



Adsorption and Photocatalytic Processes of Mesoporous SiO₂-Coated Monoclinic BiVO₄

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The silicon dioxide (SiO₂)-coated bismuth vanadate (BiVO₄) composites as visible-driven-photocatalysts were successfully synthesized by the co-precipitation method. The effects of SiO₂ coating on the structure, optical property, morphology and surface properties of the composites were investigated by X-ray diffraction (XRD), UV-visible diffuse reflectance spectroscopy (DRS), transmission electron microscopy (TEM) and Brunauer-Emmett-Teller (BET) measurements. The photocatalytic activity of monoclinic BiVO₄ and BiVO₄/SiO₂ composites were evaluated according to the degradation of methylene blue (MB) dye aqueous solution under visible light irradiation. The SiO₂-coated BiVO₄ composites showed the enhancing photocatalytic activity approximately threefold in comparison with the single phase BiVO₄.

Keywords: composite materials, photocatalysis, BiVO₄, BET isotherms, SiO₂

INTRODUCTION

Nowadays, the advanced oxidation process is known as an effective method for water purification and wastewater treatment. One of the most famous advanced oxidation process is heterogeneous photocatalysis; the contaminant (i.e., organic compounds) containing in the water and wastewater is finally degraded to carbon dioxide (Legrini et al., 1993; Mukherjee and Ray, 1999). This process can remove the organic contaminant perfectly and does not generate the second contaminant (i.e., sludge and other organic compounds) which are required the further treatment and disposal. According to the heterogeneous photocatalysis, the titanium dioxide (TiO₂) has been played a role as the important catalyst to promote the photocatalytic activity. Due to its wide band gap of 3.2 eV, the photocatalyst of TiO₂ is typically activated under the UV light (the wavelength <390 nm is required), which accounts for 45–50% of solar radiation (Linsebigler et al., 1995; Bahnemann et al., 2007; Devipriya et al., 2012). This theoretical fact becomes the limitation and non-cost-effectiveness of actual photocatalytic system for purifying the water at the site.

Another catalyst of monoclinic bismuth vanadate (BiVO₄) has been proposed to overcome the drawback of photocatalytic system using TiO₂ and together with enhance the photocatalytic activity during implementation. Since BiVO₄ has narrow band gaps of 2.4 to 2.8 eV (Kudo et al., 2001; Xie et al., 2006; Li et al., 2008), this photocatalyst can be activated by the visible light and consequences

the effective use of solar energy. However, the low specific surface area and poor surface textural property are the significant disadvantages of using BiVO₄ as the catalyst. Its low surface area and adsorption capacity cause the low efficiency of photocatalytic system for organic contaminant removal and also the long treatment period required. Therefore, the increase in specific surface area of BiVO₄ catalyst is necessary prior to imply the photocatalytic system to the actual wastewater.

Recently, alternative composite materials have been synthesized by combining metal oxide and porous materials (i.e., alumina, silica, zeolites, carbon black, charcoal) (Belessi et al., 2007; Wang et al., 2012; Xing et al., 2016) with the aim of improving the specific surface area, pore structure, and photocatalytic activity of catalysts (Gan et al., 2003; Kimura et al., 2003). For example, the enhancement of Ag-doped TiO₂ photocatalytic activity was suggested by adding the mesoporous SiO₂; the excellent efficiency of methyl orange (MO) removal was achieved by 2.5 h (Roldan et al., 2015). The increasing adsorption capacity of TiO₂ catalyst was observed when the catalyst was combined with SiO₂; the adsorption capacity was increased (Hu et al., 2012). The SiO₂ addition also enhance the separation rate of electron-hole pairs under UV excitation. Further, the deposition of gold nanoparticles (Au) on the porous SiO₂-WO₃ composite can enhance the methyl blue (MB) adsorption capacity; the adsorption capacity of Au-SiO₂-WO₃ was greater than SiO₂-WO₃ and WO₃ respectively (DePuccio et al., 2015). The complete MB removal was achieved by 300 min under visible light, and the fast kinetic of MB removal was found in Au-SiO₂-WO₃ catalyst, following by Au-WO₃ and WO₃ catalysts.

