Sample Preparation Methods

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Preventing aggregation of nanoparticles during the drying procedure for TEM sample preparation

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Nanomaterials have been promised a great future in various fields of applications. Because their physical and chemical properties depend very much on their size and shape, their characterization is of utmost importance. When nanomaterials are dispersed in a fluid an in-situ observation technique such as dynamic light scattering (DLS) is often used to probe the dispersion state of colloids. This technique is very precise in determining mean particle size and its distribution as long as it remains monomodal. However, when multimodal distributions evolve, for example, induced by aggregation the quantification becomes challenging due to the assumptions made in the applied models.

Alternatively, transmission electron microscopic (TEM) analysis can provide model-free data. However, in order to obtain TEM samples of nanoparticles in a fluid, the solvent (mostly water) must be removed. Drying effects will take place, which leads to the accumulation and aggregation and hence introduces bias or artifacts in the analysis. It is not possible to differentiate nanoparticle aggregates caused by drying effects from aggregates which were formed prior to drying (e.g. due to colloidal instability of the system). Thus, TEM has long been thought to be unsuitable for quantitative investigations of nanoparticles and colloidal aggregates of nanoparticles in liquids. Here, we present a simple protocol that circumvents the drying issue by stabilizing the colloidal system with a protein, namely bovine serum albumin (BSA). TEM samples with various ratios of proteins to nanoparticles have been prepared that resulted in different spatial distributions of nanomaterials on the TEM grid as shown in Figure 1. When a sufficient high protein concentration was used, the resulting TEM images allow for automated data collection on a large number of nanoparticles which is in very good agreement with data obtained from dynamic light scattering and thus, the actual dispersion state.

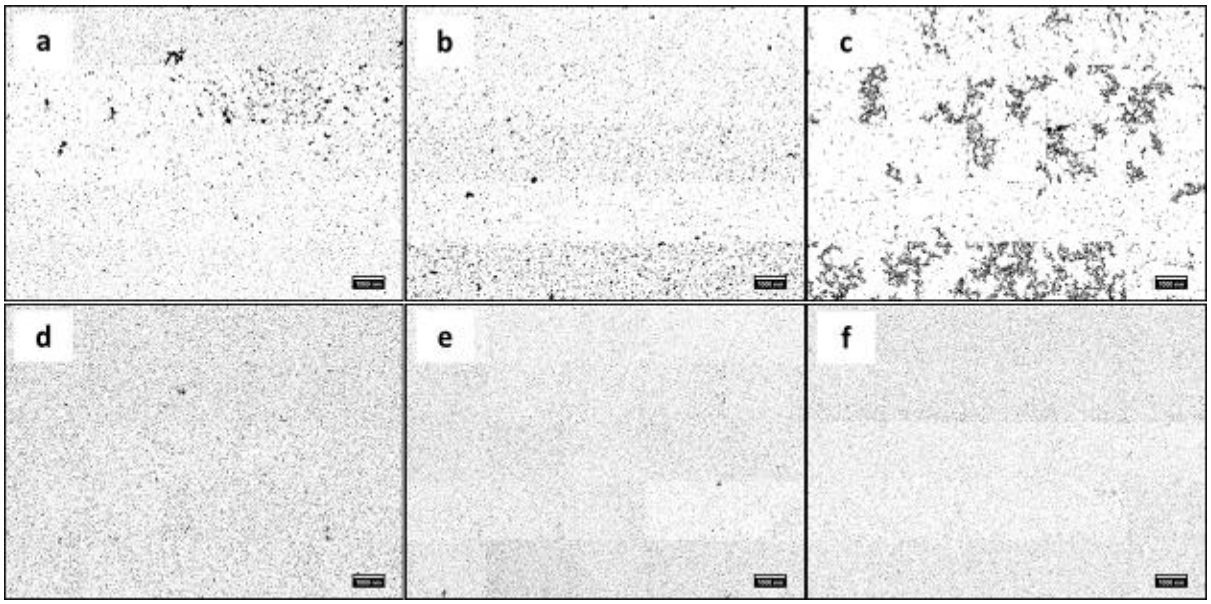

Figure 1. shows TEM mosaic images (each composed of 5x5 single images) of gold nanoparticles prepared at various concentration ratios of BSA to nanoparticles. In a) 25 images of the control sample are presented that do not contain BSA. The concentration ratio is steadily increasing from b) to f). When the control in a) is compared to e) and f) we observe an improved homogeneity in the distribution of the gold nanoparticles indicating that the accumulation of particles due to drying effects was not taking place.