Employing synthesized Zn-based photocatalysts for degradation of Rhodamine B in an aqueous environment

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Abstract
In this study, three different types of Zn-based catalysts, including ZnAl₂O₄, ZnAl₂O₄/SiO₂ and ZnAl₂O₄/CNT (CNT – carbon nanotube) photocatalysts, were synthesized by the hydrothermal method. The successful synthesize and properties of the photocatalysts were investigated by infra-red spectroscopy, X-ray diffraction, field-emission scanning electron microscopy (FESEM), X-ray scattering energy, energy-dispersive X-ray analysis, nitrogen adsorption–desorption and photoluminescence (PL) spectroscopy. Then their efficiencies on the degradation of Rhodamine B dye were studied. The characterization tests have shown that the samples are correctly synthesized. Through FESEM, it was clear that the size of ZnAl₂O₄/CNT nanoparticles was smaller than that of ZnAl₂O₄/SiO₂ and consequently its surface area was greater. Nitrogen adsorption–desorption analysis showed that the ZnAl₂O₄/CNT sample has a higher surface area compared to ZnAl₂O₄/SiO₂, which provides more improvement in photocatalytic activity. The PL test showed that in ZnAl₂O₄/CNT sample, the electron–hole pair recombination rate was lower and the separation efficiency was many times higher, which increased the photocatalytic activity of ZnAl₂O₄/CNT. Finally, it was shown that the dye was completely removed by ZnAl₂O₄/CNT along with UV-light compared to other photocatalysts after 15 min of exposure.

Keywords: Zn nanocomposite; Photocatalytic degradation; Rhodamine B; Hydrothermal method; Carbon nanotube

1. Introduction
Through the quick advance in leather, textile, paper and food industries, dye pollutants of different water resources have increased extreme distresses due to their carcinogenicity and toxicity [1]. Thus, it is essential that dye pollutants must be eliminated from aqueous media before releasing them into the environment [2]. The photocatalysts are an effective method to degrade the dye pollutant molecule [3,4]. The textile industries consume large amounts of water and are one of the largest water-consuming industries. The amount of water consumed in these industries varies between 1 to 3 cubic meters per ton of product depending on the type of production process. Dyes are a group of complex organic materials that enter the environment at various stages in the textile industry. Therefore, color pollutants are one of the primary sources of organic contaminants in the textile industry and their conversion to harmless materials is essential. Inhalation of these compounds often causes respiratory disorders

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and direct contact with them can cause local burns, eye injuries, increased sweating and mental illnesses [5,6].

Rhodamine B is a cationic dye, highly water-soluble and acidic property due to the presence of the carboxylic acid group. Due to its intense color and durability, it is widely used in the textile industry and is discharged into sewage streams. In the absence of oxygen, these dyes form toxic substances that threaten human health and the water ecosystem, thus removing these hazardous dyes to create a clean and healthy environment is important [7].

An ideal treatment method for water and wastewater involves the decomposition of available toxins which should also be cost-effective and efficient. The advanced oxidation processes (AOPs) are referred to as oxidative processes, which are mainly based on the interference of hydroxyl radicals in the reaction of pollutant decomposition. The photocatalytic process is a branch of AOPs that produces hydroxyl radicals to react with resistant pollutants [8-11].

In recent years, metal oxides such as WO₃, CdS, Fe₂O₃, SnO₂, ZnS, ZnO, and TiO₂ have received the most studies in the field of photocatalytic reactions. This can be attributed to the low cost of these oxides, the appropriate optical and electrical properties, non-toxicity and the chemical stability of these compounds [12,13]. Titanium dioxide-based heterogeneous photocatalysts have received the most attention, but it has some disadvantages. The TiO₂ bandgap is approximately 3.2 eV, thus a large bandgap prevents the absorption of visible light in photocatalytic reactions. Since only about three to five percent of sunlight forms the UV portion, inactivation by esophageal radiation is one of the significant limitations of this photocatalyst. On the other hand, electron-hole coupling recombination is performed very fast in titanium dioxide. These disadvantages limit the use of TiO₂ in photocatalytic reactions and clarify the need for alternative compounds [14-16].

