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Impregnation of activated carbon-TiO₂ composite and its application in photodegradation of procion red synthetic dye in aqueous medium

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Abstract. Synthetic dyes are often used for textile dyeing process. The dye wastewater containing hazardous materials, toxic, and also harmful to the environment. Among the existing technologies, photodegradation using semiconductor catalyst is a promising alternative method. However, the weak adsorption capacity of photocatalyst is an issue for photocatalysis process. To overcome this lack, photocatalyst material needs to combine with an adsorbent. The composite was synthesized by combining the function of activated carbon as adsorbents and TiO₂ as a catalyst. The objective of the study was to synthesize a composite of activated carbon and TiO₂ through impregnation method. The activated carbon-TiO₂ composite produced was characterized to determine the surface morphology and the elements contained in the composite by using SEM/EDX. The composite activity to photodegrade various concentration of Procion red, as a model of synthetic dye, was also investigated. To compare the activity of activated carbon-TiO₂ composite in degrading Procion red, different conditions were applied by using UV lamps, sunlight irradiation, and without irradiation (darkroom). The activated carbon-TiO₂ composite was able to photodegrade of Procion red by UV light irradiated at optimum wavelength 530 nm. The highest photodegradation percentage of 58.9% and 71.7 % were obtained when using the activated carbon-TiO₂ composite ratio of 3:7 and 1:1, respectively.

1. Introduction

Dye is an organic compound containing a conjugated chromophore group. Reactive dyes are a dye used for coloring textiles, for example Remazol brilliant orange 3R, Remazol golden yellow, procionred, and methylene blue. The dyes were often used for batik dyeing process both in large-scale industry and home industry.

The dye wastewater containing hazardous materials and toxic presence in water can block the sunlight to penetrate the aquatic environment, so that disrupting the biological processes that occur in the water body. In addition, it is also disturbing the aesthetics of the water due to the appearance of a foul odor, and also harmful to the environment [1].

It is necessary to set up wastewater treatment technology, so that the effects of pollution can be prevented and controlled. Some wastewater treatment technology with relatively low cost have been investigated to overcome the problems of waste water, synthetic dyes such as adsorption using



activated carbon from coconut shell [2], or a combination of filtration and adsorption [3]. Some other methods can be used to treat wastewater, such as by coagulation/adsorption [4], chemical and biological oxidation [5], and photodegradation by using semiconductor photocatalyst [6].

Among the existing methods, photodegradation is an alternative method that is relatively cheap and easy to apply [7]. This method requires a semiconductor material include TiO_2 , ZnO , or Fe_2O_3 and ultraviolet radiation (UV). Among semiconductor materials exist, TiO_2 is a semiconductor material availability in the market and classified the best of quality [8]. The TiO_2 is often used as a photocatalyst to decompose organic compounds such as pentaklorofenol [9], cibracron yellow, orange metal, and procion red.

Weak adsorption capacity is an issue for photocatalytic process. To cover the shortfall, photocatalyst material need to combine with an adsorbent [10]. Adsorbent which can be used include activated carbon, which is synthesized from coconut shell. Carbon which is the enhanced energy through activation of so-called activated carbon, has a sizeable surface and a good adsorption properties, so that it can be used as an adsorbent, active carbon properties can help the process of adsorption-catalytic [11].

The objective of this research was to synthesize a composite of activated carbon and TiO_2 through impregnation method and to study the application of activated carbon- TiO_2 composite in photodegradation of procion red synthetic dye. In addition, the activated carbon- TiO_2 composite produced is characterized to determine the morphology by SEM/EDX, then tested for activity to degrade the procion red dye. Activated carbon- TiO_2 composite is expected to be an effective alternative for dealing with industrial wastewater containing the dying stuff.

2. Materials and method

2.1. Materials

The activated carbon of 200 mesh, TiO_2 , aquadest, distilled water, and procion red dye were used as materials.

2.2. Procedure

Activated carbon- TiO_2 composite was varied with the composition of TiO_2 50% and 70% by weight. The composite was synthesized by dissolving the amount of TiO_2 into 100 mL of distilled water. Then the amount of activated carbon of 200 mesh was added to the TiO_2 , then stirred by using a magnetic stirrer for 5 hours, and ultrasonic for 30 minutes. Activated carbon- TiO_2 formed was dried in an oven for 5 hours with a temperature of 120°C until dry. Activated carbon that has been coated with a photocatalyst is then calcined at a temperature of 400°C for 2 hours. Composite of activated carbon- TiO_2 characterized by using SEM-EDX to determine the morphology of the composite.

