



Green synthesis and photoluminescence property of AlOOH nanoflakes



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ABSTRACT

A thorough green route (which adopts common and nontoxic reactants, and generates no by-product) was developed for the synthesis of AlOOH nanoflakes. AlOOH nanoflakes were synthesized conveniently through hydrothermal treatment of Al₂O₃ powder in deionized water at 190 °C for 12–24 h, without the use of any extra additive or template. The characterization results from X-ray diffraction, Fourier transformed infrared spectroscopy, energy dispersive X-ray spectroscopy and transmission electron microscopy confirmed the preparation of AlOOH nanoflakes, which were deficient in oxygen. Furthermore, the room temperature photoluminescence properties of the as-synthesized products were tested. It was found that all the as-synthesized products exhibited a strong emission peak centering at around 583 nm upon 291 nm excitation at room temperature, so they had potential application as a photoluminescent material.

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1. Introduction

AlOOH possesses many intriguing properties and has been studied for use as filler [1], precursor to Al₂O₃ [2,3], binder [4–6], adsorbent [7], catalyst [8], and photoluminescent material [9,10]. Nanoscaled AlOOH can display size- and morphology-dependent properties [7–11], which are important for technological applications and scientific research. In particular, two-dimensional (2D) nanomaterials such as nanobelts, nanoplates, nanosheets and nanoflakes have large surface areas, and are ideal components for fabricating functional nanodevices [10]. Hence, a lot of studies have been devoted to synthesizing 2D AlOOH so far [7–10]. For examples, Hou et al. reported a hydrothermal synthesis of lamellar γ -AlOOH architectures using hexadecyl trimethyl ammonium bromide as a structure-directing agent and precipitator [7]. Xu et al. reported a microemulsion-assisted synthesis of AlOOH nanoflakes [8]. Gao et al. reported a “molecule tailoring lamella” route for the synthesis of single-crystal γ -AlOOH nanobelts, using sodium bis(2-ethylhexyl)sulfosuccinate as the tailoring agent [9]. Chen et al. reported a hydrothermal synthesis of γ -AlOOH nanoflakes in the presence of organic bases such as ethylenediamine or hexamethylenetetramine [10]. However, the existing methods to 2D AlOOH needed the use of surfactants or organic bases, which may result in higher cost, more complex purification procedures, and even contamination of the final products and the environment.

Now, great attention has been paid to green synthesis chemistry and chemical processes [12–16], which utilize a set of principles that reduce

or eliminate the use and generation of hazardous substances in the design, manufacture and application of chemical products [14–16]. Adoption of nontoxic reactant and environmentally friendly solvent, and no release of by-product are the most important concern in a green synthesis strategy [14–16]. Herein, we report a completely “green” pathway to prepare AlOOH nanoflakes conveniently through hydrothermal treatment of Al₂O₃ powder in deionized water at 190 °C for 12–24 h. The as-synthesized products have been characterized by X-ray diffraction (XRD), Fourier transformed infrared spectroscopy (FTIR), energy dispersive X-ray spectroscopy (EDX), transmission electron microscopy (TEM) and room temperature photoluminescence spectra.

2. Experimental

1000 mg of Al₂O₃ powder was placed into a Teflon-lined autoclave of 50 mL capacity, and 40 mL of deionized water was added. After the mixture was magnetically stirred for 20 min, the autoclave was sealed and heated at 190 °C for 12 h, 18 h or 24 h. When the autoclave cooled to room temperature naturally, the resulting white precipitate was centrifuged, washed with distilled water and absolute ethanol, and finally dried in air at 80 °C for 12 h.

The products were characterized by XRD (German Bruker AXS D8 ADVANCE X-ray diffractometer), FTIR (The United States Varian Cary 670 FT-IR spectrometer), EDX (Japan Hitachi S-4800 field emission scanning electron microscopy configured with a Noran energy dispersive X-ray analyzer), TEM (The Netherlands Philips Tecnai12 microscope) and room temperature photoluminescence spectrum (Japan Hitachi F-4500 fluorescence spectrophotometer).

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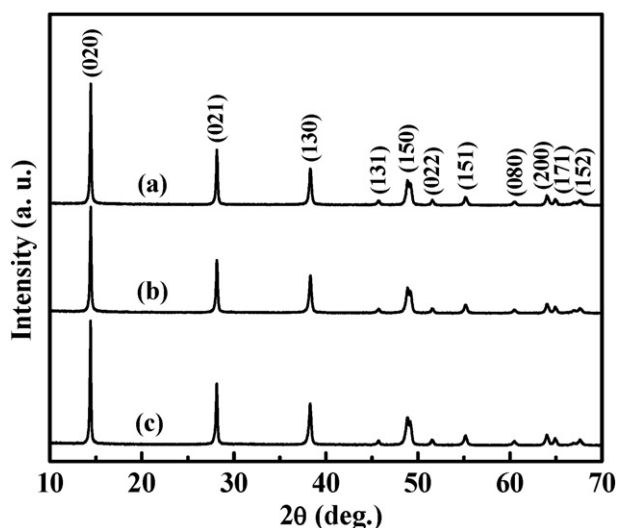


Fig. 1. XRD patterns of the products derived from hydrothermal treatment of Al_2O_3 powder in deionized water at 190°C for (a) 12 h, (b) 18 h and (c) 24 h.

3. Results and discussion

Fig. 1(a), (b) and (c) shows the XRD patterns of the products derived from hydrothermal treatment of Al_2O_3 powder in deionized water at 190°C for 12 h, 18 h and 24 h, respectively. The XRD peaks of all the products can be indexed to orthorhombic phase AlOOH, according to the Joint Committee on Powder Diffraction Standards card no. 83-1505. No obvious XRD peaks arising from Al_2O_3 were observed, indicating that the Al_2O_3 precursor had been completely transformed into AlOOH after the hydrothermal treatment in deionized water at 190°C for 12–24 h.

