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Original Research Paper

pH-dependent formation of AACH fibers with tunable diameters and their in situ transformation to alumina nanocrystals with mesoporous structure

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A B S T R A C T

Through a simple hydrothermal and thermal decomposition process of the ammonium aluminum carbonate hydroxide (denoted as AACH) precursors, we prepared mesoporous γ-Al_2O_3 fibers with tunable diameters by manipulating the amount of urea (pH-adjuster) in the reaction mixture (i.e., pH control). The experimental results show that the diameter of the obtained AACH fibers decreased obviously when the pH values of the reaction mixtures changed from 2.5 to 5.0. The as-formed precursors could transform to the morphology-remained cubic γ-Al_2O_3 with a slight shrinkage in diameter of the fibers after an annealing process. N_2 adsorption–desorption experiment indicates that the as-synthesized alumina microfibers and nanofibers have large surface area (176 and 230 m^2/g, respectively) and narrow pore-size distributions. Both the γ-Al_2O_3 microfibers and nanofibers are powerful in the removal of Congo red pollutant from waste water.

1. Introduction

Alumina are widely used as advanced catalysts, catalyst supports, adsorbents etc., [1,2]. Being a catalyst supports, the properties of Al_2O_3 are determined predominantly by crystal structure, composition, particle size and morphology. Therefore, the synthesis of Al_2O_3 with well controlled composition, size and morphology is of great significance for their applications. One-dimensional (1-D) alumina nanostructures have received considerable interest due to their novel properties, such as high dielectric constant, good thermal and chemical stability, and high mechanical modulus [3–6]. Several methods have been demonstrated to prepare of 1-D nanostructures, including thermal evaporation method [5–8], electrochemical synthesis method [9], normal and lateral stepwise anodization [10–14], sol–gel [15–18]. Especially, the introduction of hydrothermal process has provided a relatively simple and powerful method of the synthesis of 1-D nanostructures. Lee et al. [19] described a template-synthesis hydrothermal method for the growth of alumina nanotubes, nanofibers and nanorods by using poly(ethylene oxide) (PEO) template at 373 K. Zhu and co-workers [20] synthesized γ-alumina with a nanofiber morphology and large porosity in the presence of poly(ethylene oxide) surfactant as a template. Zhang et al. [21] designed a three-step synthesis route to preparation of lathlike and rod-shaped mesoporous alumina nanoparticles.

Recently, many reports studied the preparation of the morphology-controlled alumina materials via a novel AACH precursor due to its special crystal structure [22–25], which are benefits to the preparation of the morphology-controlled and mesopores-remained Al_2O_3 products. Herein, we present a facile hydrothermal and thermal decomposition route to mesoporous γ-Al_2O_3 fibers with tunable diameters under different acidic conditions. We used urea as the pH-adjuster, and the morphologies of the resulting AACH turn out to be pH-dependent. One possible formation process based on the pH-controlled nucleation and subsequently surfactant-induced growth mechanism is proposed. Finally, both of the as-obtained alumina fibers were used to adsorb Congo red from water solution.

2. Experimental

2.1. Synthesis of AACH fibers with tunable diameters

In a typical synthetic procedure for AACH microfibers: 0.1 mmol of poly-glycol (PEG)-2000 were dissolved in 8 mL deionized water to form a transparent solution. Then 5 mmol $Al(NO_3)_3{\cdot}9H_2O$ was added to the above solution under vigorous stirring. At last, urea (0.45 mol) was added into the mixture until pH value of the mixture is about 2.5. The final mixture was then transferred into a

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