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Simple hydrothermal preparation of nanofibers from a natural ilmenite mineral

Athapon Simpraditpan[a], Thanakorn Wirunmongkol[c], Sorapong Pavasupree[c,*], Wisanu Pecharapa[a,b]

[a]College of Nanotechnology, King Mongkut's Institute of Technology Ladkrabang, Ladkrabang, Bangkok 10520, Thailand

[b]Thailand and Center of Excellence in Physics (ThEP Center), Commission on Higher Education, 328 Si Ayutthaya Road, Bangkok 10400, Thailand

[c]Department of Materials and Metallurgical Engineering, Faculty of Engineering, Rajamangala University of Technology Thanyaburi, Klong 6, Pathumthani 12110, Thailand

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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.

*Corresponding author. Tel.: +66 2549 3480; fax: +66 2549 3483.
E-mail address: sorapongp@yahoo.com (S. Pavasupree).

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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 $K\alpha$ (λ=0.154 nm) irradiation at a scan rate of 0.02° 2θ 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.

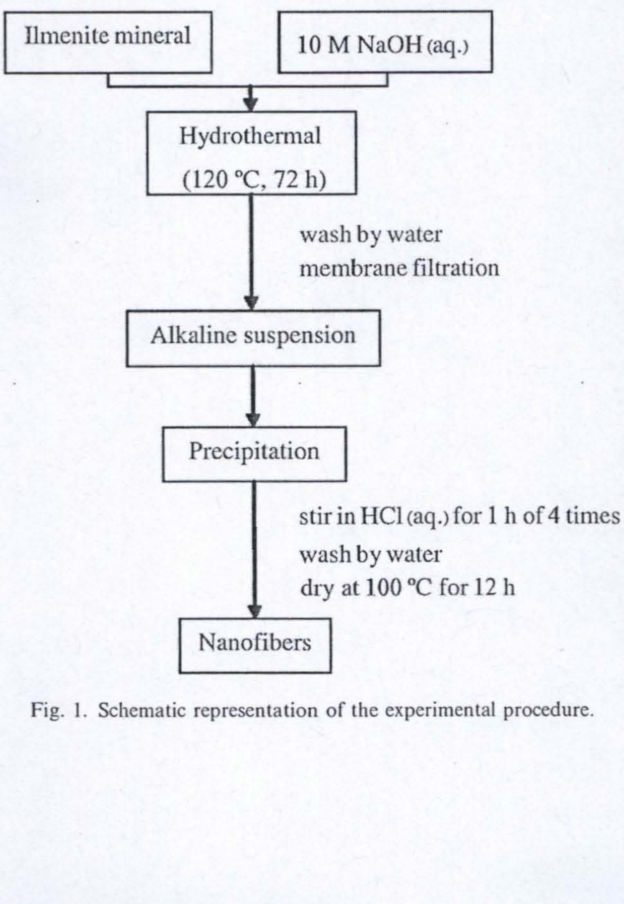

Fig. 1. Schematic representation of the experimental procedure.