Carbon nanotubes have been an essential part of nanotechnology in recent years. These nanomaterials have unique physical and chemical properties that will make them increasingly applicable. High absorption power is one of the factors for their application in environmental activities. Carbon nanotubes have a higher adsorption capacity than other compounds, such as porous graphite carbon. Another prominent feature of these materials is the high surface-to-volume ratio of these adsorbents, which has a significant impact on their use as adsorbents [17]. Multi-walled carbon nanotubes have a fullerene (ball-like) structure with large layers of carbon atoms. The surface of these nanotubes has a high tendency to react with other molecules and atoms, so these adsorbents can absorb pollutants several times as active carbon [18].

Carbon nanotubes had a high adsorption capacity to remove methyl orange and methyl blue dyes of 1 and 2 mg g⁻¹, respectively. Many studies have been done on the removal of dyes by carbon nanotubes [19].

In this study, three different types of zinc-based catalysts, including ZnAl₂O₄, ZnAl₂O₄/SiO₂ and ZnAl₂O₄/CNT (CNT = carbon nanotube) photocatalysts, were synthesized by hydrothermal method. Then their properties were investigated by infrared (IR) spectroscopy, X-ray diffraction (XRD), energy-dispersive X-ray analysis (EDX), field-emission scanning electron microscopy (FESEM), nitrogen adsorption-desorption and photoluminescence (PL) spectroscopy. Finally, the efficiencies of these photocatalysts on the degradation of Rhodamine B dye in the aqueous environment were investigated.

2. Materials and methods

2.1. Materials and equipment

Nine-hydrated aluminum nitrate, six-hydrated zinc-nitrate, sodium hydroxide, carbon nanotube, and silicon oxide were purchased from Aldrich Company in America and used to synthesize photocatalysts. The Rhodamine B dye was purchased from Merck Company in Germany. All chemicals employed were of analytical grade and used without further purification as received. The X-ray diffraction investigations were executed on a Bruker D8 ADVANCE X-ray diffractometer. Infrared spectra were logged on a Shimadzu System FTIR-8400 spectrophotometer applying the KBr pellet manner. The UV-Visible spectrophotometry is a Lambda 25 model from PerkinElmer (USA), which records the spectrum in 200–800 nm. The used FESE device was the German ZEISS SIGMA VP model. The 2001 D8 ADVANCE model of the XRD device was used to determine the properties of precursors and the synthesized catalysts in the laboratory.

The specific surface area was considered through the Brunauer-Emmett-Teller (BET) technique by N₂ adsorption-desorption tests performed at −196°C on a Micromeritics ASAP 2010. The sample was outgassed at 200°C for 3 h, before each measurement. EDX, EDX-7000/8000, prepared by the Shimadzu Company in Japan, was employed to recognize the elemental composition of synthesized catalysts.

2.2. Synthesis of ZnAl₂O₄ by hydrothermal method

A wet chemical solution method is the hydrothermal technique using low temperatures to create nanometric catalysts directly with narrow size distribution and high surface areas without the need for later thermal treatments [20]. About 10 mL of an aqueous solution containing six-hydrated Zn-nitrate (10 mmol, 2.975 g) was added to a 10 mL aqueous solution of nine-hydrated aluminum nitrate (20 mmol, 7.503 g). Then, 0.1 M sodium hydroxide solution was added to the solution containing salts. Hydroxide sodium solution was added to adjust the pH range of 9–10. The resulting mixture was stirred at 50°C for an hour and then transferred to a 100 mL hydrothermal autoclave container and incubated at 180°C for 18 h. After the hydrothermal process, the autoclave cooled to ambient temperature. The sample was then filtered and washed twice with distilled water. The resulting precipitate was dried at 100°C for 2 h.

2.3. Synthesis of ZnAl₂O₄/CNT and ZnAl₂O₄/SiO₂ composite by hydrothermal method

ZnAl₂O₄ based catalysts can be prepared by sol-gel [21], co-precipitation [22] and hydrothermal methods. First, 5 mL of nanotube carbon suspension with an initial concentration of 25 mg mL⁻¹ was subjected to ultrasonic waves for 1 h. 10 mL of aqueous solution on six-hydrated
zinc nitrate (10 mmol, 2.975 g) was added to 10 mL aqueous solution of nine-hydrated aluminum nitrate (20 mmol, 7.503 g). The dispersed nanotube carbon mixture was added to humans containing soluble salts and then 0.1 M hydroxide sodium solution drop was added. Hydroxide sodium solution was added to adjust the pH range from 9.5 to 10.0. The resulting mixture was stirred at 50°C for 1 h and then transferred to a 100 mL hydrothermal autoclave container and incubated at 180°C for 18 h. After the hydrothermal process, the autoclave cooled to ambient temperature. The sample was then filtered and washed twice with distilled water and ethanol. The resulting precipitate was dried at 100°C for 2 h.

