

Novel Synthesis of MnO₂-SiC Fiber-TiO₂ Ternary Composite and Effective Photocatalytic Degradation with Standard Dyes

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Abstract In this work, we investigated the photo-degradation performance of MnO₂-SiC fiber-TiO₂ (MnO₂-SiC-TiO₂) ternary nanocomposite according to visible light excitation utilizing methylene blue (MB) and methyl orange (MO) as standard dyes. The photocatalytic physicochemical characteristics of this ternary nanocomposite were described by X-ray diffraction (XRD), scanning electron microscopy (SEM), tunneling electron microscopy (TEM), ultraviolet-visible (UV-vis), diffuse reflectance spectroscopy (DRS), electrochemical impedance spectroscopy (EIS), photocurrent and cyclic voltammogram (CV) test. Photolysis studies of the synthesized MnO₂-SiC-TiO₂ composite were conducted using standard dyes of MB and MO under UV light irradiation. The experiments revealed that the MnO₂-SiC-TiO₂ exhibits the greatest photocatalytic dye degradation performance of around 20 % with MB, and of around 10 % with MO, respectively, within 120 min. Furthermore, MnO₂-SiC-TiO₂ showed good stability against photocatalytic degradation. The photocatalytic efficiency of the nanocomposite was indicated by the adequate photocatalytic reaction process. These research results show the practical application potential of SiC fibers and the performance of a photocatalyst composite that combines these fibers with metal oxides.

Key words silicon carbide fiber, ternary composite, photocatalytic degradation, standard dye.

1. Introduction

Most of these environmental toxins that harm our ecosystem are dumped straight into undeveloped waterways. These contaminants include, among other things, chemicals, oils, leftover metal, radioactive waste, and vegetable waste. Dyes are a vital component of our lives, adding color to a wide range of goods from textile, paper, leather tanning, hair coloring, cosmetic, photochemical cell, and furniture sectors. They also contribute significantly to water pollution.¹⁻⁴⁾ Over 100,000 commercially available dyes are used by the dyeing industry. The industries release about 15 % of these non-biodegradable dyes into water bodies annually.⁵⁻⁸⁾ The main

cause for concern is the chemically active dyes found in industrial wastewater, which have the potential to cause cancer. These dyes also lower the amount of oxygen that dissolves in water and prevent sunlight from penetrating aquatic organisms, which results in reduced photosynthetic activity. Both organic and inorganic pollutants that are harmful to the environment are found in the waste generated during the dyeing process.⁹⁻¹⁷⁾ Coagulation, membrane processes, and other physio-chemical therapies are ineffective in eliminating the color and dye ingredient concentration.¹⁸⁻²¹⁾ The development of technology for the efficient removal of dyes has been identified as a rapidly expanding field of industrial importance research. The international environ-

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mental criteria are satisfied by the efficient, cost-effective, and photocatalytic dye degradation caused by semiconductors such as metal oxides, nitrides, and sulfides.²²⁻²⁴⁾ Manganese dioxide (MnO_2) is regarded as one of these semiconductors' best photocatalysts for the oxidative elimination of both organic and inorganic contaminants because it has great chemical stability and surface reactivity.^{25,26)} MnO_2 is easily reoxidized after being exposed to dioxygen and can facilitate fast electron transfer by functioning as an electron-transfer mediator during photocatalytic oxidation.^{27,28)} According to earlier research, α - MnO_2 nanorods have been utilized for photocatalytic organic dye elimination when exposed to ultraviolet (UV) light.^{29,30)}

