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Probing nanomaterials with X-ray spectroscopy

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Nowadays, nanotechnology is considered one of the most prominent fields of science with numerous potential industrial applications. Its objects of interest are nanomaterials, often referred to as structures, whose behaviour is governed by quantum mechanics. Taking full advantage of nanomaterials' unique properties requires the development of new analytical tools for their qualitative and quantitative characterization. Preferably, such instrumentation should allow examination of samples in a natural environment, for example, in form of liquid suspensions. The utilization of bulk-sensitive and element-specific methods of X-ray spectroscopy allows probing the electronic structure of the nanomaterials, providing information about the chemical state, ligand coordination, and orbital contributions of the specimen. However, analysis of nanomaterial suspensions is significantly hindered by two aspects. Firstly, due to complicated and non-scalable synthetic procedures, it is difficult to obtain sample material in the quantities necessary for optimal measurement. Secondly, nanostructures in suspensions tend to agglomerate into bigger clusters, eventually leading to particle settlement at the bottom of a sample container in a process called sedimentation. Both factors make an examination of nanomaterials in solvents challenging, in particular for experiments requiring hours of data acquisition. To address these issues, we developed a special sample setup, designed specifically to measure low volumes of nanomaterial suspensions with continuous stirring to prevent agglomeration and sedimentation processes.1

The designed setup is based on a polyimide capillary equipped with an electrically-driven spindle that mixes nanomaterial suspensions to maintain its stability during measurements. The systems' performance was verified using ZnO nanoparticles (NPs) suspended in distilled water. A series of simultaneous Zn K-edge X-ray absorption spectroscopy (XAS) and Zn Kα X-ray emission spectroscopy (XES) measurements utilizing laboratory-based X-ray spectrometer2 were conducted to probe the ZnO NPs concentration over 90 min of setup operation.

Acquired XAS and XES data showed a stable signal throughout the entire experiment. On the other hand, in the absence of mechanical stirring, rapid sedimentation of ZnO nanoparticles was observed, occurring a few minutes after the start of the measurements with a maximum rate of 2.5 % of concentration loss in the beam field per minute. Obtained results demonstrated that the developed setup allows the acquisition of high-quality XAS & XES of nanomaterial suspensions. Presented instrumentation could be utilized in future studies of various nanosystems based on 3d metals such as plasmonic nanoparticles or semiconductor quantum dots. Studies of nanostructures could be expanded beyond the standard X-ray spectroscopy methods toward time-dependent measurements at femtosecond time scales to follow charge dynamics induced by ultrashort optical pulses. The possible application of the newly proposed concept of X-ray chronoscopy3 in the studies of nanomaterial systems will also be briefly discussed.

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References

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