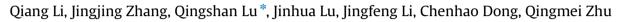
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Hydrothermal synthesis and characterization of ordered mesoporous magnesium silicate-silica for dyes adsorption



School of Physical Science and Technology and Inner Mongolia Key Laboratory of Nanoscience & Nanotechnology, Inner Mongolia University, Hohhot 010021, China

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ABSTRACT

Mesoporous materials with substantial specificities have potential applications in pollutant adsorption. In this work, ordered mesoporous magnesium silicate-silica was prepared by a facile hydrothermal method using mesoporous silica SBA-15 filled with carbon (C@SBA-15) as both assisted template and silicon source. During hydrothermal process, the carbon inside the pores of the SBA-15 supported magnesium silicate as mesoporous wall. The magnesium silicate together with the remainder silica exhibited ordered mesoporous structure, BET surface area of 432 m^2/g , and bimodal pore size distributions. The mean pore sizes were 5.46 and 55.0 nm. The ordered mesoporous magnesium silicate-silica showed a fast adsorption rate for methylene blue; meanwhile, the adsorption capacities for methylene blue and Rhodamin B were 283 and 227 mg/g, respectively.

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1. Introduction

Environmental contamination caused by organic dyes and heavy-metal ions has been attracted extensive attention [1,2]. Up to now, various strategies for the removal of these pollutants have been explored. The adsorption method is a facile and efficient way. Conventional adsorbents including activated carbon, clay mineral, zeolite, and polymer were used to remove pollutants [3–5]. To obtain adsorbents with superior adsorption property, increasing the specific surface area and controlling the porous structure are two critical strategies [6,7]. The surface is where the interaction between the pollutants and adsorption sites occurs, while the porous structure determines the mass transportation rate.

Nanostructured magnesium silicate with unique structure and composition shows fast adsorption rate and superb adsorption capacity [8–11]. Till now, various magnesium silicate nanostructures including hollow sphere [9], nanotube [12], core-shell [13], yolk-shell [14], and hierarchical structure [15] have been synthesized. As a special nanostructure, mesoporous materials are well known for their favorable regular and tunable pore size, high specific surface area, and uniform pore size distribution [16]. However, magnesium silicate with ordered mesoporous structure has not been reported. In this work, we aim to fabricate ordered mesoporous magnesium silicate for dye adsorption. Using mesoporous silica SBA-15 filled with carbon (C@SBA-15) as both silicon source and assisted template, ordered mesoporous

* Corresponding author. E-mail address: luqs@imu.edu.cn (Q. Lu).

http://dx.doi.org/10.1016/j.matlet.2016.02.029 0167-577X/© 2016 Elsevier B.V. All rights reserved. magnesium silicate-silica was prepared by hydrothermal method. The samples showed excellent adsorption property for methylene blue (MB) and Rhodamin B (RhB).

2. Experimental section

C@SBA-15 was synthesized according to the literature procedures [17]. Ordered mesoporous magnesium silicate-silica was prepared as follows: 0.3846 g of magnesium nitrate hexahydrate was dissolved in 50 mL of deionized water and 25 mL of ethanol. Then 0.15 g of the C@SBA-15 was added to above water/ethanol solution under stirring. The pH value of the suspension was adjusted to 9 using ammonia solution. The suspension was transferred into a teflon-lined autoclave, sealed at 140 °C for 12 h under hydrothermal treatment. The as-prepared products were collected by centrifugation, washed with deionized water, dried at 100 °C overnight, and calcined at 650 °C for 6 h. Finally, ordered mesoporous magnesium silicate-silica was obtained and denoted as meso-MgSiO-SiO.

The phase structure and mesostructure were investigated by X-ray diffraction (XRD) on a PANalytical Empyrean diffractometer with Cu K_{α} radiation. The morphology and microstructure were characterized by a Hitachi S-4800 scanning electron microscopy (SEM) and FEI Tecnai F20 transmission electron microscopy (TEM), respectively. N₂ adsorption-desorption isotherms were measured with a Micromeritics ASAP2020 specific surface area analyzer. Pore size distribution was calculated using Barrett–Joyner–Halenda (BJH) method based on the adsorption branch of the isotherms. The concentrations of MB and RhB were analyzed using Hitachi U-3900 UV–vis spectrophotometer.





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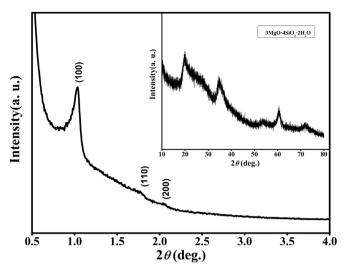


Fig. 1. Low-angle and high-angle XRD patterns of meso-MgSiO-SiO.

For the adsorption rate study, 30 mg of meso-MgSiO-SiO was added to 60 mL of MB solution with an initial concentration of 50 mg/L. After a specified contact time, the meso-MgSiO-SiO and MB solution were separated and the MB concentration was analyzed by UV-vis spectrophotometer. The dependence of normalized MB concentration on contact time was studied. For the adsorption isotherm study, 30 mg of meso-MgSiO-SiO was added to 60 mL of MB solution with different concentrations. After stirring for 6 h, the MB concentration in the remaining solution was analyzed by UV-vis spectrophotometer. The adsorption isotherm was obtained by changing the initial MB concentration. The adsorption isotherm of meso-MgSiO-SiO for RhB was carried out in a similar way.

3. Results and discussion

Fig. 1 shows the low-angle XRD pattern of meso-MgSiO-SiO. A sharp and two weak peaks are indexed as (100), (110), and (200) diffractions of mesoporous structure with a two-dimensional hexagonal

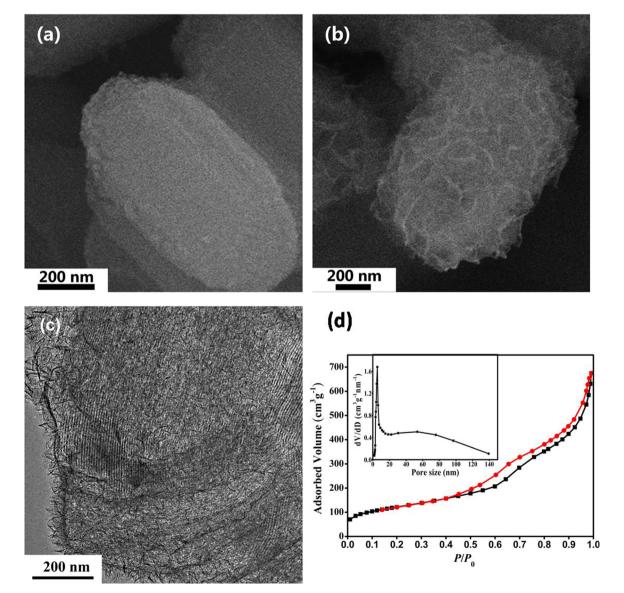


Fig. 2. SEM micrographs of C@SBA-15 (a) and meso-MgSiO-SiO (b); TEM micrograph (c) and N₂ adsorption-desorption isotherms (d) of meso-MgSiO-SiO (The inset shows the pore size distribution).

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