In addition, titanium dioxide (TiO_2) is a famous photocatalyst because of its unique chemical stability, affordability, and biocompatibility.³¹⁾ Moreover, TiO_2 broad bandgap (3.2 eV) means that it mostly absorbs UV light, which produces hydroxyl radicals, and are a potent oxidant that breaks down organic molecules.³²⁾ In addition, silicon carbide (SiC) is a viable encouragement for photocatalytic applications due to its unique features that include correct bandgap for UV light, strong chemical and thermal stability. Several research have addressed the synergistic effect of SiC inclusion to TiO_2 , and they have all shown that this leads to enhanced photocatalytic activity because SiC grains and TiO_2 nanoparticles form heterojunctions.^{33,34)} The SiC- TiO_2 plots also allowed calculating the rate constant of the methyl orange (MO) degradation reaction heterojunctions occur simply by covering the photocatalytic surface with SiC foam layers. The developed photocatalytic activity causes the charge carriers' movement between the conduction and valance band of TiO_2 and SiC.³⁵⁾ Furthermore, the combination of MnO_2 , SiC fiber, and TiO_2 can facilitate efficient charge separation and migration during photocatalysis. MnO_2 and TiO_2 are known for their ability to generate electron-hole pairs under light irradiation. SiC, being a semiconductor, can also contribute to electron transfer processes. This synergy allows for more effective separation of photo-generated electrons and holes, reducing charge recombination, which is crucial for improved photocatalytic performance. Therefore, we selected the MnO_2 -SiC- TiO_2 nanocomposite of good efficiency for photocatalytic degradation.

2. Experimental Procedure

2.1. Chemicals

Methylene blue (MB) and MO was procured from (Duck-san Chemical Co., Korea), Titanium Tetra-n-butoxide Monomer from Kanto Chemical Co. (Japan), Manganese (II) Chloride Tetrahydrate purchased from Daejung (Korea), DI water was made by self- product.

2.2. Synthesis of the nanocomposite

13.5 g SiC fiber and Titanium Tetra-n-butoxide 3.3 g were dissolved in 100 mL distil water under magnetic stirring for the synthesis of SiC- TiO_2 composite. After the mixing, this solution provided into the dryer at 100 °C for 24 h, then powder formed. Afterwards, 6.75 g SiC- TiO_2 powder and 1.94 g Manganese (II) Chloride Tetrahydrate powder were dispersed in 100 mL DI water mixed using by magnetic stirring. After well mixing, this solution was dried for 24 h at 100 °C. Then, the following synthesis chemical substances were calcined at 1,000 °C.

2.3. Characterization

X-ray diffraction (XRD, SHIMADZU XRD-6000) spectrophotometry was employed to investigate the nanocomposite crystallinity and chemical functionalization. SHIMADZU XRD-6000 equipment was utilized to gather the XRD data, along with a Cu-K α X-ray source (1.5406) fitted throughout a 2-theta range of 10–90° at 40 kV and 40 mA. Scanning electron microscopy (SEM; JSM-5600 JEOL, Akishima, Tokyo, Japan) and the surface dispersion of the nanocomposites was evaluated using transmission electron microscopy (TEM; Hitachi HT7700, Tokyo, Japan) with bright-field imaging at 100 kV. The bandgap energies of the samples were calibrated using the Kubelka-Munk function, which utilizes UV-Vis diffuse reflectance spectroscopy (DRS) data (SHIMADZU UV-2600). Using a 150 W Halogen bulb and a tabletop SLS301-stabilized Tungsten-Halogen light source, the photocurrent value was determined. Electrochemical impedance spectra or EIS were utilized to measure the sample resistivity and rate of deterioration using an electrochemical analyzer (Zahner, Germany).

3. Results and Discussion

The XRD patterns of SiC-TiO₂ and MnO₂-SiC-TiO₂ nanocomposites are demonstrated in Fig. 1. The TiO₂ coated SiC generated a thin layer of TiO₂ on SiC fibers reduces the intensity of the SiC fibers' XRD peaks, and a few additional

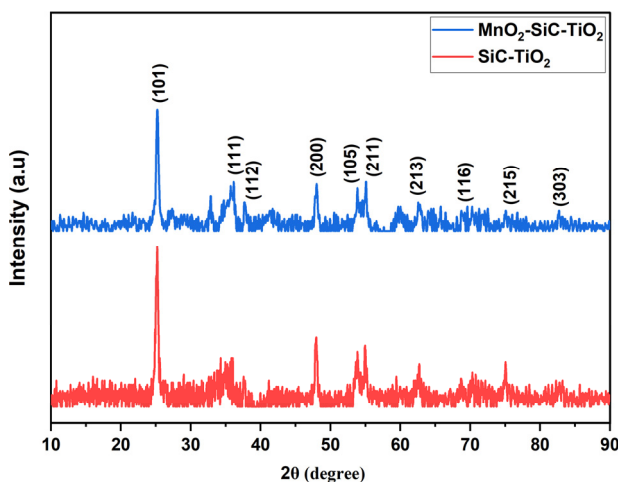


Fig. 1. XRD result of the SiC-TiO₂ and SiC-Mn-TiO₂.

