Synthesis of novel activated carbon/BiVO₄ nanocomposite photocatalysts for degradation of organic compounds in wastewater

Saranyoo Chaiwichian¹*
¹Department of Science and Mathematics, Faculty of Industry and Technology, Rajamangala University of Technology Isan Sakonnakhon Campus, Phangkon, Sakonnakhon, 47160, Thailand

*Email: saranyou530531117@gmail.com

Abstract. Novel activated carbon/BiVO₄ nanocomposite photocatalysts were straightforwardly prepared via an impregnation method. All the samples were characterized by using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Brunauer-Emmett-Teller (BET), X-ray photoelectron spectroscopy (XPS), UV-vis diffuse reflectance spectra (UV-vis DRS) and photoluminescence (PL) techniques. The photocatalytic efficiency of activated carbon/BiVO₄ nanocomposite photocatalysts was estimated by rhodamine B dye degradation under visible light illumination. Results revealed that compositied activated carbon on the surface of BiVO₄ importantly improved the photocatalytic efficiency. In addition, the compositied activated carbon was assisted to fast reduce in the recombination of photogenerated charges, resulting in enhanced photocatalytic efficiency. The amount of composited activated carbon had affected the photocatalytic efficiency. The 5.0 mol% activated carbon showed the highest photocatalytic efficiency for the degradation of rhodamine B dye under visible light irradiation. Moreover, the photocatalytic efficiency of 5.0 mol% activated carbon/BiVO₄ nanocomposite photocatalyst showed a slight loss after five runs for rhodamine B dye degradation. The radical trapping experiments and mechanism of the improved photocatalytic efficiency in the degradation of rhodamine B dye were also proposed.

1. Introduction
Rhodamine B (RhB) is a cationic dye which is significantly employed as organic chemicals in various industries such as paint, plastic, rubber, adhesives, printing ink and so on [1-4]. Moreover, Rhodamine B is regarded as one of the most significant dangerous pollutants in the present and future. Especially, very toxic pollutants are discharged into the environment and can cause the wastewater issue significantly. Furthermore, these pollutants have hazardous effects on human health and ecological system. As a result, the RhB in the wastewater should be urgently fixed and treated. Up to date, numerous methods are highly welcome for the wastewater purification and degradation of toxic organic pollutants. As one of the popular and suitable techniques for solving the wastewater problem is the applications in renewable energy sources. Heterogeneous photocatalysis reaction using oxide semiconductor-based photocatalysts has gained extensive attention which is a promising technology for degrading wastewater and hazardous contaminant in water by the utilization of solar energy [5-7].
Many oxide semiconductors with narrow band gap energy and can be utilized in the range of visible light, such as BiVO$_4$ [8], BiFeO$_3$ [9], Fe$_2$O$_3$ [10], Cu$_2$O [11] and Bi$_2$WO$_6$ [12]. Among them, Bismuth vanadate (BiVO$_4$) is one of the most important oxide semiconductors that has received great interest for water splitting and the oxidative degradation of organic pollutants due to its good properties such as short band gap energy of 2.4 eV, high photoabsorption, good chemical stability, non-toxic and outstanding photocatalytic efficiency in the range of visible light [13]. Generally, BiVO$_4$ has three main phases including monoclinic zircon, tetragonal zircon and tetragonal scheelite. Two structures of tetragonal have the band gap energy of 3.1 eV [14]. Monoclinic-BiVO$_4$ structure with its narrow band gap energy exhibits the best photocatalytic activity in comparison with both tetragonal structures under visible light irradiation [15,16]. However, it is also well-known that pure BiVO$_4$ has low photocatalytic activity owning to its relatively slow separation of photoinduced charge carriers, leading to the high recombination rate of charge carriers and decreased photocatalytic performance [17,18]. One of the choices to overcome and reduce the recombination of photogenerated electron-hole pairs is the combination of BiVO$_4$ with other materials to strongly increase photocatalytic properties for degrading water pollutants. Activated carbon (AC), a non-metal material, has been extensively used as an absorbent for degrading organic contaminants in polluted water due to their high surface area and increasing active site and microporous structure [19-22]. Therefore, the combination of the merits between activated carbon and BiVO$_4$ to construct a promising photocatalyst is one of the ways for solving the photogenerated electron-hole recombination during the photodegradation process of organic contaminants, and then its enhanced photocatalytic efficiency. Consequently, the synthesis and the application of the activated carbon/BiVO$_4$ composite photocatalyst in the photocatalytic process is a challenging task. To the best of our knowledge, the activated carbon/BiVO$_4$ nanocomposite photocatalysts synthesized by the impregnation method has been infrequently reported. Moreover, the decorating of activated carbon on the surface of BiVO$_4$ to form a composite is to develop the optical absorption in the range of visible light and improve photocatalytic activity.

