A facile synthesis of MgFe2O4/ZnS heterojunction with effectively enhanced visible light photocatalytic activity for degradation of methylene blue and crystal violet dyes

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Abstract

A novel n-MgFe$_2$O$_4$–n-ZnS heterojunction catalyst was employed via two step approach for photodegradation of organic dyes such as methylene blue (MB) and crystal violet (CV) dyes under visible light irradiation. The synthesized MgFe$_2$O$_4$/ZnS NCs were characterized using PXRD, FTIR, UV-visible spectroscopy, PL and FESEM analysis which reveals the formation of flake like structure with size as to be ~50 nm. The photocatalytic experimental result demonstrates that the MgFe$_2$O$_4$/ZnS nanocomposites (NCs) improve photodegradation performance with 98.0% and 91% decomposition of MB and CV dyes within 120 min illumination during simulated visible light irradiation. From the result, MgFe$_2$O$_4$/ZnS NCs has higher photocatalytic performance than that of MgFe$_2$O$_4$, and ZnS due to efficient separation of the photo-induced electron-hole pairs and effective electron–hole generation transfer by the formation of n-MgFe$_2$O$_4$–n-ZnS heterojunction. Hence, photodegradation performance implies that the synthesized MgFe$_2$O$_4$/ZnS NCs can be effectively utilized in waste water purification systems.

1. Introduction

Interest in research on semiconductor based photocatalysts has attracted much attention in the fields of photochemistry, catalysis, electrochemistry, and owing to their high chemical stability and photocatalytic activity [1]. Several semiconductor photocatalysts like ZnO, TiO$_2$, WO$_3$, CdO, Fe$_2$O$_3$, ZnS, MoS$_2$ and CdS etc. have been more apt for the elimination of organic pollutants contaminate in the waste water which release from the paper, leather and textile industries [2]. Among this, ZnS act as a considerable II–VI compound semiconductor material with wide band gap (3.6 eV) and large exciton binding energy (40 meV), has been studied widely as an excellent photocatalyst due to its quick photoexcited electron (e$^-$) - hole (h$^+$) pairs and highly negative reduction potentials of excited electrons [3, 4]. Numerous methods have been utilized to develop ZnS nanoparticles (NPs) like electrochemical deposition, sol-gel, solvothermal, hydrothermal, co-precipitation, pyrolysis, microemulsion, laser ablation, combustion synthesis, and vapor deposition [5]. Among them, co-precipitation technique has various reaction time, temperature, and parameters like pH. As well as, initial concentration of the solution and material, have played a crucial role on receiving ceramic powders with preferred shape and size [6].

In current decade, transition metal ferrite (ZnFe$_2$O$_4$, CdFe$_2$O$_4$, MgFe$_2$O$_4$, NiFe$_2$O$_4$, CoFe$_2$O$_4$, CuFe$_2$O$_4$, SrFe$_{12}$O$_{19}$ etc.) has been widely researched as photocatalysts due to their easy magnetic separation, photocorrosion in aqueous solutions, low cost and biocompatibility [7]. Among ferrites, magnesium ferrite (MgFe$_2$O$_4$, MFO) is an n-type semiconductor with a spinel structure, which endows the absorption of visible light owing to their narrow band gap of 2.0 eV [8]. Fascinatingly, earlier literatures revealed p-n (or) n-n type heterojunction structure for enhance the photocatalytic activity by their charge separation efficiency between photo-induced electron and hole pair’s [9]. In this aspect, Su et al. has developed MgFe$_2$O$_4$–ZnO heterojunction photocatalyst for degradation of Rhodamine B organic dye. The excellent photocatalytic activity has been achieved via interconnected heterojunction of n-MgFe$_2$O$_4$ and n-ZnO nanoparticles [10].
Therefore, in the present investigation focus to form a novel n-MgFe$_2$O$_4$/n-ZnS heterojunction for improve the photocatalytic activity owing to their efficient separation of photo-excited electron and hole pairs. The photocatalytic activity was investigated by prepared samples of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs under visible-light illumination by the photodegradation of methylene blue (MB) and crystal violet (CV) and reported for the first time.

2. Experimental Procedure

2.1. Synthesis of MgFe$_2$O$_4$ Nanoparticles

In a chemical co-precipitation process, MgFe$_2$O$_4$ NPs were fabricated, and this step-by-step technique is followed. Mg(NO$_3$)$_2$•6H$_2$O (Magnesium (II) nitrate hexahydrate) and Fe(NO$_3$)$_3$•9H$_2$O (Ferric (III) nitrate nonahydrate) are taken and gradually mixed in 200 ml of DD water in a 1:2 ratio. The solvent was stirred to produce a homogeneous solution for 30 minutes. After that, diluted NaOH (4M in 20 ml of DD water) aqueous solution was added to the above homogenous solution and stirring for 7 hours. Finally, precipitate was obtained and filtered through filter paper using ethanol as well as dried in hot air oven at 80°C for 12 hours. The dried particles are smashed and calcined at 700°C in the muffle furnace for 3 hours.

