Abstract:

Synthesis of mesoporous silica SBA-15 has been demonstrated by using copolymer as structural directing agent (SDA). The prepared materials are showing applications in which efficient diffusion of molecules is in demand.

Keywords: Mesoporous silica, Copolymer, SDA.

Introduction

Ordered mesoporous materials are attracting wide interest in fields of catalysis, selective sorbents for separation processes, regulated transport systems, and the immobilization of bio molecules.1–6 A well-documented example is mesoporous silica SBA-15 synthesized by using Pluronic P123(EO20PO70EO20) as the SDA,7–9 which has two-dimensional (2-D) hexagonal p6mm symmetry and channel-type mesopores. These materials generally have additional micropores in the silica walls resulting from the interpenetrating network of silica and hydrophilic EO chains formed during the synthesis.10–13

Experimental Procedure: Simple and facile synthesis of mesoporous silica SBA-15 materials carried out with the carboxylate-terminated tri block copolymer Pluronic P123. The syntheses of mesoporous silica SBA-15 materials were performed in the high (1.6M) or low (0.3M) acid conditions,8,9 and the tri block copolymers P123-OH and P123-COOH were used as SDAs. The SBA-15 materials synthesized with original hydroxyl-terminated P123 (abbreviated P123-OH) or P123-COOH in high (1.6M) or low acid (0.3M) conditions. We find that by using P123-COOH, the formation of hexagonally ordered SBA-15 materials is accelerated. Furthermore, the resulting materials have additional intra-particle meso-ormacro porosities, depending on the acid concentration in the synthesis mixtures.

Characterization:

XRD patterns of nanocomposites were recorded by a RigakuMiniflex X-ray diffractometer (M/S.Rigaku
Corporation, Japan) using Ni filtered Cu Kα radiation with scan speed 2° min⁻¹ and 2θ range from 10-80°. The low angle XRD patterns were obtained from Altima –IV Rigaku X-ray diffractometer (M/S. Rigaku Corporation, Japan) using Ni filtered Cu Kα radiation with scan 1° min⁻¹ and 2θ range from 0.7-5°. The scanning electron microscopy (SEM) images and energy-dispersed X-ray spectrometer (EDX) were taken on a Hitachi S-800 field-emission scanning electron microscope and S-2400 scanning electron microscope, respectively. The nanocomposite samples prepared in this project work were characterized by transmission electron microscopy (TEM) (M/s. Phillips, Netherlands, Model: Tecnai Feil2, capacity: 120 KV). The finally powdered nano composite samples were sonicated in methanol for about 15 min and placed on sample holders (made of gold) and conducted the TEM analysis.

Results & Discussion:

Characterization by XRD: The shorter induction time for the (100) reflection of SBA-15-H-C than that of SBA-15-H-H suggests that the carboxylate end groups on the micelles of P123-COOH accelerate the formation of ordered meso structures. The pattern of SBA-15-H-C, just as that of SBA-15-H-H, exhibits (100), (110), and (200) reflections attributed to a hexagonal \( p6mm \) structure. This suggests that the end-group oxidation of P123 does not affect the formation of ordered mesoscopic structure.

![Fig-1: P-XRD of SBA-15-H-H(a) and SBA-15-H-C(b).](image)

Characterization by SEM: The SEM images in Figure 2 show that while SBA-15-H-H consists of uniform and rope-like domains, just like other SBA-15-silica materials, SBA-15-H-C is composed of aggregates of particles that are irregular in shape. Such a morphological difference of the two samples may be related to the end-group oxidation of the copolymer SDA.
Characterization by TEM: the TEM images of the SBA-15-H-C, as indicated in Figure 3a, reveal the presence of cracks well distributed in SBA-15-H-C. In SBA-15-H-C, the walls are not very uniform, and one can find some breakages of the walls (Figure 5a) and cross sections of the mesopores that are not spherical in shape (Figure 3b).

**Conclusion:** Synthesis of mesoporous silica SBA-15 has been achieved with additional intra-particle porosities by using the carboxylate-terminated tri block copolymer P123 as the SDA. The prepared SBA-15 materials have ordered pore structures with better connectivity, and they are promising for practical applications in which good molecular diffusibility is one of the major concerns.

**References:**


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