Drying-SAXS: *in-situ* characterization of structural change during drying of silica suspensions

S. Kim, K. Hyun¹, K. H. Ahn and S.J. Lee

School of Chemical Engineering, Seoul National University, Seoul 151-744, Korea ¹School of Chemical and Biomolecular Engineering, Pusan National University, Jangjeon-Dong 30, Busan 609-735, Korea

Drying of particulate suspension for the film formation is of importance in various applications to produce secondary battery, display, solar cells and so on. Because dispersion stability is a crucial issue in the coating and printing process of ink and paste, many attempts have been carried out to analyze the suspension microstructure and film microstructure respectively, and try to understand the structural change during drying. Even if the solid understanding of drying behavior is important in the colloidal science and many application fields, there are only a few studies on the drying of particulate suspensions. Moreover, the methodologies developed so far were only help us to speculate the structural change and not satisfactory in obtaining the *in-situ* information about structural development during drying.

X-ray scattering is a useful technique to observe the suspension microstructure more directly than any other techniques[1-2]. In order to employ the x-ray scattering to the drying behavior of suspensions, the angle of beamline need to be vertical to ground, unlikely to the conventional SAXS experiment. In 2010, we attempted to observe the structural change during drying of silica suspension using the BW1 in HASYLAB (the schematics for the experiment is described in Figure 1) and successfully obtained the scattering image during drying. To the best of the knowledge of the authors, SAXS study on drying film so called ‘Drying-SAXS’ is conducted for the first time.

Figure 1. Schematics for Drying-SAXS experiment in BW1, HASYLAB. Suspensions for drying are simply loaded on the fixture installed in rheometer. Drying condition is controlled by the chamber which is already installed in the rheometer in BW1.

We conducted the *Drying-SAXS* experiment in BW1 from July 5 to July 7, 2010. The model system in this study is an aqueous dispersion of colloidal silica with initially 30wt% solids, (Ludox HS-30, Aldrich). Particle diameter of silica is 12 nm and it is small enough to measure the scattering image from X-ray [3]. The dispersion stability was controlled by addition of the various amount of NaCl. From the measurement of zeta potential and particle size measurement, the electrostatically stabilized silica suspension loses its stability as the amount of salt increases. Figure 2 is the result of *Drying-SAXS* experiment of silica suspension by changing the dispersion stability. In all measurement, scattering intensity decreases due to increasing salt concentration during drying. However, shape of SAXS spectra sensitively depends on salt concentration. Especially, at 0.2 M
salt concentration (Figure 2d), the sudden decrease of the intensity is observed, probably which is ascribed by the sudden aggregation during drying. Currently, we interpret the result for the clear and meaningful investigation. As we found that the Drying-SAXS in BW1 is very promising method to understand the drying behavior of particulate suspensions, we propose further study about the drying of particulate suspension with systematic parameter.

![SAXS spectra from 30 wt% silica suspension (d=12nm) with various salt concentrations during drying. Salt concentration is (a) 0 M, (b) 0.05M, (c) 0.1M, and (d) 0.2M. The unit of legend is min.](image)

Figure 2. SAXS spectra from 30 wt% silica suspension (d=12nm) with various salt concentrations during drying. Salt concentration is (a) 0 M, (b) 0.05M, (c) 0.1M, and (d) 0.2M. The unit of legend is min.

**References**

