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Facile mesoporous template-assisted hydrothermal synthesis of ordered mesoporous magnesium silicate as an efficient adsorbent

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ABSTRACT

Mesoporous materials with unique structure as well as special morphology have potential applications in pollutant adsorption. In this work, using mesoporous silica SBA-15 filled with carbon (C@SBA-15) as both silicon source and assisted template, the ordered mesoporous magnesium silicate ($Mg_3Si_4O_9(OH)_4$) has been fabricated at 140 °C by a novel and facile hydrothermal method. During the hydrothermal process, the magnesium silicate grew along the silica walls at the expense of consuming silica and deposited on the carbon surface of the C@SBA-15. Meanwhile, the rigid carbon inside the pores of the SBA-15 supported the magnesium silicate as mesoporous walls under hydrothermal condition. The obtained magnesium silicate possessed ordered mesoporous structure, high specific surface area of 446 m^2/g, large pore volume of 0.84 cm^3/g, and hierarchical structure assembled with ultrathin nanosheets of 15 nm in thickness. These characteristics endow the ordered mesoporous magnesium silicate with the fast adsorption rate and high adsorption capacity of 382 mg/g for methylene blue. In addition, this synthesis method opens a new approach to fabricate other ordered mesoporous silicates.

1. Introduction

Environmental pollution with organic dyes and heavy-metal ions has become a serious problem and been extensively concerned [1,2]. Various physicochemical methods have been proposed to solve this problem [3]. Gupta et al. [4–6] reported the photocatalytic degradation of the hazardous dyes using TiO_2 as catalyst under UV irradiation. Sekaran et al. [7] proposed the idea of degrading aromatic amine by using the heterocatalytic Fenton oxidation. Gupta and Nayak [8] studied the cadmium removal and recovery from aqueous solution by novel adsorbents. Among these methods, the adsorption technique is considered to be the most facile, efficient, and convenient way [9,10]. Conventional adsorbents such as clay minerals, polymers, and activated carbons were investigated for the adsorption of dyes and toxic metal ions [11–14]. Various waste materials such as bottom ash and deoiled soya were employed to remove toxic dyes from wastewater. These waste materials have great practical applications in environmental field [15–18]. To obtain adsorbents with high adsorption efficiency and capacity, increasing the specific surface area and controlling the porous structure are two critical factors. The surface is where the interaction between the pollutants and adsorption sites occurs, while the porous structure determines the transportation rate of reactants [19,20]. For example, alumina or manganese dioxide-coated multi-wall carbon nanotubes were used as efficient adsorbents for the treatment of lead aqueous solution [21,22]. Recently, hierarchical nanostructured materials have opened up many new application possibilities in different fields due to their combined micro-nano structure features with large specific surface area, high reactivity, weak aggregation, and easy recycling [23–25]. There have been great interests in the controlled synthesis and applications of various nanostructured adsorbents.

Silicates are composed of silicon oxygen tetrahedrons shaped anionic group with a negative four charge, while metal ions reside between layers or among chains. These unique structure and composition endow them with ideal candidates for adsorption [24]. As a typical example of silicates, magnesium silicate with fast adsorption rate and superb adsorption capacity has been proved to be a highly efficient adsorbent [26]. Numerous researches have been focused on fabricating magnesium silicate with unique structure as well as special morphology. Up to date, various nanostructured magnesium silicates including hollow sphere [27], nanotube [28], core-shell [29], yolk-shell [30], and hierarchical structure [31] have been successfully synthesized via different methods. They have shown excellent adsorption performance for removing dyes and heavy-metal ions.

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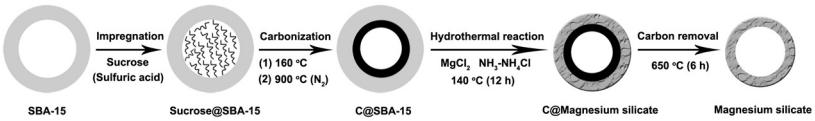

Fig. 1. Schematic illustration for the formation process of the ordered mesoporous magnesium silicate.

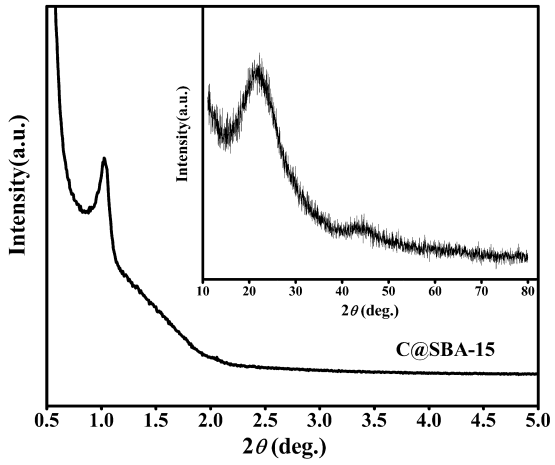

Fig. 2. Low-angle XRD pattern and high-angle XRD pattern (in the inset) of the C@SBA-15.