peaks are seen that correspond to TiO₂. The major diffraction peaks of the synthesized TiO₂ can be indexed as (101), (111), (112), (200), (105), (211), (213), (116), (215) and (303) peaks are located at 2θ of 25.28, 36.94, 38.58, 48.50, 53.89, 55.06, 62.12, 68.76, 75.03, 82.13⁰, respectively.³⁶⁾ The SEM indicated the profile of the sample that was scanned using a high-energy electron beam and exhibited the images in Fig. 2. The SiC-TiO₂ composite of SEM images were presented in Fig. 2(a). The TiO₂ had a large, differently sized particle, and the TiO₂ NPs were widely distributed. The SiC fiber has a plentiful surface with spread out TiO₂. The SiC fiber's diameter was 11.3 nm, and its surface was smooth. The SiC fiber's physical characteristics provided the ideal setting for metal oxide semiconductors to create the best SEM profiles. The mixed morphology of MnO₂ with SiC-TiO₂ composite (rod-like and spherical) is revealed in the high magnification image of Fig. 2(b). For each sample, we acquired TEM pictures at various magnifications. The MnO₂-SiC-TiO₂ and SiC-TiO₂ nanocomposite analysis applying TEM to reveal the perfect images of the particles size and structure, as

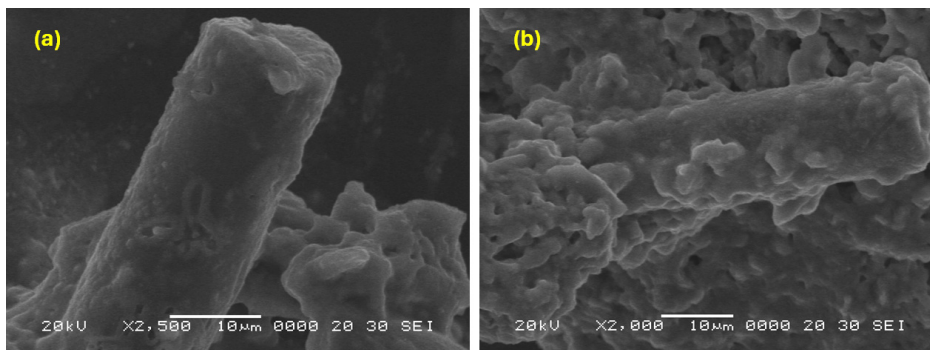


Fig. 2. SEM of the (a) SiC-TiO₂; (b) MnO₂-SiC-TiO₂.

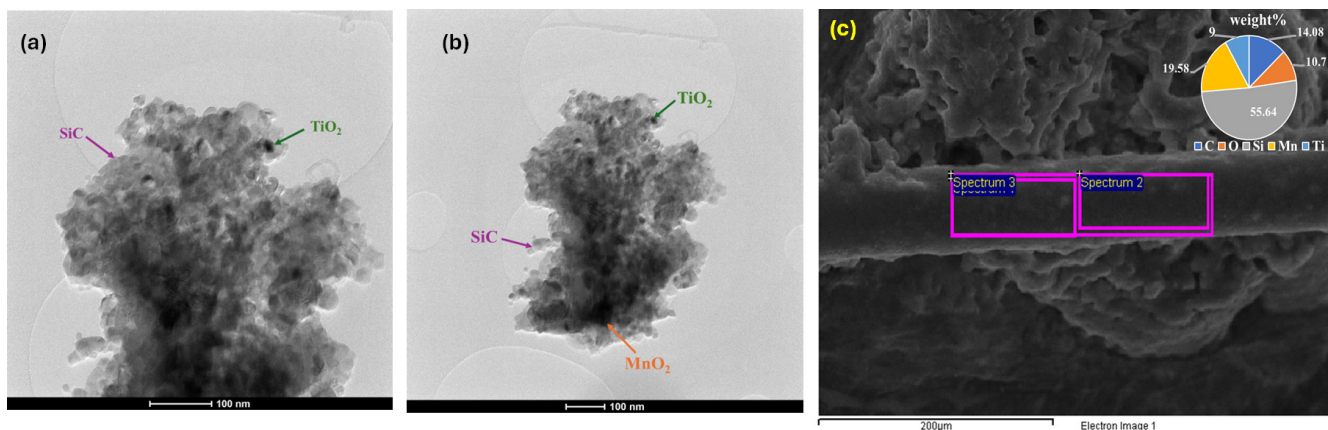


Fig. 3. TEM image of the (a) SiC-TiO₂; (b) MnO₂-SiC-TiO₂ and (c) EDS elemental analysis of C, O, Si, Mn and Ti.

