilized by electrostatic repulsion leading to low tendency of reagglomeration within a time period of a week. Stabilization can be further improved using complex building ions [5,6]. A numerical model for high pressure dispersion using computational fluid dynamics has been set up for further assessments [7].

In coorporation with the group of Prof. Pratsinis we synthesized nanoscale particles at different parameters of production [5], which result in different degrees of agglomeration. The study of the degree of agglomeration during gas phase synthesis has been conducted in former studies at the ESRF facilities using USAXS [8, 9, 10]. This experiment at the ESRF aimed to characterize the state of dispersion and dispersion process depending on the state of agglomeration induced by the production parameters.

Experimental

The silica suspensions for the experiments were prepared by high pressure dispersion and stabilized in Karlsruhe. We used the high brilliance beamline ID 02 using the pinhole SAXS and Bonse-Hart camera. Assembled scattering curves include primary particle scattering at high q's as well as agglomerate scattering within the Guinier regime at low q's with agglomerate radii up to 150 nm.

Background scattering of capillary sample holder and water was subtracted from the scattering curves and those were normalized with NIST data for the electron density of water. Data from both experimental setups were merged by normalizing the Bonse-Hart data to pinhole data for overlapping q-values. The assembled scattering curves were analyzed calculating the Porod constant, scattering invariant and fitting the Guinier plot for the agglomerates obtaining their radii of gyration.

Results and discussion

The specific surface area obtained coincides with BET- and SAXS-data (from experiment ME-628) obtained for powders before liquid dispersion [4]. An increase in the specific surface area due to high pressure dispersion is not visible. Results from different degrees of agglomeration and calcined agglomerates prove the break-up of sintering bonds at high stresses [4]. Since the surface area does not increase within experimental accuracy these sinter necks must be very small sized.

A fractal structure of the agglomerates is described by their fractal dimension. The fractal dimension of the commercially availible silica ranges from 2.0 to 2.2 indicating a diffusion limited aggregation whereas the silica particles synthesized for this study are partly non-fractal.

Further analysis of the data will include the unified fit model proposed by Beaucage [2]. The results are partly included in [4] and will be published in an additional reviewed paper.

References

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