In this study, the composite photocatalyst of activated carbon and BiVO$_4$ was successfully synthesized through an impregnation method. Then the phase structure, surface area, morphology, elemental composition and optical absorption of all the as-prepared samples were investigated via X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), Brunauer-Emmett-Teller (BET) and UV-vis diffuse reflectance spectra (UV-vis DRS) techniques. Meanwhile, the efficient separation lifetime of charge carriers on the activated carbon/BiVO$_4$ interface was carried out by photoluminescence (PL) technique. The photocatalytic properties of obtained samples were estimated by degrading the rhodamine B dye under visible light irradiation.

2. Experimental

2.1. Activated carbon synthesis
Firstly, Gigantochloa kurzii Gamble (bamboo) was made to be a charcoal form by chemical activation method, and then the as-prepared charcoal was cleaned several times with deionized water and absolute ethanol at room temperature before drying in an electrical oven at 80 °C for 24 hours. After that, charcoal and nitric acid (HNO$_3$) solution of 1:1 was mixed with vigorously continuous stirring to form pores. Finally, the prepared activated carbon particles were cleaned with deionized water and heated for 1 hour at 600 °C.

2.2. BiVO$_4$ particle synthesis
All chemicals for preparing pure BiVO$_4$ particle included bismuth nitrate pentahydrate (Bi(NO$_3$)$_3$•5H$_2$O), ammonium metavanadate (NH$_4$VO$_3$), nitric acid (HNO$_3$) and ammonium hydroxide (NH$_4$OH). Firstly, the concentration of 0.1 M for Bi(NO$_3$)$_3$•5H$_2$O and 0.1 M for NH$_4$VO$_3$ was dissolved in 2.5 M HNO$_3$ solution, and then both solutions were mixed with magnetic stirring till
homogeneous solution and yellow change. The mixtures were adjusted to neutral pH by using 6 M NH\(\text{\textsubscript{4}}\)OH solution. After that, the solution of as-prepared BiVO\(_4\) was poured into a Teflon-lined stainless steel autoclave and treated at 180 °C for 6 hours. The precipitates were then left to cool down and cleaned with deionized water three times. Finally, the as-prepared BiVO\(_4\) products were kept in an electric oven at 80 °C for 24 hours.

2.3. Activated carbon/BiVO\(_4\) particle synthesis
The impregnation method was used to prepare the activated carbon/BiVO\(_4\) particles. First of all, the pre-synthesized activated carbon powder varying from 1.0-5.0 mol% was mixed with 1.0 g of BiVO\(_4\) in crucible together with 1.0 ml of deionized water and sealed tightly. Then the sealed crucible was sonicated until homogeneous and calcined in a hot stove at 450 °C for 1 hour. Finally, the activated carbon/BiVO\(_4\) particles were grounded with mortar and pestle leading to its fine uniform particles and small particle size.

2.4. Characterization of the photocatalyst samples
X-ray diffraction (XRD, Philips X'Pert MPD) was applied to study the crystalline phase of as-prepared photocatalysts. The morphology and particle size of photocatalysts were carried out using scanning electron microscopy (SEM, JEOL JSM-6335F) and transmission electron microscopy (TEM, JEOL JEM-2010) techniques. Brunauer-Emmett-Teller (BET) (Autosorb 1 MP, Quantachrome) was used to measure the specific surface area. The details of the elemental composition of prepared samples were investigated through X-ray photoelectron spectroscopy (XPS, AXIS ULTRA\textsuperscript{BLD}, Kratos Analytical, Manchester UK). The photoabsorption properties and band gap energies of samples were evaluated using UV-vis diffuse reflection spectroscopy (UV-vis 3600, Shimadzu). The immigration and separation of photocharges during the photocatalytic process of samples were examined by using photoluminescence (PL) with an LED lamp (Oceans optics, LLS-345).