shown in Fig. 3(a, b). As we observe, the dimensions of the nanoparticles of TiO_2 powders have been shown to have simultaneously spherical and rod-shaped particles, and MnO_2 and SiC nanoparticles size is physically aggregated. As shown in Fig. 3(c), by using energy-dispersive spectroscopy (EDS) analysis, the C, O, Si, Mn and Ti components verified from the elemental mapping of $\text{MnO}_2\text{-SiC-TiO}_2$ nanocomposite. UV-Vis absorbance was utilized to measure the degradation of MB concentrations, which is applied as the contaminant.³⁷⁾ Fig. 4(a) presents an effective characterization of the greatest peak of UV-Vis absorbance, which ranges 665 nm. We examined the degradation of contaminants using the wavelength shifting of the greatest peak.^{38,39)} The UV-Vis light absorption characteristics of the produced photocatalysts are examined using DRS. The Kubelka-Munk theory is used to determine the band-gap value of semiconductors.⁴⁰⁾ In the formula $\alpha h\nu = A(h\nu - E_g)^{1/2}$, the absorption coefficient, light frequency range, balance unchanged, and band gap are denoted by the values of α , ν , A , and E_g . The plot of $(\alpha h\nu)^{1/2}$ versus energy ($h\nu$) is shown in Fig. 4(b). The band gap of SiC-TiO_2 and $\text{MnO}_2\text{-SiC-TiO}_2$ is 3.23 eV and 3.21 eV, respectively. After addition of MnO_2 the band gap is slightly smaller than SiC-TiO_2 . Thus, the exposure to visible

light increases the use of visible light and the activity of photocatalysis. As shown in Fig. 4(c), the EIS test result of the SiC-TiO_2 , $\text{MnO}_2\text{-SiC-TiO}_2$ nanocomposite electrode in the phosphate buffer solution (PBS). The high resistivity of those samples is readily visible. The $\text{MnO}_2\text{-SiC-TiO}_2$ composite has demonstrated low resistivity in comparison to the SiC-TiO_2 sample. Thus, the $\text{MnO}_2\text{-SiC-TiO}_2$ composite has a large electron transfer carrier. By using photocurrent responses, the effectiveness of charge collection was demonstrated, and the investigation focused on the excitation, separation, transfer, and recombination of photogenerated charge carriers in the samples. Using several on-off light cycles of the periodic, the photocurrent measurement versus the time of various photoelectrodes was shown in Fig. 4(d, e). As shown in Fig. 4(d) that the photocurrent response of the SiC-TiO_2 composite is higher current density than the $\text{MnO}_2\text{-SiC-TiO}_2$, and [Fig. 4(e)] nanocomposite sample due to the MnO_2 could modify band structure of the SiC-TiO_2 sample, altering the positions of the valence band and conduction band. However, the lower density of the $\text{MnO}_2\text{-SiC-TiO}_2$ nanocomposite has indicated that the increased surface area and improved light absorption to contribute the enhanced performance and efficiency of the photoelectrode applica-