As all the above mentions, this study aimed to improve the surface morphology and photocatalytic activity of BiVO₄ catalyst by coating SiO₂. Various analytical techniques including X-ray diffraction (XRD), transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET) and UV-vis diffuse reflectance spectra (DRS) were used to clarify the better property of BiVO₄/SiO₂ composite rather than BiVO₄ and SiO₂. Further, the performance of BiVO₄/SiO₂ composites on wastewater

treatment was preliminary studied in the batch test under visible light irradiation, and its performance was compared to the other two materials of BiVO₄ and SiO₂.

EXPERIMENTAL PROCEDURE

All chemicals used were of analytical grade and were used as received without any further purification. The chemicals including tetraethyl orthosilicate (TEOS), bismuth (III) nitrate pentahydrate [Bi(NO₃)₃·5H₂O], ammonium metavanadate (NH₄VO₃), methylene blue powder, sodium hydroxide pellet (NaOH), ammonia solution (28%) and nitric acid (37% HNO₃) were obtained from Sigma-Aldrich. All solutions were prepared with deionized water.

Preparation of SiO₂ Particles

SiO₂ particles were prepared by the sol-gel method. Ammonia solution (28%) was added in 100 mL of a mixed solution of absolute ethanol/DI water (80: 20 v/v) and stirred under ultrasonic dispersion for 60 min. Then, 20 mL of tetraethyl

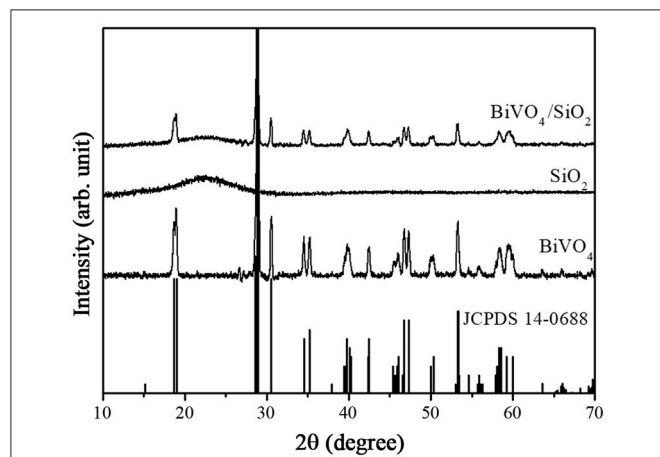


FIGURE 1 | XRD patterns of as-prepared BiVO₄, SiO₂, and BiVO₄/SiO₂.