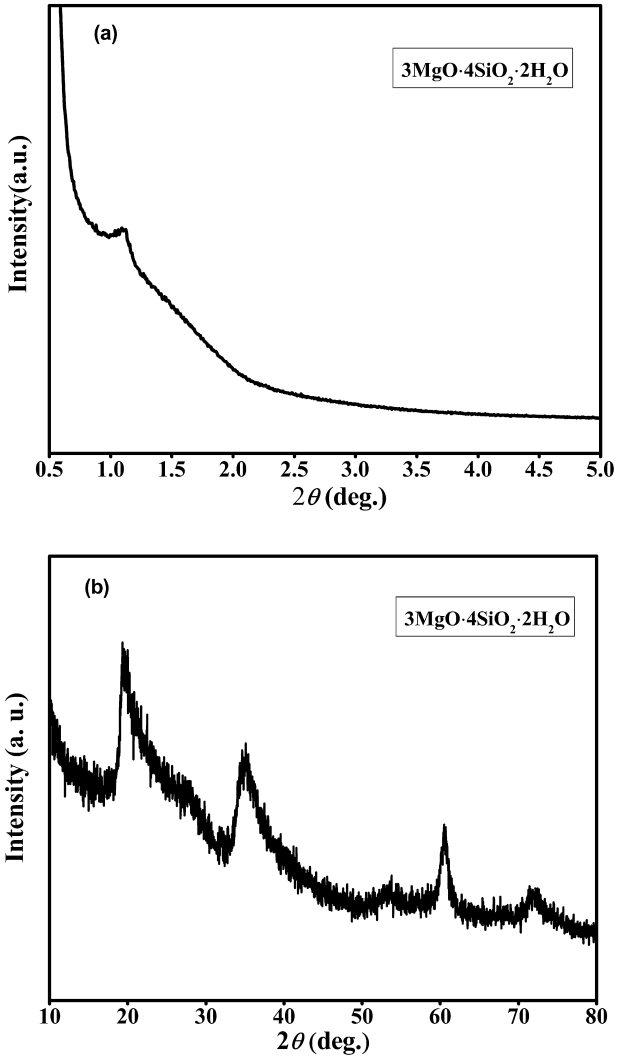

Fig. 3. Low-angle XRD pattern (a) and high-angle XRD pattern (b) of the ordered mesoporous magnesium silicate.

As a special nanostructure, mesoporous materials have attracted considerable attention and great efforts have been devoted to controlling their porous structure, specific surface area, and pore size distribution. These factors significantly influence their properties as catalysts, absorbents, gas sensors, solar cells, batteries, and electronics [32,33]. Especially, mesoporous materials with substantial specificity can meet the requirements as excellent adsorbents for removing dyes and heavy-metal ions, not only providing huge interface and large space capable of accommodating guest species, but also enhancing the mass transfer efficiency. In addition, in term of the diverse electronic structures and textural compositions, non-silica mesoporous materials such as metal oxides, carbon, silicates, and metals have wide range of applications. Even though a number of non-silica mesoporous materials have been fabricated by either the surfactant-assisted approach or nanocasting method [34,35], the preparation usually involves complicated process, low yield, poor reproducibility, and less success. Hence it is still a great challenge to date for synthesizing ordered non-silica mesoporous materials including ordered mesoporous magnesium silicate.

In this work, a mesoporous template-assisted hydrothermal method was developed to fabricate ordered mesoporous magnesium silicate using mesoporous silica SBA-15 filled with carbon (C@SBA-15) as both assisted template and silicon source. This facile and novel method was proposed for the first time. The crystalline phases, morphologies, and microstructures of the ordered mesoporous magnesium silicate were studied. The formation process of the ordered mesoporous structure was proposed. The crystalline magnesium silicate ($Mg_3Si_4O_9(OH)_4$) possessed ordered mesoporous structure, uniform pore size distribution, large specific surface area, and hierarchical structure assembled with ultrathin nanosheets, which exhibited excellent adsorption properties for methylene blue (MB).

2. Experimental

2.1. Sample preparation

C@SBA-15 was synthesized in the same way as the preparation of ordered mesoporous carbon CMK-3 except for the removal of silica. The details have been presented in literature [36]. The ordered mesoporous magnesium silicate was prepared as follows. 0.75 mmol of magnesium chloride and 10 mmol of ammonia chloride were dissolved in 50 mL of deionized water. Then 1 mL of ammonia solution (28%) was added to the above solution under continuously stirring. 77 mg of the obtained C@SBA-15 was dispersed homogeneously in 25 mL of deionized water to form suspension. The solution and the suspension were mixed together, then transferred into a teflon-lined autoclave, and sealed at 140 °C for 12 h for hydrothermal treatment. Finally, the as-prepared products were collected by centrifugation, rinsed with deionized water, dried at 100 °C overnight, and calcined at 700 °C for 2 h under N_2 atmosphere with a heating rate of 1 °C/min. The carbon was removed by calcination at 650 °C for 6 h in air. Finally, the ordered mesoporous magnesium silicate was obtained.

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