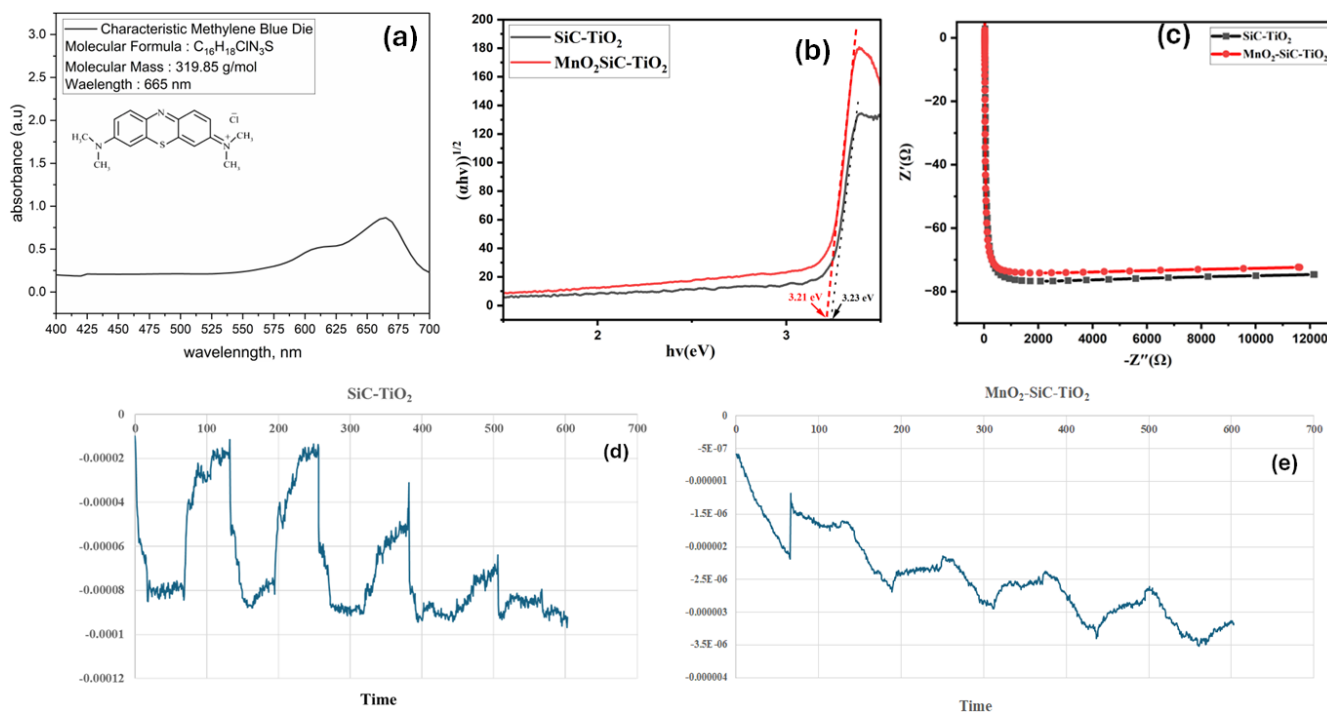


Fig. 4. (a) Methylene blue wavelength; (b) DRS plot and (c) EIS plot of the SiC-TiO_2 , and $\text{MnO}_2\text{-SiC-TiO}_2$; Photocurrent test of the (d) SiC-TiO_2 and (e) $\text{MnO}_2\text{-SiC-TiO}_2$

tions.

Fig. 5(a) illustrates the photocatalytic performance as evaluated by the MB dye degradation in the presence of visible light, which is triggered by TiO₂, SiC and MnO₂ composite. During the experiments with the MB solution of a catalyst, it was found that the decrease in MB concentration was mostly related to the photolysis effect delivered by visible light. The adsorption capacity of the MnO₂ is higher than with SiC and TiO₂ sample. Therefore, after binding MnO₂ with SiC-TiO₂ composite, its photocatalytic efficacy is higher compared with SiC-TiO₂ composite. As shown in Fig. 5(b), we observed the photocatalytic degradation efficiencies of MnO₂-SiC-TiO₂ nanocomposites and SiC-TiO₂ to learn more about the photocatalytic activity of the MB. Evidently, the MnO₂-SiC-TiO₂ nanocomposite exhibits a photocatalytic degradation efficiency that approaches almost 20 % after 120 min of irradiation, surpassing that of SiC-TiO₂ composite. According to the data above, MnO₂-SiC-TiO₂ nanocomposites have superior photocatalytic decomposition activity and increased degradation efficiency. MnO₂ may extend the light absorption range of the catalyst system. While TiO₂ primarily absorbs UV light, MnO₂ can absorb visible light. In Fig. 5(c), $\ln C/C_0$ is plotted versus t , where C denotes the MB concentration at time t and C_0 is the MB concentration at initialization. The degradation process's kinetics suit the pseudo-first-order kinetics model well. The logarithmic correlation was also found between the obtained kinetic constant and the concentration of MB and it has a good correlation of SiC-TiO₂ and MnO₂-SiC-TiO₂ nanocomposite.

