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I-SEEC 2012

Proceeding - Science and Engineering (2013) 294–301

Proceeding
Science and Engineering

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Science and Engineering Symposium
4th International Science, Social Science, Engineering and Energy Conference 2012

A study of Photocatalytic Activity of TiO_2 Films on Water Wheel for MB Degradation

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Abstract

This work, the decolorization of methylene blue (MB) due to water wheels that were coated with titanium dioxide films was investigated. The decolorization occurred under photocatalytic process of the sunlight. TiO_2 were prepared by sol–gel technique from titanium tetraisopropoxide (TTIP) and iso-propanal (IPP) of 1:10 (V/V) then, TiO_2 sol-gel was used for coating on grade-304 stainless steel in dimension of 10.0×10.5×0.01cm3 by dip coating of 20 cycles. The coated substrates were used as the paddles of water wheel. After coating, TiO_2 films were treated at annealed temperature and annealed time of 500 ◦C and 1 hour, respectively. The 24 paddles were used for construction of water wheel. XRD Pattern showed that TiO_2 films were crystalline structures in pure anatase phase. SEM images showed films morphologies, grain sizes showed in the range of 20-200 nm. The photocatalytic activity was tested by degradation of methylene blue (MB) under the sunlight over 3 daylights, in the period of 9.00am-5.00pm. Initially, concentration of MB was adjusted at 0.05 mmole in the glass-container of 35 L. The angular speed of water wheel was kept constant at 17 rev/min. that were controlled by microcontroller box set. To compare photocatalytic activity, uncoated TiO_2 - water wheel (UTWW) was run in the same condition of coated TiO_2 - water wheel (CWW). The result showed that the coated TiO_2 water wheel was higher in efficiency than uncoated TiO_2 water wheel by about 20 percent.

Keywords: TiO_2; Water wheel; Photocatalytic activity; Decolorization; Films

1. Introduction

In developing country, industrial wastewater and domestic wastewater are very important problems that we have to solve. Several methods have been used for water treatment, for example, physical treatment, chemical treatment and biological treatment. Nevertheless, water treatment techniques have to be developed to treat the

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various kinds of wastewater, diminish treatment time and economical consumption. In the last decade, the photocatalyst has been developed to use in organic composed process. The famous photocatalyst is TiO_2 that has high photocatalytic activity. However, researchers have still developed the photocatalyst to produce higher photocatalytic activities. Meanwhile, the techniques for using photocatalysts in water treatment field have been developed. In this work, photocatalyst (TiO_2) was coated on the paddles of water wheel to study “how to treat wastewater and develop treatment technique by using photacatalytic process with water wheel”.

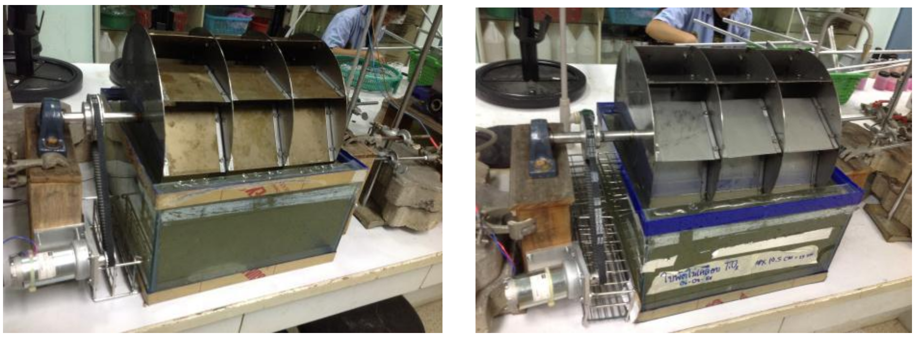
Nomenclature

A	substrate (Grade-304 stainless)
B	TiO_2-Substrate without annealing
C	TiO_2-Substrate with annealing of 500°C
MB	methylene blue
mag.	magnification
TTIP	titanium tetra-isopropoxide
IPP	iso-propanal
V/V	volume/volume
UTWW	uncoated TiO_2 -water wheel
CTWW	coated TiO_2 -water wheel

