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One-step hydrothermal synthesis of Cu/Bi/$CuBi_2O_4$ nanocomposite and investigation of its photocatalytic performance in methylene blue degradation and dibenzothiophene desulfurization

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ABSTRACT

In this research, for the first time, Cu/Bi/$CuBi_2O_4$ (copper/bismuth/copper bismuth oxide) nanocomposite was synthesized through a one-step hydrothermal method using hydrazine. Synthesized product was characterized using XRD, DRS, FT-IR, EDS, and FESEM techniques. Diffuse reflectance spectrum analysis showed that Cu/Bi/$CuBi_2O_4$ nanocomposite has much higher light absorption than pure $CuBi_2O_4$, especially in the visible light range. The photocatalytic efficiency of Cu/Bi/ $CuBi_2O_4$ nanocomposite in the degradation of methylene blue (MB) as well as in the desulfurization of dibenzothiophene (DBT) was 93% and 87%, respectively, which shows a significant increase compared to pure $CuBi_2O_4$ with an efficiency of 40% (for the destruction of methylene blue) and 39% (for desulfurization). Examining the effect of pH showed that the photocatalyst has a higher efficiency (93%) in an acidic environment (pH = 4) and the efficiency decreases with increasing pH, so that at pH = 7 and pH = 9, the efficiency was 83% and 69%, respectively. Furthermore, the reusability tests demonstrated that the synthesized Cu/Bi/ $CuBi_2O_4$ photocatalyst exhibited excellent stability and recyclability for DBT desulfurization and MB degradation, maintaining its efficiency even after five reaction cycles.

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1. Introduction

There have been an increasing number of concerns regarding the contamination of water sources and soil as a result of the release of hazardous industrial chemicals such as surfactants, solvents, organic dyes, toxic cations, and pesticides over the past few decades [1-3]. It is possible for these pollutants to pose a threat to human life and other organisms if they are discharged into the environment without being treated [4]. As a result of their intricate nature and inability to degrade, extensive efforts are made to treat these pollutants effectively [5]. Environmental pollutants are themselves classified as hazardous chemicals when they are used in traditional treatment techniques. Thus, more efficient and environmentally friendly treatment methods are needed in place of conventional methods [6, 7]. There have been recent proposals for new approaches to dispose of organic pollutants, such as photocatalytic treatment [8].

A major concern for the oil refining industry is the presence of sulfur, which can result in corrosion of process equipment such as flow lines, pumps, and tanks [9]. On the other hand, when these compounds are combusted, oxide gases are released, causing acid rain, damages to buildings, as well as other problems.

Due to the acidification of the soil, pastures are lost, fresh water sources disappear, and multiple ecosystems are irreparably damaged [10]. A cutting-edge approach within the field of oxidative desulfurization, photocatalytic oxidative desulfurization stands out among other desulfurization methods [11]. There are several reasons for this method's popularity, including its low energy consumption, environmental friendliness, the use of sunlight as a power source and the overall cost-effectiveness of the process [12]. As a method of removing aromatic sulfur compounds from liquid fuels, photocatalytic desulfurization has demonstrated promise [13]. An active semiconductor photocatalyst is activated by incident light beams with a wavelength equal to its bandgap by photocatalytic processes. The primary components of highly oxidative species are electrons and holes (e-/h+) that are generated during activation. Mineralization of complex organic pollutants is capable of converting the pollutants into products that are more biodegradable and less toxic [14]. Photocatalysts can also be utilized to remove pollution caused by dyes such as methylene blue

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(MB) [15]. Due to its toxicity and effectiveness even at low concentrations, methylene blue is used in a wide variety of industries, including textiles, paper making, and pharmaceuticals [16]. A photocatalytic process involves breaking down and oxidizing methylene blue molecules by using catalysts as well as light (usually sunlight or visible light) [17, 18]. During this process, catalysts on their surfaces absorb electrons from water or oxygen under the influence of light, and these electrons are then transferred to methylene blue molecules, causing their oxidation and destruction. The use of light energy and the use of high-efficiency catalysts make this process an efficient and clean method for removing pollutants from water, including methylene blue. In order to be an effective photocatalyst, it must possess certain characteristics, including non-toxicity, optical stability, activity in the visible region, a high optical activity, and a substantial surface area for accurate pollutant absorption [19]. An attractive characteristic of copper bismuth oxide as a visible light photocatalyst is the large pore size and high surface area of the catalyst, which facilitates good contact between the active sites and the substrate [20].