The degradation of the MO solution was chosen, as seen in

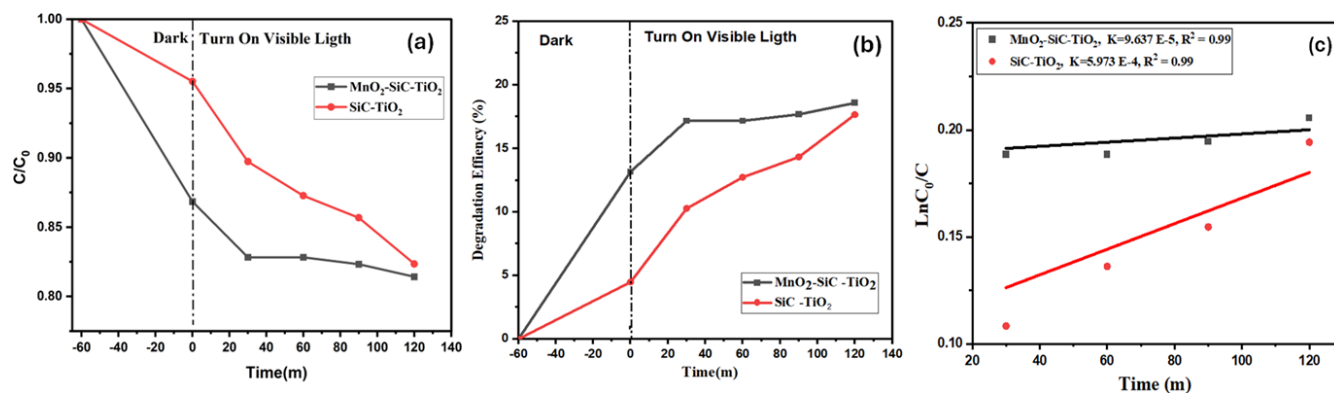


Fig. 5. (a) and (b) Photocatalytic degradation efficiency of methylene blue (MB) of the SiC-TiO₂, and MnO₂-SiC-TiO₂ under ultraviolet light irradiation; (c) Linear fittings of pseudo-first-order kinetics.

Fig. 6(a), the distinctive absorption of the MO solution at a wavelength 465 nm was used to track the adsorption and photocatalytic degradation processes. The photocatalytic efficiency and the change in organic pollutant concentration (C/C_0) together with the irradiation time over the SiC-TiO₂ and MnO₂-SiC-TiO₂ composite in visible light. Regarding the irradiation time, C_0 is the starting concentration and C is the current concentration. The outcomes are shown in Fig. 6(b); it can be observed that the SiC-TiO₂ composite decreased in MO concentration within 120 min compared to the MnO₂-SiC-TiO₂ nanocomposite. The photocatalytic activities of photocatalysts under light irradiation were assessed using MO solution. As demonstrated in Fig. 6(c), after 20 min the SiC-TiO₂ composite has changed in the MO adsorption rate, which indicates the highest photocatalytic efficiency, approximately 15 % than the MnO₂-SiC-TiO₂ composite. Fig. 6(d) illustrates that a good linear correlation of $\ln(C/C_0)$ against time ($0.99 < R^2 < 1$) were obtained, implying that pseudo-first-order kinetics govern the catalytic process. The plots performed it possible to determine the MO degradation reactions rate constant as well for the reaction catalyzed by SiC-TiO₂ and MnO₂-SiC-TiO₂ composite.

In addition, photocatalytic activity of the MnO₂-SiC-TiO₂ nanocomposite was determined by different recycling analyzing the degradation of MB in the solution under UV illumination. Fig. 7(a) shows the relationship between Abs_{max} (at 660 nm) and time (h) in the decomposition cycle test for the decomposition of MB by MnO₂-SiC-TiO₂ nanocomposites. From these graphs, it can be seen that the maximum value of absorbance decreases as the number of cycle tests increases. This can be explained by the blocking of pores on the photo-

