

Organofunctionalized mesoporous silicas SBA-15

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Abstract – The SBA-15 mesoporous silica of hexagonal-ordered pore structure was synthesized and functionalized with the organosilanes, (3-aminopropyl)trimethoxysilane, *N*-[3-(trimethoxysilyl)propyl]ethylenediamine and (3-glycidyloxypropyl)trimethoxysilane. XRD pattern of calcined, shows three diffraction peaks in the region of $2\theta = 1.5\text{--}4.0$. SEM images reveal that the sample consists of many rope-like domains with relatively uniform sizes. ²⁹Si HPDEC NMR spectra of SBA-15 functionalized with amino groups showed two resonance peaks up-field corresponding to Q⁴ and Q³, and two peaks down-field, assigned to T³ and T². The presence of T peaks confirms the incorporation of the aminosilane moieties as a part of the silica wall structure.

The SBA-15 mesoporous silica of hexagonal-ordered pore structure, synthesized using amphiphilic triblock copolymers as the pore-directing agents and under acidic conditions, have received great attention in recent years because of its relatively large pore diameter and high hydrothermal stability in comparison to MCM-41, its analog in the M41S family [1]. In the past few years, organic-functionalized mesoporous silica materials have been extensively studied due to the variety of functionality incorporated. In this trend, the SBA-15 mesoporous silica was synthesized and functionalized with the organosilanes, (3-aminopropyl)trimethoxysilane, *N*-[3-(trimethoxysilyl)propyl]ethylenediamine and (3-glycidyloxypropyl)trimethoxysilane.

In the typical synthesis, tetraethoxysilane was introduced to an aqueous HCl solution containing triblock copolymer Pluronic P123 (EO₂₀PO₇₀EO₂₀) and hydrolyzed for 1 h under stirring at 35 °C. The resulting solution was kept in a static condition at 35 °C for 20 h. The mixture was followed by hydrothermal treatment at 100 °C for 48 h. The solid product was filtered off and then washed with deionized water repeatedly. After drying at room-temperature in air, the products were calcined at 550 °C for 6 h. SBA-15 modified was synthesized by using the calcined SBA-15 as starting material. Organosilanes was mixed with the calcined SBA-15, in a weight ratio of 4–1, in toluene under stirring at 80 °C for 24 h. The solid product was obtained by filtration, washed with toluene and acetone, dried in air at room temperature [2].

The XRD pattern of calcined SBA-15, as shown in Fig. 1, presented three diffraction peaks in the region of $2\theta = 1.5\text{--}4.0$, which can be indexed to the (100), (110), and (200) diffractions. The peaks can be indexed in agreement to two dimensional hexagonal *p6mm* symmetry, indicating a well defined SBA-15 mesostructure. Scanning electron microscopy (SEM) images (Fig. 2) reveal that the synthesized SBA-15 sample consists of many rope-like domains with relatively uniform sizes, which are aggregated into wheat-like macrostructures. ²⁹Si HPDEC NMR spectra (Fig. 3) of functionalized SBA-15 with amino groups showed two resonance peaks up-field corresponding to Q⁴ ($\delta = 111$ ppm) and Q³ ($\delta = -102$ ppm), and two peaks down-field, assigned to T³ ($\delta = -68$ ppm) and T² ($\delta = -59$ ppm), respectively. The presence of T peaks confirms the incorporation of the aminosilane moieties as a part of the silica wall structure, (Fig. 3).

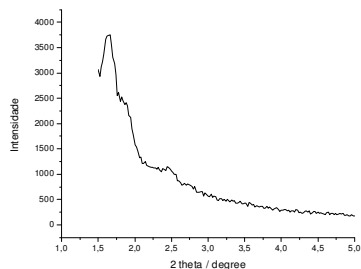


Figure 1: XRD patterns of the calcined SBA-15

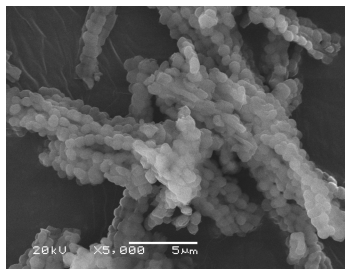


Figure 2: SEM images of SBA-15.

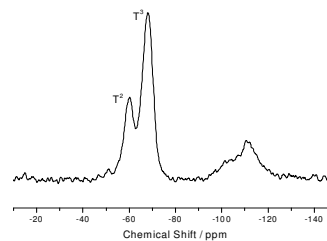


Figure 3: ²⁹Si HPDEC NMR spectra of SBA-15.

References

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