ESRF	**Experiment title:** Shared-solvent Colloid–Polymer Interfaces	**Experiment number**: SC-4688
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Report:

Summary

We have performed for the first time X-ray reflectivity (XRR) experiments on the fluid–fluid interface of phase-separated colloid–polymer mixtures. These interfaces are composed of a single solvent and their fascinating structural properties remain largely unexplored. Our results serve foremost as a proof of concept, showing that it is indeed possible to measure structural interfacial properties of these kinds of systems using XRR. Further, our results indicate that the interfacial width is of the order of the diameter of the colloidal particles. Our experiments pave the way toward systematic structural investigation of these interfaces in the future.

Introduction

In recent years, a lot of attention has been given to the use of XRR for structural investigations of solid–liquid, liquid–air, or oil–water interfaces. However, there are many examples of liquid–liquid interfaces that share the same solvent, which are found for example in food formulations and biology. We have studied a model interface comprising a single solvent, which has never been studied using XRR before and which has largely unexplored structural properties. XRR is a unique technique for the characterization of these interfaces, as it is non-invasive and allows structural characterizations with nanometer resolution.

The system consists of sterically stabilized stearyl-silica particles ($d \approx 29$ nm) mixed with non-adsorbing polymer (PDMS, $R_g \approx 15$ nm), dispersed in cyclohexane. Phase separation into coexisting colloidal liquid and gas phases, sharing the same solvent, is observed due to the so-called depletion interaction [1]. Several theoretical predictions exist regarding the structure of the colloidal gas–liquid interface in such systems and our experiments can be seen as a first test of these predictions.

Setup

Experiments were carried out at ID10-EH1 at a photon energy of 22 keV and a Mythen 1K detector. Due to the small density differences involved, the critical angles are rather small (~0.02°–0.05°). This necessitates the use of rather elongated samples in view of the beam footprint. We therefore used home-made sample cells of varying lengths to find optimal conditions. The cells had lengths of 5.0, 7.5 and 10 cm, a width of 0.5 cm, and a height of 1.0 cm, fitted with kapton windows on the long ends of the sample cell.

Initial results

Foremost our results indicate that it is indeed possible to perform X-ray reflectivity experiments on the colloidal gas–liquid interface with a shared solvent. An example of a measurement showing specular reflection is shown in Fig 1 (left panel, bright vertical 'streak').

Our experiments indicate that a sample cell of 7.5 cm offers a good compromise between transmittance and access to small angles.

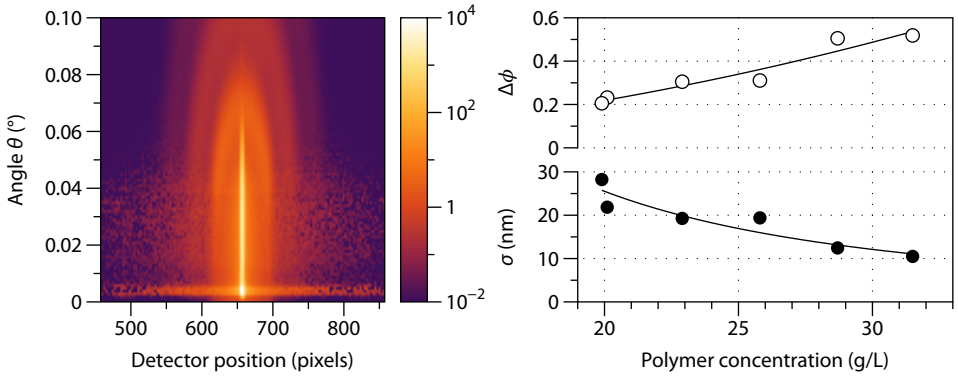

Fig 1. (left) Example of signal of Mythen 1K line detector as a function of the specular angle. (right) Preliminary results on the density difference ($\Delta\phi$) and interfacial thickness (σ) as a function of polymer concentration as obtained from various measurements as those in the left panel.

Preliminary data analysis (Fig 1, right panel) shows that the density difference between the colloidal liquid and gas phase $\Delta\phi$ increases as expected with the polymer concentration. Further the interfacial thickness decreases with increasing polymer concentration. The thickness is of the order of the diameter of the colloidal particles.

References

[1] H. N. W. Lekkerkerker and R. Tuinier, *Colloids and the Depletion Interaction* (Springer, 2011).