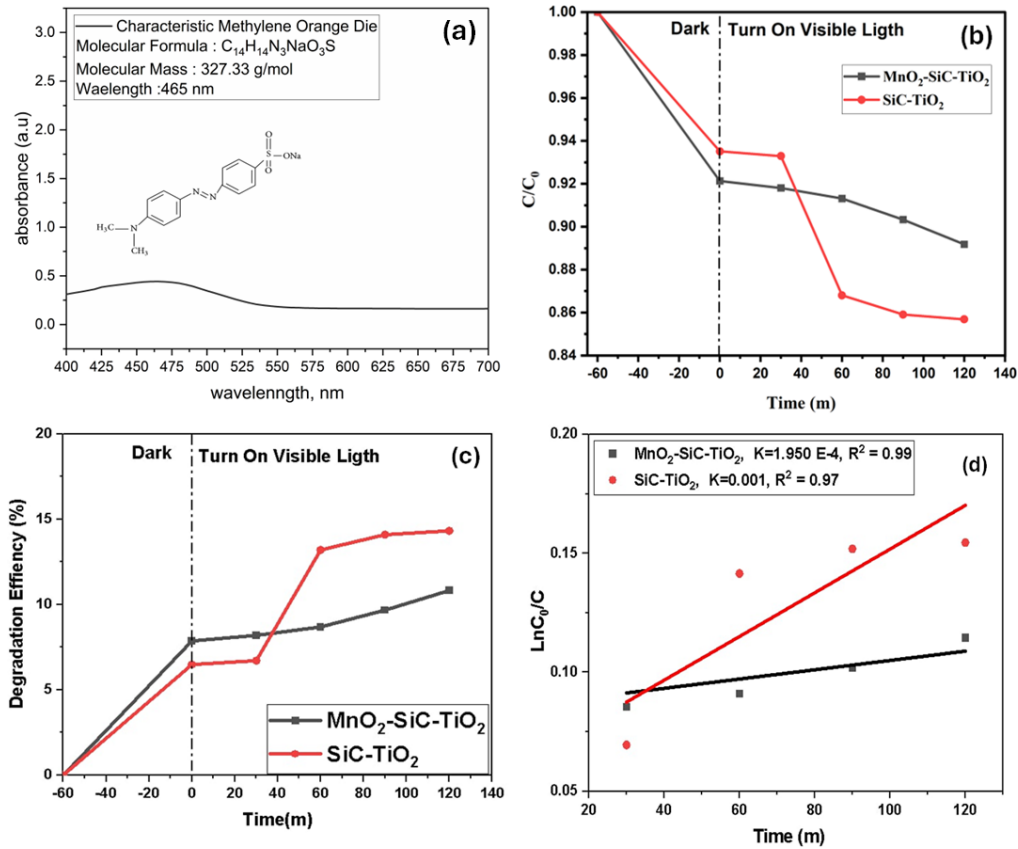


Fig. 6. (a) Methyl orange (MO) wavelength; (b, c) Photocatalytic experiment performed using SiC-TiO₂ and MnO₂-SiC-TiO₂ nanocomposite with MO; (d) Apparent first-order linear transform of MO degradation kinetic plots for SiC-TiO₂ and MnO₂-SiC-TiO₂ composites.

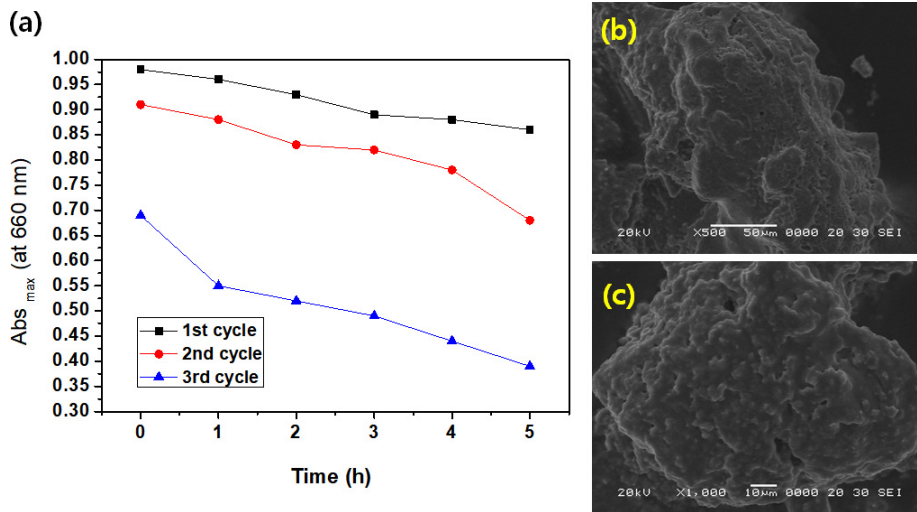


Fig. 7. Degradation cycle test (a) Abs_{max} (at 660 nm) vs. time (h) graph for the degradation of methylene blue (MB) by the MnO₂-SiC-TiO₂ nanocomposite. (b) and (c) SEM images after 3 cycle degradation for the methylene blue.