2. Experiment

2.1 Water wheel

Fig. 1 showed water wheel that was used in this work, a) coated TiO_2 -water wheel: CTWW and b) uncoated TiO_2 -water wheel: UTWW. Water wheel consisted of the axle, the paddles and the circle-case. The axle was fixed in the bearing and connected to control motor by using timing belt. Angular speed was controlled by micro-controller unit that was programed to change speed. However, in this work, the angular speed was kept at 17 rev/min.



a) Coated TiO2-Water Wheel :CTWW   b) Uncoated TiO_2-Water Wheel: UTWW

Fig. 1. Water wheels were used in this work, a) CTWW and b) UTWW

2.2 Film preparation

TiO_2 films were coated on the grade 304-paddles- stainless steel. TiO_2 was prepared from titanium tetraisopropoxide (TTIP): isopropanal (2PP) in the volume ratio of 1:10(V/V). The pH of TTIP:IPP solutions were adjusted at 2 by using hydrochloric acid (HCl). The solutions were kept to set gel around 24 hrs. After that, the paddles were coated by dip coating and heat at 100 $^{\circ}$C to release organic substance from the films. Dip coated-cycle was kept constant at 20 cycles. The 24 paddles were used to create water wheel. To compare photocatalytic activity, the 24-without film-paddles were used to create other water wheel.

However, to study crystallinity and surface morphology, the samples were prepared to be 3 cases that consist of:

A: Substrate (Grade-304 stainless)
B: TiO_2-Substrate without annealing
C: TiO_2-Substrate with annealing of 500°C

2.3 Characterization

Film crystallinity was investigated by X-ray diffraction (XRD). Surface morphology was observed by scanning electron microscope (SEM) and Photocatalytic activity test was observed by MB decolorization. The concentration of MB solutions was kept constant at 0.05 mmol. MB solution was filled in the glass-container of 35 L. After that, water wheel was immersed in the container that already contained MB solution. The sunlight was used as UV sources to generate photocatalytic process, shined over MB-container with the water wheels. After treatment, MB solutions were measured absorbance by using UV-VIS spectrophotometer (wavelength 190-800nm) to observe degradation.

3. Result and Discussion

3.1 Films Crystallinity

The films crystallinity was observed by Bruker D8 Advanced X-ray diffractometer (XRD) technique. X-ray beam was emitted from Cu-Kα source that generated wave length of 1.54056 Å . 2-theta of diffraction angles was recorded from 10-90 degree. Current and voltage of power supply were 30 mA and 40 kV; respectively. Fig.2 showed XRD pattern of substrate (grade-304 stainless), TiO_2 films on substrate without annealing and TiO_2 film on substrate with annealing at temperature of 500C. The result showed TiO_2 film without annealing was amorphous structure that XRD pattern showed crystalline structure of substrate; thus, x-ray beam could penetrate to substrate that was under TiO_2 film, while TiO_2 film with annealing at temperature of 500 ºC showed crystalline phase in anatase phase. The main plane of anatase phase was A(101).The other anatase planes were A(004), A(200)**,** A(105), A(211) and A(204) that appeared on XRD pattern. The crystalline size from Scherrer'equation for A(101) was about 26 nm.