Several investigations have demonstrated that the introduction of contaminating compounds such as bismuth oxide in metals such as copper not only inhibits electron and hole recombination, but also narrows the band gap, thus facilitating the extension of absorption into the visible spectrum [21]. As a result, the overall efficiency of the process is enhanced. According to Zhang et al. [22], $CuBi_2O_4$/$CdMoO_4$ nanocomposites were synthesized with different mass ratios to investigate the photocatalytic activity of these materials and enhance their properties. $CuBi_2O_4$ and $CdMoO_4$ compounds were combined in this study to form a heterogeneous Z-scheme structure. In this configuration, the energy gap is minimized to broaden the range of wavelengths that are absorbed, encompassing wavelengths ranging from ultraviolet to visible. $CuBi_2O_4$/$CdMoO_4$ nanocomposite (10%) showed an optimal photocatalytic performance with a high degradation rate of 95.36% for methylene blue exposed to visible light. A nanocomposite composed of Cu/Cu_2O/$BiVO_4$/Bi_7VO_{13} was synthesized by Mousavi-Kamazani [23], exhibiting a cube shape and a particle size of about 50 nm. Initially, thiophene was photocatalytically desulfurized under visible light conditions in order to assess its efficacy. As a result of the specified research, it has been established that doping photocatalysts with copper compounds resulted in enhanced conductivity when incorporated into nanocomposites. In turn, the energy gap was decreased, resulting in more efficient electron transfer processes, thereby improving the light absorption properties of the nanocomposites. As a result, the photocatalytic process can be significantly improved through the application of this methodology. The efficiency of the system achieved a remarkable 92% after 150 minutes of visible light irradiation. A unique flower-like morphology was observed when Sokhansanj et al. [24] synthesized $Bi_2O_2CO_3$-$CuBi_2O_4$ nanocomposite by utilizing a hydrothermal method and ultrasonic waves as an assisting factor. Our primary objective was to evaluate the effectiveness of $Bi_2O_2CO_3$-$CuBi_2O_4$ nanocomposite as a photocatalytic agent for degradation of malachite green when irradiated under visible light. The synergistic effects of combining the two aforementioned phases in this investigation were explored throughout in order to increase the effectiveness of the photocatalyst. In order to prepare samples with varying weight ratios of $CuBi_2O_4$-$Bi_2O_2CO_3$, the $Bi_2O_2CO_3$ phase was applied as a coating on $CuBi_2O_4$. In addition, further investigation revealed that, as a result of the petal-shaped structure's ability to capture additional light through two-sided surface areas, separating charge carriers becomes more efficient. Thus, the decreased recombination rate of electron-hole pairs can result in increased photocatalytic efficiency, as Bhakar et al. [25] were able to achieve 98.48% degradation efficiency of methylene blue using a $CuBi_2O_4$@$CdFe_2O_4$ nanocomposite. The mentioned nanocomposite was attributed to photocatalytic activity due to the synergistic effects of heterogeneous interfacial contacts, which enhanced the light absorption range and enhanced the photocatalytic activity of the nanocomposite as well as facilitated carrier separation and transfer.

In the present study, with the aim of improving photocatalytic efficiency by increasing surface adsorption and increasing light absorption in the visible range, Cu/Bi/$CuBi_2O_4$ nanocomposite has been synthesized for the first time by a one-step hydrothermal method. Hydrazine as both a reducing agent and a source of hydroxide ion production provides the conditions for a one-step synthesis. Additionally, diffuse reflectance spectroscopy (DRS), field emission scanning electron microscopy (FESEM), x-ray diffraction (XRD), energy dispersive spectroscopy (EDS), and Fourier transform infrared spectroscopy (FT-IR) analyses were employed to determine the nanocomposite's morphology and structure. Finally, the performance of this nanocomposite was evaluated for the degradation of methylene blue as a representative of pollutants. Dibenzothiophene was evaluated as a model of petroleum derivatives. This research can be valuable considering that hydrothermal is a cost-effective method and inexpensive reactive materials are used for one-step synthesis of Cu/Bi/$CuBi_2O_4$ photocatalyst with high stability.

