Direct Synthesis of Mesoporous SBA-15 with Long Alkyl Chains and Its Application on Chromatographic Columns

Chi-Feng Cheng (鄭吉豐)

Department of Chemistry, Chung Yuan Christian University, Chungli, Taiwan

In recent years, hexagonal mesoporous molecular sieve SBA-15 has attracted much interest because of their high surface area (~800 m^2/g), thicker wall (~ 5 nm), large and uniform pore size (6.0~30 nm), and thus they are potential candidates for catalysis,separation and nano-material template. Silica materials with functional groups have been widely used as solid phase in high performance liquid chromatography (HPLC) and solid phase extraction (SPE).

Silane-functionalized mesoporous silicas have been synthesized by co-condensation of alkylsilanes and tetraethoxysilane (TEOS) in acidic medium with the block copolymer Pluronic 123 as the structure directing agent. The influence of synthesis parameters, the molar ratio of X = silane /(silane + TEOS) was studied. SBA-15 materials surface-modified by co-condensation with long alkylsilanes were used as HPLC stationary phase and show better separation effect than traditional calcined SBA-15 materials surface-modified with silanes.

The XRD patterns in Fig. 1 show XRD patterns of SBA-15 with alkyl chains prepared by the co-condensation of dodecyltrimethoxysilane(DTMS) and TEOS at different ratios of silane (X) and following extraction by acidic ethanol solution. The main Bragg reflections in the range of $0.50< 2\theta <3$ can be indexed as (100), (110) and (200) hexagonal diffraction patterns. The XRD patterns of samples have shown that the 100 reflection intensity was similar when DTMS /TEOS molar ratios increased from 5% to 40%. It can be observed in Fig. 1, all samples prepared with different DTMS/TEOS ratio have two sharply well define (110) and (200) reflections. It seems like that increasing the addition amounts of DTMS did not deteriorate the mesostructure of functionalized SBA-15.

The successful incorporation of DTMS in functionalized SBA-15 mesoporous materials is confirmed by NMR spectroscopy in Fig. 2. ^{13}C CP MAS solid state NMR spectra demonstrated that the resonance intensity of functionalized SBA-15 increases with increasing the DTMS amounts added.

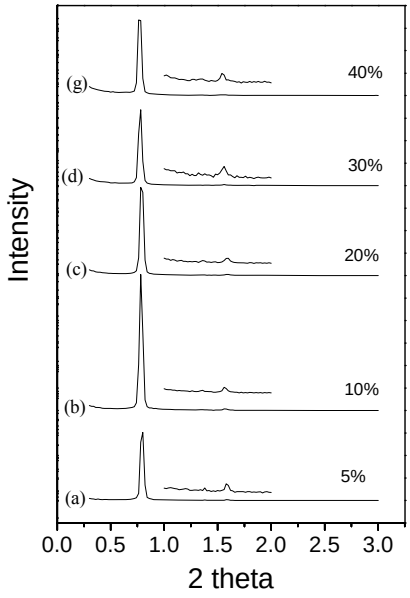

Fig. 1: Powder XRD patterns of SBA-15 with alkyl chains prepared by the co-condensation of DTMS and TEOS at different ratios of silane (X) and following extraction by acidic ethanol solution. (a) 5%, (b) 10%, (c) 20%, (d) 30% and (e) 40%

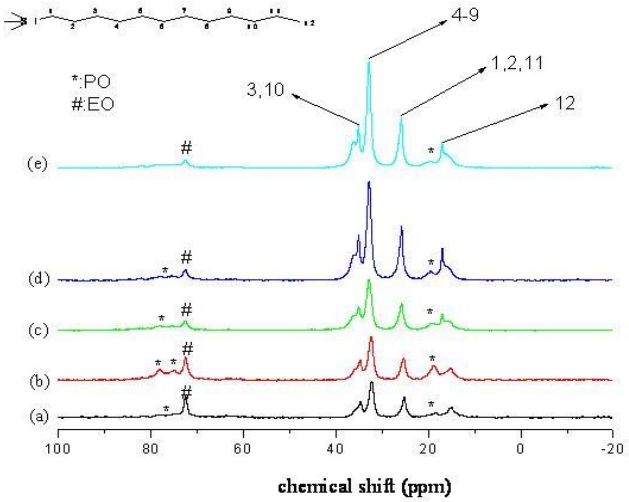

Fig. 2: ^{13}C CP MAS solid state NMR spectra of SBA-15 prepared by the co-condensation of DTMS and TEOS at different ratios of silane (X) and extraction by acidic alcohol solution (V_{C2O5OH} : V_{HCl}=97 : 3).(a)5% (b)10% (c) 20%(d)30% (e)40%.