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Enhanced Photocatalytic Activity of Boron Doped Silicon Carbide Nanofibers and Its Composite with Polyvinyl Borate

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ABSTRACT

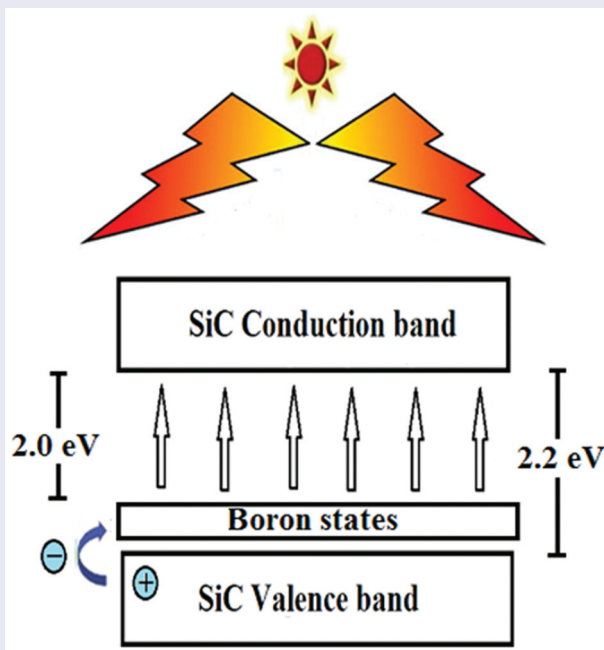
Boron doped silicon carbide (B-SiC) and its composite with polyvinyl borate (PVB) were studied due to the role of boron acting as an electron-acceptor, which can reduce the recombination rate of the photoinduced electron-hole pair on SiC upon UV light irradiation. The structure and morphology of SiC and B-SiC, and their PVB composites were characterized by FTIR, X-ray diffraction spectroscopy and field emission scanning electron microscopy. The optical property and the band gap energy of the as prepared samples were evaluated from UV-Vis absorption spectroscopy. When compared to SiC and PVB/SiC, boron doped SiC and its PVB composite exhibited enhanced photocatalytic activity toward the model dye (methylene blue) degradation owing to the effective separation of the photoinduced electron-hole pair on the photocatalyst nanofibers.

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1. Introduction

Silicon carbide (SiC), which is among the third generation of semiconductor materials, has diverse scientific and industrial applications due to superior thermal conductivity and chemical stability, high hardness and strength properties.^[1,2] Various forms of SiC such as nanofibers, nanotubes, nanofibers and mesoporous structures are utilized as a fundamental building block component of catalytic systems, electrical devices,

protective coatings and polymer composites.^[1,2] Especially, one-dimensional nanoscale SiC has attracted great attention as a component of electronic and optoelectronic devices because of its blue and yellow luminescence. In fact, SiC can be regarded as a potential alternative for the replacement of silicon in the electronic industry.^[3] In addition, SiC is considered as the most promising photocatalyst owing to the high reduction

potential of photoinduced electrons in the conduction band of SiC. In other words, the potentials of the conduction band and the valence band are suitable for the photolysis of organic materials. The use of UV light or sunlight for photocatalytic degradation of organic compounds in wastewater can be considered as a green technique, protecting the environment.^[2]

Wastewater, containing organic dye, is mainly discharged by dye processing and textile industries. With the growth of the dye processing industry, this type of wastewater has become an environmental problem.^[4] Among variety of treatment techniques, photocatalytic degradation by semiconductor photocatalysts can remove organic dye from the wastewater effectively. Almost all organic molecules harmful to the environment and the living things can be decomposed to harmless H₂O and CO₂ molecules by photocatalytic degradation technique.^[4] Similar to other semiconductor photocatalysts, SiC exhibits relatively low photocatalytic activity in the absence of a cocatalyst. The reason for the limited activity is the high recombination rate of the photoinduced electron-hole pair. Mostly, the photoinduced charge carriers tend to recombine before they diffuse to the photocatalyst surface for the catalytic degradation reactions.^[2] To suppress the recombination of the photoinduced electron-hole pair, metal or non-metal doping was applied to the semiconductor photocatalysts.^[2] Various metal atoms such as Pd,^[5] Ir,^[5] Fe^[6] and Ag^[11] have been studied as dopant with SiC to improve the photocatalytic activity. According to the literature, electron transfers were occurred between the metal atoms and the SiC photocatalyst due to the Mott-Schottky effect, which could improve the separation of photoexcited mobile charge carriers as well as the photocatalytic activity of the catalyst.^[1,5,6] On the other hand, it was reported that the incorporation of nonmetal atoms played a critical role in changing the electronic structure of SiC and improved light absorption feature. Hence, doping SiC with nonmetal atoms is one of the most leading methods to improve the catalytic activity and obtain higher efficiency.^[7] In this context, SiC was doped with C atoms,^[8] S atoms^[7] and B atoms.^[9] Yang et al.^[9] synthesized boron doped SiC nanowires. The effect of the boron doping on the photocatalytic hydrogen production mechanism of SiC was researched in the specified study. It was reported that boron atoms were substituted by Si atoms during the doping reaction, resulting in a narrower band gap. Therefore, the conduction band edge potential of B-SiC was more negative than that of undoped SiC and the photo excited electrons of B-SiC could easily leave from the conduction band of the photocatalyst, which decreased the

recombination rate of the photoexcited charge carriers and enhanced the photocatalytic activity.^[9]