2. Materials and methods

2.1. Materials and instruments

All substances utilized in this investigation, encompassing copper (II) nitrate (Cu $(NO_3)2.3H_2O$), bismuth (III) nitrate (Bi $(NO_3)3.5H_2O$), bismuth (III) nitrate ($Bi_5H_9N_4O_{22}$), sodium hydroxide (NaOH), hydrazinium hydroxide (N_2H_2OH 100%), normal hexane (C_6H_{14}), nitric acid (HNO_3), methylene blue (MB), and dibenzothiophene (C_8H_6S) were acquired from Merck and Sigma-Aldrich suppliers and used without any purification. DRS was performed using a Shimadzu UV3600Iplus. Using a Zeiss Sigma300-HV instrument, FESEM images were recorded. A Philips-X'PertPro instrument was used to examine XRD patterns, which were filtered with Ni-filters and then emitted with Cu Kα radiation. Magna-IR devices-specifically a Nicolet 550 spectrometer-were used to carry out FT-IR (Fourier transform infrared) analysis. The range of resolution was 0.125 cm on KBr tablets covering 400 to 4000 cm^{-1}. The experiment was carried out using an X-ray scattering apparatus from Philips XL30 for EDS. Measurements of sulfur content were made with a sulfur analyzer in oil model Horiba-SLFA-20.

2.2. Synthesis of Cu/Bi/$CuBi_2O_4$ nanocomposite

First, 5 ml of hydrazine was dissolved in 25 ml of distilled water and stirred on a stirrer for 3 min to prepare solution A. Solution B was prepared by dissolving 0.241 g of copper nitrate salt and 2.922 g of bismuth salt ($Bi_5H_9N_4O_{22}$) in 50 ml of distilled water in a separate beaker. After adding solution B to solution A, the suspension was mixed on a stirrer for 5 min and then transferred to a stainless-steel autoclave. Upon cooling the autoclave to room temperature, the precipitate was collected and washed several times with distilled water and ethanol. After drying at 60 °C for 12 hours, the product was obtained.

Using the method described by Chen et al. [15], pure $CuBi_2O_4$ was synthesized in order to compare its performance to that of the as-synthesized nanocomposite. For this purpose, solution A was prepared by dissolving 1.940 g of bismuth salt (Bi $(NO_3)3.5H_2O$)

in 3 ml of nitric acid solution. Solution B was prepared by dissolving 0.4832 g of copper nitrate salt in 30 ml of distilled water, and then the two solutions were mixed together. In the final step, 30 ml of sodium hydroxide 0.6 M, which was prepared separately, was added to the mixing solution and then heated at 120 °C in an autoclave for 16 hours. After the sample was removed from the oven, it was washed twice with distilled water and twice with alcohol and dried for 12 hours at 60 °C.

2.3. Methylene blue degradation

First, 100 ml of methylene blue (MB) aqueous solution with a concentration of 10 ppm was prepared. Then, 50 mg of the synthesized photocatalyst was added to it and the resulting suspension was stirred for 40 minutes on a stirrer in the dark until the adsorption-desorption equilibrium was established. Then the suspension was exposed to visible light for 40 minutes with a 400 W Osram lamp. At intervals of 10 minutes, 5 ml of the irradiated solution was removed and the photocatalyst was separated by centrifugation. Then, the concentration of the aqueous solution was measured by an ultraviolet-visible spectrophotometer at the maximum absorption wavelength (663 nm) of methylene blue. After finishing the process, the photocatalyst was separated from the solution by centrifugation, washed several times with distilled water, and dried at 70 °C for a few min. The photodegradation efficiency (η) of MB was calculated as follows:

$$\eta\ (\%) = (1 - C/C_0) \times 100 \quad (1)$$

where C_0 and C are the initial concentration and the irradiated concentration of MB solution, respectively.