3. Results and discussion

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

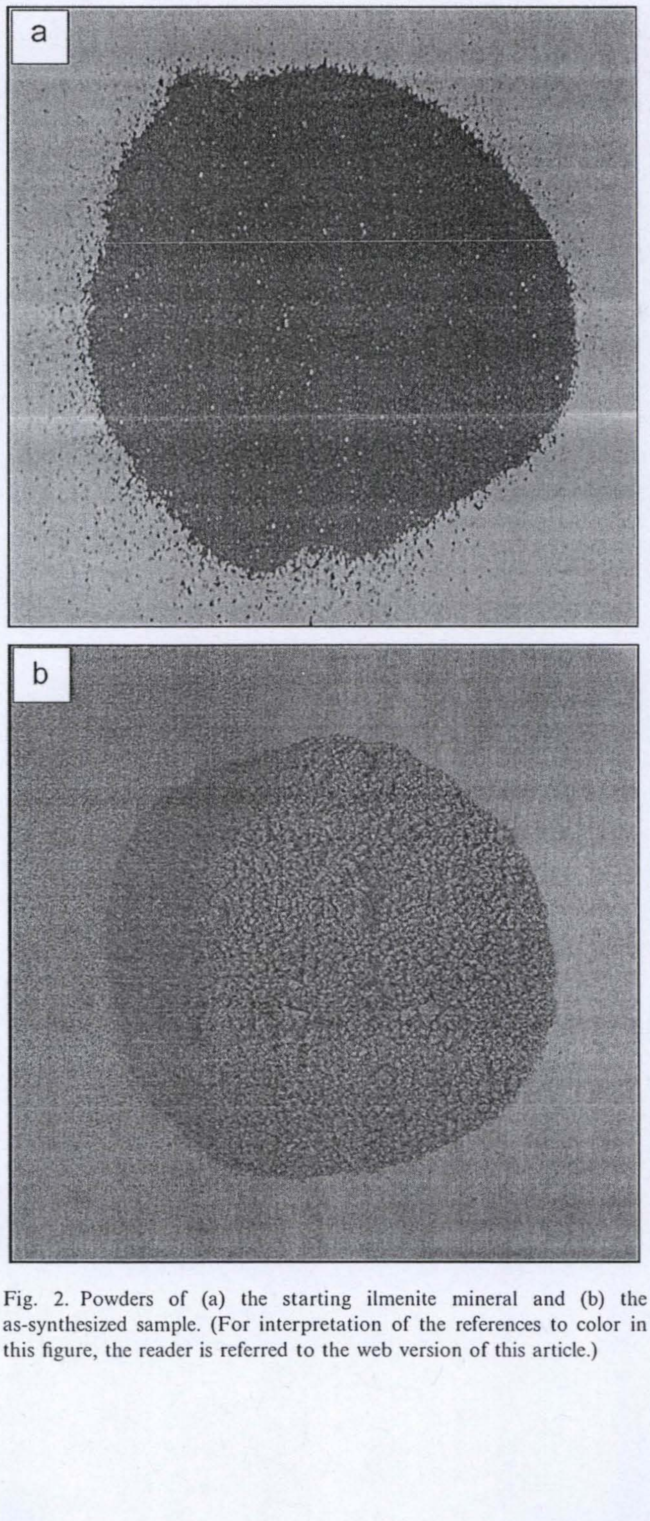

Fig. 2. Powders of (a) the starting ilmenite mineral and (b) the as-synthesized sample. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

Table 1
Chemical composition of the ilmenite mineral and an as-synthesized sample.

Oxide	Ilmenite mineral (wt%)	As-synthesized sample (wt%)
TiO_2	66.99	76.21
Fe_2O_3	24.01	21.80
Al_2O_3	3.38	0.12
SiO_2	2.11	0.30
MnO	0.82	0.68
ThO_2	0.64	0.01
ZrO_2	0.62	0.12
MgO	0.27	0.09
Cr_2O_3	0.26	< 0.01
P_2O_5	0.25	< 0.01
SO_3	0.15	0.05
Y_2O_3	0.09	–
ZnO	0.21	< 0.01
Nb_2O_5	0.24	0.15
CaO	0.16	0.08

indicates that a large portion of Fe impurities were removed by the NaOH (aq.) hydrothermal treatment and the neutralization/washing processes [38]. The chemical compositions of the ilmenite mineral and of the as-synthesized samples found using X-ray fluorescence are shown in Table 1. During the hydrothermal process, the quantities of impurities, such as Fe_2O_3, Al_2O_3, SiO_2, and MnO, decreased while the TiO_2 content increased from 66.99 to 76.21 wt% This may be due to higher solubility of the impurities in the NaOH and HCl aqueous solutions during the preparation process [46,47]. The doping of Fe^{3+} in the nanofiber matrix leads to a significant red shift in the optical response toward the visible spectrum caused by a reduction in the band gap energy [48], resulting in the brown-color of the as-synthesized samples. The nanofibers doped with Fe^{3+} could be an alternative, economically efficient material used as a photocatalyst in hydrogen production, dye-sensitized solar cells and the decomposition of organic dyes.

The XRD patterns of the starting ilmenite mineral and the as-synthesized sample are shown in Fig. 3. The crystalline structure of the starting ilmenite mineral appears to be of the rutile phase, while the crystalline structure of the as synthesized nanofibers demonstrated a layered titanate $H_2Ti_xO_{2x+1}$ structure, most likely trititanate ($H_2Ti_3O_7$), indicating the existence of hydrogen in the prepared nanofibers [38–41]. No diffraction peaks of other impurities (such as starting rutile and NaCl) are observed. This result is due to the reduction of the Na content in the nanofibers from repeated HCl leaching and water washes [36,49]. However, when compared with titanate nanotubes, the nanofibers contain more residual Na ions under the same ion exchanging conditions because of the geometry of the nanofibers, i.e., their solid and thicker structure. In addition, alkali-metal hexatitanates ($A_2Ti_6O_{13}$, A = Na, K, and Rb) tend to form stable solid fibrous structures that do not leach out easily during aqueous HCl treatments at