As an alternative to the specified study and the literature, boron doping was applied to SiC nanofibers in this study and doped nanofibers were immobilized within polyvinyl borate (PVB) matrix through synthesizing the boron-containing polymer in the presence of the photocatalyst nanofibers. PVB is a cross-linked polymer, which contains boron oxide structures along its chains.^[10] According to the previous studies performed by myself, PVB was a mechanically and thermally stable polymer and the contact of the boron oxide structures with the semiconductor photocatalysts enhanced the photocatalytic activity.^[10,11] Within the scope of this study, the photocatalytic activity of B-SiC and PVB/SiC was analyzed and compared with undoped SiC and PVB/SiC composite by monitoring the degradation of an organic dye in aqueous solution, exposed to UV light irradiation. To the best of our knowledge, the study on the effect of boron doping on the photocatalytic activity of SiC, immobilized in PVB matrix, has not been reported so far.

2. Experimental

2.1. Doping of SiC and Preparation of PVB Composites

SiC nanofibers ($D < 2.5 \mu\text{m}$, $L/D \geq 20$, Sigma-Aldrich) were used as photocatalyst. Boric acid (H₃BO₃), from Sigma-Aldrich, was used as boron atom (B) source. Polyvinyl alcohol (PVA), whose molecular weight changes in the range from 89000–98000 g/mol with a degree of hydrolysis greater than 99 mol.%, and boric acid were used to synthesize polyvinyl borate (PVB). Methylene blue, provided from Sigma-Aldrich, was used as a model dye to monitor the photocatalytic activity of SiC nanofibers. Boron doped SiC (B-SiC) nanofibers were obtained through the wet impregnation method.^[6,12] For this purpose, appropriate amount of boric acid and SiC nanofibers (4 g) were added in ethanol-distilled water mixture (100 ml) and then the solution was kept under stirring for 3 hours. The as-prepared slurry was separated from the solution and rinsed several times with ethanol-distilled water mixture. Then the slurry was dried at 100°C for 24 hours. After drying, the obtained product was calcined at 400°C for 3 hours. Finally, boron doped SiC (B-SiC) nanofibers were obtained. Boron atom content of SiC nanofibers was adjusted as 1 wt%.^[6,12]

PVB/SiC and PVB/B-SiC composites were prepared through the condensation reaction of PVA and boric acid in a B–OH: PVA–OH molar ratio of 1.4:1 (Figure 1).^[10,13]

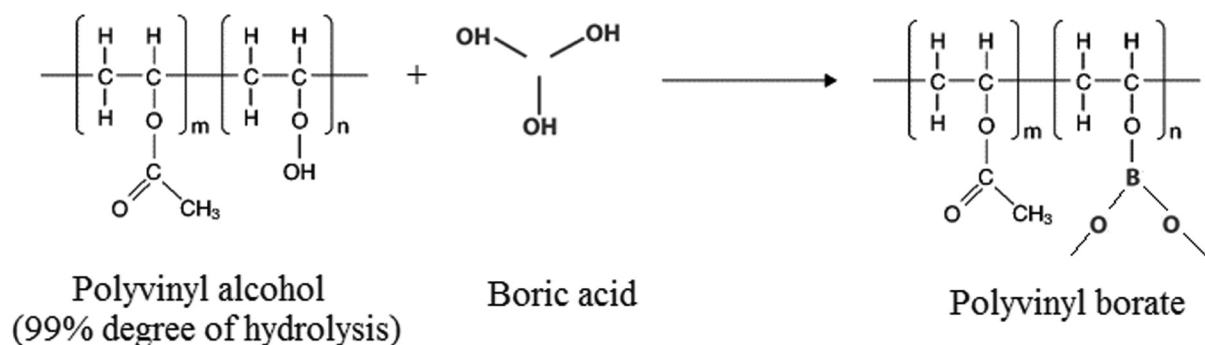


Figure 1. Reaction pathway between PVA and boric acid to form PVB.

For this purpose, PVA (2 g) was dissolved in distilled water (50 ml) by heating at 80°C with constant stirring. Appropriate amount of the photocatalyst (SiC or B-SiC) was added to the PVA solution and dispersed using the ultrasonic bath. At the same time, boric acid (2 g) was dissolved in distilled water (50 ml) under stirring and added into the PVA solution, including the photocatalyst nanofibers (SiC or B-SiC). The as-prepared solution was kept under stirring for 30 minutes. Then the obtained composite (PVB/SiC or PVB/B-SiC) was separated from the solution and dried at 120°C for 24 hours. PVB composites, containing 20 wt.% SiC or B-SiC, were prepared in this way.^[10,13]

2.2. Methods of Characterization

SiC, B-SiC, PVB/B-SiC were subjected to FTIR spectroscopy to examine the chemical structure of the as-prepared photocatalyst systems by using a Nicolet 380 (Thermo Scientific) spectrophotometer in the region of 4000–400 cm⁻¹. The crystal structure of SiC and B-SiC were characterized by XRD analysis through Rigaku Ultima IV X-ray diffractometer over a 2θ range from 20° to 80° using Cu Kα radiation (1.54 Å). The morphology of all samples was monitored by a field emission scanning electron microscope (FE-SEM, QUANTA 400 F). The optical absorption ability of all samples was investigated by UV-Vis absorption spectroscopy using a Genesys 10S (Thermo Scientific) spectrophotometer in the wavelength range from 200 nm to 800 nm.