Procion red solution with a concentration of 150, 200, 250, 300 mg/L was added with activated carbon- TiO_2 composite at a contact time of 2 hours, 4 hours, 6 hours and 8 hours with 3 different conditions: with UV light, sunlight and darkroom at various time intervals of 2 hours between 10 am until 2 pm for 2 days. The mixture was filtered to separate activated carbon- TiO_2 composite from procion red wastewater.

The absorbance value of procion red that has been photodegraded or adsorbed by activated carbon- TiO_2 composite was measured by using a UV-Vis spectrophotometer at the maximum wavelength. From these absorbance values, the concentration of red procion can be calculated, as well as the percentage of photodegradation and adsorption of procion red.

2.3. Characterization and analysis

The intensity of sunlight was measured by using a Light meter (Luxtron Lx-103, Lux, Taiwan) at various time intervals of 2 hours between 10 am until 2 pm. The intensity of sunlight was 123.9 W/m^2 . The intensity of UV light was 15.3 W/m^2 . And the intensity of darkroom was 0.5 W/m^2 . Composite of activated carbon- TiO_2 characterized using Scanning Electron Microscope-Energy

Dispersive X-ray (SEM-EDX) to determine the morphology of the composite and to identify the elemental composition of materials.

3. Results and discussion

3.1. Characterization of activated carbon-TiO₂ composite by SEM/EDX

Activated carbon-TiO₂ (AC-TiO₂) composite were characterized by SEM/EDX to see the surface morphology and the elements contained in the composite. Figure 1 and 2 shows the results of SEM.

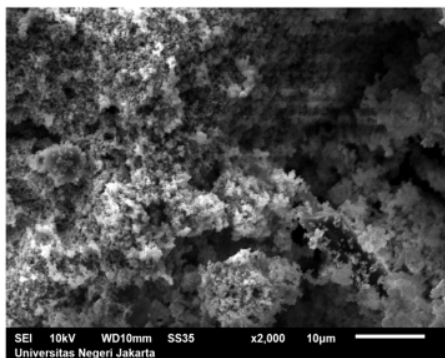


Figure 1. SEM images of AC-TiO₂ (3:7).

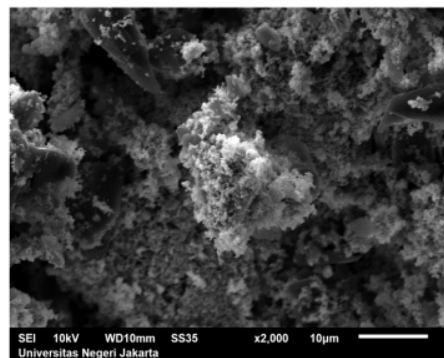


Figure 2. SEM images of AC-TiO₂ (1:1).

From the figure, it can be seen that TiO₂ with the characteristics of white powder are dispersed evenly cover the surface of activated carbon. The results show the texture of activated carbon-TiO₂ composite having an aggregate structure, but the morphology of the surface looks uniform. The SEM results of activated carbon-TiO₂ composites are then compared with the results of another researcher. The research of Tang, *et al* (2012) shows similarities to the surface morphology of activated carbon-TiO₂ composite in this research [12]. This means that the activated carbon-TiO₂ composites have been successfully synthesized.

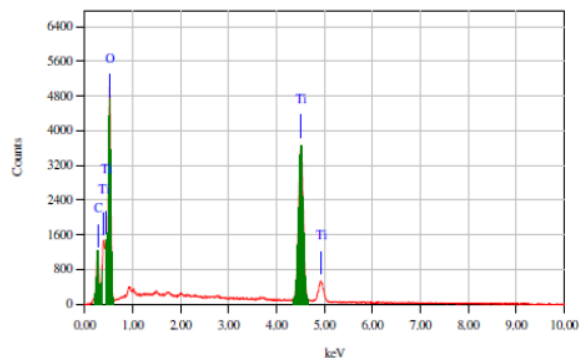


Figure 3. EDX images of AC-TiO₂ (3:7).

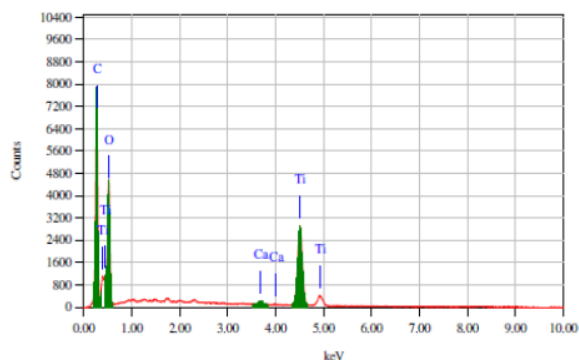


Figure 4. EDX images of AC-TiO₂ (1:1).

Characterization of activated carbon-TiO₂ by using EDX was shown in figure 3 and 4. The EDX analysis was used to determine which chemical elements are present in the composite. The EDX Spectra showed the peaks of Ti and O. It means the composite comprises of TiO₂ photocatalyst.

