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Simple hydrothermal preparation of nanofibers from a natural ilmenite mineral

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Abstract

Titanate nanofibers were synthesized by a simple hydrothermal method using a natural ilmenite mineral as the starting material. The chemical composition, crystalline structure, shape, size, and specific surface area of the prepared samples were characterized by X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and the Brunauer–Emmett–Teller analysis (BET). The crystalline structure of the as-synthesized nanofibers demonstrated a layered titanate form, $H_2Ti_xO_{2x+1}$. The length of the prepared nanofibers ranged from 2 to 7 μm with diameters ranging from 20 to 90 nm. The as-synthesized nanofibers were solids with BET surface areas of approximately 50 m^2/g. This synthetic method provides a simple route for the fabrication of one-dimensional (1-D) nanostructured materials from a low-cost natural mineral.

Keywords: B. Nanofibers; C. Hydrothermal; D. Ilmenite; D. Titanium dioxide

1. Introduction

One-dimensional TiO_2 nanostructures including nanowires, nanorods, nanowhiskers, nanotubes and nanofibers have been intensively studied and researched due to their exceptional properties including chemical stability [1], biocompatibility [2,3], high photocatalytic reactivity [1,4], and cost-effectiveness. TiO_2 is one of the most attractive metal oxides for a versatile range of potential and novel applications [4–9], such as humidity sensors [10], optoelectronic devices [11], lithium ion batteries [12–14], hydrogen storage [15,16], dye sensitized solar cells (DSSC) [17–19], water treatment materials, catalysts, and gas sensors [20–25]. Low-dimensional TiO_2-related nanomaterials can be synthesized by various methods including electrospinning [26], hydrogen treatment [27], anodic porous alumina templating [28,29], carbon nanotube inner templating [30], supramolecular assembly templating [31], anodic oxidation of a titanium sheet [32], and hydrothermal NaOH (aq.) treatment [33,34]. Among these methods, the hydrothermal method for the synthesis of TiO_2 nanotubes, first proposed by Kasuga et al. [33,34], has been widely exploited for low-dimensional nanostructures [35–37]. The hydrothermal method is a straightforward synthesis that is cost effective and environmentally innocuous [38–41]. Furthermore, this technique can also be applied to the preparation of a wide range of low-dimensional TiO_2 nanostructures, such as nanoparticles [42], nanowires [43], nanofibers [38,39,41] and nanoribbons [43]. Ilmenite ($FeTiO_3$) is a natural source of low titanium content TiO_2 (usually approximately 50–60%) [44,45]. In our previous work, nanofibers were prepared by a simple hydrothermal method from a leucoxene mineral [41].

In this work, the direct synthesis of nanofibers from an ilmenite mineral is first reported. The nanofibers are prepared by the simple hydrothermal method using a low-cost ilmenite mineral as the starting material. Characterization of the prepared nanofibers is also reported.

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2. Experimental

2.1. Synthesis

Titanate nanofibers are synthesized by the hydrothermal method using a natural ilmenite mineral (Sakorn Minerals Co., Ltd., Thailand) as the starting material. These materials are made from 5 g of the black granules of ilmenite mineral (used without purification) are placed in a Teflon-lined stainless steel autoclave. To the autoclave was then added 200 mL of 10 M NaOH (aq.), followed by heating at 120 °C for 72 h with stirring. This process resulted in the formation of solid nanowires and long nanofibers [41]. After the autoclave was allowed to cool to room temperature, the resulting product was washed several times with an HCl (aq.) solution and then several times with distilled water, followed by drying with hot air. The experimental procedure is schematically shown in Fig. 1.

2.2. Characterization

The chemical compositions of the as-synthesized samples are analyzed by X-ray fluorescence (XRF, Philips, PW-2404, 4 kW). The phase and crystallinity of the samples were characterized by X-ray diffraction (XRD, X'Pert PRO MPD model pw 3040/60, PANalytical) with Cu $K\alpha$ ($\lambda = 0.154$ nm) irradiation at a scan rate of 0.02° $2\theta\ s^{-1}$ and a 2θ range of 10–90°. The microstructure of the as-synthesized product was analyzed by scanning electron microscopy (SEM, JEM-6510, JEOL), with accelerating voltages of 5–20 kV and transmission electron microscopy (TEM, JEOL JEM-2010 Electron Microscope). The distribution of the sizes of the nanofiber diameters was analyzed by SEM. Nitrogen adsorption measurements (Quantachrome Instruments, Autosorb-1) are used to determine the Brunauer–Emmett–Teller (BET) specific surface area.