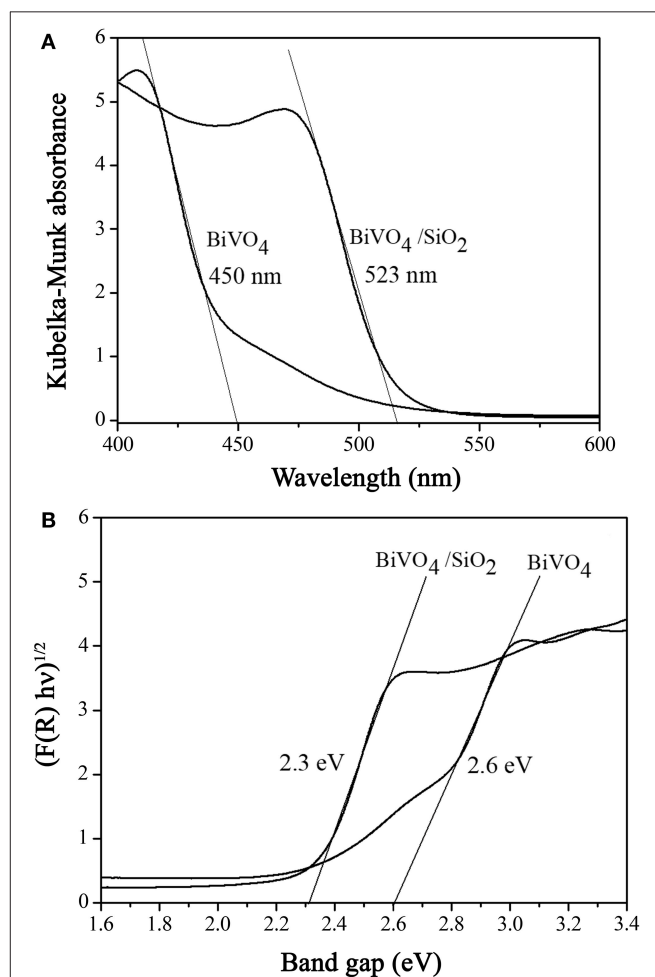


FIGURE 2 | (A) Diffuse reflectance UV-visible spectra and (B) the plot of adsorption function vs. photon energy for determination of band gap (E_g).

orthosilicate (TEOS) was added drop by drop to the mixed solution and stirred for 120 min at room temperature. After the reaction was homogenized, the fine particles were separated by centrifugation with typical rotating speed of 6,000 rpm for 15 min, washed by DI water and dried at 80°C for 24 h in a hot air oven. Fine particles of SiO₂ were obtained as a white powder following heat treatment at 500°C for 1 h in ambient.

Preparation of Monoclinic BiVO₄ and SiO₂-Coated BiVO₄ Composites

Monoclinic BiVO₄ were obtained by the co-precipitation method. Firstly, 12 mmol of bismuth (III) nitrate pentahydrate [Bi(NO₃)₃·5H₂O] and the same volume of ammonium metavanadate (NH₄VO₃) were dissolved in 100 mL of 2 M nitric acid (HNO₃) under vigorous stirring. The pH of the mixed solution was adjusted to 9 by adding 3 M sodium hydroxide (NaOH). The yellow precipitate was then separated by centrifugation at 6,000 rpm for 15 min, washed thoroughly with distilled water and ethanol and finally dried in a hot air oven at 80°C for 24 h. Crystalline monoclinic BiVO₄ was formed after calcination at 550°C for 4 h.

BiVO₄-coated SiO₂ composites were also prepared by the same method for comparison with an additional step of adding SiO₂ powder to 100 mL of 2 M HNO₃.

Photocatalytic Reaction

Photocatalytic activities of the BiVO₄, SiO₂ and BiVO₄/SiO₂ composites were evaluated through degradation of methylene blue (MB) dye as a model organic pollutant under visible light.

A total of 0.20 g of photocatalyst was added to 100 mL MB aqueous solution (initial concentration C₀ = 20 ppm) under magnetic stirring in darkness for 60 min to achieve adsorption-desorption equilibrium. The system was irradiated by three 18 W halogen lamps (Essential MR, Philips, Thailand) to investigate photocatalytic degradation. Reduction of MB concentration over time (C_t) was recorded every 15 min by measuring the intensity change of the characteristic absorption peak at 664 nm using UV-vis double beam spectroscopy (UV-6100, Mapada).

Characterisation

Crystal phase and structure of the prepared samples were characterized by powder X-ray diffraction (XRD, Philips X'Pert MPD) using Cu K_α (λ = 1.54056 Å) radiation. Morphological changes in the composite materials were monitored by transmission electron microscopy (TEM, JSM-2010, JEOL). Brunauer-Emmett-Teller (BET) measurements (Adtosorb 1 MP, Quantachrome) were performed to compare the specific surface area of the BiVO₄ and BiVO₄/SiO₂ composites. Measurement of UV-vis diffuse reflectance spectroscopy (DRS UV-vis, Shimadzu UV-3101PC) was carried out at room temperature to detect reflectance and absorbance spectra.