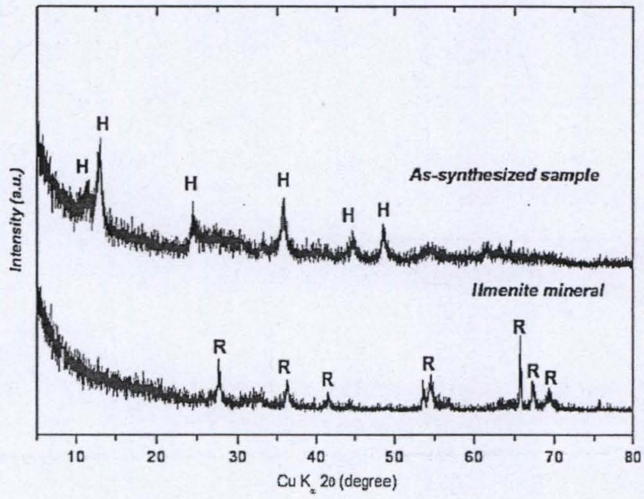

Fig. 3. XRD patterns of the starting ilmenite mineral and the as-synthesized sample, H = hydrogen titanate and R = rutile TiO_2.

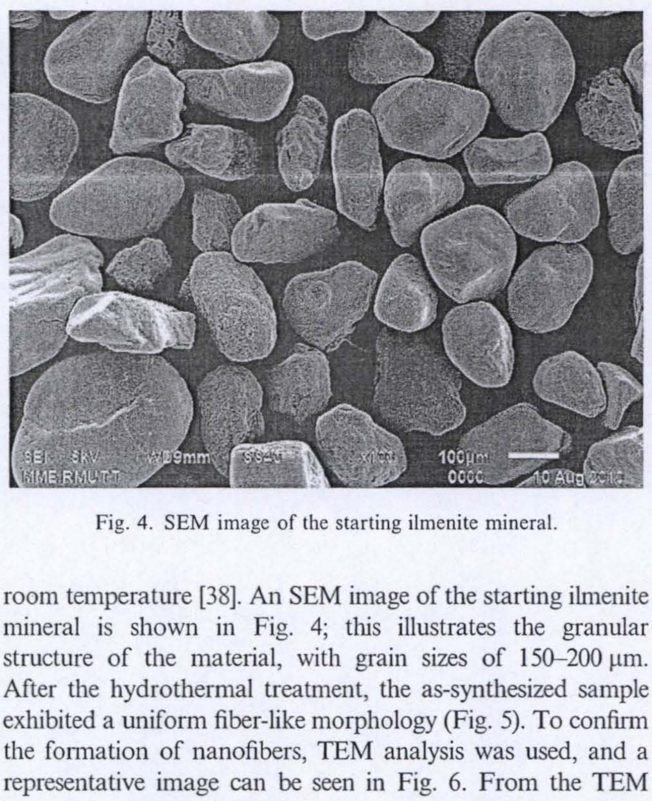

Fig. 4. SEM image of the starting ilmenite mineral.

room temperature [38]. An SEM image of the starting ilmenite mineral is shown in Fig. 4; this illustrates the granular structure of the material, with grain sizes of 150–200 µm. After the hydrothermal treatment, the as-synthesized sample exhibited a uniform fiber-like morphology (Fig. 5). To confirm the formation of nanofibers, TEM analysis was used, and a representative image can be seen in Fig. 6. From the TEM images, it can be observed that the as-synthesized nanofibers are solid rather than hollow.

The nanofibers tend to form bundles; thus some of the nanofibers look thicker than others. The prepared nanofibers had lengths from 2 to 7 µm with diameters of 20–90 nm (Fig. 6). The nanofiber formation can be explained by the coarseness of the ilmenite granules, which retarded their dissolution in the NaOH solution, suppressing nucleation and assisting preferential crystal growth along the 010 direction of the trititanate [38]. The diameters (Fig. 7) of the as-prepared nanofibers were found to

