APPLICATION OF TiO_2 NANOSTRUCTURE USING HYDROTHERMAL METHOD FOR WASTE WATER TREATMENT

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ABSTRACT

Titanium dioxide (TiO_2) can be used as a photo-degradation material. TiO_2 nanostructured has been successfully fabricated by hydrothermal method. The objective of this study is to optimize the TiO_2 nanostructures for waste water treatment. Titania Degusa P25 and sodium hydroxide (NaOH) were treated hydrothermally with different concentrations of NaOH i.e. 1, 5 and 10 M. The morphology of the samples were characterized using FESEM and XRD for structural analysis. All the samples were observed to have clumps of nanowires based on the FESEM images. XRD analysis showed that the samples produced were anatase phase. Later, the TiO_2 nanostructures were applied to waste water treatment.

Keywords: titanium dioxide, hydrothermal, waste water.

INTRODUCTION

Titanium dioxide (TiO_2) is a promising semiconductor that had been vastly studied by researchers. The special characteristics of TiO_2 include being inert, has high reflectivity, thermal stability and environment friendly (Chang, Ellis *et al.* 2009). Practically, TiO_2 can be found in our daily life as white pigments in paint, UV sunscreen, food coloring, plastics and papers (Markowska-Szczupak, Ulfig *et al.* 2011, Luttrell, Halpegamage *et al.* 2014).

The structure of TiO_2 can be divided into brookite, anatase and rutile. Brookite is polymorphous compared to anatase and rutile which are crystalline. Anatase is metastable which means it can be transformed to rutile when applied high heat whereas rutile is the most stable structure (Eder, Kinloch *et al.* 2006, Hanaor and Sorrell 2011).

The band gap of TiO_2 is quite wide with 3.2 and 3.0 eV for anatase and rutile respectively (He, Cai *et al.* 2013, Luttrell, Halpegamage *et al.* 2014). The electrons from TiO_2 can be excited using UV light with the photon energy in range of 3.10 to 3.94 eV (Habibi and Nasr-Esfahani 2007). Besides, TiO_2 also has high refractive index. Thus, TiO_2 is eligible to be a photo-catalytic material and be used in photo-degradation for waste water treatment.

Photo-catalytic reaction on a TiO_2 started upon the exposure of light. It will generate hole-electron pairs. The electrons at valence band will be excited to conduction band when the light energy was equivalent or overcome the band gap energy. Thus, the processes of oxidation and reduction might occurr when the photo-generated electron reaches the reaction sites (Kreysa, Ota *et al.* 2014).

TiO_2 can be fabricated using various techniques such as sol-gel method (Habibi and Nasr-Esfahani 2007), spray pyrolysis deposition technique (Ranga Rao A. 2007), hydrothermal method (Fen, Han *et al.* 2011), and chemical vapour deposition (CVD) (Chang, Ellis *et al.* 2009). In this study, hydrothermal method was used to synthesize the TiO_2 nanostructures. Hydrothermal is a process done at low temperature and in atmospheric pressure in a closed-system. This method may produce crystallite structure of TiO_2 (Funda Sayilkan 2007, Mali, Kim *et al.* 2013).

Improper waste water management generate by-products to the water source. These contaminated water is not safe for domestic use. TiO_2 can be used in degradation of polluted water due to its photocatalytic properties. The TiO_2 nanostructure produced by hydrothermal method is used in the application of water waste treatment.

METHODOLOGY

The reagents used in this study were Degusa P25, acetic acid, sodium hydroxide (NaOH), and deionized water. All the reagents are used as purchased. NaOH were weighted 8 , 40 and 80 g to vary the NaOH concentration of 1, 5 and 10 M respectively. Then, the NaOH were diluted in 200 ml deionized water with magnetic stirrer until the NaOH pallets are all diluted. 1 g of Degusa P25 was added into the solution and the stirring continued until it has mixed well. After that, the solution was put into Teflon- lined stainless steel autoclave and was heated in the oven at 150 °C for 24 hours. The autoclave was let to cool down to room temperature.

Then, the solution was filtered using filter paper and is let to dry at room temperature for one day. Then, the precipitate obtained were grinded using mortar with 5.5 ml of acetic acid and deionized water until it mixed well. Later, the solution was filtered again and dried overnight. Lastly, the sample was dried in the oven at 100 °C for 30 minutes. The samples were characterized by field emission scanning electron microscopy (FESEM) and x-ray diffractometer (XRD) to analyze the structure and crystallinity respectively.