The photocatalytic activity of the samples was evaluated by monitoring the degradation of the model dye, methylene blue, in aqueous solution using a Genesys 10S UV-VIS spectrophotometer (Thermo Scientific). A 12 W UVA lamp (320 nm – 400 nm) was used as the light source to degrade the dye molecules. To determine the photocatalytic activity, 0.1 g of a photocatalyst sample was added to 100 ml of methylene blue solution (10 mg/l). Before irradiation, the dye solution including

the photocatalyst sample was held in the dark for half an hour to attain adsorption/desorption equilibrium between the dye molecules and the photocatalyst particles. The methylene blue solution was irradiated with the specified UVA lamp for 300 minutes. At certain time intervals (60 minutes), 3 ml of the methylene blue solution was sampled and then separated from the photocatalyst particles for the photocatalytic activity test. The photocatalytic degradation efficiency of the samples was analyzed by determining and comparing the intensity of the optical absorption peak of methylene blue at 663 nm.

3. Results and Discussion

3.1. FTIR Analysis

FTIR spectrum of pure SiC nanofibers is shown in Figure 1a. The absorption bands are shown at around 3448 cm⁻¹ and 1630 cm⁻¹, which were attributed to the -OH stretching vibration and the O-H deformed vibration, respectively.^[14] The reason for these weak peaks might be slight water content in the nanofibers. The absorption peak between 1010 cm⁻¹ and 670 cm⁻¹ was related to the stretching vibration of the Si-C bond (Figure 2a).^[14] FTIR spectrum of B-SiC nanofibers also exhibits the weak absorption bands of the -OH stretching vibration and the O-H deformed vibration at around 3450 cm⁻¹ and 1625 cm⁻¹, respectively (Figure 2b). The absorption peak of the Si-C bond shifted slightly toward the lower wavenumber region and the intensity of this peak was more than that of the pure nanofibers due to boron doping. No other peaks could be seen in the FTIR spectrum of B-SiC nanofibers (Figure 2b). On the other hand, the broad band between 3650 cm⁻¹ and 2960 cm⁻¹ in the FTIR spectrum of PVB was related to the -OH group of polyvinyl alcohol, formed complexes with boron-containing oxyanions.^[13] The absorption peaks at 1280 cm⁻¹ and 1118 cm⁻¹ were attributed to the stretching vibrations of the B-O-C bonds, which

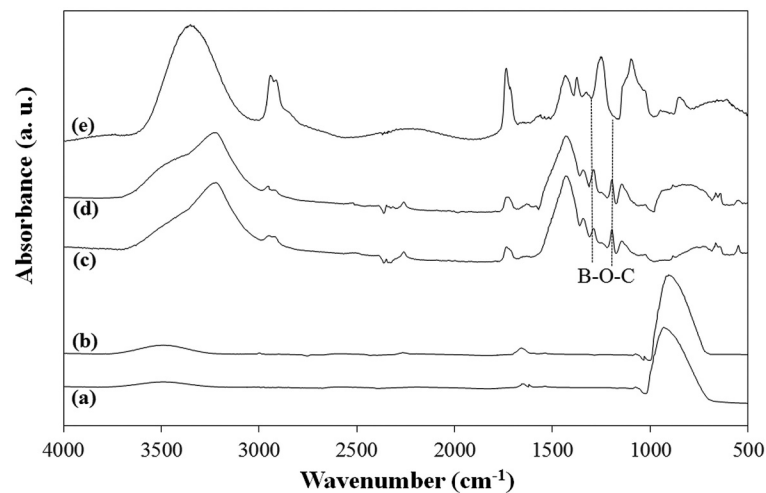


Figure 2. FTIR spectra of (a) SiC, (b) B-SiC, (c) PVB, (d) PVB/B-SiC and (e) PVA.

confirmed the reaction between polyvinyl alcohol and boric acid to form polyvinyl borate.^[13] FTIR spectrum of PVA did not contain these characteristic absorption peaks of PVB (Figure 2e). The absorption peaks seen at 2902 cm^{-1} , 1708 cm^{-1} , 1411 cm^{-1} and 1332 cm^{-1} were assigned to the C = O, C-H, B-O and C-O bonds, respectively (Figure 2c).^[13] Despite having a high degree of hydrolysis (99%), the absorption peak observed at 2902 cm^{-1} on the spectrum PVA and PVB composites was thought to be due to the reactant material, polyvinyl acetate, used to produce PVA. FTIR spectrum of PVB/B-SiC was similar to the spectrum of the pure polymer. The absorption peaks present at 1280 cm^{-1} and 1118 cm^{-1} were also present on the spectrum of PVB/B-SiC, confirming the formation of PVB chains in the presence B-SiC (Figure 2d). The characteristic peak of the Si-C bond also appeared on the spectrum of PVB/B-SiC between 990 cm^{-1} and 660 cm^{-1} , indicating the presence of B-SiC nanofibers in PVB matrix. This characteristic absorption peak of Si-C shifted toward lower wavenumbers. This situation reveals the interaction between PVB and B-SiC in the composite.^[15]

3.2. XRD Analysis

Pure SiC nanofibers have diffraction peaks at the angles of 34.92° , 40.96° , 60.38° , 71.84° and 75.39° corresponding to (111), (200), (220), (311) and (222) peaks characteristic to the cubic crystal structure of SiC (Figure 3a).^[16] All the XRD peaks correspond to the different facets of β -SiC (JCPDS, No. 29-1129).^[17] With boron contribution, the diffraction peaks shifted slightly toward larger diffraction angles (35.12° , 41.30° , 60.58° , 72.08° and 75.12°) (Figure 3c). The shift in the diffraction angle values was related to the distortion in the

