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FABRICATION OF SAND/ZINC OXIDE/TITANIUM DIOXIDE NANOCOMPOSITE AS PHOTOCATALYST

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Abstract

In this work, a nanocomposite photocatalyst was fabricated by growing zinc oxide (ZnO) and titanium dioxide (TiO_2) on the sand as a substrate. The initial sand/ZnO was fabricated via sol-gel immersion method for 4 h at 95℃. Furthermore, the sand/ZnO/TiO_2 was fabricated using hydrothermal method for 5 h at 150℃. Based on field emission scanning electron microscopy (FESEM) analysis, the fabricated sand/ZnO/TiO_2 consists of random formation of hexagonal ZnO nanorods and two pyramidal spindle ends of TiO_2 nanorods. The addition of TiO_2 on top of ZnO nanorods increased the number of active sites which enables more contaminants to be absorbed thus enhanced the photocatalysis process. Moreover, based on the micro-Raman spectra, the synthesized TiO_2 was in rutile phase and the ZnO peak was unobservable due to the overlapping with TiO_2 peak. Based on its morphological and structural properties, the fabricated sand/ZnO/TiO_2 nanocomposite was potential to be applied as photocatalyst.

Key Words: Sand, Zinc Oxide, Titanium Dioxide, Sol-Gel Immersion, Hydrothermal

INTRODUCTION

One of the major contributor of dye pollution is from textile industries which released plenty types of organic dye which are hazardous and harmful [1-2]. About 10-15% of waste dye product from textile industries are released into the environment, causing serious pollution and affecting aquatic life and human health [3]. Dyes have stable molecular structure which difficult to degrade by traditional methods such as flocculation, coagulation, reverse osmosis, membrane filtration and biodegradation [4,5]. One of the most effective way for dye degradation is heterogenous photocatalysis as it degraded the dye into carbon dioxide and water which are non-harmful to the environment [6].

ZnO and TiO_2 are the most common semiconductors to be utilized as photcatalyst. These were due to their unique characteristics such as high stability, eco-friendly and cheap [7, 8, 9]. However, photocatalysis performance of individual ZnO and TiO_2 were limited due to their large band gap energy and high rate of recombination between electron and hole [10, 11,12]. In addition, the incomplete dye removal by TiO_2 and self-oxidation of ZnO also lead to the inefficient photocatalysis performance [10]. In order to overcome these drawbacks, the combination of ZnO and TiO_2 was done to enhance its performance. It has been reported that the coupling between these two semiconductors showed excellent photocatalysis performance [6, 13, 14 ,15].

Mass production of photocatalyst is essentially needed in photocatalysis process. However, its powder form is inefficient as it is produced milky solution and easily dispersed [16]. Furthermore, powder form of catalysts are easily to aggregate and difficult to separate due to its nanosize [16]. In order to hinder these drawbacks, the photocatalyst must be supported on a substrate in order to immobilize it in the solution. There are several substrates that are commonly used such as zeolites, glass, clay, silicon, sand and quartz [4]. In this work, sand is selected as a substrate due to its availability, high density, inexpensive and chemically inert [17]. Basically, there are various methods that can be used to synthesize TiO_2 such as microemulsion, sonochemical, sol gel, thermal treatment method, chemical vapour deposition (CVD), and electrodeposition [18]. Among these methods, hydrothermal method presents a simple, save energy, and eco-friendly [19]. Meanwhile, ZnO was synthesized via sol-gel immersion method as this method is simple, less production steps and low synthesis temperature [10, 20].

In this work, the sand-based photocatalyst was fabricated by combining the sol-gel immersion and hydrothermal methods. The structural properties of sand/ZnO and sand/ZnO/TiO_2 were characterized by FESEM, and micro-Raman spectroscopy.

METHOD

0.05 M of ZnO solution was prepared by mixing hexamethylenetetramine (HMT), zinc nitrate and DI water into a schott-capped bottle. The prepared solution was sonicated in an ultrasonic cleaner for 30 minutes followed by stirring process for 2 hours at room temperature. Then, the solution was left for 24 hours for aging process at room temperature. For the next day, the cleaned sand was put into the ZnO solution and placed into the water bath to perform sol-gel immersion method for 4 hours at 95℃. After the process, the sand was taken out and rinsed by DI water and directly dried in the electric oven for 10 minutes at 150℃ followed by annealing process at 500℃ for 1 hour.

For TiO_2 growth, the prepared sand/ZnO was put into the hydrothermal solution which was butoxide (TBOT) and stirred for another 15 minutes. Then, the prepared hydrothermal solution was poured into an autoclave followed by sand/ZnO and heated at150°C for 5 hours to perform hydrothermal process. After the process, the autoclave was allowed to cool down at room temperature. Then, the sand was taken out and rinsed by DI water. The sample was heated for 5 minutes at 150°C and annealed for 1 hour at 450°C.