RESULTS AND DISCUSSION

In **Figure 1**, the broad XRD peak at 2θ = 22–23° corresponded to the amorphous SiO₂. The XRD pattern of BiVO₄ without SiO₂ was assigned to the standard monoclinic BiVO₄ (JCPDS no. 14-0688) (Gotić et al., 2005). After coating BiVO₄ with

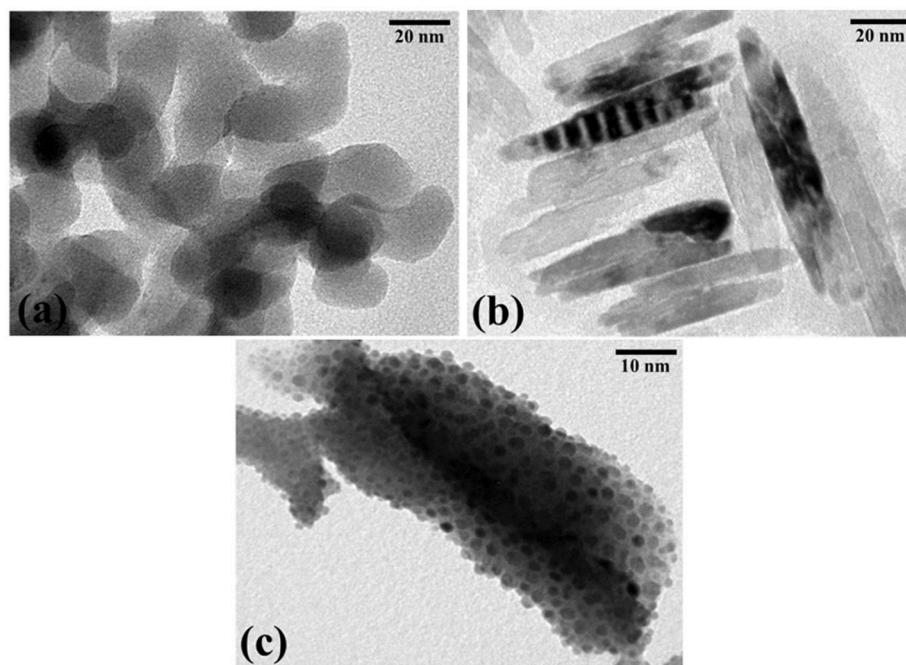
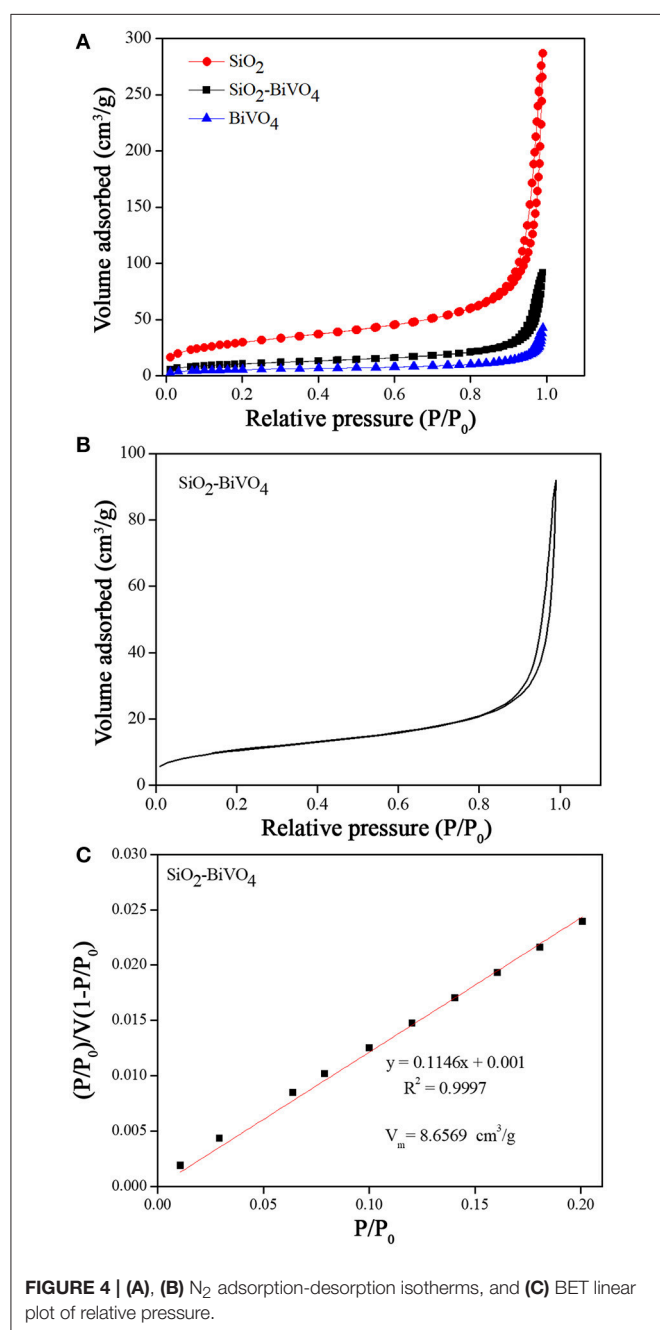