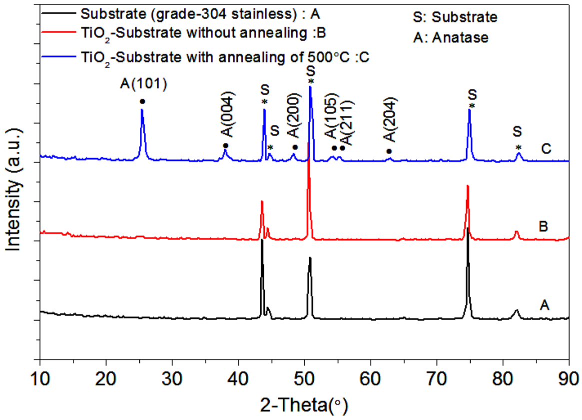


Fig. 2. XRD pattern of (A) substrate –grade 304 stainless, (B) TiO_2-Substrate without annealing and (C) TiO_2-Substrate with annealing of 500 ºC

3.2 Surface Morphology

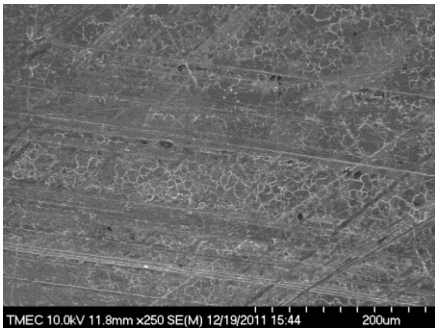
SEM images were used for observation surface morphology. Fig. 3 (a) showed enlargement of surface morphology of A: substrate at 250 magnifications that showed web net of the scratched trace due to being polished. For fig. 3 (b) showed surface of substrate at 2,500 magnifications which could be seen in different size and shape of the grain that were in the range of 20-100 µm. Each grain was separate off and showed sharp grain boundary trace. In fig. 3 (c)-(d) showed B: TiO_2-Substrate without annealing, film was coated on substrate and covered all over the place of substrate. The images showed the fracture of the film due to being stressed on the film. However, the films were still strongly adhered on substrate. In fig.3 (e)-(h) showed surface of C: TiO_2-Substrate with annealing of 500°C, in fig.3 (e), TiO_2 film was dispersed all over the place of substrate. However, film on substrate could still reveal substrate morphology that showed web net texture. Fig. 3 (f) and (h) showed enlargement of C at 50,000 and 100,000 magnifications; respectively. After annealing at 500˚C, TiO_2 film formed grain that looked like knobby shape. Nano-grain sizes were slightly different, showed in the range of 10-50 nm. Nano-structure of films surface morphology gave higher surface area to produce higher photocatalytic activity. The grain structure occurred due to thermal annealing that could adjust atomic network and set up the crystal structure in anatase phase. Besides, thermal annealing could change surface morphology from flat shape of B to knobby shape of C. In Fig. 3 (g) showed the trace of fracture of C that showed TiO_2 film was coated in the trace. The grains of TiO_2 film that appeared on trace surface and films still adhered on substrate.

3.3 MB degradation

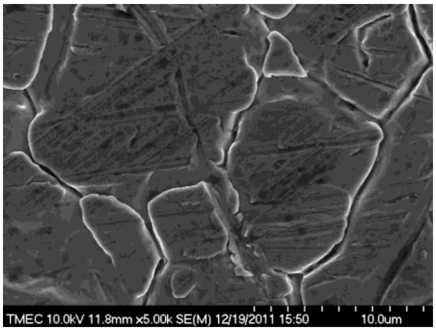
The MB degradation under sunlight with CTWW and UTWW was used to indicate ability of wastewater treatment. The half of water wheel was immersed in the container that already contained MB solution. The sunlight shined over MB solution container with water wheel over daytime of 3 days. The systems were run from 8.30am to 4.30pm. Thin plastic films were used to cover MB solution container and water wheel to protect evaporation of MB solution. In fig.4 (a) and (b) showed absorbance of MB solution after being treated over 24 hrs. for CTWW and UTWW; respectively. Both of CTWW and UTWW showed absorbance decreased as treatment time increased, which means they could degrade MB solution. However, in fig.5 showed absorbance of MB solution after treated over 24 hrs. for CTWW and UTWW at wavelength of 664 nm. The result showed that CTWW could degrade MB solution higher than UTWW about 16%.