3.2. Photodegradation of procion red by activated carbon-TiO₂ composite

Photodegradation process conducted at a contact time of 8 hours, and by varying the concentration of procion red namely 150, 200, 250, 300 mg/L with 100 mg weight of composite irradiated by UV Light 254 nm. The intensity of UV light was 156 W/m². Activities photodegradation of procion red dye by activated carbon-TiO₂ composite irradiated by UV Light can be seen from the percentage value of photodegradation shown in figure 5.

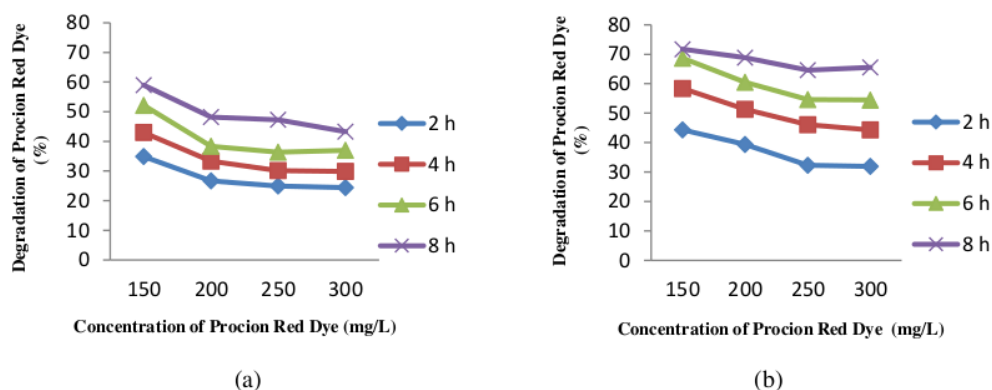
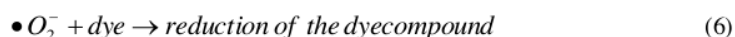
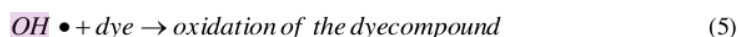


Figure 5. The relationship between concentration and percentage of procion red photodegradation by UV light by a composite of: (a) AC-TiO₂ (3:7); (b) AC-TiO₂ (1:1).

Procion Red were used as controls irradiated with UV light has a photodegradation percentage of 4.6%; 3.3%; 2.7% and 1.9% for each concentration procion red 150, 200, 250, and 300 mg/L, respectively. Photodegradation of procion red by activated carbon-TiO₂ composite ratio of (3:7) and (1:1) irradiated by UV light produce photodegradation percentage respectively 58.9% and 71.2%.

In the photocatalytic process, when TiO₂ absorbs light energy equal to or greater than the band gap energy there will be a charge separation or photo excitation in the semiconductor. Electrons excited into the conduction band leaving a positive hole (H⁺) in the valence band. The positive hole has a high affinity for oxygen in H₂O molecules adsorbed on the surface of TiO₂, so it will react into OH⁻ and H⁺. Hydroxyl radicals are highly reactive species that attack organic molecules and degrade into CO₂ and

H₂O, and halide ions if organic molecules containing halogen atoms. The oxidation of organic molecules are not selective. The reaction is written in the equation below :



Photodegradation of procion red was done by sunlight for 8 hours. The intensity of sunlight was measured using Light meter (Luxtron Lx-103, Lux, Taiwan) at various time intervals of 2 hours between 10 am and 2 pm for 2 days. The first day was done 0-4 hours and the second day 4-8 hours. The mean intensity of sunlight irradiation was 123.9 W/m². Figure 6 shows the relationship between concentration and percentage of photodegradation of procion red irradiated by sunlight by activated carbon- TiO₂ composite (3: 7) and activated carbon- TiO₂ (1: 1).

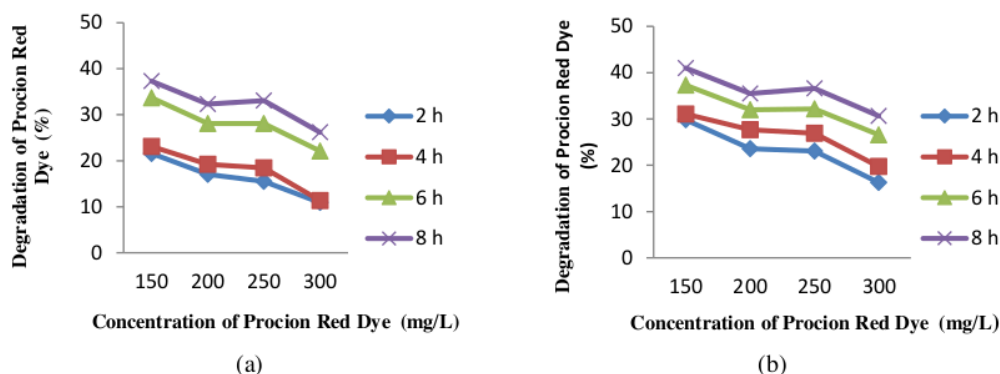


Figure 6. The relationship between concentration and percentage of photodegradation of procion red by sunlight by composite of : (a) AC-TiO₂ (3: 7), (b) AC-TiO₂ (1: 1).