FIGURE 3 | TEM images of (a) SiO₂, (b) BiVO₄, and (c) SiO₂-coated BiVO₄.

SiO₂, the diffraction peaks matched well with the pure phase monoclinic BiVO₄ and no peaks of any other phases or impurities were recorded. However, the diffraction intensity of BiVO₄ decreased after coating SiO₂, because the amorphous substance had the negative effect on crystallinity. Alternatively, self-doped Si⁴⁺ ions in the BiVO₄ crystal structure might cause the decreasing crystallinity of BiVO₄/SiO₂ composites, and resulted in the broader peaks of the composite samples, which are similar to those reported by Phanichphant et al. (2016) for the binary composite CeO₂/SiO₂ photocatalysts and Kumar et al. for TiO₂/SiO₂ nanocomposites in solar cell applications (Arun Kumar et al., 2012).



As shown in **Figure 2a**, the BiVO₄ demonstrated the absorption edge of the visible region at 450 nm, corresponding to the optical band gap (E_g) of 2.60 eV which was calculated by the Kubelka–Munk function (see **Figure 2b**) (Sirita et al., 2007). Compared to BiVO₄/SiO₂ composites, the value of the graph intercept was estimated at 2.30 eV, corresponding to the strong absorption edge in the visible region at 523 nm. The band gap energy of BiVO₄ decreased from 2.60 to 2.30 eV in the composite materials, due to the influence of Si⁴⁺ ions doping into the lattice of BiVO₄ which created the abundant doping energy levels. The estimated band gap values in this study was similar to those of BiVO₄ reported by Jiang et al. (2012), who prepared the BiVO₄ photocatalysts with different morphologies using the hydrothermal method. Liu et al. (2015) observed that the band gap energy of BiVO₄/SiO₂ catalyst estimated to be 2.32 eV, which was almost the same as that of calculate by this study.

The TEM images of SiO₂, BiVO₄ and BiVO₄/SiO₂ composites are presented in **Figure 3**. The SiO₂ image shows the aggregation of spherical-shaped particles with diameters ranging of 20–30 nm (**Figure 3a**), while **Figure 3b** shows the rod-like nanostructures of monoclinic BiVO₄ with the diameter of 10 nm and the length of 60 nm. Typical TEM images are used for characterizing the composite materials and proving the heterojunction formation between BiVO₄ and SiO₂, which demonstrated that the rod-like BiVO₄ core was covered by the SiO₂ particles growing on the surface (**Figure 3c**).