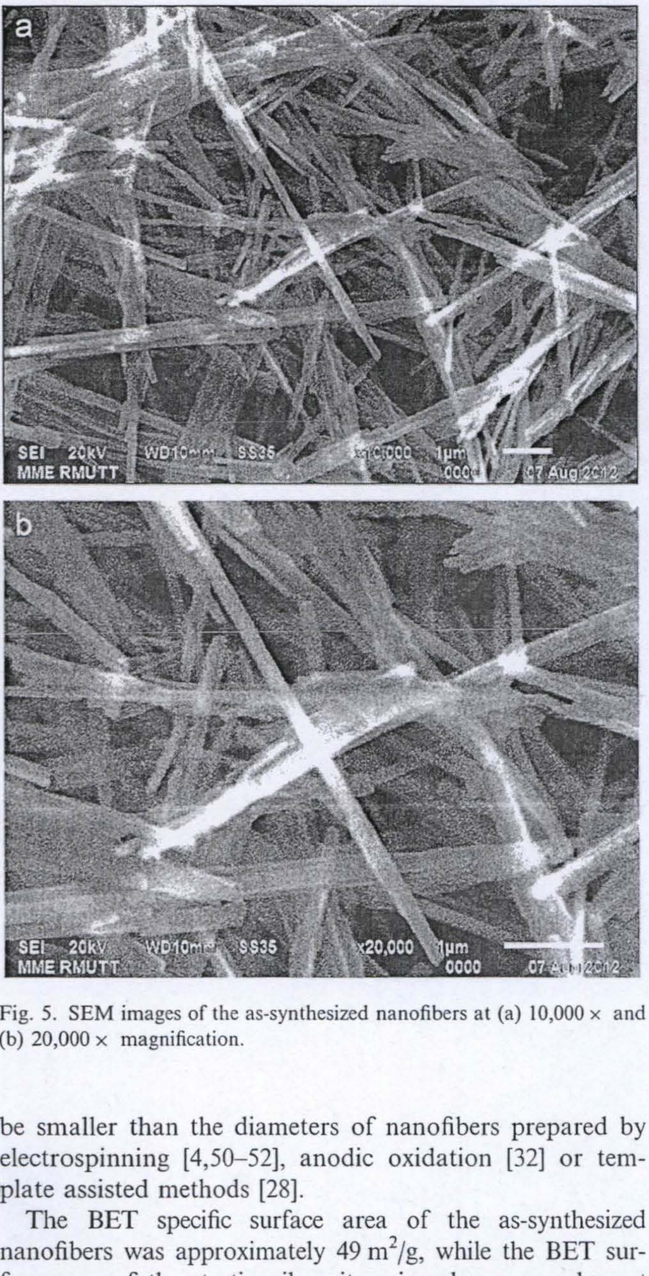

Fig. 5. SEM images of the as-synthesized nanofibers at (a) 10,000 × and (b) 20,000 × magnification.

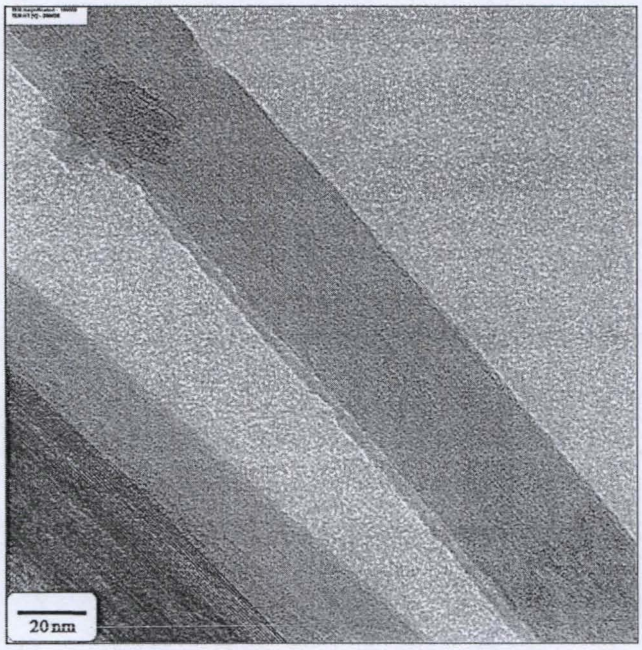

Fig. 6. TEM image of the as-synthesized nanofibers at 100,000 × magnification.