2.4. Photocatalytic desulfurization of dibenzothiophene

In the first step, 500 ml of a solution containing 800 ppm of sulfur were obtained from dibenzothiophene. Afterwards, 100 ml of standard solution and 0.1 g of the photocatalyst powder were introduced into the beaker, and the beaker was then positioned within the reactor. In order to ensure full equilibrium between desorption and adsorption, the mixture was agitated on a stirrer under aeration for 30 min and then desulfurized under visible light (400 W Osram visible lamp). For the measuring of sulfur, 15 ml of sample was collected at designated time intervals and subsequently separated using a centrifuge and so on washing was done with acetonitrile. Finally sulfur content in the samples was determined using a sulfur-in-oil measuring instrument. Desulfurization efficiency (η) is estimated by the following equation:

$$\eta\ (\%) = (1 - S/S_0) \times 100 \quad (2)$$

where S and S_0 are sulfur content after and before light irradiation. A schematic of synthesis and desulfurization methods is displayed in Scheme1. Scheme 1 Photocatalytic desulfurization mechanism from dibenzothiophene by Cu/Bi/$CuBi_2O_4$ nanocomposite.

3. Results and discussion

3.1. Characterization

According to the XRD pattern presented in Fig. 1, the product mainly contains Bi with rhombohedral phase (JCPDS No. 01-085-1329, and lattice parameters a = b = 4.546 Å, and c = 11.862 Å).

The existence of additional peaks confirms the presence of $CuBi_2O_4$ compounds with a tetragonal phase (JCPDS No. 01-079-1810, and lattice parameters a = b = 8.4875 Å, and c = 5.7901 Å) and Cu with a cubic phase (JCPDS No. 00-004-0836 and cell constant a=3.6150 Å). Hydrazine as a hydroxide ion agent causes the production of $CuBi_2O_4$ and as a reducing agent causes the production of bismuth and copper metals. The average crystallite size of Cu, Bi, and $CuBi_2O_4$ structures in the nanocomposites were calculated to be about 26, 37, and 33 nm based on Scherrer's equation [26, 27].

Fig. 2 shows the FTIR spectrum of the as-synthesized Cu/Bi/$CuBi_2O_4$ nanocomposite. As expected, no peak is seen in the fingerprint area, which reveals that no hydrazine contamination has remained on the surface of the nanocomposite. The peaks of 432 cm^{-1} and 563 cm^{-1} indicate the presence of Bi-O and Cu-O bonds in the composition of copper bismuth oxide [23, 28, 29]. FESEM images with different magnifications of Cu/Bi/$CuBi_2O_4$ nanocomposite are presented in Fig. 3. The presence of different morphologies is due to the presence of various materials including copper nanoparticles, copper bismuth oxide nanoparticles, and bismuth microstructures in the composite. In fact, spherical $CuBi_2O_4$ nanoparticles as well as spherical Cu nanoparticles are distributed on the surface of rectangular cubic Bi microstructures.

In order to better investigate the structure and composition, an EDS spectrum and EDS mapping of Cu/Bi/$CuBi_2O_4$ nanocomposite was prepared and presented in Fig. 4. According to Fig. 4a, which corresponds to the EDS spectrum of the as-synthesized nanocomposite, only bismuth, copper, and oxygen elements are visible, and there are no other impurities. X-ray mapping of the Cu/Bi/$CuBi_2O_4$ nanocomposite (Fig. 4b) shows a good distribution of elements.

