10th Eco-Energy and Materials Science and Engineering Symposium


On December 5-8, 2012
Suenee grand hotel,
Ubon-ratchathani

Organized by

Co-organized by
PREFACE:
Message from the President of Rajamangala University of Technology Thanyaburi

Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is please to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This international conference is not only giving an opportunity for Thai and foreign researchers to present and discussion their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Our program consists of six research tasks: (1) Energy Technology, (2) Environmental and Social Impact, (3) Nanotechnology and Materials Science, (4) Energy Economics and Management, (5) New Energy Technology and (6) Nuclear Technology.

I would like to take this opportunity to express our sincere gratitude to our two distinguished Plenary Speakers for kindly accepting our invitation. I deeply appreciate of the very strong support given by Kyoto University. Thanks to the tireless works of the Organizing Committee, the Technical Program Committee, the invited speakers and paper and poster contributors, and excellent program been assembled to cover a broad spectrum of interesting topic.

We warmly welcome you to the 10th EMSES on December 5-8, 2012, Ubon Ratchathani, Thailand.

Numyoot SONGTHANAPITAK, Ph.D.
President of Rajamangala University of Technology Thanyaburi and Conference Chairman of 10th EMSES 2012
PREFACE:
Message from the Director of
Institute of Advanced Energy, Kyoto University

It is my great pleasure to have the 10th Eco-Energy and Materials Science and Engineering Symposium (EMSES) with Rajamangala University of Technology Thanyaburi (RMUTT) under the long-term collaboration between RMUTT and Kyoto University. The 1st EMSES was held in 2001 in Thailand and the symposium has been expanded in its scientific contents as well as the academic network. I believe that the 10th EMSES gives a good opportunity to all participants to exchange their knowledge and idea to realize eco-friendly energy system in society. I would like to express my welcome to all participants and sincere thanks to the 10th EMSES organizing committee and all supporting organizations to make us having this symposium. I hope that the symposium will be successful and lead to further progress in energy science and technology and also in friendships of participants.

Professor Yukio Ogata, Ph.D.
Director of Institute of Advanced Energy, Kyoto University
PREFACE:
Message from the Former Dean of
Graduate School of Energy Science, Kyoto University
Program Leader,
Global COE “Energy Science in the Age of Global Warming”

I want to express my hearty welcome to all participants of Eco-Energy and Materials Science and Engineering Symposium (10th EMSES). This symposium is aiming the realization of importance of energy and materials technology through the academic, science and technology network among the world communities. The symposium gives an opportunity for researchers to discuss their research works and also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the further cooperation work will be developed.
I would like also extend my sincere thanks to all who made the meeting possible, including the 10th EMSES organizers, the SEE forum committee members, and the Japanese Government, JSPS, for their kind support. I am looking forward to seeing you in Ubon Ratchathani, Thailand.

Professor Takeshi YAO, Ph.D.
Former Dean of Graduate School of Energy Science, Kyoto University
and Program Leader, Global COE “Energy Science in the Age of Global Warming”
Rajamangala University of Technology Thanyaburi (RMUTT), in conjunction with Kyoto University, is pleased to host the 10th Eco-Energy and Materials Science and Engineering Symposium (10th EMSES).

RMUTT has a major mission on encouraging and supporting all areas of research. One of the key reasons is to assist in developing capability in science and technology in order to cope with recent rapid change in this field. We have jointly set up an academic symposium on the 10th EMSES with the perception on the significance of exchanging knowledge and research experiences between researcher in the field of energy, materials technology and environmental science. This symposium is not only giving an opportunity for Thai and foreign researcher to present and discuss their research works and update their expertise but also to initially stimulate the development of research works on eco-energy and materials science and engineering. Once the cooperation among researchers has been created, the closer future cooperation incorporate with joint-research works will be developed. Thus, to support the aforesaid role, the symposium working committee would like to invite you to participate in this academic symposium.

I would like to express our sincere thanks to the organizing committee, participants and contributors for your kind corporation to this symposium. I wish this symposium proceeding will be a useful reference for future scientific research development.

