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Preparation of High Photocatalyst Mesoporous TiO_2 from Nanosheets Using Autoclave Unit (Thai Made)

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Abstract

The aim of this study is to prepare mesoporous TiO_2 from high specific surface area nanosheets by simple hydrothermal method at 120 °C of 12 h using autoclave unit (Thai made). The shape, size by TEM, crystalline structure by XRD, BET-specific surface area and photocatalytic activity of the prepared samples were investigated. The XRD result revealed that the prepared nanosheets were amorphous phase. The specific surface area, average pore diameter and pore volume were 360.28 m^2/g, 3-5 nm and 0.275 cm^3/g, respectively. The crystalline structure of calcined nanosheets at 300-600 °C was anatase TiO_2 with decreasing in the specific surface area. The intensity of anatase TiO_2 structure increased when the calcination temperature was increased. Moreover, the photocatalytic activity of the calcined nanosheets was higher than those of the as-synthesized nanosheets and commercial nano TiO_2 (P-25, JRC-01, JRC-03). This preparation method provided a simple routh to fabricate high photocatalyst mesoporous TiO_2 using autoclave unit (Thai made).

Keyword: Hydrothermal; Nanosheets; Photocatalytic activity; Titanium dioxide

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1. Introduction

The titania materials (TiO_2) and TiO_2-related materials are of importance for utilizing solar energy and environmental purification. TiO_2 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_2 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 Procedure

2.1. Preparation of nanosheets

Nanosheets were prepared via hydrothermal method using titanium (IV) butoxide mixing with acetylacetone in autoclave. Teflon-lined stainless steel autoclave was designed and built of Rajamangala University of Technology Thanyaburi (RMUTT) (Fig. 1). Distilled water (80 ml) and NH_3OH were added into the solution and stirred about 5 minutes, then the autoclave was heated at 120 °C and stirring for about 12 h. 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 h. The prepared sample was heated at 300, 400, 500, 600, 700 and 800 °C for 2 h.

2.2. Characterization

The crystalline structure of the prepared 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).

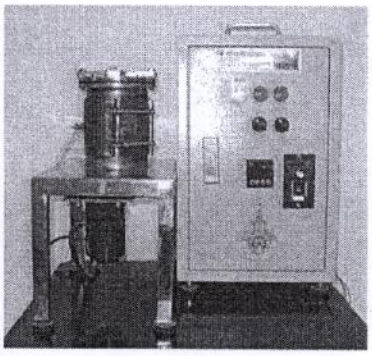

Fig. 1. Teflon-lined stainless steel autoclave.

2.3. Photocatalytic activity measurment

Photocatalytic activity was measured though the concentration of I_3^- that generated from photo-oxidation reaction of I which transformed into I_2 in excess of I condition [16] following Eqs. (1) and (2).

$$2\,I^- \longrightarrow I_2 + 2e^- \quad (1)$$

$$I_2 + I^+ \longrightarrow I_3^- \quad (2)$$

The 50 mg of TiO_2 powders and potassium iodide solution were 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 h then 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 4.0×10^4 cm mol/l.

3. Results 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 h were anatase phase. The anatase TiO_2 structure increased when the calcination temperature was increased. The peaks were rather sharp, which indicated the calcined nanosheet TiO_2 had relatively high crystallinity.The peaks corresponding to rutile TiO_2 appeared at 700 °C and almost showed rutile TiO_2 structure at 800 °C.

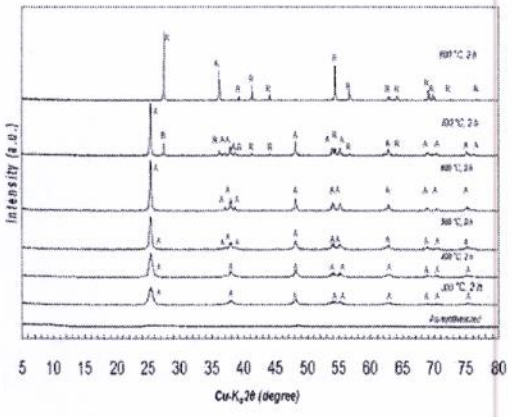

Fig. 2. The XRD patterns of as-synthesized nanosheets and calcined nanosheets at various temperatures (A: anatase and R: rutile).

The BET specific surface area, pore diameter and pore volume of nanosheet were about 360.28 m^2/g, 3-5 nm and 0.275 cm^3/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_2 crystallites [17-18] (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^2/g, respectively. The BET specific surface area decreased with increasing calcination temperature, resulting in the increasing of pore diameter and decreasing of pore volume. The nanosheets structure after calcinations were destroyed and changed to nanoparticles composite at high temperature [15, 19-20].

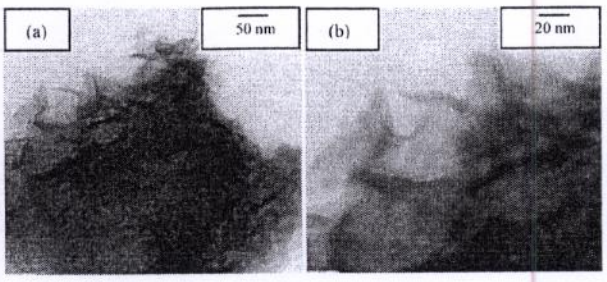

Fig. 3. TEM images of as-synthesized nanosheets (a) x 40,000 and (b) x 100,000 magnified.