2.5. Photocatalytic study of the rhodamine B dye degradation
The photoactivity efficiency of all photocatalysts for rhodamine B dye degradation under visible light irradiation was investigated using a photoreactor. The halogen lamp of 100 W with a 400 nm cut-off filter was used as a light source for the irradiation in the photocatalytic process. A 100 ml solution containing 0.1 g of photocatalyst and the concentration of 0.2 M rhodamine B dye was homogeneously mixed with vigorously magnetic stirring and then kept in the dark for 24 hours to make sure about an adsorption and desorption equilibrium between the photocatalysts and rhodamine B dye molecules. During the irradiation, 3 mL samples of the suspension were kept every 15 minutes together with centrifuging to separate the photocatalysts out of rhodamine B dye solution. The absorbance intensity of as-degraded rhodamine B solution was measured through UV–vis spectrophotometer (SHIMADZU, UV-1700).

3. Results and Discussion

3.1. XRD and BET analysis
To study the crystal structure of pure activated carbon, pure BiVO\(_4\) and activated carbon/BiVO\(_4\) nanocomposite photocatalysts, XRD technique was employed and shown in Figure 1. The diffraction peaks of pure activated carbon located at the position of 23.55° for (002) plane and 43.44° for (100) plane in graphite carbon [23,24], in the part of the XRD peaks of pure BiVO\(_4\) appeared at 29.20°, 30.90°, 34.96°, 35.53°, 42.77° and 46.27° corresponding to the (-121), (040), (200), (020), (051) and (240) planes, respectively, which indexed as monoclinic BiVO\(_4\) crystal (JCPDS file no. 14-0688). For the activated carbon/BiVO\(_4\) nanocomposite, the diffraction peaks were similar to the pure BiVO\(_4\) phase and no other impurities were measured, confirming the diffraction peaks of all samples had a high purity phase. In addition, the doping of activated carbon on the surface of BiVO\(_4\) did not affect the change of BiVO\(_4\) structure and not appear the activated carbon peaks, which might be attributed to
a very low amount of doped activated carbon into BiVO$_4$. The nitrogen adsorption BET technique was used to detect the specific surface area of all samples. The BET specific surface area of pure activated carbon, pure BiVO$_4$ and activated carbon/BiVO$_4$ nanocomposites with different mole (activated carbon) concentrations of 1.0, 2.0, 3.0, 4.0 and 5.0 was 361.12 m$^2$/g, 24.71 m$^2$/g, 17.57 m$^2$/g, 23.32 m$^2$/g, 32.68 m$^2$/g, 33.77 m$^2$/g and 35.24 m$^2$/g, respectively. From the BET analysis results, it was observed that increasing content of activated carbon added into the BiVO$_4$ surface affected the increased surface area and more active sites, which was a key factor to improve its photocatalytic efficiency.

![Figure 1](image1.png)

**Figure 1.** XRD patterns of pure activated carbon, pure BiVO$_4$ and activated carbon/BiVO$_4$ with different mole (activated carbon) concentrations.