Sommai PIVSA-ART, Ph.D.
Dean of Faculty of Engineering, RMUTT
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Preparation of Nanotubes from Natural Ilmenite Mineral by Hydrothermal Method

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Abstract—Titanate nanotubes have been successfully prepared by adopting ilmenite mineral and sodium hydroxide (NaOH) as the starting material via the simple hydrothermal method at 105 °C for 24 h using Thai autoclave unit. The chemical composition, shape, size, crystalline structures and specific surface area of the as-prepared samples were characterized by x-ray fluorescence (XRF), scanning electron microscopy (SEM), transmission electron microscopy (TEM), x-ray diffraction (XRD), and Brunauer-Emmett-Teller (BET) surface area. The prepared titanate nanotubes had an average inner diameter around 6-8 nm and outer diameter around 10-20 nm. The length of the prepared titanate nanotubes was approximately 0.1-0.5 μm. The BET specific surface area and pore volume of the prepared titanate nanotubes were about 96.198 m²/g and 0.990 cm³/g, respectively. This preparation method provides a simple route to fabricate nanotubes with low-cost mineral using Thai autoclave unit.

Keywords—Nanotubes, Ilmenite mineral, Hydrothermal, Titanate, TiO₂.

1. INTRODUCTION

Titanium dioxide (TiO₂) is one of the most important materials. TiO₂ as n-type II-VI compound semiconductor with a wide direct-band gap of 3.2 eV, has attracted more and more attention over the past few years because it can fine application in various fields, such as a semiconductor in dye-sensitized solar cell, water treatment materials, catalysts, transducers gas sensor, transparent high power electronics, piezoelectric transducers, and so on [1-7]. However, in order to obtain TiO₂ powders with appropriate chemical, electrical and optical properties specific for their intended applications, purity and particle size during their synthesizing process is important. Up to now, TiO₂ is known to exist in three natural polymorphs, i.e., rutile, anatase, and brookite, with different properties and so three, had been prepared via different synthesis method or under different preparation conditions [8]. Different routes such as sol-gel, electrodeposition, electrospinning and hydrothermal process have been utilized for preparing TiO₂ powder [9-11]. The hydrothermal synthetic route has advantages to obtain high-crystallized powders with narrow grain size-distribution and high purity without the expensive precursors, elaborate apparatus and heat treatment at low temperature [9-16].

This study was aimed at the preparation of nanotubes by hydrothermal method using inexpensive-natural ilmenite mineral as the starting material. The use of ilmenite mineral in this study is < US$ 1/kg, which is 1/50 - 1/100 of other works [30]. The characterization of the prepared nanotubes will be reported.

2. EXPERIMENTAL PROCEDURE

2.1 Synthesis

The hydrothermal method was basically similar to that in the previous reports for preparation nanofibers, nanotubes and nanowires [17-33]. In the typical manner, 8 gram natural ilmenite granules 90-250 μm (Fig. 1) was added into NaOH aqueous solution (10 M, 2,000 ml). Then, the solution was stirred at room temperature for 5 min. After kept stirring, the solution was put into a teflon-lined stainless steel autoclave (Fig. 2). The autoclave was heated at 105 °C for 24 h with stirring condition. After the autoclave was allowed to cool to room temperature. There existed some redder membrane-like float on the NaOH solution and brownish hard precipitate at the bottom of the vessel, which were not observed for the hydrothermal process using pure TiO₂ source [26-27]. The float and precipitate, presumably caused by the impurities in natural ilmenite, can be easily separated from the main product, i.e., This phenomenon implies the hydrothermal treatment for natural rutile may be used not only as a reaction step but also as a purification step. 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 Figure 3.

2.2 Characterization

The chemical composition of the ilmenite mineral was analyzed by x-ray fluorescence (XRF, PW-2404, Philips, Netherlands). The shape and size of the prepared samples were observed by scanning electron microscope (SEM, JSM-6510, JEOL, Japan) and transmission electron microscope (TEM, JEM-2100, JEOL, Japan). The phase and crystallinity of the samples were characterized by x-ray diffraction (XRD, X'Pert PRO MPD, PANalytical, Netherlands). The Brunauer-Emmett-Teller (BET, BELSORP-Mini, Rubotherm) specific surface area and pore structure of the samples were characterized by the nitrogen adsorption-desorption isotherm.

This work was supported by National Research Council of Thailand (NRCT).
2.3 Photocatalytic activity measurement

The photocatalytic activity was measured through the formation rate of \( I_3^- \) due to the oxidation photo reaction of \( I^- \) to \( I_2 \) in excess \( I^- \) conditions [6, 34]. A reaction system was set up by adding 50 mg of a sample powder into 10 ml of 0.2M of potassium iodide (KI) aqueous solution then stirred and irradiated with UV light with a maximum emission at about 365 nm at room temperature. After the irradiation of 15, 30, 45, and 60 min, the suspension was withdrawn and centrifuged. After the clear supernatant was diluted 10 times, the concentration of liberated \( I_3^- \) ions was monitored by the absorbance at 288 nm, using an UV–vis spectrophotometer (Shimadzu UV 2450). The molar extinction coefficient was determined to be \( 4.0 \times 10^4 \) (cm mol/l)\(^{-1}\). For reference, four different commercially available nanoparticles \( \text{TiO}_2 \) powders such as P-25 (Nippon Aerosil Co., Ltd., Japan), JRC-01, JRC-03 (The Catalysis Society of Japan), and white pigment \( \text{TiO}_2 \) were tested.