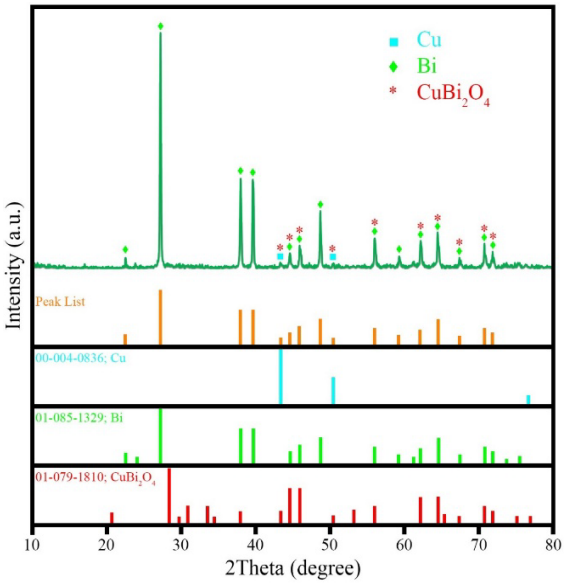


Fig. 1. XRD patterns of the as-synthesized Cu/Bi/CuBi2O4 nanocomposite

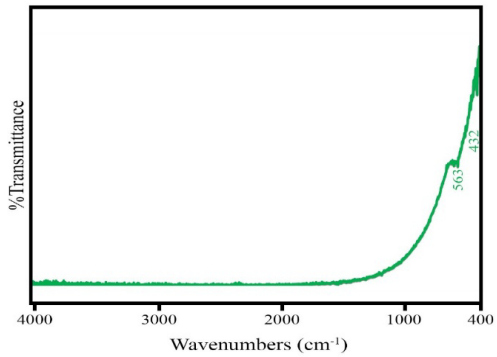


Fig. 2. FT-IR spectrum of the as-synthesized Cu/Bi/CuBi2O4 nanocomposite.

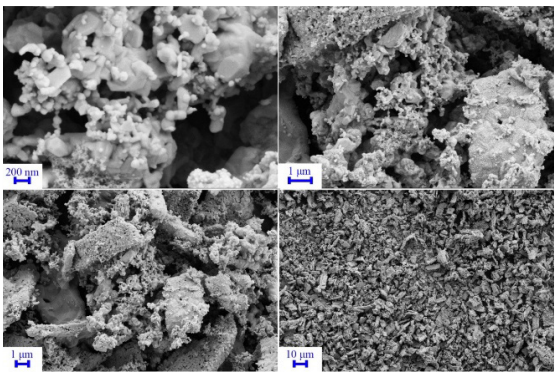


Fig. 3. FESEM images of the as-synthesized Cu/Bi/CuBi2O4 nanocomposite.

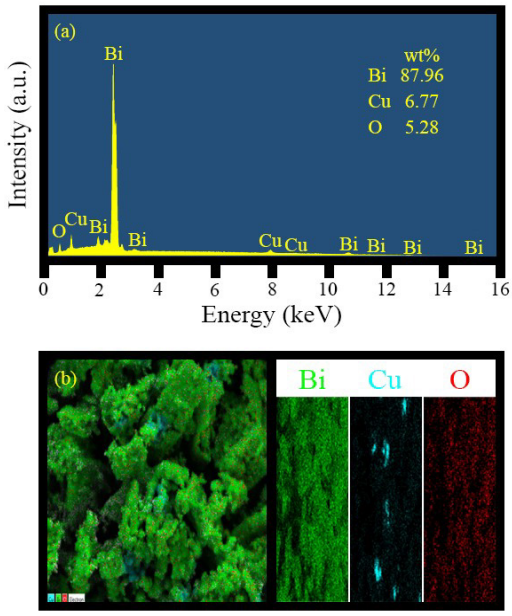


Fig. 4. (a) EDS spectrum and (b) EDS mapping of the as- synthesized Cu/Bi/$CuBi_2O_4$ nanocomposite.

According to the map, copper, bismuth, and oxygen elements are distributed throughout the microstructures. In fact, it can be said that CuBi2O4 nanoparticles are well distributed on the surface of bismuth microstructures and copper nanoparticles are formed in some parts. This evidence is consistent with the results of FTIR, FESEM, and XRD analyses. In order to investigate the optical properties as an important factor in the photocatalytic process, the UV–vis diffuse reflection spectrum (DRS) of Cu/Bi/$CuBi_2O_4$ nanocomposite is presented in Fig. 5 and compared with pure $CuBi_2O_4$ absorption spectrum. As seen in Fig. 5, Cu/Bi/$CuBi_2O_4$ nanocomposite has wider light absorption in the ultraviolet and visible regions pure $CuBi_2O_4$, and this promises much better photocatalytic performance.

