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SYNTHESISAND CHARACTERIZATION OF MESOPOROUSSILICASBA-15

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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 immobiliza-tionofbio 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 (abbreviatedP123-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 K_α radiation with scan speed 2ºmin^{-1} and 2θ range from 10-80º.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 1ºmin^{-1} and 2θ range from 0.7-5º. 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-Hsuggests 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.

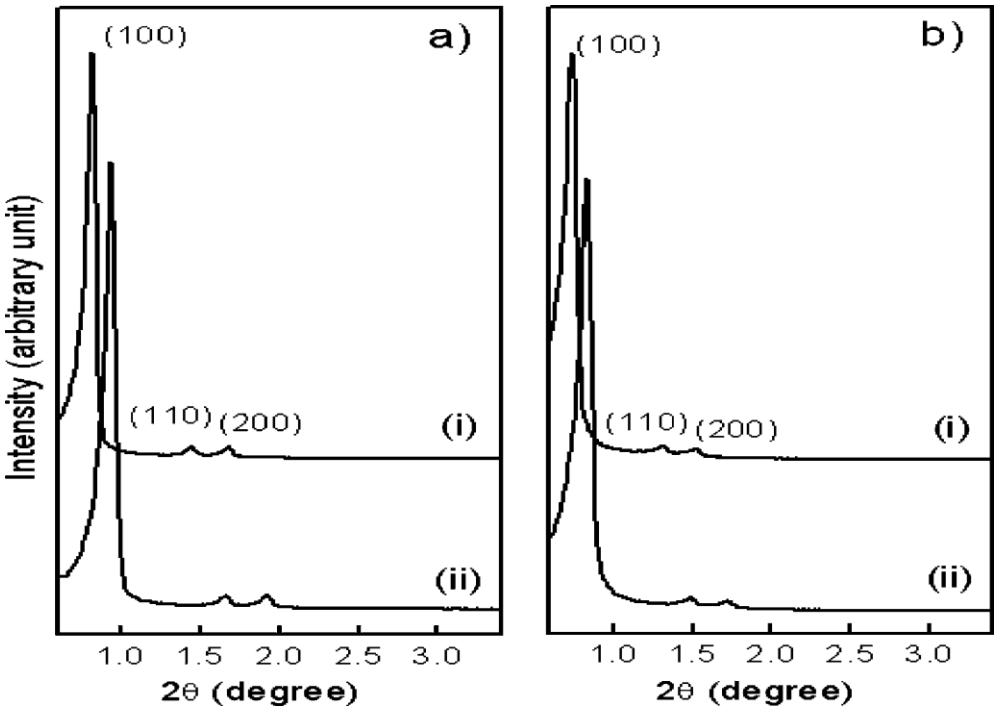

Fig-1: P-XRD ofSBA-15-H-H(a)andSBA-15-H-C(b).

Characterization by SEM: The SEM images in Figure2 show that while SBA-15-H-H consists of uniform and rope-like domains, just like other SBA-15-silica materials, SBA-15-H-Ciscomposed 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.

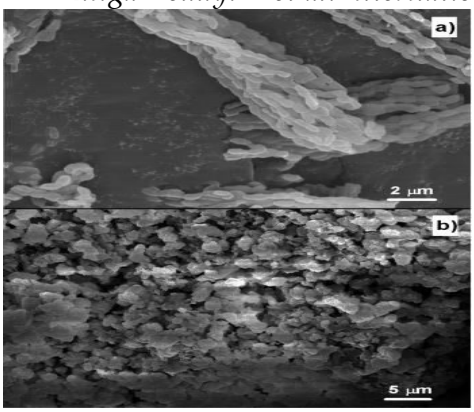

Fig-2: SE Mimagesof(a)SBA-15-H-Hand(b)SBA-15-H-C.

Characterization by TEM: theTEMimagesoftheSBA-15-H-C, as indicated in Figure3a, reveal the presence of cracks well distributed in SBA-15-H-C. in SBA-15-H-C is not very uniform, and once an find some break ages of the walls (Figure5a) and cross sections of the mesopores that are not spherical in shape (Figure3b).

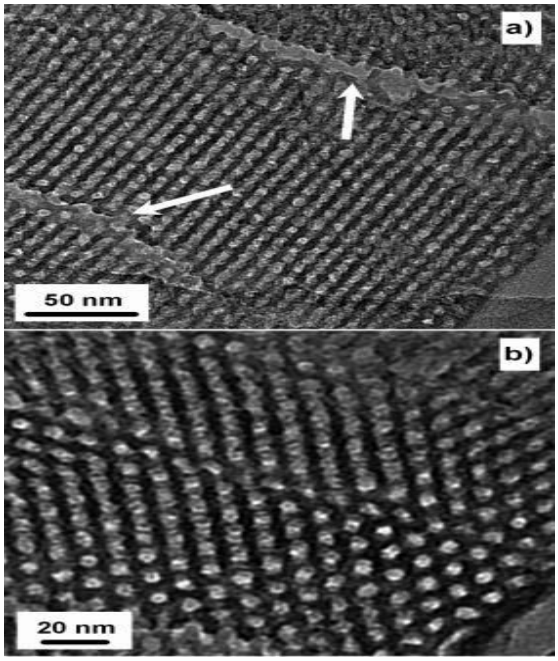

Fig-3: TEM images of (a) along the axis of the hexagonal pores (b). The arrows indicate the slit-type cracks in the sample.

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.

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