The N₂ adsorption-desorption isotherms (**Figure 4a**) show that the N₂ adsorption of BiVO₄/SiO₂ composites were relatively higher than that of the pure BiVO₄, however the value was much lower than that of the SiO₂. The specific surface areas of SiO₂, BiVO₄/SiO₂ composites, and BiVO₄ were found to be 106.9959, 37.6851, and 19.4964 m²/g, respectively. In the meanwhile, the pore size was calculated by using the BJH method, and the results were 9.0316, 11.0776, and 11.8111 nm for SiO₂, BiVO₄/SiO₂, and BiVO₄ respectively (as summarized in **Table 1**). The surface area and pore size are positively related to the photocatalytic activity, therefore the photocatalytic activity of BiVO₄/SiO₂ composites were higher than that of pure BiVO₄. Even though the surface area of SiO₂ was higher than the BiVO₄/SiO₂ composite, the adsorption of pollutant by SiO₂ with high specific surface area have only the ability to transfer pollutants to alternative phases, but not completely get rid of them. Therefore, the photocatalytic process based on using the hydroxyl radicals is required in this study.

TABLE 1 | Surface properties of the prepared samples.

Sample	Specific surface area (m ² /g)	Average pore size diameter (nm)
SiO ₂	106.9959	9.0316
BiVO ₄ /SiO ₂	37.6851	11.0776
BiVO ₄	19.4964	11.8111

Figure 4b shows the N₂ adsorption-desorption isotherms of BiVO₄/SiO₂ composites in the relative pressure (P/P₀) range 0.00–1.00. The curve exhibited Type IV isotherm characteristic

with a small hysteresis loop at the relative pressure of 0.80–1.00. This indicated the existence of mesopores in the sample with the pore diameter ranging of 2–50 nm (Brunauer et al., 1940; Bae et al., 2010).

The information from the isotherm can be used to determine the specific surface area from the mathematical relations in Equation (1) and Equation (2) below (Itodo et al., 2010; Thommes et al., 2015)

$$\frac{P/P_0}{V(1 - P/P_0)} = \frac{1}{V_m C} + \frac{(C - 1) P}{V_m C P_0} \quad (1)$$

where,

P₀, Initial pressure of N₂; P, Equilibrium pressure of N₂ adsorption; V_m, Monolayer capacity; V, Amount of N₂ adsorbed at standard temperature and pressure (STP).

$$\text{Specific Surface area} = \frac{V_m N_a \times A}{m \times 22400} \quad (2)$$

where,

A, Cross-sectional area of the adsorbed N₂; m, Adsorbate molecular weight; N_a, Avogadro's number.

The intercept and slope of the plot in **Figure 4c** were used to calculate the maximum volume of gas adsorbed at the monolayer (V_m), it was 8.6569 cm³/g. The specific surface area was also calculated via the V_m value (see Equation 2). The result showed that the surface area of BiVO₄/SiO₂ composites was 37.6851 m²/g.

Figure 5a presents the degradation efficiency of MB as a function of C_t/C₀ and visible irradiation time. The C₀ was the initial concentration of MB before irradiation and C_t was the MB concentration at the interval irradiation time (t, min). For using the SiO₂ as catalyst, the MB was removed of 83% under the dark adsorption, and only 5% of MB was further degraded under the visible light. For using the single phase monoclinic BiVO₄, the MB was removed around 10% under the dark adsorption, and 40% of MB was further degraded under the visible light irradiation. When the BiVO₄/SiO₂ composites was used, the MB removal efficiency reached 35 and 86% under