The synthesis method of ZnAl₂O₄/SiO₂ composite is similar to that of the previous one, using 5 mL of silicon oxide suspension containing 25 mg mL⁻¹ of silicon oxide in catalyst synthesis.

3. Results and discussion

3.1. Identification of synthesized catalysts

Different identification methods were used to identify the synthesized photocatalysts. Understanding the characteristics of the built-in catalysts is important for investigating their work. To study the structure and morphology of catalysts, identification techniques including IR spectroscopy, XRD, scanning electron microscopy (SEM), X-ray scattering energy and nitrogen adsorption–desorption were used.

3.2. Infrared spectroscopy of the samples

A series of absorption peaks in the range of 400–4,000 cm⁻¹ were showed by Fourier-transform infrared spectroscopy (FTIR) spectra. The functional groups present in the catalysts can be realized due to the specific frequencies of the absorption peaks. The molecular structure characteristics of the samples were identified by FTIR spectra. The infrared spectrum of functionalized carbon nanotubes is illustrated in Fig. 1a. The peak at 3,434 cm⁻¹ corresponds to the tensile vibration of the hydroxyl group. The 2,924 and 2,857 cm⁻¹ peaks are related to the tensile vibration of the –CH₂ group and the 1,390 and 1,457 cm⁻¹ peaks are related to the flexural vibration of the –CH₃ group. The peaks of 1,630 and 1,113 cm⁻¹ are related to the tensile vibrations of C=C and C–O carboxylic acid groups, respectively [23].

According to the FTIR spectrum of the ZnAl₂O₄ sample, the peak around 1,630 cm⁻¹ and three peaks in the 1,670–470 cm⁻¹ area are related to the HOH bending vibration of the absorbed water, the symmetric tensile Al–O vibration, the symmetric tensile Al–O vibration, and the asymmetric Al–O tensile vibration (Fig. 1b). These peaks are triple peaks of the regular spinel structure and the aluminum centers with octahedral coordinates which present in all synthesized photocatalyst samples [24,25].

The spectrum of ZnAl₂O₄/CNT is shown in Fig. 1c which is a combination of the infrared spectra of ZnAl₂O₄ and CNT. As it can be seen from Fig. 1d, the FTIR spectrum of the ZnAl₂O₄/SiO₂ sample showed the peak in the 1,670–470 cm⁻¹ area that is related to Al–O symmetric tensile vibration, and Al–O symmetric flexural vibration. The peak at 440 to 450 is associated with the bending vibrations of the O–Si–O bond and at 790 to 800 is to the symmetric tensile vibrations of the O–Si–O bond [26].

3.3. X-ray diffraction study of synthesized photocatalysts

The crystal structure of the samples was investigated by X-ray diffraction via Cu Kα radiation (λ = 0.15418 nm). The specimens were examined in angular range (2θ = 10°–90°). All patterns matched the standard templates.

The X-ray diffraction pattern of ZnAl₂O₄ showed a good agreement with the X-ray diffraction pattern of card

Fig. 1. (a) IR spectrum of carbon nanotube.
Fig. 1. (b) IR spectrum of ZnAl₂O₄, (c) IR spectrum of ZnAl₂O₄/CNT, and (d) IR spectrum of ZnAl₂O₄/SiO₂.
number \([1961-073-01]\) on aluminum oxide. The X-ray diffraction pattern of \(\text{ZnAl}_2\text{O}_4/\text{CNT}\) showed a good match with the X-ray diffraction pattern of card number \([0669-005-00]\). Also, the X-ray diffraction pattern of \(\text{ZnAl}_2\text{O}_4/\text{SiO}_2\) showed a good agreement with the X-ray diffraction pattern of card number \([1036-082-01]\). As presented by other researchers, the ordered preparation and crystallinity of the synthesized sample can be explored over XRD results \([27]\).