2.2. Synthesis of ZnS nanoparticles

To achieve ZnS nanoparticles, Zn(NO$_3$)$_2$•6H$_2$O (Zinc nitrate hexahydrate) was dispersed in 100 ml of DD water. Subsequently, 0.5 M of sodium sulfide (Na$_2$S) was added slowly into the above formulation mixture and stirred vigorously for 7 hours. The attained precipitate was collected and subsequently rinsed using ethanol. Finally, washed precipitate was dried at 80°C for 12 hours in hot air oven. The substance was grinding with a mortar to produce a fine powder.

2.3. Synthesis of MgFe$_2$O$_4$/ZnS nanocomposite

In order to achieve a complete dispersion of nanoparticles, 1 mg of MgFe$_2$O$_4$ NPs was dissolved in 100 ml of ethanol and sonicate for 45 minutes. After that, CTAB (0.15 M) was mixed and vigorously stirred. Subsequently, Zinc nitrate hexahydrate (0.5 M) salt was gradually mixed with that solution and stirred well. After that, 0.5M of sodium sulfide was added gradually and vigorously stirring for 7 hours. Finally, the collected precipitate was rinsed several times with ethanol and dried for 12 hours at 80°C in a hot air oven.

2.4. Characterization of as prepared MgFe$_2$O$_4$/ZnS NCs

The structural characterization of the synthesized nanomaterials was analyzed using XRD pattern made by Bruker AXS D8 Advance diffractometer. The attained stretching and bending vibrations of prepared MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs were recorded using FT-IR (Perkin-Elmer spectrum), in the range of 400-4000cm$^{-1}$. UV-visible absorption spectrum of the synthesized nanoparticles were analyzed using
Jasco7800 at the wavelength ranging from 200-800nm. Morphology and topographical view of the synthesized nanomaterials were determined using FESEM analysis (JSM-7600F Japan) equipped with energy dispersive X-ray (XL 30 Philips instruments).

2.5. Experimental Setup of Photocatalytic activity

In order to evaluate the photocatalytic function of the prepared nanoparticles and nanocomposites, a commercial photo-reactor unit with built-in reflectors as well as a 200 W tungsten halogen light with range of wavelength from 320 nm to 850 nm was utilized. Organic dyes were taken as sample contaminants, such as Crystal Violet (CV) and Methylene Blue (MB). Each dye was taken by 100 ml of 20 mg/L solution with catalyst (1 mg) was applied to decay the dye solution. The deterioration mechanism of dye solutions was monitored in UV-Visible spectrophotometer by concentration changes at each 20 minutes for up to 2 hours. The photocatalytic degradation ratio (DR) was determined for MB and CV dyes by the given equation

\[ DR = \left[ \frac{(C_0 - C_t)}{C_0} \right] \times 100\% \]

Where, \( C_0 \) & \( C_t \) are the initial dye concentration and concentration of dyes at the irradiation time \( t \).

3. Results And Discussion

3.1 Structural analysis

The Powder X-ray diffraction (PXRD) gives details about the crystallographic structure and phase detection of crystalline materials. The PXRD patterns of as-prepared MgFe\textsubscript{2}O\textsubscript{4}, ZnS and MgFe\textsubscript{2}O\textsubscript{4}/ZnS NCs exhibit in Fig. 1 (a-c). The primary diffraction (2\( \theta \)) intensity peaks values of MgFe\textsubscript{2}O\textsubscript{4} NPs at 18.22\( ^\circ \), 30.09\( ^\circ \), 35.43\( ^\circ \), 37.30\( ^\circ \), 43.30\( ^\circ \), 53.70\( ^\circ \), 57.16\( ^\circ \), 62.76\( ^\circ \), 74.75\( ^\circ \) were corresponds to \( (hkl) \) planes of (111), (220), (311), (222), (400), (422), (511), (440), and (503), are in good agreement with the cubic spinel phase of MFO NPs (JCPDS card No. 88-1943) and lattice planes of (111), (220) and (311) respectively for ZnS (JCPDS card no. 05-0566) [11, 12]. The average crystallite size (D) of the prepared materials was evaluated by Scherrer formula [13–15].

\[ D = \frac{0.94\lambda}{\beta \cos \theta} \]

Where, \( \lambda \) signifies the wavelength of Cu K\( \alpha \) radiation (1.5405Å), \( \beta \) and \( \theta \) are full width at half maximum of broadened characteristic peaks and Bragg diffraction angle, respectively. The average particle size of as-synthesized MgFe\textsubscript{2}O\textsubscript{4}, ZnS and MgFe\textsubscript{2}O\textsubscript{4}/ZnS NCs was determined to be 33 nm, 47 nm, and 55 nm.