3.2. Photocatalytic activity

3.2.1. Degradation of methylene blue

The results of methylene blue degradation through photocatalytic process are presented in Fig. 6. As shown in Fig. 6a, in the absence of photocatalyst, the efficiency of adsorption and degradation is negligible. Using Cu/Bi/$CuBi_2O_4$ nanocomposite, 93% efficiency has been achieved after 40 minutes of visible light irradiation, which is a significant result.

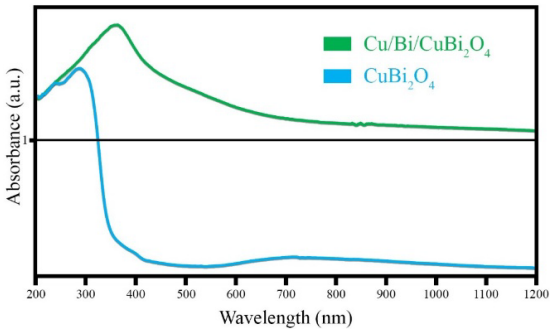


Fig. 5. UV–Vis diffuse reflectance spectra of Cu/Bi/$CuBi_2O_4$ nanocomposite and $CuBi_2O_4$

The performance of the Cu/Bi/$CuBi_2O_4$ nanocomposite in the absence of light is also excellent, and as can be seen, 83% of methylene blue has been adsorbed by 0.05 g of the nanocomposite within 40 minutes. It can be said that the synthesized Cu/Bi/$CuBi_2O_4$ nanocomposite is a good adsorbent. However, one of the characteristics of a good photocatalyst is that it has strong adsorption. In order to better determine the capability of the synthesized nanocomposite, the photocatalytic efficiency of pure $CuBi_2O_4$ was also evaluated. As seen in Fig. 6a, the adsorption of methylene blue by $CuBi_2O_4$ (in 40 minutes of darkness) was insignificant and after 40 minutes of irradiation, only 40% of methylene blue could be removed.

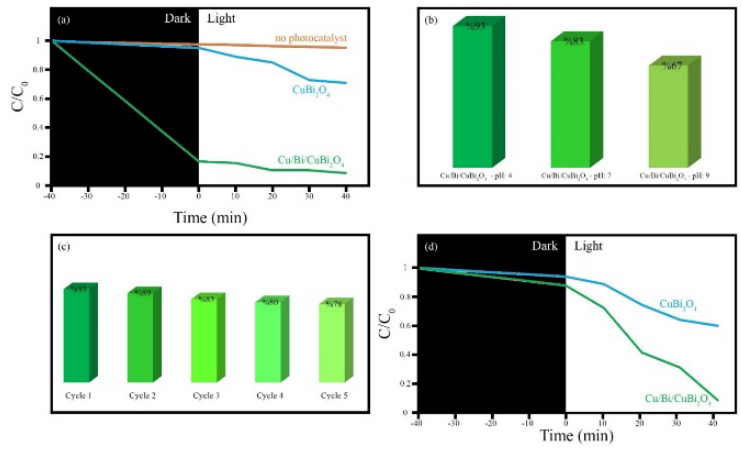
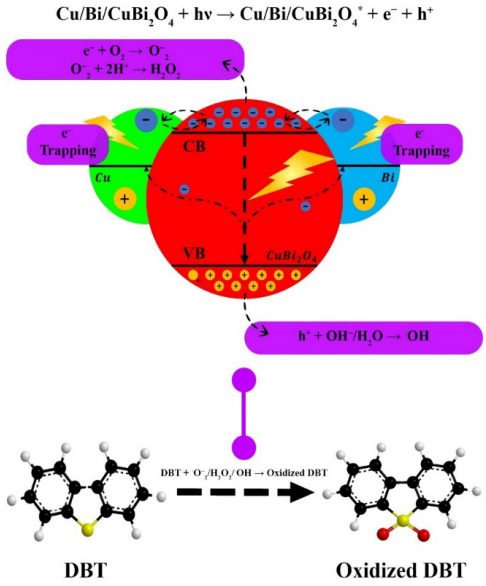