3.2. **SEM and TEM analysis**

The SEM technique was used to analyze the morphologies of pure activated carbon, pure BiVO$_4$ and 5.0mol% activated carbon/BiVO$_4$. The morphology of pure activated carbon was a sheet-like structure with many pores, which exhibited an average diameter of approximately 50-100 nm as displayed in Figure 2a. In the case of pure BiVO$_4$, the morphology was a rod-like structure with 100-200 nm in length as shown in Figure 2b. The morphology of 5.0mol% activated carbon/BiVO$_4$ nanocomposite was composed of rod and sheet-like structures matching with pure BiVO$_4$ and activated carbon, respectively, as represented in Figure 2c. To more confirm details of the actual size and in the presence of activated carbon in activated carbon/BiVO$_4$ nanocomposites, the TEM technique was used. From the Figure 2d and 2e, TEM images of pure activated carbon and pure BiVO$_4$ showed sheet and rod-like structures, respectively. In the part of activated carbon/BiVO$_4$ nanocomposites, TEM image exhibited both of rod-like structure for BiVO$_4$ and sheet-like structure for activated carbon, as illustrated in Figure 2f. Therefore, it can be concluded that in the presence of activated carbon and BiVO$_4$ in the composite, with the average particles of about 100-200 nm.
3.3. XPS analysis
The surface chemical state of all elements in 5.0mol% activated carbon/BiVO₄ nanocomposite was measured by XPS technique, as displayed in Figure 3. The 5.0mol% activated carbon/BiVO₄ nanocomposite constituted Bi 4f, V 2p, O 1s and C 1s ions respectively. In Figure 3a, the XPS spectrum of the Bi 4f peak centered at 158.68 eV and 164.04 eV regions matching with Bi 4f⁷/₂ and Bi 4f⁵/₂, respectively, which was attributed to the Bi³⁺ of BiVO₄ in the activated carbon/BiVO₄ nanocomposite [25-27], while the V 2p peak showed two signals at the binding energies of 516.64 eV for V 2p⁴/₂ and 524.26 eV for V 2p¹/₂, which was indexed as V⁵⁺ in the activated carbon/BiVO₄ nanocomposite as presented in Figure 3b [28]. In the case of XPS peak of the O 1s, the characteristic peaks were composed of three peaks at the binding energies of 532.68 eV, 533.18 eV and 534.15 eV respectively, as shown in Figure 3c. All the peaks were ascribed to the chemisorbed oxygen and hydroxyl groups from the BiVO₄ particle [29,30]. The XPS spectra peak of the C 1s signal of activated carbon in activated carbon/BiVO₄ nanocomposite was split into five peaks. These peaks took place in the region of about 284.99-289.30 eV, as illustrated in Figure 3d. The position of XPS peak located at the binding energies of 284.99 eV, attributing to the C-C in activated carbon [31,32], in the part of the binding energies at 285.70 eV, 286.73 eV, 287.89 eV and 289.30 eV corresponding to the C-O, C=O, C-N and O-C=O, respectively [31,32].
Figure 3. XPS spectra of (a) Bi 4f, (b) V 2p, (c) O 1s and (d) C 1s in 5.0mol% activated carbon/BiVO₄ nanocomposite.

3.4. UV-vis DRS analysis

In Fig. 4, pure activated carbon, pure BiVO₄ and 5.0mol% activated carbon/BiVO₄ nanocomposite samples were studied the optical property using UV-vis DRS technique. From testing results, the activated carbon showed the absorption edge in the UV-light range of approximately 253 nm as displayed in Fig. 4a. The pure BiVO₄ exhibited strong absorption in the region of visible light with a wavelength of about 497 nm, as represented in Fig. 4b. Moreover, in which activated carbon deposited on the surface of the BiVO₄ nanorod, resulting in the optical absorption shifted toward a longer visible light region of around 499 nm, as depicted in Fig. 4c. This might be attributed to the tightly chemical bonding between activated carbon and BiVO₄ [33]. In addition, the BiVO₄ doping with activated carbon was helped to increase the optical absorption area, which was a key factor that leads to the improvement of photocatalytic activity in the degradation of organic dyes under visible light. The position of band gap energies of pure activated carbon, pure BiVO₄ and 5.0mol% activated carbon/BiVO₄ nanocomposite samples was assessed by using Tauc’s equation:

\[(\alpha h\nu)^n = A (h\nu - E_g)\]  

where, \(\alpha\), \(h\nu\), \(A\) and \(E_g\) are the absorption coefficient, the photon energy of the semiconductor, the constant and the band gap energy of the semiconductor, respectively. From the calculation results, the band gap energies of pure activated carbon, pure BiVO₄ and 5mol% activated carbon/BiVO₄ nanocomposite photocatalysts were evaluated to be 4.36 eV, 2.53 eV and 2.46 eV, respectively. Therefore, it was inferred that 5.0mol% activated carbon had more optical absorption range and suitable band gap energy for the utilization in the photocatalytic process.
3.5. Photodegradation process of rhodamine B dye