catalyst surface within a short time. Regarding these results, SEM images after 3 cycles of decomposition on MB are shown in Fig. 7 (b, c). From these images, the surface shows a rough surface state that is completely different from the

surface state before the photocatalytic test.

Fig. 8 shows the relationship between Abs_{max} (460 nm) and time (h) in the decomposition cycle test for the decomposition of MO by MnO₂-SiC-TiO₂ nanocomposites. From

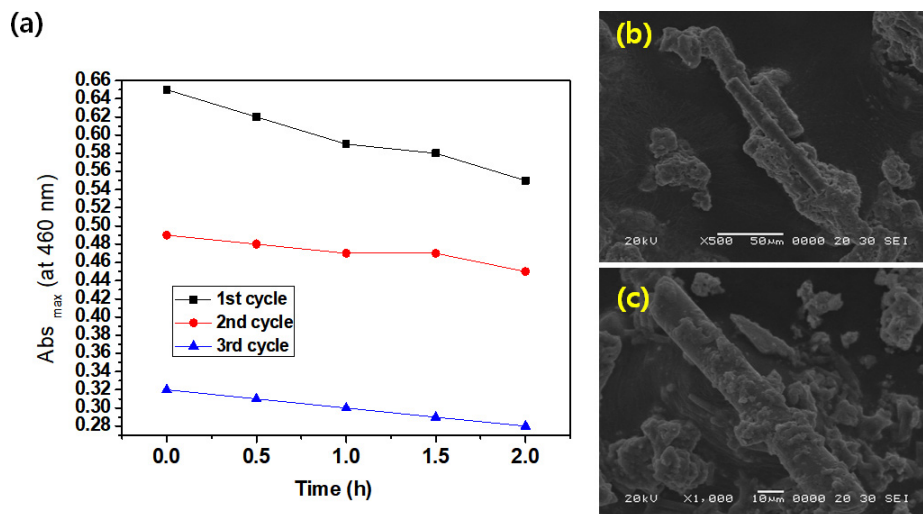


Fig. 8. Degradation cycle test (a) Abs_{max} (at 460 nm) vs. time (h) graph for the degradation of methyl orange (MO) by the MnO₂-SiC-TiO₂ nanocomposite. (b) and (c) SEM images after 3 cycle degradation for the methyl orange.

these graphs, it can be seen that as the number of cycle tests increases, the maximum value of absorbance decreases rapidly. This can also be explained by the blocking of pores on the photocatalyst surface within a short time. In addition, the MnO₂-SiC-TiO₂ nanocomposites show poor efficiency in MO decomposition compared to MB decomposition. Regarding these results, SEM images after 3 cycles of decomposition for MO are shown in Fig. 8 (b, c). These can also be confirmed from the presented images of the rough surface state, which was completely different from the surface state before the photocatalytic test.

4. Conclusion

In this research, the MnO₂-SiC-TiO₂ nanocomposite used the hydrothermal method. The optimized photocatalysts were evaluated by photocurrent testing, UV-Vis DRS, XRD, TEM, EDS and SEM. The MnO₂-SiC-TiO₂ composite enhanced the photoelectric efficiency and visible light response. The photocatalytic degradation of MB and MO was selected as a standard reaction to compare the photocatalytic activities of MnO₂-SiC-TiO₂ and SiC-TiO₂ composite, with finding showed that the MnO₂-SiC-TiO₂ nanocomposite exhibited the high efficiency in conveniently degrading of MB than that of MO. The additional characteristics of MnO₂ make it an appealing photocatalyst that reacts to visible light. Therefore, we selected the MnO₂-SiC-TiO₂ nanocomposite for the photocatalytic degradation of these hazard dyes.

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