Effect of Salt on Self-assembly of SBA-15 Studied by In Situ Small-angle Synchrotron X-ray Scattering

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Mesoporous silica materials possessing high surface areas and ordered mesoporous structures show potential applications in separation, enzyme immobilization, catalysis, nanocasting and electro-optical devices. Among the mesoporous materials, SBA-15 of tunneling pores in 4-10 nm arranged in 2D-hexagonal *p6mm* structure has received great attention in the past decade because of its relatively large pore size and high hydrothermal stability in comparison to other mesoporous silica materials, such as MCM-41, its analogue in M41S family [1]. Recent studies have indicated that the addition of salt, such as NaCl, KCl and so on, seems to facilitate the self-assembly of P123 micelles and silicate, and lead to highly ordered and stable mesoporous structures [2]. However, the salt ions were not incorporated into the SBA-15 framework based on element analysis. The salt effect on synthesis of SBA-15 is still ambiguous. In this annual report, the salt effect on self-assembly of SBA-15 was investigated by in situ small-angle synchrotron X-ray scattering (SAXS) experiments performed at beamline 17A of the National Synchrotron Radiation Center (NSRRC) facility, Hsinchu, Taiwan. The standard operating conditions were around 1.5 GeV and 300 mA. The wavelength (λ) of synchrotron X-ray radiation was 1.334431 Å. The gelled sample was pumped into a Teflon sample cell by a wriggle pump. In situ SAXS patterns were collected by image plate detection (120 s per one).

In situ SAXS pattern of the conventional SBA-15 material prepared by following the procedure of Zhao et al [1] is shown in Fig. 1(A). The SAXS patterns were recorded before and after the addition of tetraethyl othorsilica (TEOS) precursor. No scattering peak was found in the low-angle region prior to adding TEOS, implying that no ordered pore structure exists in the synthesis solution containg P123 micelles. As TEOS was added, a broad scattering peak was found at 2 θ ~ 0.78 corresponding to a d-spacing of 9.8 nm. That is probably due to the short-range ordered arrangment of TEOS adsorbed P123 micelles. After TEOS was added for 40 nm, a broad scattering peak at 2 θ ~ 0.78 disapeared and the other two scattering peak at 2 θ ~ 0.62 and 1.1 corresponding to d-spacings of 12.3 and 7.0 nm gradually observed. As the self-assembly period was prolonged, the scattering peaks of (100), (110), (200), and (210) planes could be clearly observed, indicating that the long-range ordered pore arrangement of 2D hexagonal *p6mm* structure is formed. When NaCl was added in the synthesis solution containing P123micelles, no scattering peak was found, implying that no ordered structure is present in P123 micellar solution with NaCl. With adding TEOS, a broad scattering peak was formed due to the aggregation of TEOS adsorbed P123 micelles. A serious of scattering peaks due to the arrangement of P123 micelles into ordered pore structure was quickly observed after TEOS was only added 15 min. Moreover, after 30 min, at least four diffraction peaks of (100), (110), (200), and (200) planes were observed, indicating that highly-ordered *p6mm* pore structure is already formed. This result clearly demonstrated that the self-assembly of P123 micells and TEOS was accelerated by adding NaCl in the synthesis solution resulting in highly-ordered pore structure of SBA-15.

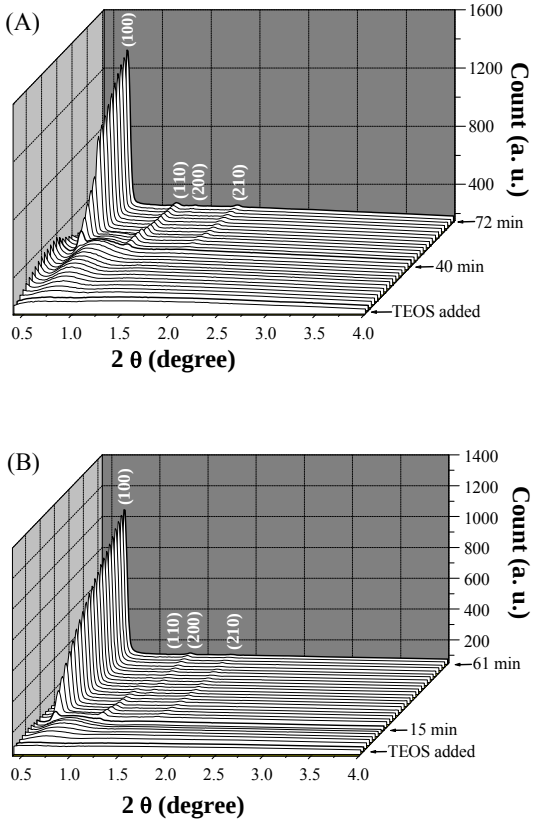

Fig. 1: In situ SAXS patterns of (A) conventional SBA-15, (B) SBA-15 prepared with a NaCl/Si ratio of 2.

References

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