As shown in Figure 5a, rhodamine B dye was employed as the model test dyes for evaluating the photodegradation efficiency of pure activated carbon, pure BiVO₄ and activated carbon/BiVO₄ nanocomposite photocatalysts with different mol (activated carbon) concentrations varying from 1.0, 2.0, 3.0, 4.0 and 5.0 under visible light irradiation. The test results of rhodamine B photolysis without nanocomposite particles were found to be 3%. In the part of the photodegradation efficiency of rhodamine B dye in the presence of pure activated carbon, pure BiVO₄ and activated carbon/BiVO₄ nanocomposite photocatalysts with different mole (activated carbon) concentrations varying from 1.0, 2.0, 3.0, 4.0 and 5.0 was shown to be 6%, 7%, 19%, 29%, 53%, 42% and 67%, respectively. It was found that all nanocomposites showed better efficiency than other pure activated carbon and pure BiVO₄. In addition, the content of 5mol% activated carbon decorated on the BiVO₄ showed the highest photodegradation efficiency of rhodamine B dye within 180 minutes under the selfsame conditions when compared with other samples. The best efficiency of 5.0mol% activated carbon/BiVO₄ might be attributed to the introduction of activated carbon into BiVO₄ could restrain the photoinduced charges recombination and help the long-time separation of charge carriers, causing the increased photodegradation efficiency. Moreover, the doping of activated carbon into BiVO₄ was led to its lower band gap energy and high specific surface area, which were important factors for the better photodegradation performance of rhodamine B dye. Figure 5b shows the photocatalytic reaction kinetics for the degradation of rhodamine B dye under visible light irradiation. The kinetic rate constants can be expressed by using the pseudo-first-order kinetic equation as follows: 

\[ \ln \left( \frac{C_0}{C_t} \right) = kt \]

where \( C_0 \), \( C_t \) and \( k \) are the initial concentration of rhodamine B dye at irradiation.
time 0, the concentration of rhodamine B dye at irradiation time $t$ and the pseudo-first-order kinetic rate constant [35], respectively. The kinetic rate constants can be determined by the linear plot between $\ln\left(\frac{C_0}{C_t}\right)$ versus irradiation time. From the experiment, it was found that the kinetic rate constant ($k$) for the rhodamine B dye photodegradation over the 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst showed the best among other samples. The kinetic rate constant ($k$) of 5.0mol% activated carbon/BiVO$_4$ nanocomposite was reached up 0.00632 min$^{-1}$. It was concluded that the proper activated carbon concentration was 5.0mol% which resulted in the best photodegradation efficiency of rhodamine B dye under visible light irradiation. In addition, the amount of upper or lower activated carbon would cause a decrease in the photodegradation performance.

The reusable efficiency of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst for the degradation of rhodamine B dye under visible light illumination was investigated by five cycles and displayed in Figure 5c. From the five tests, it was found that the photodegradation efficiency of rhodamine B dye exhibited slight loss, concluding that the 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst had high stability and recyclability during rhodamine B degradation process under visible light irradiation.

![Figure 5](a) photoactivity of rhodamine B dye degradation, (b) kinetic rate constants of rhodamine B dye degradation and (c) recycling test of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst in the degradation of rhodamine B dye.