3.4. Using SEM test for the synthesized catalysts

In SEM analysis, a sample was scanned with an electron beam to create a magnified image for analysis. The morphology of the synthesized photocatalysts is shown as carbon nanotubes as rods on the substrate in Fig. 2. The photocatalyst was prepared through the hydrothermal method with a small particles size. The micrographs of \(\text{ZnAl}_2\text{O}_4\) and \(\text{ZnAl}_2\text{O}_4/\text{CNT}\) attained by SEM are presented in Fig. 3a–d, respectively. The micrograph of \(\text{ZnAl}_2\text{O}_4\) (Fig. 3a and b) exposed agglomerated particles with the morphology of shaped plate type and minor aggregates on the surface of larger clusters \([28]\). The SEM images of \(\text{ZnAl}_2\text{O}_4/\text{CNT}\) and \(\text{ZnAl}_2\text{O}_4/\text{SiO}_2\) (Fig. 3e and f) showed the presence of plate-like aggregates with porous structures and uneven surfaces. The morphology of the catalysts firmly rests on the used synthesis process. The \(\text{ZnAl}_2\text{O}_4\) powders prepared with polyhedral morphology with the solid-state method by Du et al. \([29]\) and zinc aluminate powders with rod-like-needles morphology synthesized by citrate sol–gel were investigated by Motloung et al. \([30]\).

The SEM pictures of the samples showed that the particles of \(\text{ZnAl}_2\text{O}_4\) based catalysts are nearly spherical with narrow particle size distribution. By comparing the images, it is evident that the size of the carbon nanotube nanoparticles is smaller than \(\text{SiO}_2\) and thus its surface area is more significant, which increases the carbon nanotube photocatalytic performance. This is confirmed by the BET results that are presented in the next section. Both synthesized \(\text{ZnAl}_2\text{O}_4\) powders prepared with polyhedral morphology with the solid-state method by Du et al. \([29]\) and zinc aluminate powders with rod-like-needles morphology synthesized by citrate sol–gel were investigated by Motloung et al. \([30]\).

3.5. Elemental analysis of the synthesized catalysts by EDX method

The EDX technique was used for chemical characterization and qualitative elemental analysis of samples \([31,32]\). The \(\text{Zn}\)-based catalysts were composed of \(\text{Zn, Al, C, Si, and oxygen}\). The EDX spectra of \(\text{ZnAl}_2\text{O}_4/\text{CNT}\) and \(\text{ZnAl}_2\text{O}_4/\text{SiO}_2\) nanocomposites are presented in Figs. 4 and 5. It can be seen the presence of carbon, oxygen, aluminum and zinc elements in the \(\text{ZnAl}_2\text{O}_4/\text{CNT}\) nanocomposite and the silicon, oxygen, aluminum and zinc elements in the \(\text{ZnAl}_2\text{O}_4/\text{SiO}_2\) nanocomposites indicating the synthesis of these compounds and, more importantly, the absence of other compounds.

3.6. BET results of synthesized samples

The pore volume and specific surface area of the synthesized catalysts were determined by \(\text{N}_2\) adsorption at \(-196^\circ\text{C}\).

