

# Preparation of High Photocatalyst Mesoporous TiO<sub>2</sub> from Nanosheets Using Autoclave Unit (Thai Made)

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**Abstract**— The aim of this study is to prepare mesoporous TiO<sub>2</sub> from high specific surface area nanosheets by simple hydrothermal method at 120 °C of 12 hrs using autoclave unit (Thai made). The shape, size (TEM), crystalline structure (XRD), BET-specific surface area and photocatalytic activity of the prepared samples were investigated. The XRD results revealed that the prepared nanosheets were amorphous phase. The specific surface area, average pore diameter and pore volume were 360.28 m<sup>2</sup>/g, 3-5 nm and 0.275 cm<sup>3</sup>/g, respectively. The crystalline structure of calcined nanosheets at 300-600 °C was anatase TiO<sub>2</sub> with decreasing in the specific surface area. The intensity of anatase TiO<sub>2</sub> structure increased when the calcination temperature was increased. Moreover, the photocatalytic activity of the calcined nanosheets was higher than the as-synthesized nanosheets and commercial nano TiO<sub>2</sub> (P-25, JRC-01, JRC-03). This preparation method provided a simple route to fabricate nanosheets using autoclave unit (Thai made).

**Keywords**— Hydrothermal, Nanosheet, Photocatalytic Activity, Titanium Dioxide

## 1. INTRODUCTION

Titania materials or TiO<sub>2</sub> and TiO<sub>2</sub>-related materials are of importance for utilizing solar energy and environmental purification. TiO<sub>2</sub> has been widely used for various applications such as a semiconductor in dye-sensitized solar cell, water treatment materials, catalysts and gas sensors [1-6].

From the earlier study exhibited two types only of crystalline (rutile and anatase) measured in photocatalytic, the rutile was not adequate in order to catalyst because high recombination of electron and hole than anatase as a result the photocatalytic activity was poor [7-14]. In our previous work, high specific surface area nanosheets were synthesized via hydrothermal method [15].

In this study, the preparation of anatase mesoporous TiO<sub>2</sub> from high specific surface area nanosheets by hydrothermal method using Teflon-lined stainless steel autoclave (Thai made) was investigated. The shape, size, crystalline structure, BET-specific surface area and photocatalytic activity of the prepared samples were characterized.

## 2. EXPERIMENTAL PRECEDURE

### 2.1 Preparation of nanosheets

Nanosheets were prepared via hydrothermal method using titanium (IV) butoxide mixing with acetylacetone in a Teflon-lined stainless steel autoclave that was designed and built of Rajamangala University of Technology Thanyaburi (RMUTT) (Fig.1). Distilled water (80 ml) and NH<sub>3</sub>OH were added into the solution and stirred about 5 minutes, then the autoclave was heated at 120 °C and stirring for about

12 hrs. After that, the mixture was cooled at room temperature. In the last, the product was filtered and washed by the 0.1 M of HCl and the distilled water for several times.

The synthetic material was dried in the oven at 100 °C for 12 hrs. The prepared sample was heated at 300, 400, 500, 600, 700 and 800 °C for 2 hrs.

### 2.2 Characterizations

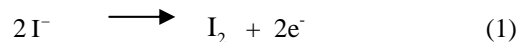
The crystalline structure of the samples was evaluated by X-ray diffraction (XRD, X'Pert PRO MPD model pw 3040/60, PANalytical). The microstructure of the prepared materials was analyzed by transmission electron microscopy (TEM, JEM-2100, JEOL). The Brunauer-Emmett-Teller (BET, BELSORP-Mini, Rubotherm) specific surface area was determined by the nitrogen adsorption (BELSORP-Mini, Rubotherm).



Fig.1. Teflon-lined stainless steel autoclave.

### 2.3 Photocatalytic activity measurement

Photocatalytic activity was measured the concentration of I<sub>3</sub><sup>-</sup> that generated from photo-oxidation reaction of I which transformed into I<sub>2</sub> in excess of I condition [16] following Eqs. (1) and (2).