Photodegradation of procion red by activated carbon-TiO₂ composite ratio (3:7) and (1:1) irradiated sunlight produces photodegradation percentage respectively 37.3% and 41%.

To compare the activity of activated carbon-TiO₂ composite in degrading procion red, the condition without irradiation (darkroom) was applied. In the activated carbon-TiO₂ composite that is not exposed to UV light and sunlight, the photodegradation process does not occur, but only the adsorption process of composite activated carbon-TiO₂ as adsorbent. Adsorption of Procion red by activated carbon- TiO₂ composite ratio (3:7) and (1:1) in a darkroom reaches percentage 23.4% and 24.3%.

Activated carbon-TiO₂ composite having a good photodegradation activity to procion red, by UV light and sunlight. Comparison of the activity of photodegradation and adsorption on activated carbon-TiO₂ composite can be seen in figure 7 below.

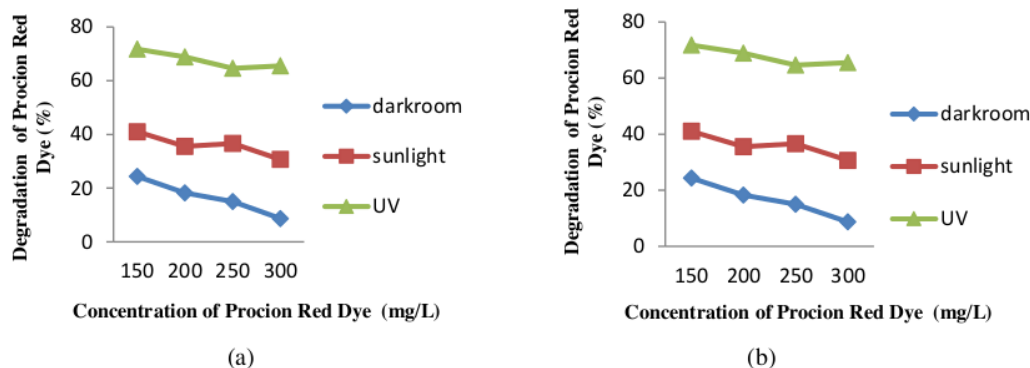


Figure 7. The relationship between procion red concentration and percentage of photodegradation and adsorption procion red by composite of: (a) AC-TiO₂ (3:7); (b) AC-TiO₂ (1:1).

From figure 7 (a), adsorption percentage of activated carbon-TiO₂ composite (3:7) to procion red reached 23.4 %, while photodegradation percentage by UV light and sunlight reached 58.9 % and 37.3% for 8 hours irradiation, respectively. And from figure 7 (b), adsorption percentage activated carbon- TiO₂ composite of (1:1) to procion red reached 24.3 %, while photodegradation percentage by UV light and sunlight reached 71.7 % and 41%, respectively, for 8 hours irradiation. Activated carbon-TiO₂ (1:1) has a higher value than activated carbon-TiO₂ composite (3:7) because of the difference in the composition. The amount of activated carbon to the activated carbon-TiO₂ composite (1:1) more so as to assist the adsorption capacity of the weak which is a problem for the photocatalytic process.

4. Conclusion

Activated carbon-TiO₂ composite has been succeeded in impregnating 86.5% for activated carbon-TiO₂ (3:7) and 84% for activated carbon-TiO₂ (1:1) with a gray powder texture. Photodegradation of procion red by activated carbon-TiO₂ composite ratio of (3:7) and (1:1) irradiated by UV light produce photodegradation percentage of 58.9% and 71.7%, respectively. Whereas photodegradation of procion red by activated carbon-TiO₂ composite ratio of (3:7) and (1:1) irradiated by sunlight produces photodegradation percentage of 37.3% and 41%, respectively. Adsorption of procion red by activated carbon-TiO₂ composite ratio of (3:7) and (1:1) in a darkroom, reaches the photodegradation percentage of 23.4% and 24.3%, respectively.

5. Acknowledgment

The authors would like to thankful the Waste Treatment Technology Laboratory, Chemical Engineering Department, Faculty of Engineering, Universitas Sriwijaya, for laboratory support.

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