3.7. Photoluminescence spectroscopy analysis

The PL analysis is a valuable tool for investigating the electrical, optical, and photoelectric properties of
Fig. 3. SEM images of the synthesized photocatalysts: (a,b) ZnAl₂O₄ photocatalyst, (c,d) ZnAl₂O₄/CNT photocatalyst, and (e,f) ZnAl₂O₄/SiO₂ photocatalyst.
semiconductor nanomaterial and a valuable method for understanding the processes occurring on the material surface. The improvement in the activity of photocatalysts and other nanocompound originated from a single up-conversion of graphene quantum dots or carbon has been investigated by many researchers [33,34]. Aleksandrzak et al. [35] investigated the alteration of graphitic carbon nitride by graphene oxide and reduced graphene oxide and their photocatalytic and photoluminescent was studied. Based on PL analysis, the recombination rate of the electron–hole pairs produced during the photocatalytic reaction can be studied. It can be noticed that the peak intensities recorded in this test were lower, so the recombination rate of the electron-cavity pairs will decrease and the separation efficiency will increases. This will lead to an increase in photocatalytic degradation of the pollutant. The PL spectrum of ZnAl2O4/SiO2 and ZnAl2O4/CNT nanocomposites with excitation wavelength at 340 nm shows a sharp peak at 650 nm wavelength, which is higher in ZnAl2O4/SiO2 nanocomposites than in ZnAl2O4/CNT, meaning that the ZnAl2O4/CNT sample has a lower electron-cavity recombination rate and higher separation efficiency. This would lead to increased photocatalytic degradation of the contaminant by the ZnAl2O4/CNT sample. It should be noted that the PL spectra of pure ZnAl2O4 are not shown in Fig. 6, but according to the results of other researchers [36], it was clear that it has a sharper peak than the peaks of ZnAl2O4/SiO2 and ZnAl2O4/CNT sample at 650 nm, which showed that it has a higher electron-cavity recombination rate and consequently lower photocatalytic activity than the modified ones.

3.8. Effect of different photocatalysts on RhB

The UV-Vis absorption spectrum of Rhodamine B in aqueous solution in the presence of ZnAl2O4, ZnAl2O4/SiO2 and ZnAl2O4/CNT nanocomposites showed that with an increase in irradiation time for all samples, the absorption intensity of dye at 554 nm was decreased. However, complete disappearance for ZnAl2O4/CNT occurred after 15 min of illumination (Fig. 7), but in the case, with ZnAl2O4 and ZnAl2O4/SiO2 photocatalysts, the degradation efficiency after an hour was 40% and 47%, respectively. Since no new peak was observed in the UV-Vis spectrum, it can be concluded that in ZnAl2O4/CNT technique, all intermediate products formed have been destroyed.

The degradation efficiency is calculated by the following equation:

\[
\text{Degradation efficiency} = \left( \frac{A_0 - A}{A_0} \right) \times 100
\]

where \(A_0\) is the initial color concentration and \(A\) is the color concentration at time \(t\). The color absorption capacity, bandgap energy, surface and structural properties of the catalyst, as well as the cavity-electron coupling energy produced in the photocatalytic process and their separation, are some of the critical parameters that influence the
photocatalytic performance. The degradation of Rhodamine B on the CNT-nanocomposite sample is higher than that of other samples due to the intensive interactions between Rhodamine B and the carbon nanotube.

In the photocatalytic test, about 0.4 g of catalyst was mixed with a 500 mL RhB solution (1 × 10⁻⁵ M) at pH of 7, and an adsorptive test mainly continued in the dark for 30 min to reach desorption–absorption equilibrium of RhB on the catalyst surface before irradiation. After an interval of the arranged time, 3 mL of dye solution was withdrawn for concentration analysis, which was checked by UV-Vis spectrophotometer.

The degradation efficiencies of RhB for various samples under the UV-light were investigated and the results are presented in Fig. 8. Compared with the ZnAl₂O₄/SiO₂ particle, the improved photocatalytic property of ZnAl₂O₄/CNT is related to the expansion of specific surface area to obtain more active sites which can play an important role in the absorption of UV-light. These findings are in agreement with the results of other researchers such as Xie et al., that they found out the photocatalytic activity of T/Z-5 is nearly 30% greater than that of T/Z-0 ones, showing that the combination of TiO₂ into ZrO₂ origins a slow in recombination of electron-hole pair and rapid electron transfer [37].

3.9. Investigation of the intermediate reaction

Degradation of the organic pollutants by photocatalysts mostly can occur via the absorption of photo and generation of holes and electrons on the catalyst, the transmission of charge transporters, and the application of the charge transporters with reagents. ZnAl₂O₄/SiO₂ particle, the improved photocatalytic property of ZnAl₂O₄/CNT is related to the expansion of specific surface area to obtain more active sites which can play an important role in the absorption of UV-light. These findings are in agreement with the results of other researchers such as Xie et al., that they found out the photocatalytic activity of T/Z-5 is nearly 30% greater than that of T/Z-0 ones, showing that the combination of TiO₂ into ZrO₂ origins a slow in recombination of electron-hole pair and rapid electron transfer [37].

