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Facile hydrothermal synthesis of Ag@AgCl@AlOOH hollow microspheres and their characterizations

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ABSTRACT

In this paper, Ag@AgCl@AlOOH hollow microspheres were successfully synthesized with $AgNO_3$ and $AlCl_3 \cdot 6H_2O$ in NaOH/urea solution by a facile hydrothermal method. NaOH/urea solution acted as structure-directing agent here. Scanning electron microscopy (SEM) and X-ray powder diffraction (XRD) were used to characterize the as-prepared samples. The effects of hydrothermal heating temperatures on the phases and morphologies of samples were investigated. The size distributions analysis showed that the samples were Ag@AgCl@AlOOH microspheres with a diameter of 5.48 ~ 6.77 μm. With higher reaction temperatures, more Ag@AgCl@AlOOH microspheres with hollow structures appeared, and the size of Ag@AgCl@AlOOH microspheres decreased. The formation process of Ag@AgCl@AlOOH hollow microspheres was explored and discussed in detail.

1. Introduction

Recently, the visible-light-driven photocatalysis are attractive, due to their excellent properties and widely applications [1–3]. Wen et al. reported the synthesis of MoS_2/g-C_3N_4 nanocomposites with enhanced visible-light photocatalytic activity for the removal of nitric oxide [4]. Ag@AgX (X=Cl, Br, I) particles have also attracted much attention due to the high photocatalytic activities [5–10]. Hollow Ag@AgCl micro/nanostructures have been the most popular because of their excellent photocatalytic activities [11–14]. In order to improve their photocatalytic efficiency and stability, many ternary composite photocatalysts have also been developed, such as Ag@AgCl@TiO_2 [15], Ag@AgCl@ZnO [16], and Ag@AgCl@C_3N_4 [17]. However, as far as we know, the synthesis of Ag@AgCl@AlOOH hollow microspheres has not been reported yet. In many cases, hollow microspheres structure could effectively improve the catalytic efficiency due to their big specific surface area, lower density, and low combination rate of photo-generated electron-hole pairs.

Herein, we reported a facile hydrothermal method for synthesizing Ag@AgCl@AlOOH hollow microspheres. The formation process of Ag@AgCl@AlOOH hollow microspheres was discussed in detail.

2. Experimental

All chemicals were of analytical grade and used as received without further purification. All experiments were conducted under air atmosphere. A typical synthesis experiment occurred as follows: 0.236 g $AgNO_3$ and 0.337 g $AlCl_3 \cdot 6H_2O$ were added into the deionized water (20 mL) under vigorous stirring for 10 min. 7 g NaOH and 12 g urea were added into the deionized water (100 mL) under vigorous stirring to form NaOH/urea solution. Then, the above two solution were mixed together and were transformed into a 50-mL Teflon-lined stainless steel autoclave. The autoclave was maintained at 140, 160, and 180 °C for 12 h. The product was separated from the solution by centrifugation, washed with water and ethanol three times and dried at 60 °C for further characterization.

3. Results and discussion

The phase and crystal structure of the synthesized samples were characterized by XRD (Rigaku D/max 2550, Cu Kα radiation). Fig. 1**a-c** showed XRD pattern of samples synthesized at 140, 160, and 180 °C, respectively. All samples exhibited similar diffraction peaks with mixed phases of crystallized Ag with a cubic structure (marked with *, JCPDS No.04-783) and well-crystallized AgCl with a cubic structure (JCPDS No.31-1238). The other diffraction peaks marked with "#" can be assigned to AlOOH (JCPDS No.083-1505).

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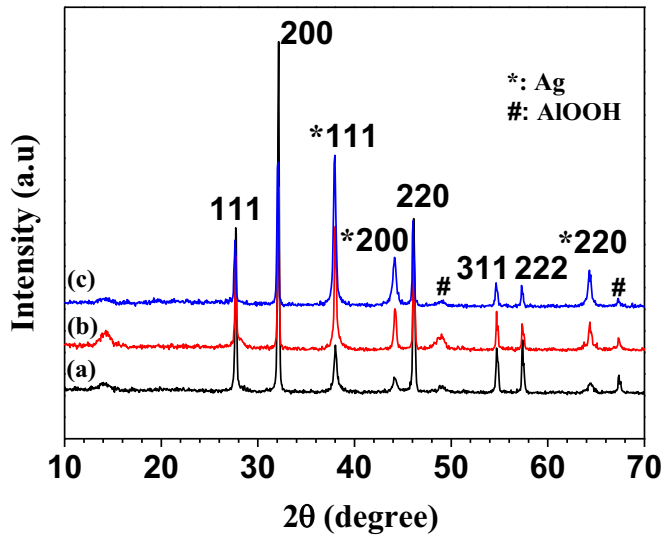

Fig. 1. XRD patterns of samples synthesized at (a) 140 °C; (b) 160 °C; and (c) 180 °C, respectively.

The results confirmed that Ag@AgCl@AlOOH was successfully synthesized in NaOH/urea solution via a hydrothermal method.

