Drying Particle-Polymer suspensions
In-situ monitored by Vertical Small Angle X-Ray Scattering

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Drying of colloidal dispersions to create thin solid film is a common process for various applications. When a liquid film solidifies during drying, the concentration not only of particles but also of other components increases and the corresponding particle interaction changes over time. As a result, the structure of the solid film may be different from expectation derived from the original composition of the suspension. Therefore, the drying process needs to be understood to control the structure and properties of the final product. In-situ synchrotron X-ray has proven to be one of the most promising tools

in understanding the non-equilibrium phase behavior of colloids. However, conventional synchrotron beams are horizontal to the ground, thus they are not able to pass perpendicular through the colloidal suspension which intrinsically settles down in a horizontal plane due to gravity. Because of this limitation, scattering has been applied only for a limited number of cases in drying research, for which horizontal SAXS can be applied.

In the present work, we report a first application of novel *vertical* SAXS to investigate the drying process of a colloidal suspension by overcoming gravity related restrictions. We employed a unique rheo-SAXS setup which has been developed to allow a synchrotron X-ray beam to pass vertically through the plate/plate geometry of a rheometer. After demonstrating the methodology with charge-stabilized colloidal silica[1], we explore the drying behavior of silica/polyvinyl alcohol suspension where liquid microstructure is controlled by pH. The scattering intensity during drying shows that non-adsorbed polymer is found to introduce the depletion attraction during drying, which turns the stabilized suspension into porous solid film with particle aggregates. Adsorbed polymer allows the particle in the secondary minimum, which stabilizes the suspension against depletion attraction during drying, leading the suspension to the dense solid film without aggregates. This allows us to successfully understand a questioned drying behavior of the suspension in our previous study[2]

[1] Kim, S.; Hyun, K.; Kim, Y. S.; Struth, B.; Clasen, C.; Ahn, K. H., Drying of a Charge-Stabilized Colloidal Suspension in Situ Monitored by Vertical Small-Angle X-ray Scattering. *Langmuir* **2013,** 29, (32), 10059-10065.
[2] Kim, S.; Sung, J. H.; Ahn, K. H.; Lee, S. J., Drying of the Silica/PVA Suspension: Effect of Suspension Microstructure. *Langmuir* **2009,** 25, (11), 6155-6161.