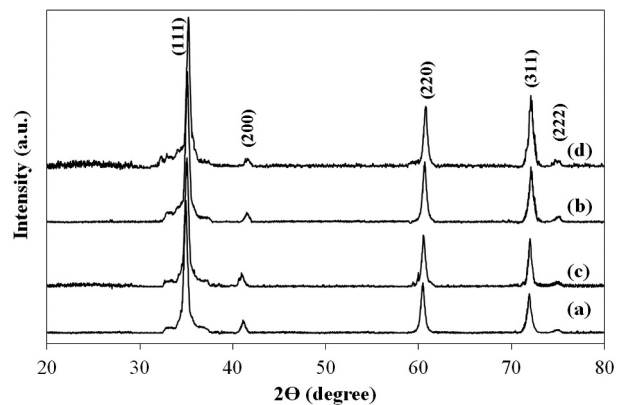


Figure 3. XRD spectra of (a) SiC, (b) PVB/SiC, (c) B-SiC and (d) PVB/B-SiC.

cubic crystal structure due the substitution of boron atoms (0.095 nm) by silicon atoms (0.134 nm) in SiC crystal lattice. In addition to the specified diffraction peaks, no other peaks were seen on the XRD spectrum of B-SiC, supporting the claim that boron atoms integrated into SiC crystal structure.^[16,17]

The mean size of crystalline SiC domains in the pure nanofibers and the composites were calculated using the Debye-Scherrer formula:

$$D = k\lambda/\beta\cos\theta \quad (1)$$

where k is the dimensionless shape factor (0.9), λ is the X-ray wavelength; β is the full width at half the maximum intensity of the main diffraction peak and θ is the Bragg angle.^[18] For SiC and B-SiC nanofibers, the average crystallite size was 21.8 nm and 20.9 nm, respectively. The average crystallite size of SiC reduced slightly due to the distortion effect of the boron doping.

The same diffraction peaks of SiC and B-SiC were also observed on the XRD spectrum of the composites

(Figures 3b and 3d). Apart from that, XRD spectrum of PVB/SiC and PVB/B-SiC exhibited one broader peak between $2\theta = 20^\circ$ - 30° , which was attributed to the amorphous structure of PVB. Within the composite structure, no significant changes were observed in the diffraction angle and the intensity of the SiC and B-SiC peaks. The peak intensity of SiC and B-SiC shifted slightly toward larger diffraction angles, confirming the formation of the PVB layers around the SiC nanofibers.^[18] For the PVB composites, the average crystallite size of SiC and B-SiC were 21.3 nm and 20.2 nm, respectively. No significant changes were observed in the crystal structure of SiC and B-SiC nanofibers within the composite structure.

3.3. Morphological Analyses

The SEM image in Figure 4 reveals that both SiC and B-SiC nanofibers have a nearly cylindrical shape with an average diameter, ranging from 500 nm to 1000 nm. It was clearly observed that the introduction of boron atoms did not affect the morphology of SiC nanofibers. According to the SEM image in Figure 5, SiC and B-SiC nanofibers were dispersed uniformly in PVB matrix and the composites had dense structure. SiC and B-SiC nanofibers exhibited relatively good adhesion to the

PVB matrix. PVB encapsulated SiC nanofibers. In certain regions of the matrix, micron sized voids can be observed at PVB-SiC interfaces. The hydrophobic structure of SiC nanofibers might cause the specified micron sized voids within the matrix.

3.4. UV-Vis spectroscopy

Optical absorption behavior of SiC, B-SiC, PVB/SiC and PVB/B-SiC was studied by UV-Vis spectroscopy. All samples exhibited wide absorption from UV light to visible light (Figure 6). Different from the UV-Vis spectrum of SiC, B-SiC had an absorption band at around 390 nm, which might be attributed to B-C stretching vibrations.^[16,17] Boron atoms might replace by silicon atoms in the SiC crystal lattice and this replacement might cause the specified band in the spectrum. PVB enhanced the absorption of SiC and B-SiC between 250 nm and 800 nm. Any effect to enhance the absorption of irradiating photons could improve the subsequent photocatalytic activity,^[19] which was also confirmed by the methylene blue degradation experiments.

The band gap energy of the samples was estimated from the optical absorption measurements by using the Tauc equation^{[17]:}

