

A Study on Formation Mechanism of Mesoporous Silica SBA-15 Platelet with Perpendicular Nanochannels

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The Mesoporous silica SBA-15 platelet with perpendicular nanochannels was prepared by using ternary surfactant system of CTAB-SDS-P123 as the template via sol-gel process. We further study its formation mechanism. To elucidate the microstructures in the CTAB-SDS-P123 surfactant system, we resorted to Small-Angle X-ray Scattering at BL23A of NSRRC. The instrumental settings in beam-time 2009-2 and 2009-3 were used cover the wavelength range $0.0055 \text{ \AA}^{-1} < Q < 0.2853 \text{ \AA}^{-1}$ and $0.0017 \text{ \AA}^{-1} < Q < 0.0887 \text{ \AA}^{-1}$, respectively. Scattering intensity versus scattering angle ($I(Q)$ vs Q) curves were fit to various inter-particle form factor models using the macro functions provided by NIST for use with Igor Pro software.

The scattering curve of P123 solution showed a Porod slope of -1.5 at low Q and a scattering hump around $0.05 \text{ \AA}^{-1}$. It revealed that the P123 spherical micelles with radius $\sim 11 \text{ nm}$ would aggregate as fractal objects. The scattering curve of CTAB-SDS solutions displayed a sharp rise at low Q and a scattering hump around $0.013 \text{ \AA}^{-1}$. At low Q , the sharp rise, a Porod slope between -2 and -3, indicated the presence of large fractal structures. The scattering hump around $0.013 \text{ \AA}^{-1}$ reflected spherical micelles or vesicle with radius $\sim 39.8 \text{ nm}$. In 2009-2 experiment, the scattering curve of CTAB-SDS-P123 solution showed inter-particle interference at low Q , a broad scattering peak around $q \sim 0.08 \text{ \AA}^{-1}$ and the q^{-4} decay at high q . It revealed that larger

aggregates, lamellar phase, and small complex exist in at the ternary surfactant system. The scattering profiles suggested that the P123 spherical micelles would be unwrapped by CTAB-SDS complexes and the mixed ternary surfactant system would appear lamellar phase. In 2009-3 experiment, we performed CTAB-SDS-P123 surfactant system with different P123 content to investigate the P123 copolymer effect in the ternary surfactant template. However, the scattering curves showed poor trend in this experiment. There were two types of SAXS patterns in 2009-3 experiment. One appeared a Porod slope between -2 and -3 at low Q and a scattering hump around $0.09 \text{ \AA}^{-1}$. It revealed that spherical micelles or vesicles with radius $\sim 52.3 \text{ nm}$ aggregated into fractal objects. The other displayed nearly plateau behavior at low Q and smooth decay curve. The complex scattering pattern could be described by polystacking disks with diameter $150\text{-}200 \text{ nm}$ and $\sim 9 \text{ nm}$ thickness or three structural levels with $R_g \sim 70 \text{ nm}$. The latter would be in good correspond to the FF-TEM results.

Based on the SAXS results, we found that the surfactant samples appeared to be unstable or poor micelles dispersion in bulk solution. It suggested that the surfactant system in dynamic equilibrium dispersed inhomogeneously in the bulk solution. Thus, the sampling conditions in SAXS experiment significantly influenced the scattering patterns.