RESULTS AND DISCUSSION

FESEM images of sand/ZnO and sand/ZnO/TiO_2 nanocomposite are presented in Figure 1. Based on Figure 1 (a), the morphology of the fabricated sand/ZnO shows smooth surface formation of ZnO nanorods represented by the red arrows with various diameter and length. These ZnO nanorods are well-dispersed with perfect hexagonal shape which are caused by the presents of HMT. Meanwhile, the morphology of sand/ZnO/TiO_2 nanocomposite are presented in Figure 1 (b). The synthesized TiO_2 and ZnO nanorods are shown by white and red arrows, respectively. It can be seen that the formation of the sand/ZnO/TiO_2 nanocomposite are randomly arranged and closely packed together. TiO_2 nanorods possess two pyramidal spindle ends compared to the flat ends of ZnO nanorods. The addition of TiO_2 into ZnO nanorods increased the number of active sites which enables more contaminants to be absorbed thus enhanced the photocatalysis performance [2, 21]. Moreover, the sand/ZnO/TiO_2 nanocomposite has longer lifetime of electron/hole pair recombination and extension of light absorption range as compared to sand/ZnO which make it potential to be applied as a photocatalyst [19].

prepared by mixing 60 ml of DI water and HCl for 5 minutes followed by dropping 3 ml of titanium

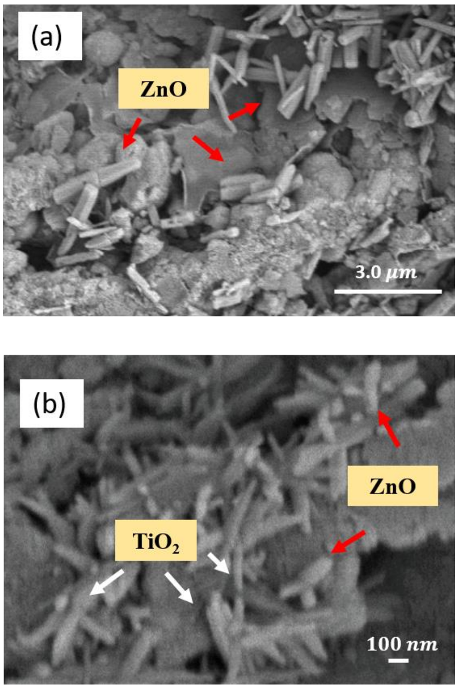

Figure 1. FESEM images of the fabricated (a) sand/ZnO and (b) sand/ZnO/TiO_2 nanocomposite

Micro-Raman spectroscopy was then used to determine the crystallinity and structural properties of the fabricated sand-based material. Figure 2. presents the micro-Raman spectra of sand/ZnO and sand/ZnO/TiO_2 nanocomposite. Based on micro-Raman spectra, sand/ZnO shows an intense peak at 438 cm^{-1} that corresponding to E_2 (high) which confirmed the the formation of wurtzite phase of ZnO nanorods [18, 22].

The spectra of sand/ZnO/TiO_2 shows four peaks in the range of 100 cm^{-1} to 800cm^{-1}. The weak peak at 143 cm^{-1} is related to B_{1g} mode of rutile TiO_2 [23]. Meanwhile, the dominant peaks at 448 cm^{-1} and 610 cm^{-1} were related to E_g and A_{1g} modes of rutile TiO_2, respectively. These several peaks confirmed the presence of rutile phase in the nanocomposite sample [6,24]. Moreover, peak at 237 cm^{-1} represent the multiple phonon of rutile TiO_2. The ZnO peak was unobservable in the nanocomposite sample due to its overlapping with TiO_2 peak.

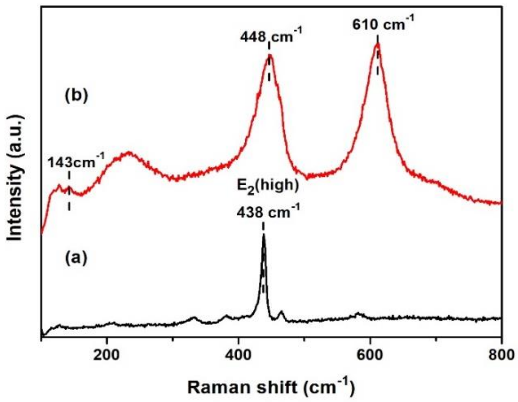

Figure 2. Micro-Raman spectra of the fabricated (a) sand/ZnO and (b) sand/ZnO/TiO_2 nanocomposite

CONCLUSION

The sand/ZnO/TiO_2 has been successfully fabricated as photocatalyst for future photocatalysis application. The structural properties are being analyze by using FESEM, and micro-raman spectroscopy. The FESEM images show the formation of hexagonal ZnO and TiO_2 nanorods with two pyramidal spindle ends. Then, the micro-Raman spectra shows the structure of sand/ZnO/TiO_2. Thus, based on the morphology and structural properties it showed that sand/ZnO/TiO_2 can be applied as photocatalyst for photocatalysis application.

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