

Exposé des geplanten Dissertationsvorhabens

The current advances in nanomaterial development require the use of accurate and traceable characterization techniques, such as Small-angle X-ray Scattering (SAXS). SAXS determines the size distribution and shape of the nanoparticles (NPs) in suspension by collecting X-ray photons elastically scattered off the electrons in the sample, at detection angles below 5° in forward direction. This technique is suitable for the traceable size determination of particles in the range of from about 5 nm to 200 nm and allows the characterization of a NPs with core-shell structures.

The project is based in the investigations of nanoparticles in suspension using the SAXS setup at the four-crystal monochromator beamline of PTB at the synchrotron radiation facility BESSY II. Through the high brilliance and collimation of current synchrotron radiation sources, scattering curves of diluted samples can be recorded with short exposure time and good resolution.

Nevertheless, the inner structure present in many low-density nanoparticles cannot be probed easily with single-contrast SAXS. Therefore, complementary information must be acquired by the method of the solvent contrast variation. In SAXS, the solvent contrast variation technique is achieved by adding a suitable contrast agent to the suspending medium (e.g. sucrose) and recording the scattering data as a function of the adjusted solvent density.

My work develops a novel approach to contrast variation in SAXS based on the constitution of a solvent density gradient in a glass capillary in order to choose *in situ* the most appropriate contrast and to acquire extensive datasets in a short time interval. The measurement of SAXS curves at different capillary heights allows the tuning of the solvent contrast within the provided density range, resulting in a virtually continuous solvent contrast variation.

By examining the scattering curves measured at different aqueous sucrose densities, information about the internal morphology of the nanoparticles as well as their size distribution can be obtained. Additionally an estimation of the particle density can be determined focusing on the Guinier region of the curve. A comparison between the continuous contrast variation approach and other techniques (DCS and imaging methods) can validate the results obtained for polymeric colloids across a wide spectrum of polymers.

The continuous contrast variation technique can also be employed to characterize

relevant materials for the nanomedicine community, such as human lipoproteins or the first approved nanodrug Doxil (R), a PEGylated liposomal formulation of doxorubicin. This study is focused on the isoscattering point position and the model-free analysis of the scattering curves and highlights the advantages in comparison to widely used characterization techniques as DLS and TEM.

A part of the project studies the response of the nanocarriers to increasing solvent osmolality and compares the different response of PEGylated and plain liposomes to osmotic pressure depending on their size. By focusing on the phospholipid bilayer feature observed at large q -values, information about the lamellar structure of the liposomes can be obtained and an insight into the shape variation of the lipid vesicles under osmotic stress can be investigated.