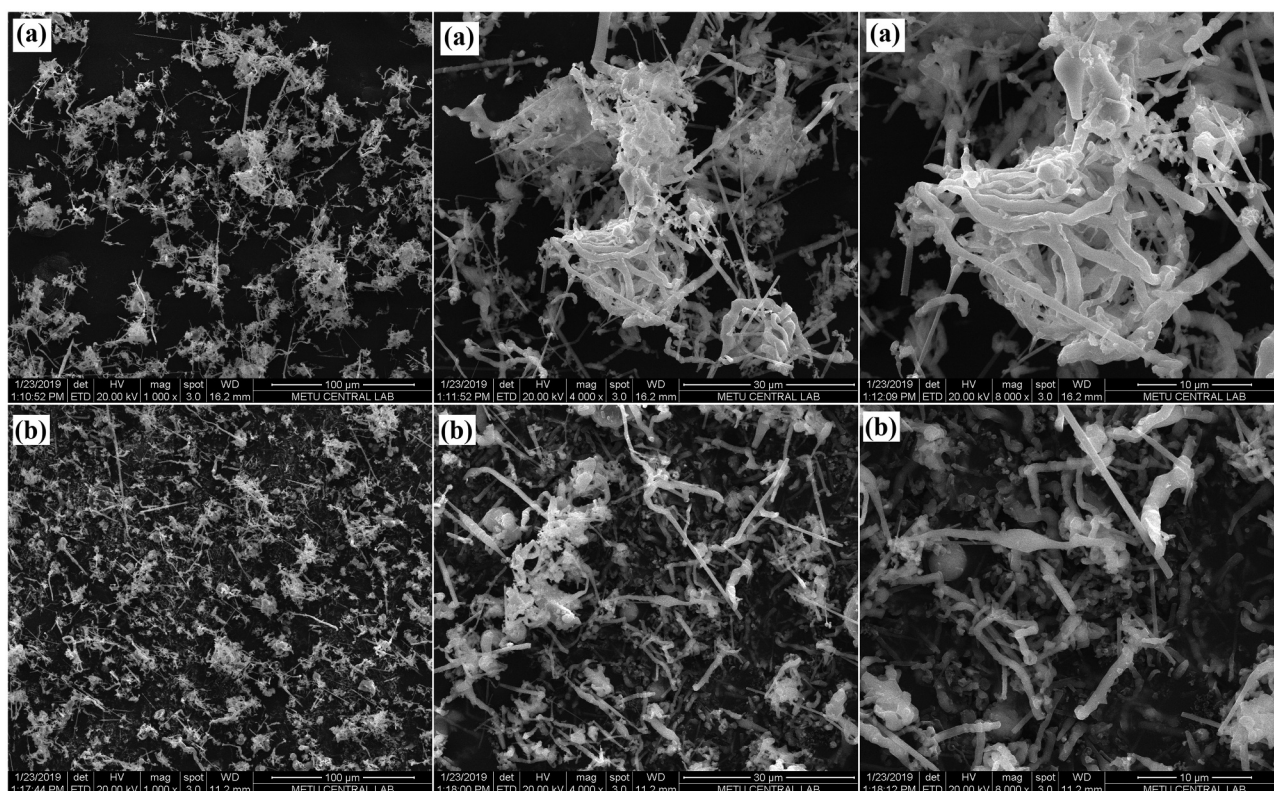


Figure 4. SEM images of (a) SiC and (b) B-SiC.

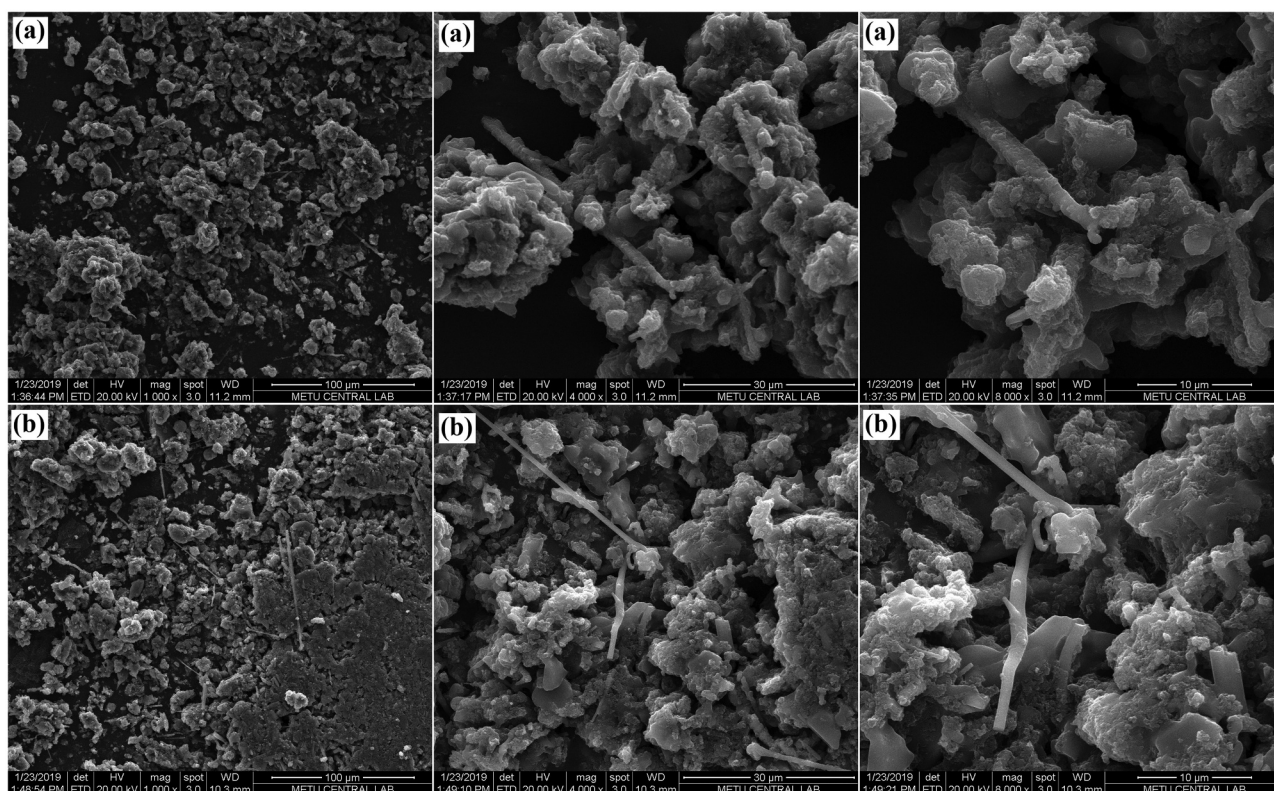


Figure 5. SEM images of (a) PVB/SiC and (b) PVB/B-SiC.