Fig. 2 showed SEM (Hitachi 3400 N) images and size distributions of Ag@AgCl@AlOOH synthesized at different temperatures. The Ag@AgCl@AlOOH microspheres were observed with a lot of nanosheets attached to their spherical surfaces (Fig. 2**a**). Most of the Ag@AgCl@AlOOH microspheres showed a solid structure with a mean diameter of ~5.73 μm. More Ag@AgCl@AlOOH hollow microspheres with a diameter of ~5.94 μm appeared at 160 °C, magnified images gave an insight on the big hole in the interior of the microsphere (Fig. 2**b**). Increased the temperature to 180 °C, large numbers of hollow solid microspheres with a mean diameter of ~6.30 μm were observed (Fig. 2**c**). Based on the SEM results, one can conclude that higher temperatures favored for the formation of hollow structures of Ag@AgCl@AlOOH and the growth of their particle size. Higher temperature could provide energy to form more nucleation sites, fast growth of Ag, AgCl, and AlOOH crystals, inducing the synthesis of Ag@AgCl@AlOOH microspheres with increasing size. Usually, the size of samples could affect photocatalytic activity due to the change of void space in hollow structure, as well as the surface contact area with dye molecules.

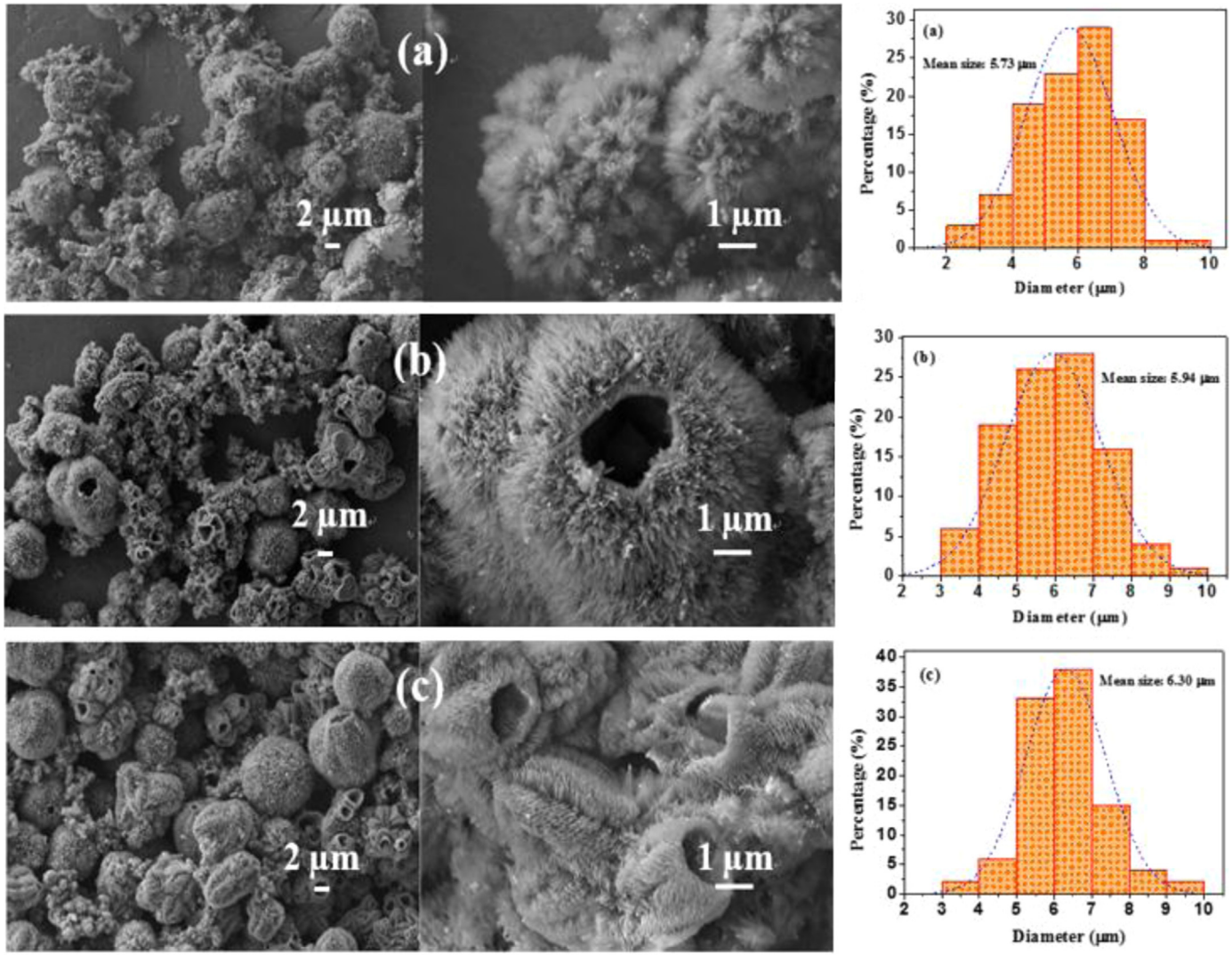

Fig. 2. SEM images and histograms of size distributions of same samples as in Fig. 1.