4. Conclusions

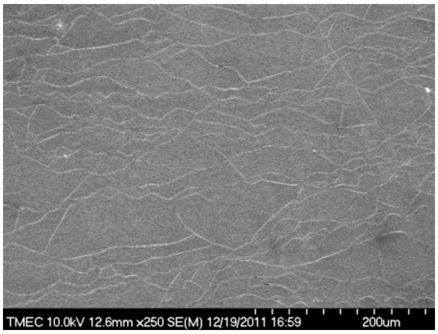
The CTWW was prepared from TiO_2 films coated on the grade 304-paddles- stainless steel. TiO_2 was prepared from titanium tetraisopropoxide (TTIP): isopropanal (2PP) in the volume ratio of 1:10(V/V). Under thermal annealing at 500 °C, films transformed from amorphous to crystalline structure in anatase phase with crystalline size about 26 nm. Surface morphology of TiO_2 film formed grain in nano-grain size in the range of 10-50 nm. Under sunlight with photocatalysis process over 24 hrs, CTWW could degrade MB solution higher than UTWW about 20%.



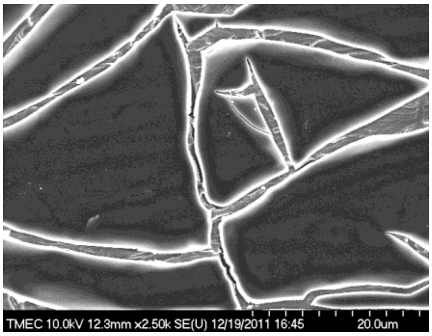
(a) A ×250 mag.



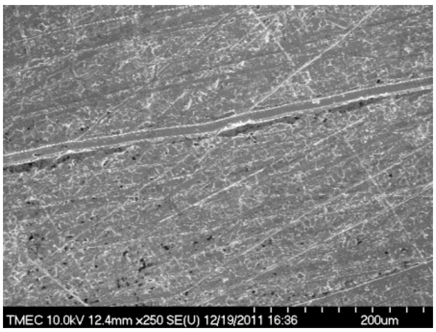
(b) A ×5,000 mag.



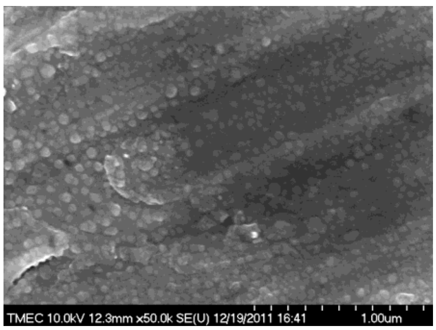
(c) B ×250 mag.



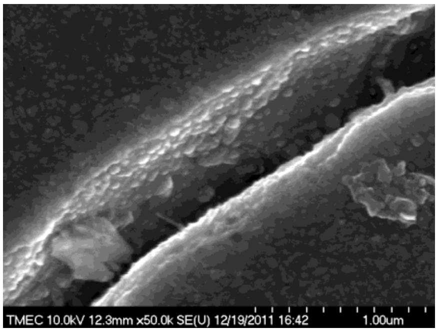
(d) B ×2,500 mag.



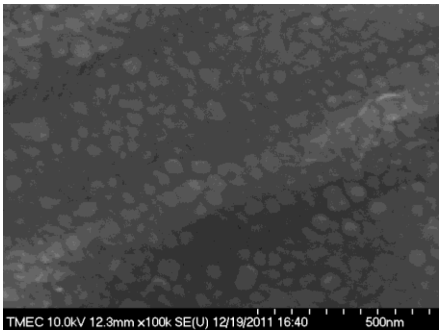
(e) C ×250 mag.



(f) C ×50,000 mag.