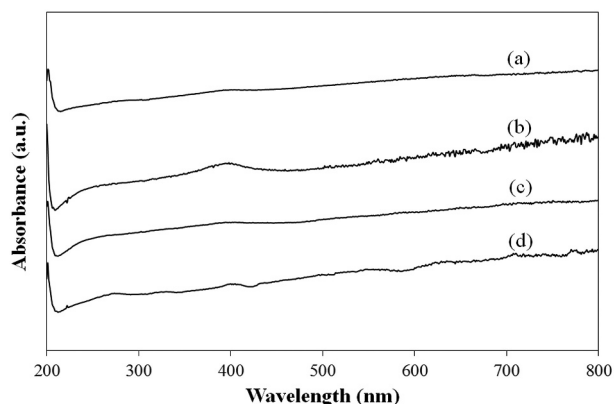


Figure 6. UV-Vis absorbance spectra of (a) SiC, (b) B-SiC, (c) PVB/SiC and (d) PVB/B-SiC.

$$\alpha hv = A(hv - E_g)^{1/2} \quad (2)$$

where A , α , E_g and $h\nu$ are a proportionality constant, the absorption coefficient, the band gap energy and the photon energy, respectively. From the plot of $h\nu$ vs. $(\alpha h\nu)^2$, the band gap energy was estimated by extrapolating the linear part of the curve to $(\alpha h\nu)^2 = 0$ (Figure 7).^[17] The band gap value of SiC decreased from 2.2 eV to 2.0 eV with boron doping. Boron atoms might induce acceptor energy states above the valance band of SiC,

which gave rise to narrowed band gap.^[17] As expected, the band gap of PVB/B-SiC (1.9 eV) was narrower than that of PVB/SiC (2.1 eV) due to boron doping.

3.5. Photocatalytic activity

Photocatalytic activity of all samples was evaluated through the model dye, methylene blue, degradation experiments. Figure 8 illustrates the absorbance of methylene blue in the presence of the photocatalyst samples as a function of irradiation time. As irradiation time increased, the absorption intensity decreased for all samples. To make the photocatalytic performance of the samples more clearly, the curves of C_t/C_0 versus irradiation time were plotted and shown in Figure 9. The model dye, methylene blue, was degraded 10.3% by SiC within 300 minutes. When compared with SiC, B-SiC exhibited high photocatalytic activity. Methylene blue was degraded 16.3% within 300 minutes by B-SiC. According to the literature boron atoms might diffuse through the interstitial sites of SiC and/or substitute for Si atoms in the crystal lattice. The diffusion of boron atoms through the interstitial sites of SiC could reduce the recombination rate of the photoexcited charge carriers by trapping the photoinduced electrons on the conduction band of the photocatalyst nanofiber.^[20] On

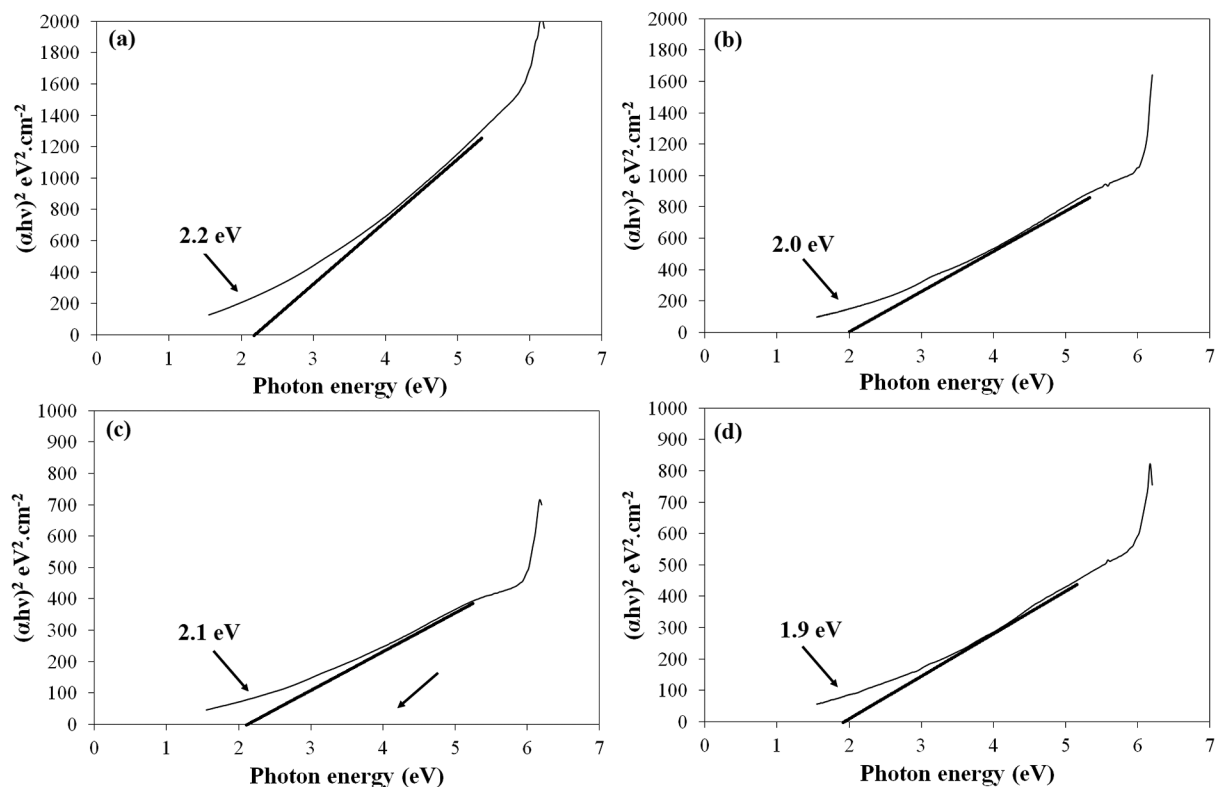


Figure 7. Tauc's plot for (a) SiC, (b) B-SiC, (c) PVB/SiC and (d) PVB/B-SiC.