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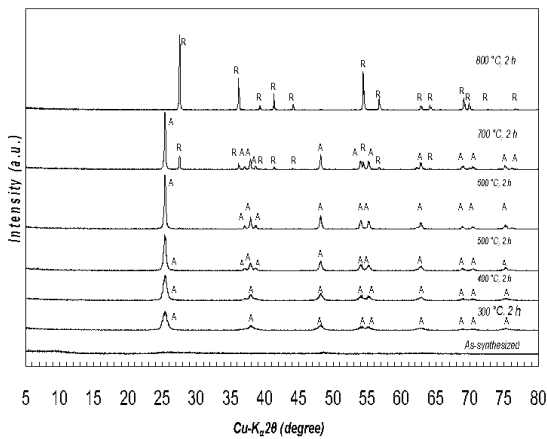
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The 50 mg of TiO<sub>2</sub> powders and potassium iodide solution was filled into a cylindrical vessel. After that, it was placed on obscure condition, and 15 W of UV light was illuminated with stirring condition at room temperature for 1 hour after that the solution was separated by centrifuge method and it was diluted for 10 times order to measured of ion by light absorption of 288 nm using UV-vis spectrometer, the coefficient of the intensity from the experimental was 4.0x10<sup>4</sup> cm mol/l.

### 3. RESULT AND DISCUSSION

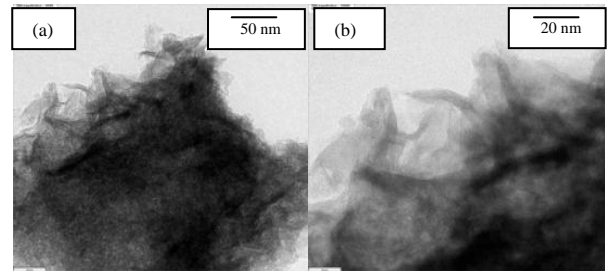
#### 3.1 Characterization

The XRD pattern of the prepared sample showed amorphous-like structure (Fig. 2). The calcined samples of 300-600 °C of 2 hrs were anatase phase. The anatase TiO<sub>2</sub> structure increased when the calcination temperature was increased. The peaks were rather sharp, which indicated the calcined nanosheet TiO<sub>2</sub> had relatively high crystallinity. The peaks corresponding to rutile TiO<sub>2</sub> appeared at 700 °C and almost showed rutile TiO<sub>2</sub> structure at 800 °C [17].

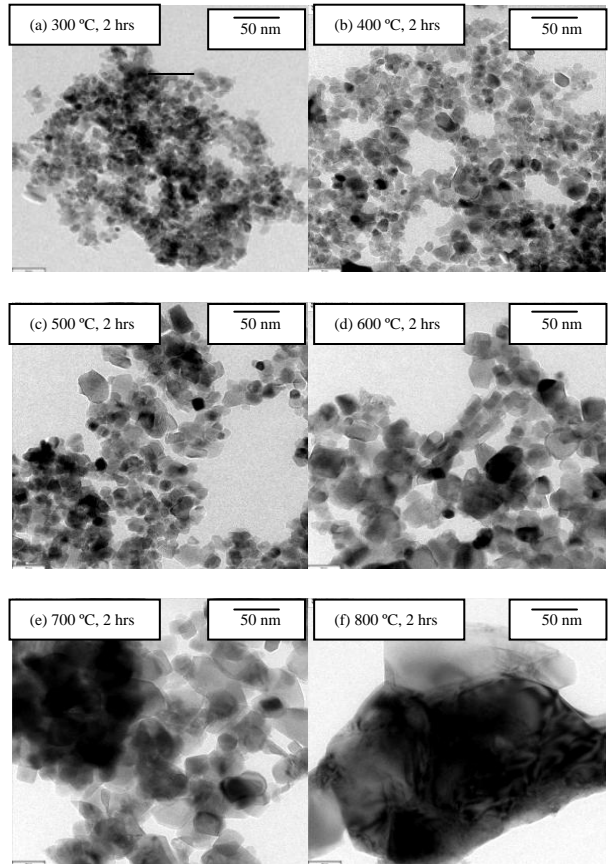


**Fig.2. The XRD patterns of as-synthesized nanosheets and calcined nanosheets at various temperatures (A: anatase and R: rutile).**

