Dispersion stability and drying behavior of colloidal silica

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Introduction

Colloidal suspensions containing nano-sized particles are used in numerous applications such as batteries, inks and paints, and also used as precursors to a wide variety of industrial materials. The ability to control the microstructure during drying is important for many potential applications. When it comes to solar cell technology, controlling the microstructure plays a critical role ranging from thin film solar cell (where no void in the film is desirable) to dye-sensitized solar cell (where void is desired to produce small pores). For instance, Copper-Indium-Selenide solar cell, or more commonly known as CIS solar cell requires smooth surfaces and uniform microstructure. In those applications non-uniform particle aggregation is a critical defect to be avoided. In this sense, drying process of colloidal suspension is a crucial step that often determines final coating quality.

While there have been many studies on liquid states and final film structures of colloidal system, the mechanism of drying process is not fully understood yet. In this study, we investigate drying process of colloidal suspension by examining its microstructure.

Experiment

Ludox is spherical shaped particles and the zeta potential of Ludox is substantially large and negative. By changing zeta potential, we can easily control the dispersion stability of suspension. Therefore Ludox is appropriate as a model system to investigate dispersion stability and drying behavior of colloidal system. Silica particles were supplied by Aldrich (Ludox HS30, Ludox SM) in the form of a 30 wt % electrically stabilized aqueous silica suspension.

The dispersion stability was examined by means of zeta-potential measurement using ELS-8000 (Photal Otsuka Electronics, Japan). The stability of electrostatically stabilized systems is sensitively influenced by variation of the ion concentration. An increase of the ion concentration provokes a decrease of both the Debye screening length and the zeta-potential. As ionic strength of medium is increased, eventually only attractive force is effective due to the absence of an energy barrier between particles. Figure.1 depicts this effect in terms of zeta-potential and different sodium chloride concentrations.

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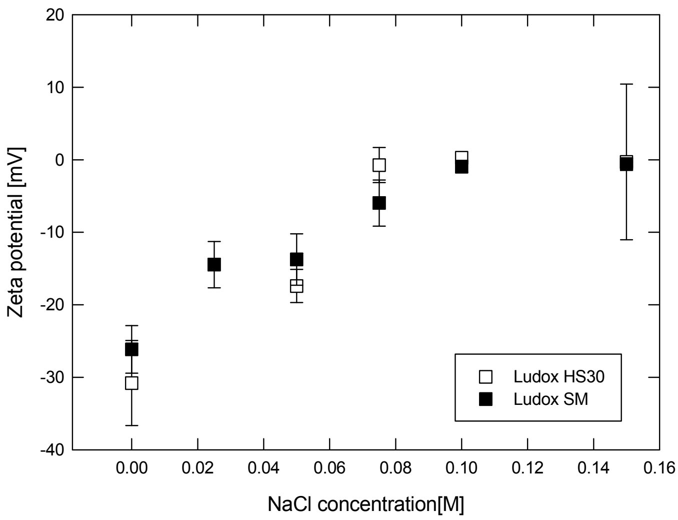

Figure 1. Zeta potential of Ludox HS30 & Ludox SM with different NaCl concentration

Before drying when the particles are far from each other, the dispersion looks much the same. But as drying proceeds, the distance between particles becomes closer and they will go through different drying mechanism. Once particulate film was formed after drying, it showed pronounced differences in crack behavior for the differently stabilized suspensions (Figure 2). Singh *et al.* provided some estimates of the crack behavior of electrostatically stabilized colloidal particles [1]. The amount of crack thickness can be estimated from the differences between the dried packing densities. In this study experimental results are consistent with previous work.

Figure 2. Crack Behavior of Ludox SM with increasing NaCl concentration from 0M to 0.12M

Like the macroscopic crack behavior has observed, the micro-structural differences between films are obtained by FE-SEM (Figure 3). As the ion concentration is increased, non-uniform particle aggregation and surface roughness is observed. Structural differences could be explained by strength of the particle interaction force. Attaining sufficiently low repulsive forces during the drying process lead to rougher and more porous films whereas highly repulsive forces cause dense particle packing.