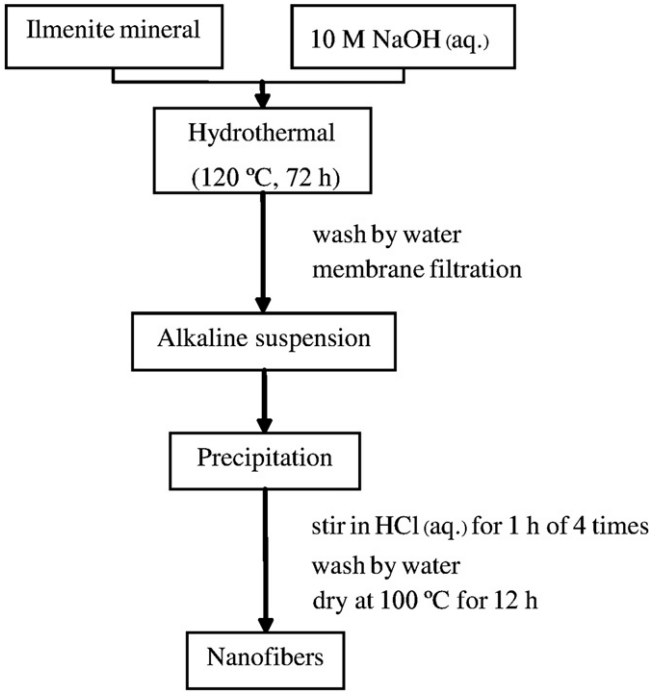

Fig. 1. Schematic representation of the experimental procedure.

3. Results and discussion

The as-synthesized sample was brown, whereas the starting ilmenite mineral was black (Fig. 2). This result

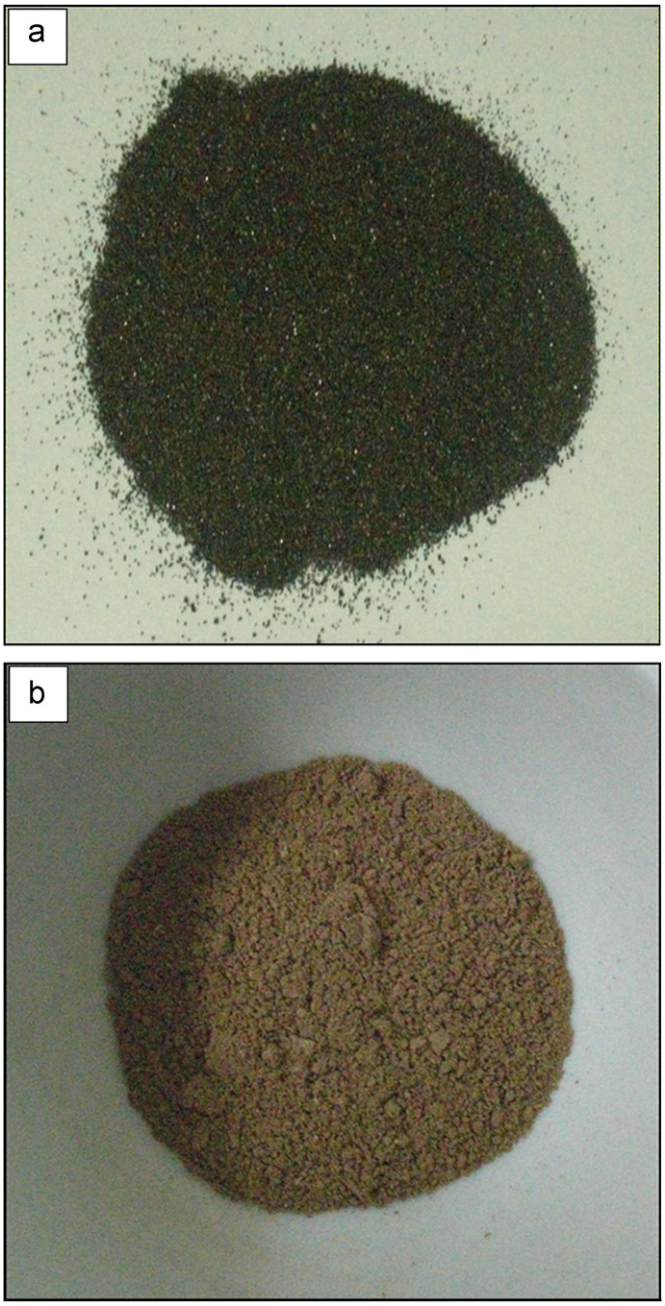

Fig. 2. Powders of (a) the starting ilmenite mineral and (b) the as-synthesized sample. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)