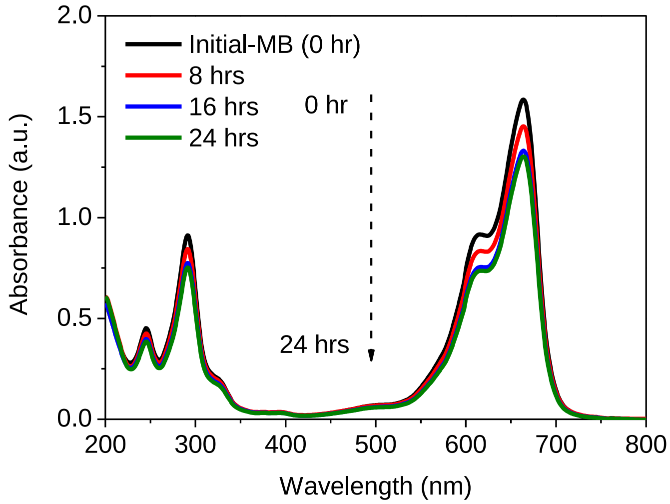


(g) C ×50,000 mag.

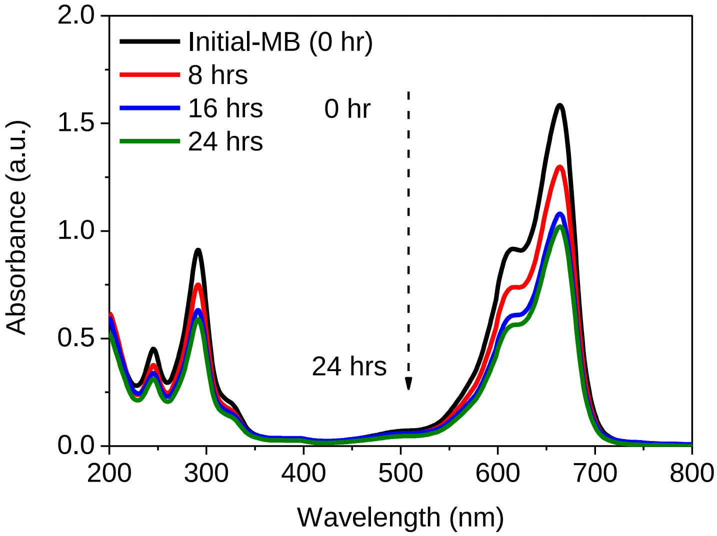


(h) C ×100,000 mag.

Fig. 3. SEM images of (a)-(b): A substrate, (c)-(d): B TiO_2-Substrate without annealing and (e)-(h): C TiO_2-Substrate with annealing of 500°C



(a) MB degradation for UTWW



(b) MB degradation for CTWW

Fig. 4. Absorbance of MB solution after treatment under sunlight over 24 hrs. a) MB degradation for UTWW and b) MB degradation for CTWW

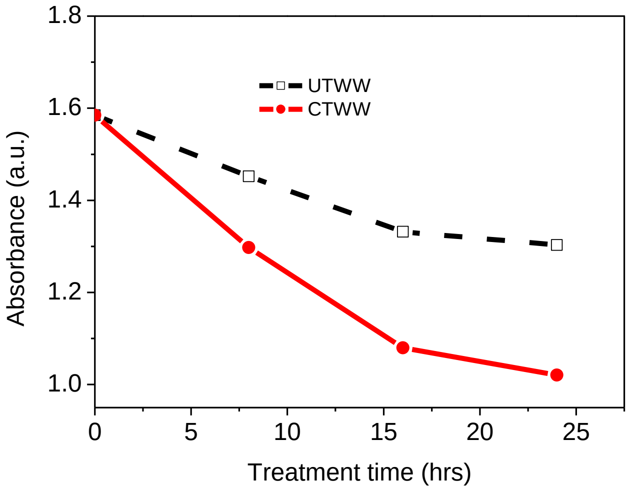


Fig. 5. Absorbance of MB solution under treatment for CTWW and UTWW at wavelength of 664 nm

Acknowledgements

The author would like to thank national research council of Thailand and office of the higher education commission for providing research fund.

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