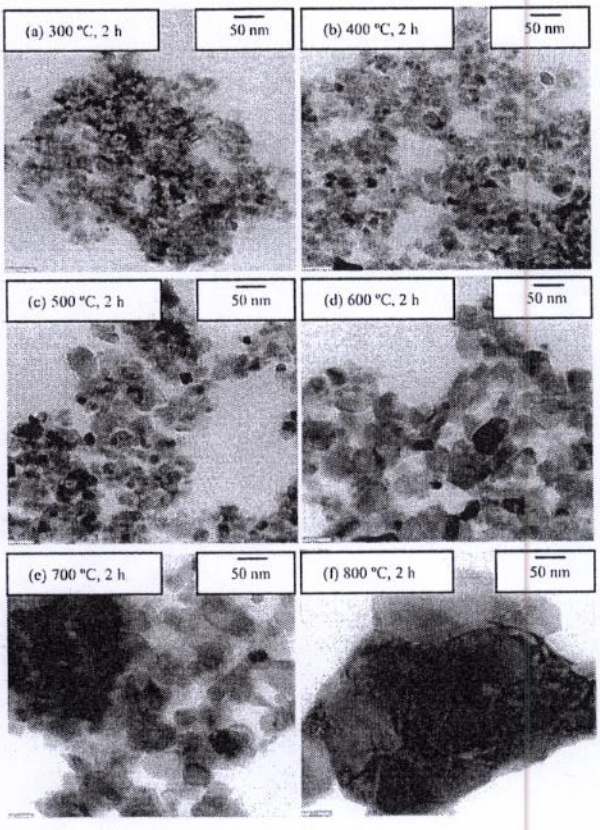

Fig. 4. TEM images of as-synthesized nanosheets TiO_2 calcined at various temperatures for 2 h at (a) 300 °C, (b) 400 °C, (c) 500 °C, (d) 600 °C, (e) 700 °C and (f) 800 °C.

3.2. Photocatalytic activity

In Figure 5, the calcined nanosheets TiO_2 (excepted at 800 °C) showed higher I_3^- concentration than the as-synthesized sample. The calcined nanosheets TiO_2 at 300 °C for 2 h showed the highest activity and also higher than commercial nanoparticle TiO_2 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].

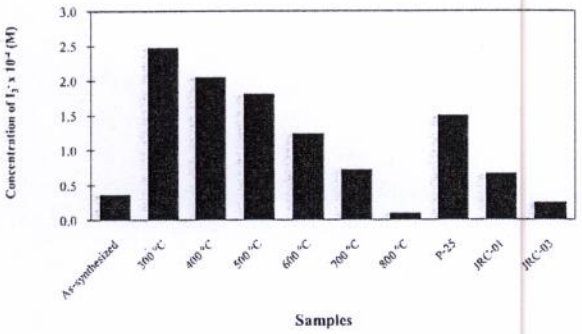

Fig. 5. Photocatalytic activity (I_3^- concentration) of the as-synthesized nanosheets, the calcined nanosheets and commercial nano TiO_2 samples (P-25, JRC-01, JRC-03).

4. Conclusion

Nanosheet TiO_2 was prepared by hydrothermal method at 120 °C for 12 h with a Teflon-lined stainless steel autoclave (Thai made). The as-synthesized nanosheets TiO_2 showed amorphous structure. The BET specific surface area, pore diameter and pore volume of the nanosheets TiO_2 were about 360.28 m^2/g, 3 nm and 0.275 cm^3/g, respectively. The calcined nanasheets showed higher photocatalytic activity than those of the as-synthesized sample and the commercial nano TiO_2 powders (P-25, JRC-01, JRC-03). This preparation method provided a simple routh to fabricate nanostructures TiO_2 with high photocatalytic activity.

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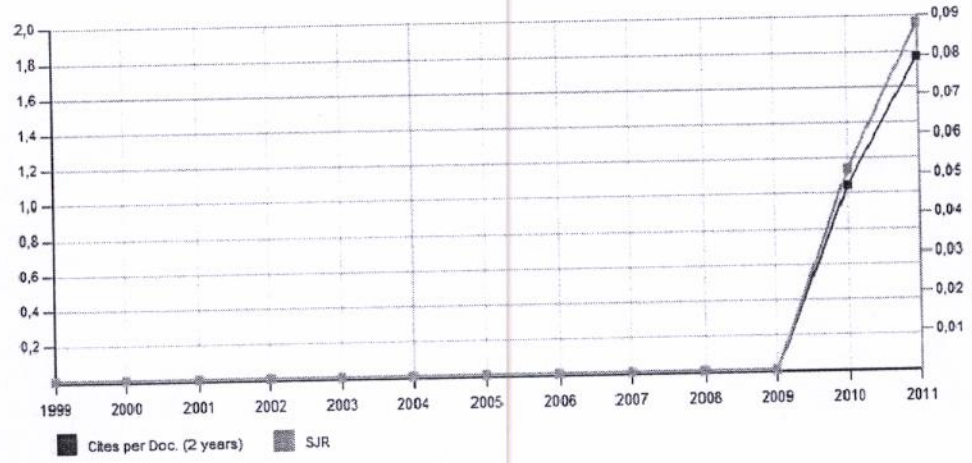

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