The BET specific surface area, pore diameter and pore volume of nanosheet were about 360.28 m<sup>2</sup>/g, 3-5 nm and 0.275 cm<sup>3</sup>/g, respectively. The result of microstructure showed that the as-synthesized sample was nanosheets-like structure. The prepared nanosheets were roll, twist and agglomerate (fig.3). The nanosheets transformed to nanoparticles (fig. 4 (a)) when heated at 300-800 °C. The increasing of calcination temperatures, indicating the grain growth of TiO<sub>2</sub> crystallites [18] (fig. 4 (b)-(f)). The BET specific surface area of the calcined nanosheets at 300, 400 and 500 °C were about 108.93, 71.24 and 46.77 m<sup>2</sup>/g, respectively. The BET specific surface area decreased with increasing calcination temperature, resulting in the increasing of pore diameter and decreasing of pore volume, became the nanosheets structure after calcinations were destroyed and changed to nanoparticles composite at high temperature [15, 19-20].



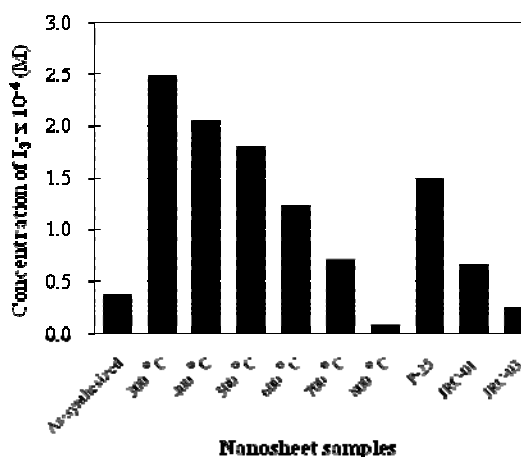
**Fig.3. TEM images of as-synthesized nanosheets TiO<sub>2</sub> (a) x 40,000 and (b) x 100,000 magnified.**



**Fig.4. TEM images of the nanosheets TiO<sub>2</sub> calcined at various temperatures for 2 hrs (a) 300 °C, (b) 400 °C, (e) 500 °C, (d) 600 °C, (e) 700 °C and (f) 800 °C.**

#### 3.2 Photocatalytic activity

The calcined nanosheets TiO<sub>2</sub> (excepted at 800 °C) showed higher I<sub>3</sub><sup>-</sup> concentration than the as-synthesized sample. The calcined nanosheets TiO<sub>2</sub> at 300 °C for 2 hrs showed the highest activity and also higher than commercial nanoparticle TiO<sub>2</sub> samples (P-25, JRC-01, JRC-03) due to the exist of mesoporous structure with high specific surface area and anatase phase [7, 18, 21].



**Fig.5. Photocatalytic activity (I<sub>3</sub><sup>-</sup> concentration) of the as-synthesized nanosheets, the calcined nanosheets and commercial nano TiO<sub>2</sub> samples (P-25, JRC-01, JRC-03).**

## 6. CONCLUSION

Nanosheet TiO<sub>2</sub> was prepared by hydrothermal method at 120 °C for 12 hrs with a Teflon-lined stainless steel autoclave (Thai made). The as-synthesized nanosheets TiO<sub>2</sub> showed amorphous structure. The BET specific surface area, pore diameter and pore volume of the nanosheets TiO<sub>2</sub> were about 360.28 m<sup>2</sup>/g, 3 nm and 0.275 cm<sup>3</sup>/g, respectively. The calcined nanosheets had higher photocatalytic activity than the as-synthesized sample and the commercial nano TiO<sub>2</sub> powder (P-25, JRC-01, JRC-03). This preparation method provided a simple route to fabricate nanostructures TiO<sub>2</sub> with high photocatalytic activity.

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