All the products displayed almost the same FTIR spectra as shown in Fig. 2. The absorption peaks at about 3283 and 3092 cm^{-1} can be ascribed to the asymmetric and symmetric stretching vibrations of (Al)O–H [17–20], respectively. The absorption peaks at about 2093 and 1971 cm^{-1} correspond to the combination bands [17,18]. The absorption peak at about 1650 cm^{-1} represents the bending mode of

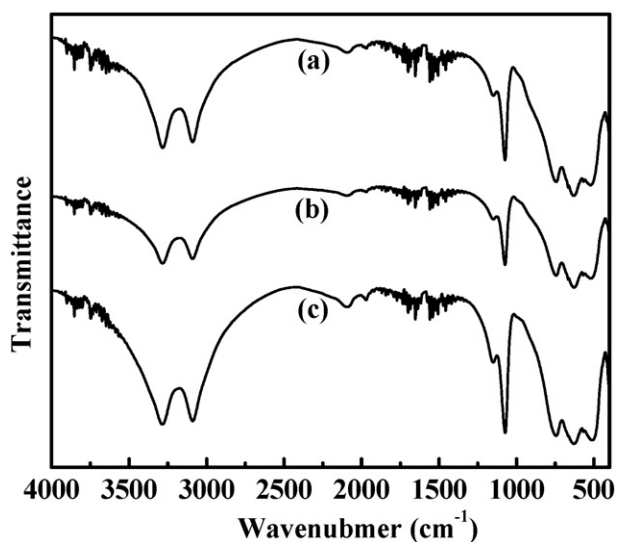


Fig. 2. FTIR spectra of the products synthesized using the reaction time of (a) 12 h, (b) 18 h and (c) 24 h.

adsorbed H_2O [17–20]. The absorption peaks at around 1152 and 1078 cm^{-1} belong to the asymmetric and symmetric stretching frequencies of Al–O–H [17–20], respectively. The absorption peaks at about 748 , 635 and 531 cm^{-1} can be assigned to the vibration modes of AlO_6 [17–20]. The FTIR spectra of our products resembled those of AlOOH reported in the previous literatures [17–20]. Thus, the FTIR characterization results further confirmed the preparation of AlOOH by the present green hydrothermal route.

Fig. 3(a), (b) and (c) shows the EDX spectra of the products derived from hydrothermal treatment of Al_2O_3 powder in deionized water at 190°C for 12 h, 18 h and 24 h, respectively. It can be seen from Fig. 3(a), (b) and (c) that all the products were made of Al and O elements (EDX cannot analyze the hydrogen element in samples). The atomic ratios of Al/O in the products synthesized using the reaction time of 12 h, 18 h and 24 h were about $36.0/64.0$, $36.6/63.4$ and $34.3/65.7$, respectively, which suggested that the as-synthesized AlOOH products were deficient in oxygen.

Fig. 4(a), (b) and (c) shows the TEM images of the AlOOH products synthesized using the reaction time of 12 h, 18 h and 24 h, respectively. All the AlOOH products consisted of mainly nanoflakes, but the size of the nanoflakes became bigger with the increase of the reaction time.

From the above XRD, FTIR, EDX and TEM results, it can be concluded that AlOOH nanoflakes had been synthesized conveniently by hydrothermal treatment of Al_2O_3 powder in deionized water at 190°C for 12–24 h, in the absence of any extra surfactant or additive as a

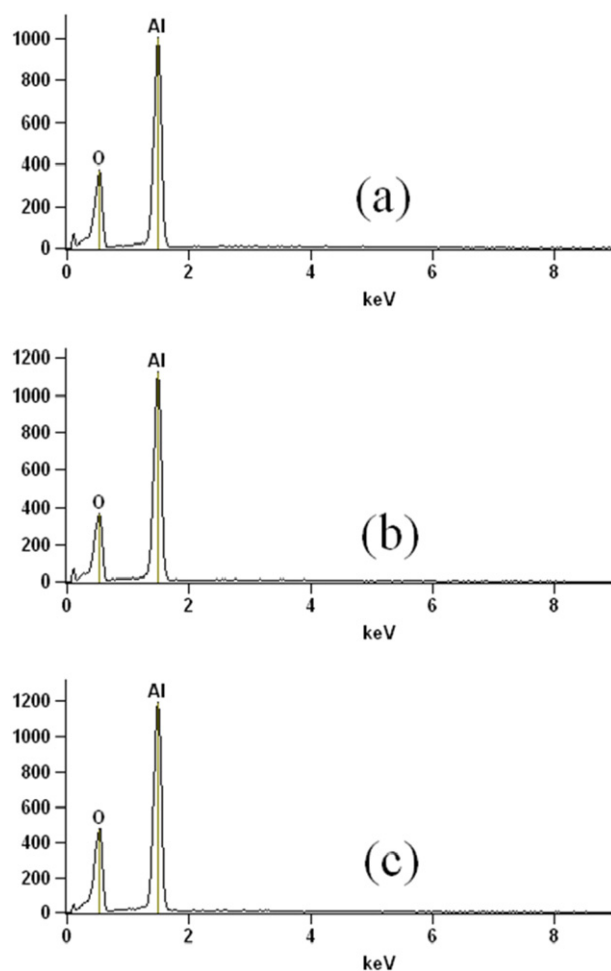


Fig. 3. EDX spectra of the products synthesized using the reaction time of (a) 12 h, (b) 18 h and (c) 24 h.

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