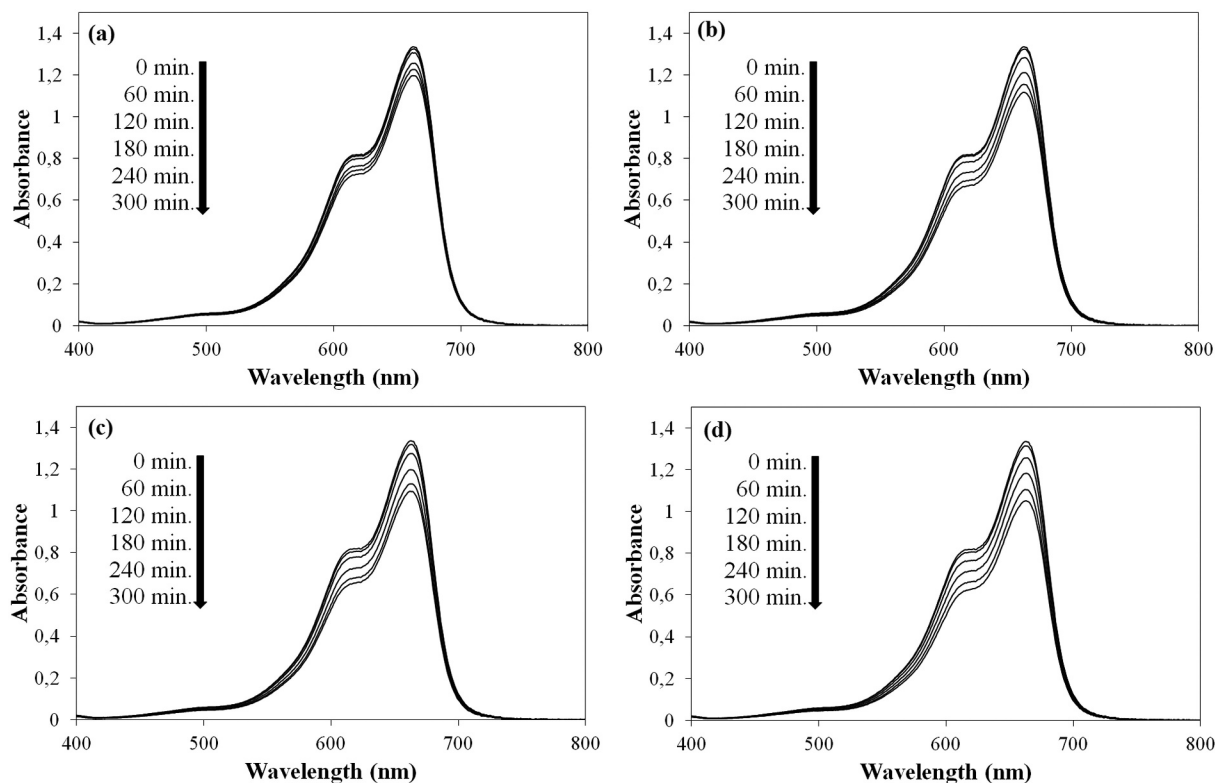


Figure 8. The variation in the UV-visible spectrum of the dye solution including (a) SiC, (b) B-SiC, (c) PVB/SiC and (d) PVB/B-SiC.

the other hand substituting boron atoms for silicon atoms might convert Si^{4+} ion to Si^{3+} ion in the crystal

lattice, promoting the charge separation by acting as photoexcited electron traps.^[20] Hence more charge

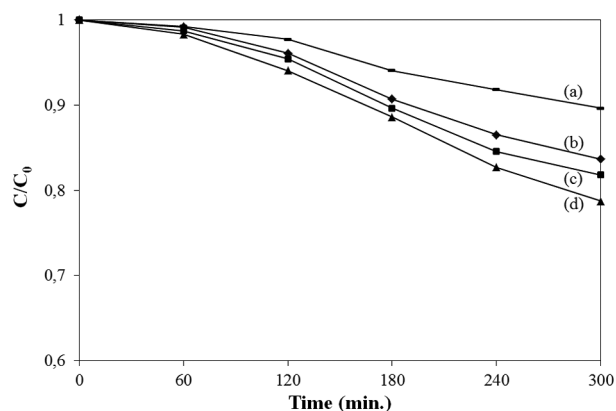


Figure 9. Degradation efficiency of the model dye, methylene blue, over (a) SiC, (b) B-SiC, (c) PVB/SiC and (d) PVB/B-SiC under UVA light irradiation.

carriers could be generated by visible light in boron doped photocatalyst and the photogenerated charge carriers could transfer in the photocatalyst with the lowest impedance and recombination rate, which could favor the subsequent photocatalytic degradation reaction.^[21]