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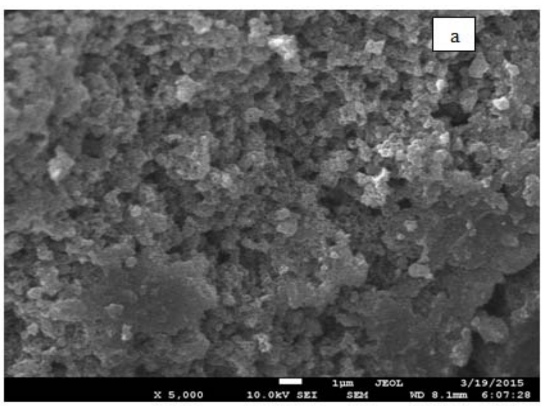
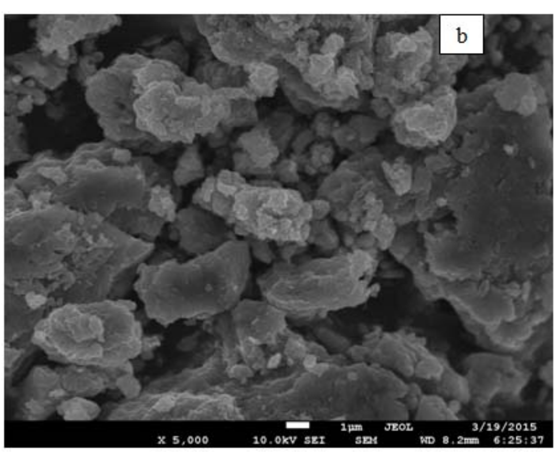
RESULTS AND DISCUSSION

Figure-1 showed the results from FESEM analysis. There were three different structures which had been synthesized with different NaOH concentration i.e. 1, 5 and 10 M. Figure-1 (a) showed the clump of nanowires structure when treated with 1M NaOH. According to Armstrong and colleagues, they discovered that synthesis by hydrothermal method would get nanotubes (Armstrong, Armstrong *et al.* 2005).

The dimension of the sample was unable to be measured due to the agglomeration of TiO_2 nanostructure. Mastan had reported that the clump was due to from the high content of sodium (Mastan A.A.K. 2011). It had been supported by Chin Wei which stated that the sample formed was sodium titanate (Lai, Bee Abd Hamid *et al.* 2015).

Figure-1 (b) was the sample done by using 5M NaOH which showed lamella structures that rolled up to form cylindrical structure while Figure-1 (c) from using 10 M NaOH was observed as sheets formed of nanowires. We observed more clumping and aggelomeration of sodium titaniate as the concentration of NaOH increased. Thus, the concentration of NaOH does affect the surface morphology.





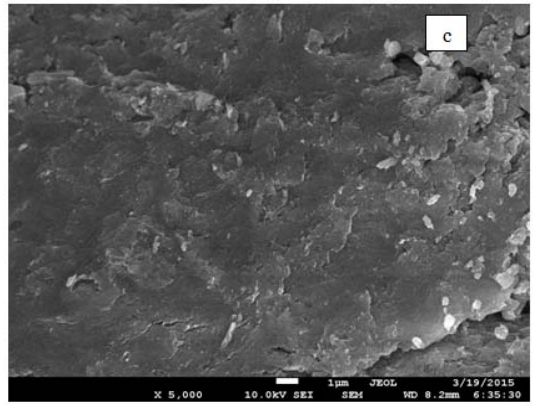


Figure-1. FESEM images of TiO_2 fabricated by using different concentration of NaOH hydrothermally (a) 1, (b) 5 and (c) 10 M.

Figure-2 showed the X-ray diffraction pattern of all three samples with different concentration of NaOH (1, 5 and 10 M). The pattern was taken from reference no. ICSD 98-008-2080 for 1 M NaOH and ICSD 98-016-1908 for 5 and 10 M NaOH. It was seen that diffractometer obtained matches TiO_2 anatase structure. The highest peak observed was at 2 theta 25.34° corresponded to [001] plane for 1 M NaOH. Other peaks were observed at 37.92°, 38.02°, 47.74° and 54.81 corresponding to the planes [013], [112], [020] and [121] respectively. As we could see in Figure 2, the intensity of TiO_2 peaks decreased and the peaks became wider and disappear as concentration of NaOH increased. It is because at 10 M NaOH, small amount of TiO_2 and more sodium titanate was formed as supported in previous research (Lai, Bee Abd Hamid *et al.* 2015).

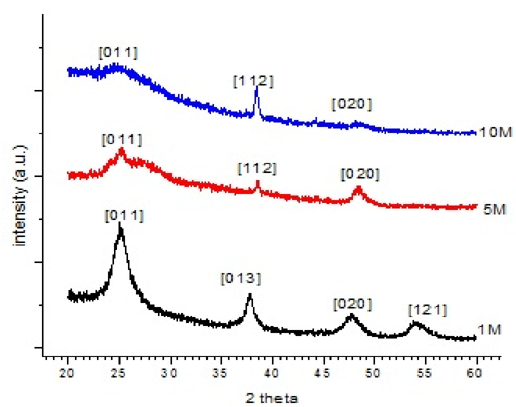


Figure-2. XRD patterns for TiO_2 fabricated by different concentration of NaOH by htdrothermal method.

CONCLUSIONS

TiO_2 nanostructures were successfully synthesized using hydrothermal method with different concentrations of NaOH. An increase of the concentration will increase the formation of sodium titanate and cause the structure to be more aggelomerated. The crystallinity

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of anatase decreases as the concentration of NaOH increases. Anatase TiO_2 that should be applied to waste water treatment must has smaller size so that the surface over volume ratio will increase and the author found that the sample with 1 M NaOH is the best sample to be tested. The TiO_2 anatase nanostructure could be obtained by reducing the concentration at 0.1 M so that it will be less aggelomerated.

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