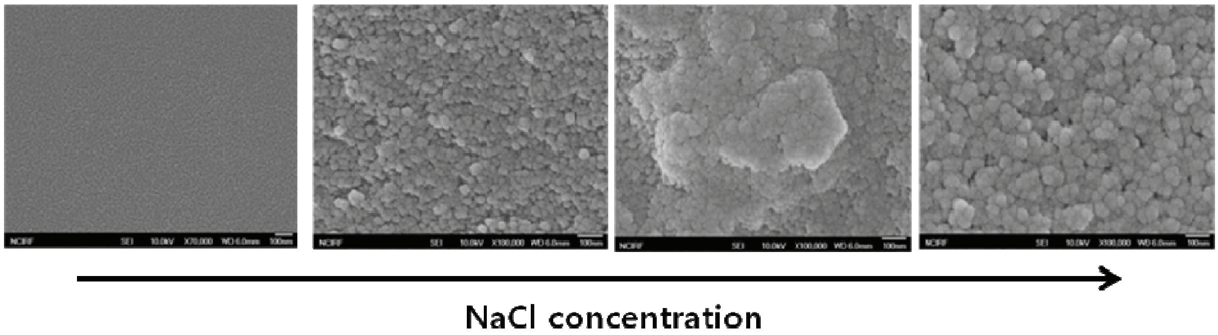

Figure 3. FE-SEM image of Ludox SM with increasing NaCl addition from 0M to 0.12M

In this study characterization of microstructure after drying was mainly done by light scattering method. Light scattering is a very powerful tool to investigate the colloidal system because it gives the structural information of nano-sized colloidal particles. Small angle X-ray scattering gives valuable information such as form factor and structure factor [2]. Form factor provides the information of size and shape of particle, and structure factor gives interactions between particles.

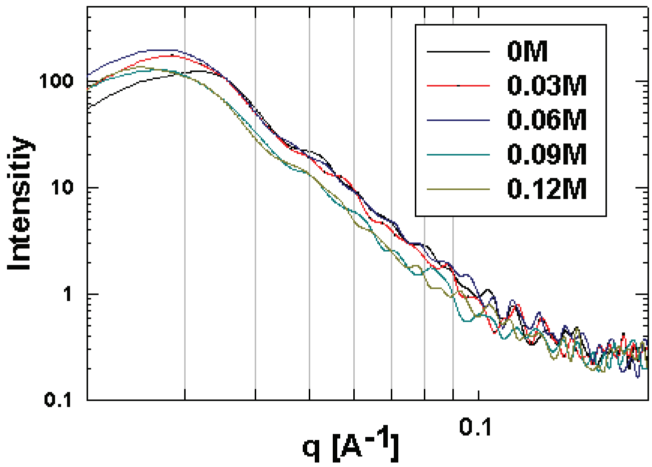

Figure 4. Scattered intensity of Ludox SM as a function of q as a function of q from Ludox SM

Small-angle X-ray scattering measurement were performed using the pinhole SAXS Instrument (NanoSTAR, BrukerAXS). In the present study sample-to-detector distance was kept at 107 centimeters, and small pinholes were used, giving a range of scattering vector as $0.006 < q$ [$Å^{-1}$] < 0.1, where $q = (4\pi/\lambda) \sin(\theta/2)$ is the modulus of the wave vector, θ is the scattering angle, and $\lambda = 1.542$[$Å^{-1}$] is the X-ray wavelength (Figure 4).

The microstructure of dried film is directly accessible by scattering experiments. The structure formation in the drying of silica suspension is strongly influenced by the ion concentration. A plot of maximum q value [$Å^{-1}$] vs. sodium chloride concentration is shown in Figure 5. We observed that maximum q value is decreased as sodium chloride concentration is increased.

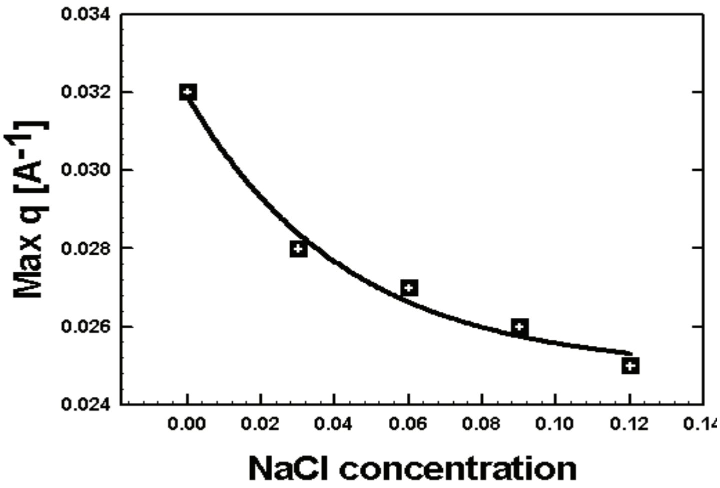

Figure 5. Maximum q value as a function of NaCl concentration

Conclusions

In this study we investigate the dispersion stability and drying behavior of colloidal silica by using various methods. A better understanding of the interrelationship between drying mechanism and film microstructure could expand the number of processing routes for producing materials in energy industry, where the controlling microstructure is the key factor to determine product quality. The ability to describe drying mechanism also would be useful in the other industrial application, where well-defined microstructure and related macroscopic properties are highly demanded.

Acknowledgements

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References

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