PVB/SiC composite could degrade 18.2% of the original methylene blue via photocatalytic activity. On the other hand, 21.3% of the methylene blue molecules were degraded on PVB/B-SiC after 300 minutes of UV irradiation. The mechanism for enhanced photocatalytic activity of both SiC and B-SiC after combining with PVB might be complex. Under UV light irradiation, electrons from the valance band of the photocatalyst induced to the conduction band, resulting photoinduced electrons and holes in the conduction band and in the valance band, respectively. The photoinduced charge carriers can combine in a very short time if they are not utilized for reaction with the dye molecules. The transfer of the photoinduced charge carriers is also difficult so that the photocatalytic degradation reaction can occur mainly on the surface of the photocatalyst nanoparticles.^[21] It was known that there are mainly two active species, hydroxyl radicals and superoxide radicals, which were formed during the photocatalytic degradation reaction induced by UV light irradiation. The photogenerated active species can react with almost all organic compounds by direct electron transfer or hydrogen atom transfer.^[22] An effective reaction between the active species and the organic pollutant depends on fast transfer of the dye molecules from the aquatic medium to the surface of photocatalyst nanoparticles. According to the literature, dispersion of the photocatalyst nanoparticles in the polymer matrix is beneficial to both absorption of irradiating photons and adsorption of the dye molecules from the aquatic medium, which could also favor the subsequent

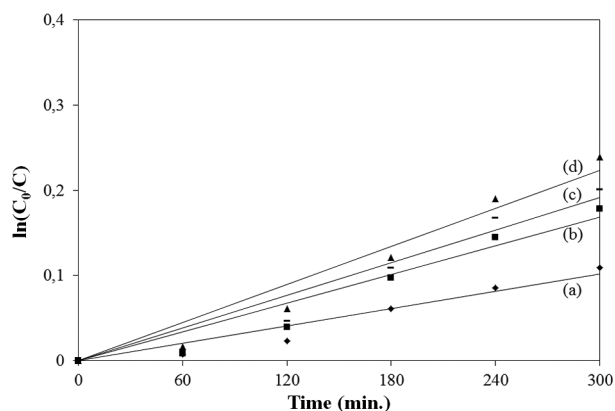


Figure 10. Kinetic plot of degradation reaction of the model dye, methylene blue, over (a) SiC, (b) B-SiC, (c) PVB/SiC and (d) PVB/B-SiC under UVA light irradiation.

photocatalytic degradation reaction.^[21] For PVB composites, boron oxide (B_2O_3) (Figure 1) structures along the chains of the polymer could serve as an electron acceptor of the photoexcited electrons of both SiC and B-SiC, which might enhance the photocatalytic activity of the photocatalyst nanofibers by reducing the recombination rate of the charge carriers.^[11,23] Hence more proportion of the photoinduced charge carriers could remain both on the valence band and the conduction band of the photocatalyst nanofibers to generate active radicals, which were necessary for the photocatalytic degradation of the model dye. The synergic contribution of boron oxide structures on the PVB chains and SiC nanofibers might improve the photocatalytic activity of the composites compared to pure SiC and B-SiC.^[11,23] Methylene blue degradation results indicated that both SiC and B-SiC in the PVB matrix have high photocatalytic activity for degrading the model dye under UVA irradiation.

In order to determine the kinetics of the photocatalytic degradation reactions, the following pseudo-first order kinetic model was utilized^[24,]

$$\ln(C_0/C_t) = kt \quad (3)$$

where C_0 and C_t are the concentration of the model dye before and after UV irradiation, t is the time and k is the degradation rate constant, which was determined from the slope of the plot of $\ln(C_0/C_t)$ vs. irradiation time t (Figure 10). The determined rate constants are

Table 1. The discoloration rate of methylene blue for SiC, B-SiC, PVB/SiC and PVB/B-SiC composites.

Sample	k (min^{-1})	R^2
SiC	0.0003	0.9437
B-SiC	0.0006	0.9406
PVB/SiC	0.0006	0.9460
PVB/B-SiC	0.0007	0.9537

illustrated in Table 1. PVB/B-SiC exhibited the highest rate constant value among the photocatalyst samples analyzed in this study. According to the degradation rate constants, the photocatalytic efficiency of SiC nanofibers was enhanced by 2 times by introducing boron atoms.

4. Conclusions

B doped SiC was prepared by using the wet impregnation technique and the photocatalyst nanofibers were immobilized within PVB matrix through the condensation reaction. Boron atoms substituted silicon atoms during the doping process, resulting in a narrower band gap. Under UVA light irradiation, B-doped SiC exhibited an enhanced photocatalytic activity and PVB/B-SiC provided the best performance for the photocatalytic degradation of methylene blue. PVB/B-SiC provided almost 1.2 times enhancement in the reaction rate constant of the photocatalytic degradation compared with that of the PVB/SiC composite. As a conclusion, B doped SiC and its composite with PVB could be considered as a promising photocatalyst to degrade organic dyes within the wastewater.

Biographical note

Dr. H. Nagehan Koysuren was born in Konya (Turkey) in 1982. She received her B.S. degree from Selcuk University (Konya, Turkey) in 2004 and Ph.D. degree from the same University in 2013. Dr. Koysuren started her academic career as a research assistant in the Environmental Engineering department of Selcuk University in 2006 and she is working as Asst. Prof. in the Environmental Engineering department of Ahi Evran University (Kirsehir, Turkey). Dr. Koysuren's research interests include photocatalytic activity, wastewater treatment, polymer composites and heavy metal adsorption.

Conflicts of interest

The author declares that she has no conflicts of interest regarding the publication of this paper.

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