mineralization of RhB. Perhaps the including reactions are as the succeeding [Eqs. (2)–(10)]:

$$\text{ZnAl}_2\text{O}_4 + h\nu \rightarrow e_{CB}^- + h_{Rb}^+$$  \hspace{1cm} (2)

$$e_{CB}^- + h_{Rb}^+ \xrightarrow{\text{Recombination}} \text{heat}$$  \hspace{1cm} (3)

$$h_{Rb}^+ + \text{RhB} \rightarrow \text{Intermediates} \rightarrow \text{CO}_2 + \text{H}_2\text{O}$$  \hspace{1cm} (4)

$$h_{Rb}^+ + \text{H}_2\text{O}_{\text{ads}} \rightarrow \text{OH}^-_{\text{ads}} + \text{H}^+$$  \hspace{1cm} (5)

$$h_{Rb}^+ + \text{OH}^-_{\text{ads}} \rightarrow \text{OH}^+_{\text{ads}}$$  \hspace{1cm} (6)

The conduction band of photocatalyst has the same potential as oxygen regeneration potential. In many photocatalytic reactions, the adsorbed oxygen acts as a trap for the electrons in the conduction band and avoiding recombination of electrons and holes and subsequently decreasing active sites [Eq. (3)]. The transfer of electrons to oxygen is the speed limiting step in photocatalytic reaction [Eq. (7)], therefore the effect of oxygen concentration on this step was removed in all experiments using stirring, providing oxygen thru air pump or shallow selection of solutions inside reactors [38].

In heterogeneous photocatalytic processes, the cavities in the VB of the catalyst oxidize the water molecules of the hydroxide absorbed on the catalyst's surface and produce hydroxyl radicals [Eq. (5)]. Oxidation of RhB molecules may occur both indirectly, through surface-bound hydroxyl radicals, and directly through the cavity of the VB before being trapped inside or at the surface of the catalyst. The electrons produced by the photocatalytic process at the CB may react with oxygen to form superoxide radicals (O₂⁻•) [Eq. (7)], and these radicals may form the peroxide of RhB or hydrogen peroxide [Eqs. (8)–(9)]. Intermediate compounds also react with hydroxyl radicals to form final mineral and nontoxic products such as water and carbon dioxide.

$$e_{CB}^- + \text{O}_2 \rightarrow \text{O}_2^\cdot$$  \hspace{1cm} (7)
$O_2^+ + RhB \rightarrow RhB – OO^*$

$O_2^+ + OH^+ + H^+ \rightarrow O_2 + H_2O_2$

$OH_\text{ads}^+ + RhB_{\text{ads}} \rightarrow \text{Intermediates} + OH_\text{ads} \rightarrow CO_2 + H_2O$

4. Conclusion

This study was performed to evaluate the photocatalytic degradation of Rhodamine B. Single-phase ZnAl$_2$O$_4$ spinel-type catalysts have been prepared by the direct method through hydrothermal technique without calcination. The ZnAl$_2$O$_4$/CNT powder showed a larger BET surface area and smaller pore size and pore volume than ZnAl$_2$O$_4$/SiO$_2$. The results showed an improvement in the photocatalytic activity of composites compared to pure ZnAl$_2$O$_4$. It was clear from FESEM that the size of ZnAl$_2$O$_4$/CNT nanoparticles was smaller than that of ZnAl$_2$O$_4$/SiO$_2$ and consequently its surface area was greater.

The presence of reduced CNT improved the photocatalytic activity of ZnAl$_2$O$_4$. The use of CNT as a catalyst substrate increased the surface area and subsequently catalyst performance. BET analysis showed that the larger surface area provides more adsorption sites during a photocatalytic reaction and thereby improves the photocatalytic activity.

The PL test showed that in the ZnAl$_2$O$_4$/CNT sample, the recombination rate of the electron–hole pairs was lower and the separation efficiency was many times higher, which increased the photocatalytic efficiency.

The UV-Vis absorption spectrum of Rhodamine B in an aqueous solution showed that the dye was completely removed by ZnAl$_2$O$_4$/CNT compared to the other photocatalysts, after 15 min of reaction. While in ZnAl$_2$O$_4$ and ZnAl$_2$O$_4$/SiO$_2$ runs, about 40% and 47% of the dye were removed after 1 h of reaction.

References