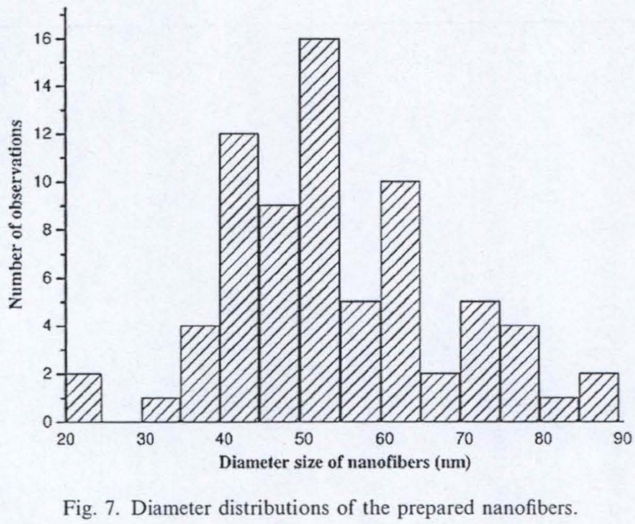

Fig. 7. Diameter distributions of the prepared nanofibers.

be smaller than the diameters of nanofibers prepared by electrospinning [4,50–52], anodic oxidation [32] or template assisted methods [28].

The BET specific surface area of the as-synthesized nanofibers was approximately 49 m^2/g, while the BET surface area of the starting ilmenite mineral was very low at approximately 0 m^2/g (Table 2). The BET specific surface area of the starting ilmenite mineral was similar to that of leucoxene [41] and rutile minerals [38,39]. The increase in the BET specific surface area is a result of the starting ilmenite mineral being completely converted into hydrogen titanate nanofibers during the hydrothermal process. Although the nanotube structure is attractive due to its high surface area, titanate nanotubes with free-alkali ions are usually unstable at high temperatures (at $\sim$500 °C) and convert into anatase particles [36,39,53,54]. To maintain 1-D nanostructures at higher temperature (typically at 500–800 °C), solid nanowires or nanofibers forms should be more favorable [36,39,53,54]. The absorption spectra of the as-synthesized nanofibers and commercially available nanostructured TiO_2 (ST-01) are illustrated in Fig. 8. The absorption spectra of the as-synthesized nanofibers exhibit a significant enhancement in the wavelength region of 300–500 nm due to the natural Fe-doping characteristic of the ilmenite mineral. Further studies on the synthesis and characterization of this material are currently being performed.

4. Conclusion

In summary, titanate nanofibers are synthesized by a hydrothermal method using a low-cost ilmenite mineral as the starting material. After the hydrothermal synthesis, solid nanofibers showed an increased TiO_2 content were obtained. Analysis of the crystalline structure of the

Table 2
The BET specific surface area of the starting ilmenite mineral and the as-synthesized nanofibers.

Samples	Bet surface area (m^2/g)
Starting ilmenite mineral	0
As-synthesized	~49

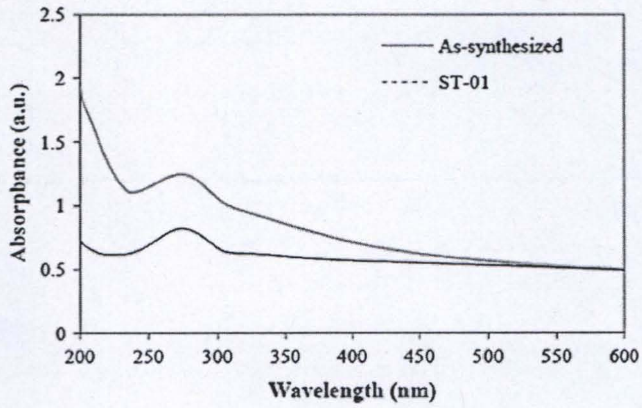

Fig. 8. UV–vis absorbance spectra of the as-synthesized nanofibers and commercial grade TiO_2 nanoparticles (ST-01).

as-synthesized nanofibers demonstrated a layered titanate $H_2Ti_xO_{2x+1}$ structure, most likely in the form of trititanate ($H_2Ti_3O_7$). The prepared nanofibers showed lengths of 2–7 μm with diameters of approximately 20–90 nm and a corresponding BET specific surface area of approximately 49 m^2/g. These Fe^{3+} doped nanofibers may show utility as a novel photocatalyst material for hydrogen production, dye-sensitized solar cells and the decomposition of organic dyes.

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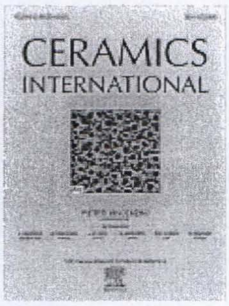

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