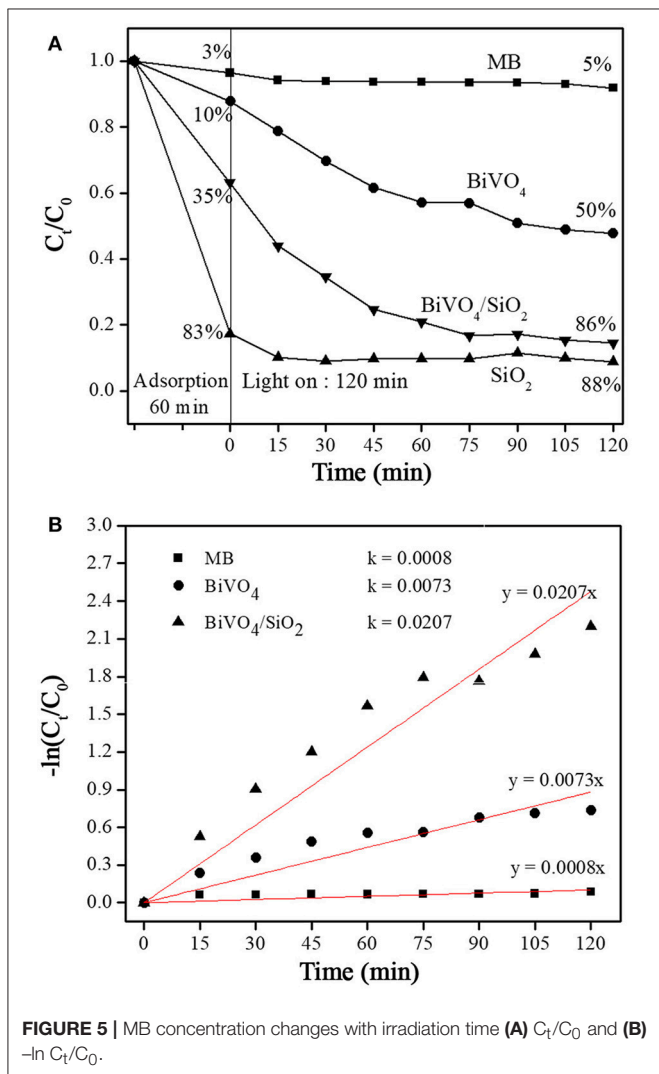


FIGURE 5 | MB concentration changes with irradiation time (A) C_t/C₀ and (B) -ln C_t/C₀.