![Fig. 1 SEM image of natural ilmenite granules with the size of 90-250 µm](image)

![Fig. 2 Teflon-lined stainless steel autoclave unit.](image)

Fig. 3 Schematic representation of the hydrothermal method of the nanotubes preparation.

3. RESULTS AND DISCUSSION

The as-synthesized sample was brown-colored while as the starting ilmenite mineral was black-colored (Fig. 4).

![Fig. 4 (a) Photo images of the natural ilmenite mineral and (b) the as-synthesized sample.](image)
This result indicates that a large portion of Fe impurities were removed by the NaOH (aq.) hydrothermal treatment and the neutralization/washing processes [30]. 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 Fe₂O₃, Al₂O₃, SiO₂, and MnO, decreased while the TiO₂ content increased from 66.99 to 70.58%wt. This may be due to higher solubility of the impurities in the NaOH and HCl aqueous solutions during the preparation process [35-36]. The doping of Fe³⁺ in the nanotubes matrix leads to a significant red shift in the optical response towards the visible spectrum caused by a reduction in the band gap energy [37], resulting in the brown-color of the as-synthesized samples. The nanotubes doped with Fe³⁺ could be an alternative, economically efficient material used as a photocatalyst in hydrogen and biogas production, dye-sensitized solar cells, water purification and decomposition of organic dyes.

Table 1 Chemical analysis of the natural ilmenite mineral and the as-synthesized sample.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Ilmenite mineral (%wt)</th>
<th>As-synthesized sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>TiO₂</td>
<td>66.99</td>
<td>70.58</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>24.01</td>
<td>21.17</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.38</td>
<td>0.43</td>
</tr>
<tr>
<td>SiO₂</td>
<td>2.11</td>
<td>0.46</td>
</tr>
<tr>
<td>MnO</td>
<td>0.82</td>
<td>0.86</td>
</tr>
<tr>
<td>ThO₂</td>
<td>0.64</td>
<td>0.04</td>
</tr>
<tr>
<td>ZrO₂</td>
<td>0.62</td>
<td>0.37</td>
</tr>
<tr>
<td>MgO</td>
<td>0.27</td>
<td>0.11</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>0.26</td>
<td>0.20</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>0.25</td>
<td>0.05</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.15</td>
<td>0.03</td>
</tr>
<tr>
<td>Y₂O₃</td>
<td>0.09</td>
<td>0.05</td>
</tr>
<tr>
<td>ZnO</td>
<td>&lt;0.01</td>
<td>0.07</td>
</tr>
<tr>
<td>Nb₂O₅</td>
<td>0.24</td>
<td>0.11</td>
</tr>
<tr>
<td>CaO</td>
<td>0.16</td>
<td>0.26</td>
</tr>
<tr>
<td>Na₂O</td>
<td>&lt;0.01</td>
<td>4.38</td>
</tr>
</tbody>
</table>

Fig. 5 shows the X-ray diffraction pattern of the prepared samples. The peaks of starting ilmenite mineral were rather sharp, which indicated the rutile phase (Fig. 5a). After synthesized at 105 °C for 24 h the sample showed titanate phase (H₂Ti₃O₇−x−y) probably trititanate (H₂Ti₃O₇), indicating the hydrogen (from water) in the prepared nanotubes [8, 30, 31, 38]. TiO₂ crystals become amorphous by the Ti-O is broken and form to the Ti-O-Na or Ti-OH bonds following treatment in aqueous NaOH, and nanotubes titanate are generated after the treatment of TiO₂ acidic solution and water. In addition, the layers of titanate nanotubes are formed depending on the synthesis conditions and residual Na which could be H₂Ti₂O₇ or Na₂H₂Ti₃O₇ and no crystallization of contaminants such as sodium chloride (NaCl) and rutile (Fig. 5b). In addition, it is expected that a new type of titanate nanotube having new properties will be formed by controlling the amount of residual Na⁺ ions and by replacing residual Na⁺ ions with other ions [39].

Fig. 5 XRD patterns of (a) the natural ilmenite mineral and (b) the as-synthesized sample.