3.6. Trapping experiment and Photodegradation mechanism

To clarify the photodegradation mechanism of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst, the trapping experiments were used to search for major scavenger species in the photodegradation process of rhodamine B dye under visible light irradiation as shown in Figure 6.
Three scavenger species, such as $O_2^-$, $OH^-$ and $h^+$ were produced by photogenerated electron-hole pairs. Therefore, benzoquinone (BQ), potassium iodide (KI) and isopropanol (IPA) were used as the $O_2^-$, $OH^-$ and $h^+$ scavenger species respectively, to add into the photodegradation process of rhodamine B dye in the presence of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst. From experimental results, the introduction of KI into the photodegradation process of rhodamine B dye had a high photodegradation rate, showing that the holes were not leading scavenger species for the degradation of rhodamine B dye. For the additions of BQ and IPA into the photodegradation process of rhodamine B dye, the photodegradation rate was rapidly decreased. It was concluded that $O_2^-$ and $OH^-$ were important scavenger species in the degradation of rhodamine B dye under visible light irradiation. Therefore, a likely mechanism of the degradation of rhodamine B dye under visible light irradiation was carried out as displayed in Figure 7. When the visible light was illuminated on the surface of activated carbon/BiVO$_4$ nanocomposite photocatalyst, the electrons on the valence band potential of BiVO$_4$ were excited to transfer on the conduction band potential of BiVO$_4$ and then move to the surface of activated carbon. Therefore, the activated carbon surface would act as an electron trap to protect the reunion of electron-hole pairs [36]. The photogenerated electrons on the activated carbon surface would react with $O_2$ molecules to form $O_2^-$. Simultaneously, the photogenerated holes on the valence band potential could then capture with $H_2O$ and $OH^-$ molecules to finally form $OH^-$. The as-produced $O_2^-$ and $OH^-$ were powerful scavenger species for oxidizing rhodamine B dye molecules in wastewater. After the complete process, the final degradation products were $H_2O$ and $CO_2$ [37,38], respectively. Moreover, the activated carbon doped on the BiVO$_4$ photocatalyst was led to more active sites, which could increase the trap of $O_2$ and $H_2O$ molecules resulting in the increment of radical generation and more adsorption of rhodamine B dye molecules on the surface of activated carbon/BiVO$_4$ nanocomposite photocatalyst [39,40]. Therefore, it was concluded that the existence of activated carbon in activated carbon/BiVO$_4$ nanocomposite was a good way to significantly help to lead to its enhanced photodegradation and adsorption abilities.

![Figure 6](image_url)

**Figure 6.** Radical trapping experiments of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst in rhodamine B dye degradation under visible light irradiation.
3.7. PL analysis
To confirm the evidence of separation and recombination of electron-hole pairs during the photodegradation process of pure BiVO$_4$ and 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst, the photoluminescence (PL) spectroscopy was used as represented in Figure 8. Results were observed that the PL intensity of BiVO$_4$ exhibited a higher emission, while the PL intensity of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst had less emission than that of pure BiVO$_4$. It was inferred that the presence of activated carbon in the composite would reduce the charge carrier's recombination resulting in the improved photodegradation performance of rhodamine B dye.

![Figure 7](image)

**Figure 7.** a probable photodegradation mechanism of rhodamine B dyes with activated carbon/BiVO$_4$ nanocomposite photocatalysts.

![Figure 8](image)

**Figure 8.** photoluminescence spectroscopy of pure BiVO$_4$ and 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalysts.

4. Conclusions
In summary, the synthesis of novel activated carbon/BiVO$_4$ nanocomposite photocatalysts was achieved by an impregnation method. The photodegradation capability of as-synthesized activated carbon/BiVO$_4$ nanocomposite photocatalysts was better than pure activated carbon and pure BiVO$_4$ samples. Among all photocatalysts, 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst was a suitable mole concentration that showed the best photoactivity in the rhodamine B dye degradation under similar conditions. Moreover, the combination of activated carbon with a high surface area on the surface of BiVO$_4$ was caused the augmentation of active sites for adsorption of rhodamine B dye molecules, and not only that, activated carbon in the composite could obstruct the electron-hole pairs' reunion resulting in the amended photodegradation capacity. The reusable results
of 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst showed high stability after five recycles, indicating that the 5.0mol% activated carbon/BiVO$_4$ nanocomposite photocatalyst was an appropriate photocatalyst for wastewater purification. Radical scavenger species derived from trapping experiments. The results were found that superoxide and hydroxyl radicals were a considerable oxidizing agent for degrading rhodamine B dye in wastewater.

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