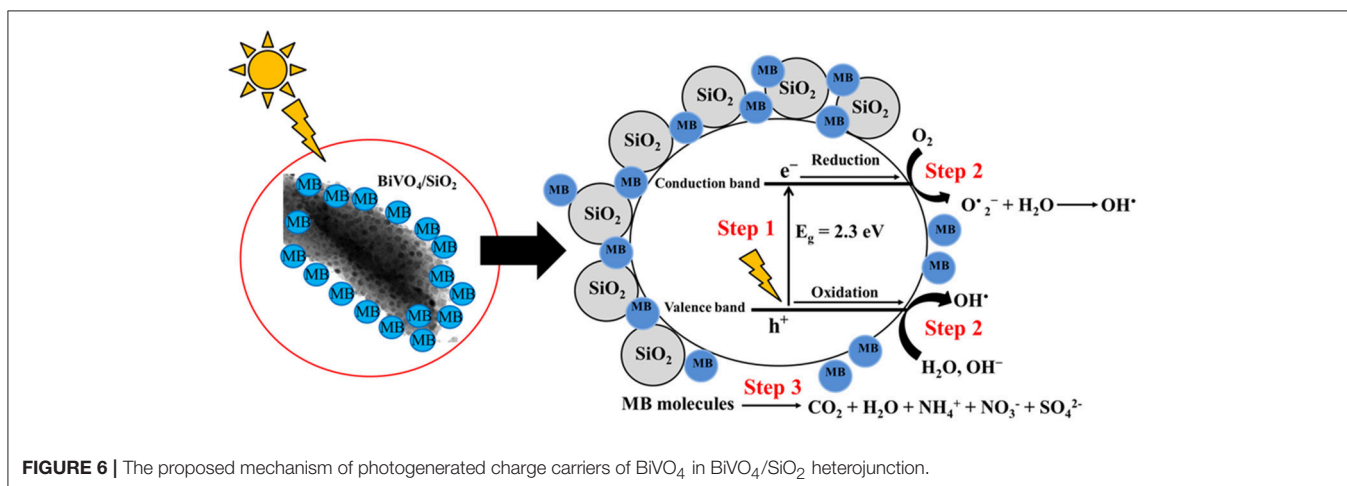


FIGURE 6 | The proposed mechanism of photogenerated charge carriers of BiVO₄ in BiVO₄/SiO₂ heterojunction.

the dark adsorption and visible light irradiation. As above explanation, the specific surface area of BiVO₄/SiO₂ composites were increased from BiVO₄, due to the SiO₂ coating. The increasing specific surface area resulted in the high adsorption of MB molecules during 60 min of the darkness, and then the adsorbed MB was continuously degraded by photocatalytic activity during visible light. These results illustrated that the photocatalytic activity of BiVO₄ was enhanced by coating the SiO₂ particles.

The kinetics of MB degradation was analyzed using the pseudo-first order model, which was given in Equation (3) (Yetim and Tekin, 2017). In **Figure 5b**, the correlation of $-\ln C_t/C_0$ and t were positive with linear equation; the kinetic constant (k) were 0.0073 min⁻¹ for BiVO₄ and 0.0207 min⁻¹ for BiVO₄/SiO₂ composites. The kinetic constant of MB degradation using BiVO₄/SiO₂ composites was approximately threefold higher than that using the single phase BiVO₄.

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

where k is the apparent rate constant of the pseudo-first order reaction (min⁻¹).

Since the photocatalytic degradation of dyes is associated with dye adsorption onto the surface of BiVO₄/SiO₂. Furthermore, photocatalytic degradation occurs at or near the surface of the catalyst rather than in the bulk solution. Thus the higher photocatalytic activity of BiVO₄/SiO₂ is consistent with the higher adsorption of MB on the surface of BiVO₄/SiO₂ photocatalyst. Mesoporous SiO₂ adsorbent enriches the MB molecules around the BiVO₄ surface as shown in **Figure 6** and the visible-light photocatalytic activity of the BiVO₄ interface in the composite materials is then excited to generate electrons (e⁻) and holes (h⁺). Subsequently, photoexcited electrons in the valance band and hole in the conduction band of BiVO₄

react with oxygen, water and hydroxide ions to produce free superoxide radicals (O₂^{-•}) and hydroxyl radicals (OH[•]) as the main active oxidizing species, which then react with MB molecules during the photocatalytic process (Lin et al., 2014; Zhou et al., 2014). The final products of MB aqueous solution photocatalytic degradation are oxidized to CO₂, H₂O, CO₂, NH₄⁺, NO₃⁻, and SO₄²⁻ (Houas et al., 2001; Luan and Hu, 2012).

CONCLUSIONS

BiVO₄/SiO₂ composites consisting of spherical SiO₂ particles coated on BiVO₄ nanorods were successfully prepared by co-precipitation. The composites exhibited higher photocatalytic activity compared to single monoclinic BiVO₄ by degrading MB under visible-light irradiation due to the greater surface area of mesoporous SiO₂. Fabrication of heterogeneous semiconductors using mesoporous materials can produce promising alternative photocatalysts for wastewater treatment under light irradiation by combining adsorption and photocatalytic processes.

AUTHOR CONTRIBUTIONS

DC designed and performed the experiments and wrote the manuscript. SP, AN advised the data analysis and edited manuscript. PJ and WK advised the data analysis. All authors reviewed the approved the manuscript.

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Conflict of Interest Statement: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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