Fig. 6 SEM images of the as-synthesized nanotubes at (a) × 5,000 magnified (b) × 10,000 magnified and (c) × 20,000 magnified.
was used, and the images can be seen in Fig. 7 at difference magnified. From the TEM images, it can be observed that the as-synthesized sample showed tubular structure. The prepared nanotubes had the lengths from 0.1 - 0.5 μm with inner and outer diameter of 6-8 and 10-20 nm, respectively (Fig. 7). The nanotubes formation can be explained as follows: Firstly, the crystallites are exfoliated into layered crystalline sheets when treated the raw ilmenite mineral in NaOH aqueous solution at 105 °C, and then the single sheets formed along with the (010) lattice planes and paralleled to the sheet surfaces. Secondly, the sheets gradually rolled up to reduce the number of surface dangling bonds and decrease system energy (both sides of these single-layer sheets have many dangling bonds that should be saturated in the solution). As a result, the single sheet rolled up into tubular shape. This process is in accord with the reported of B.D. Yao et al. [17-18, 40].

The BET specific surface area of the as-synthesized nanotubes was approximately 96.198 m²/g, while the BET surface area of the starting ilmenite mineral was very low at approximately 0 m²/g (Table 2). The BET specific surface area of the starting ilmenite mineral was similar to that of leucoxene [8] and rutile minerals [30-31]. The increasing in the BET specific surface area is a result of the starting ilmenite mineral being completely converted into hydrogen titanate nanotubes during the hydrothermal process [17-22].

![Fig. 8 N₂ adsorption–desorption isotherms of the as-synthesized sample.](image)

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

<table>
<thead>
<tr>
<th>Samples</th>
<th>BET surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ilmenite mineral</td>
<td>~ 0</td>
</tr>
<tr>
<td>Nanofibers titanate</td>
<td>49</td>
</tr>
<tr>
<td>Nanotubes titanate</td>
<td>96</td>
</tr>
<tr>
<td>Commercial TiO₂ (P25)</td>
<td>50</td>
</tr>
</tbody>
</table>

Fig. 8 shows the N₂ adsorption isotherms of the as-prepared samples which is a typical IV isotherm according to the IUPAC classification [42, 43]. An absorption-desorption hysteresis loop is observed at $P/P_0$.
\[ \approx 0.65, \text{ indicating capillary condensation of the liquid N}_2 \text{ inside the samples. The pore volume from the total amount absorbed at related pressures is } 0.5531 \text{ cm}^3\text{g}^{-1}. \]

![Graph showing photocatalytic activity (Iᵢ₃ concentration) of the as-synthesized nanotubes, the calcined nanotubes, commercial TiO₂ nanoparticles (P-25, JRC-01, JRC-03 and White pigment)](image)

Fig. 9 Photocatalytic activity (Iᵢ₃ concentration) of the as-synthesized nanotubes, the calcined nanotubes, commercial TiO₂ nanoparticles (P-25, JRC-01, JRC-03 and White pigment)

The photocatalytic activity (Iᵢ₃ concentration) of the as-synthesized nanotubes, nanotubes calcined at 300 °C for 2 h and commercially grade nanoparticles TiO₂ P-25, JRC-01, JRC-03 and white pigment is shown in Fig. 9. It was found that the photocatalytic activity of the as-synthesized nanotubes almost equal to P-25 but higher than JRC-01 and JRC-03, because of large specific surface area of the as-synthesized nanotubes [8], and the photocatalytic activity was almost proportionnal to the BET specific surface area. However the nanotubes calcined at 300 °C was higher than these, (Iᵢ₃ concentration) of as-synthesized, nanofibers form ilmenite mineral, commercially grade nanoparticles TiO₂ i.e., P-25, JRC-01, JRC-03 and white pigment due to the high specific surface area with TiO₂ (B) structure [8, 28].

4. CONCLUSION

In summary, titanian nanotubes were synthesized by a hydrothermal method using a low-cost natural ilmenite mineral as the starting material. After the hydrothermal treatment, the as-synthesized sample exhibited a uniform tubular-like morphology and showing an increased TiO₂ content were obtained. Analysis of the crystalline structure of the as-synthesized nanotubes demonstrated a layered titanian H₂Ti₄O₁₁ structure, most likely in the form of trittitanate (H₂Ti₄O₁₁). The prepared nanotubes showed lengths of 0.1-0.5 μm with inner and outer diameter of 6-8 and 10-20 nm, respectively, and a corresponding BET specific surface area of approximately 96.198 m²/g. These Fe⁺⁺ doped nanotubes may show utility as a novel photocatalyst material for hydrogen production, dye-sensitized solar cells, transducers gas sensor, water treatment catalysts, catalyst and the decomposition of organic dyes.

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