Fig. 6. (a) Photocatalytic degradation efficiency of methylene blue by Cu/Bi/$CuBi_2O_4$ nanocomposite and $CuBi_2O_4$, (b) the effect of Fig. 6. pH on the degradation of methylene blue by Cu/Bi/$CuBi_2O_4$ nanocomposite, (c) the photocatalytic recyclability of the as-synthesized Cu/Bi/$CuBi_2O_4$ nanocomposite for 5 cycles, and (d) percentage of photocatalytic oxidative desulfurization by Cu/Bi/$CuBi_2O_4$ nanocomposite and $CuBi_2O_4$.

According to DRS results, Cu/Bi/ $CuBi_2O_4$ nanocomposite has high light absorption and this can be the most important reason for its better photocatalytic efficiency than pure $CuBi_2O_4$. The presence of metal nanoparticles in the nanocomposite facilitates electron conduction and reduces electron-hole recombination, which can be another reason for the higher photocatalytic capability of Cu/Bi/ $CuBi_2O_4$ nanocomposite [23]. pH is one of the factors affecting the performance of a photocatalyst. The pH of the prepared methylene blue solution was acidic and equal to 4. Therefore, the performance of the photocatalyst was investigated in neutral (pH = 7) and alkaline (pH = 9) environments. As Fig. 6b shows, the highest efficiency has been obtained at acidic pH, and the photocatalytic activity has decreased with increasing pH. Fig. 6c shows the recyclability of the photocatalyst, and as it is clear, after 5 cycles, the efficiency has decreased by only 15% (from 93% to 78%), which is a favorable result. Photocatalytic desulfurization efficiency of Cu/Bi/$CuBi_2O_4$ nanocomposite and pure $CuBi_2O_4$ from dibenzothiophene is presented in Fig. 6d. As it is obvious, the Cu/Bi/$CuBi_2O_4$ nanocomposite with 87% sulfur removal has a higher efficiency than pure $CuBi_2O_4$ with 39% efficiency, which as mentioned above can be due to the higher light absorption of the nanocomposite. The mechanism of desulfurization of dibenzothiophene by Cu/Bi/$CuBi_2O_4$ nanocomposite is presented in Scheme 1.



Scheme 1. Photocatalytic desulfurization mechanism from dibenzothiophene by Cu/Bi/$CuBi_2O_4$ nanocomposite.

4. Conclusion

Cu/Bi/$CuBi_2O_4$ nanocomposite was synthesized through a one-step and controllable hydrothermal method, and its photocatalytic performance was evaluated in methylene blue decomposition and sulfur removal from dibenzothiophene. XRD, FT-IR, DRS, EDS, and FESEM analyzes were used to characterize the synthesized nanocomposite. Cu/Bi/$CuBi_2O_4$ nanocomposite showed a high photocatalytic ability, so that it could remove 93% of methylene blue and 87% of sulfur from dibenzothiophene in 40 minutes under visible light irradiation. Pure $CuBi_2O_4$ had 40% and 55% efficiency in methylene blue degradation and dibenzothiophene desulfurization, respectively. According to DRS results, Cu/Bi/$CuBi_2O_4$ nanocomposite has high light absorption in the visible region, and it is also easier due to the presence of electron-conducting metals in it. Therefore, increasing its photocatalytic performance can be expected. The effect of pH on the ability of the synthesized photocatalyst was also investigated, and it was found that Cu/Bi/$CuBi_2O_4$ has better efficiency in acidic environment. The recyclability of the photocatalyst was checked after 5 cycles, and it was found that the Cu/Bi/$CuBi_2O_4$ nanocomposite has good stability.

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Author contributions

Mohammad Reza Tahmasbi: Data curation, Formal analysis **Mehdi Mousavi Kamazani:** Project administration, Investigation, Methodology, Conceptualization, Writing– review & editing, **Mohammad Ghodrati:** Conceptualization, Writing – original draft, **Sanaz Alamdari:** Conceptualization, Writing – review & editing.

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Conflict of interest

The authors declare no conflict of interest.

Data availability

No data is available.

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