3.2 Spectroscopic Studies

FTIR spectroscopy reveals various functional groups with metal-oxygen bonds existence in the prepared MgFe\textsubscript{2}O\textsubscript{4}, ZnS and MgFe\textsubscript{2}O\textsubscript{4}/ZnS NCs materials, which was depicts in Fig. 2. The bands arising at 3536, 3451, 3368 cm\(^{-1}\) and 1626 cm\(^{-1}\) are attributed owing to stretching and bending vibration of hydroxyl O-H
and H-O-H functional groups of water content occurrence on the exterior of the sample [16]. The bands at 422 cm$^{-1}$ and 534 cm$^{-1}$ are assigned to the stretching vibrational metal oxide modes of Mg–O and Fe–O bonds in MFO, which indicating the successful formation of spinel magnetic ferrite structure are present in the synthesis of MgFe$_2$O$_4$ NPs and MgFe$_2$O$_4$/ZnS NCs [16, 7]. The peak at 654 cm$^{-1}$ is attributed owing to symmetric bending vibration of ZnS [17].

3.3. Optical Analysis

3.3.1 Optical absorption studies

The UV–Vis absorption spectrum of the synthesized MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs were shown in Figure.3. The ZnS respond strong UV-light absorption (200–450 nm) and MgFe$_2$O$_4$ act as more visible light region (600–700 nm) [17, 18]. The energy band gap (E$_g$) of the prepared materials was estimated based on the optical absorption edge attained from the UV–Visible absorption spectra by Tauc's relation:

$$(\alpha h\nu)^2 = A (h\nu - E_g)$$

Where, ‘A’ signifies as constant depending on the type of transition, ‘$\alpha$’ and ‘$h\nu$’ signifies as an absorption coefficient and discrete photon energy [19]. The band gap of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs was determined to be 1.8 eV, 2.94 eV and 2.05 eV, respectively. The optimal absorption of MgFe$_2$O$_4$/ZnS NCs sample shift towards visible light wavelength region compare to ZnS, which implies that ZnS enhances the visible light absorption capability of MgFe$_2$O$_4$ NPs. Therefore, the as prepared MgFe$_2$O$_4$/ZnS NCs act as crucial role to improve photocatalyst during visible light irradiation.

3.3.2 Photoluminescence (PL) studies

The PL spectrum is used to study about the charge transfer, charge carrier trapping efficiency, and immigration as well as oxygen vacancies and surface defects. PL spectra demonstrate near band edge emissions peak occur at 359, 362, 368 nm. In addition, blue and green emission peak arising at 412, 415 nm and 577, 583 nm as well as some more blue emission peaks take place at 458 nm and 493 nm could be attributed to the electron move from sulfur vacancies to zinc defects and zinc vacancies for ZnS and MgFe$_2$O$_4$/ZnS NCs (Fig. 4) [17]. Green emission may generate due to radiative recombination of a photo-induced hole and an electron occupying oxygen vacancy as well as Zn interstitial sites transfer to the deep accepter levels of oxygen vacancies which existence in the ferrite species of MgFe$_2$O$_4$/ZnS NCs [20].

3.4 Morphological Analysis

FESEM analysis is an important tool which provides information about the morphology, topography and composition of the synthesized MgFe$_2$O$_4$/ZnS NCs. Figure 5 (a-b) shows FESEM images of prepared MgFe$_2$O$_4$/ZnS NCs material, whereas the synthesized material exhibits flake like structure and average
particle size of the MgFe$_2$O$_4$/ZnS NCs lies between 50 and 100 nm. The EDAX analysis was used to examine the chemical composition of the MgFe$_2$O$_4$/ZnS NCs and it is reveals in Fig. 5(c). The uniform arrangement of Mg, Zn, Fe, S, and O elements exist in the prepared MgFe$_2$O$_4$/ZnS NCs.nanocatalysts.

3.5 Photocatalytic activity of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs

The photocatalytic activities of the as-prepared samples of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs were evaluated by the degradation of model organic pollutants (MB and CV dyes) under visible-light photocatalytic irradiation, respective photocatalytic results are depicts in Fig. 6 (a & b). The major two absorption peaks are arising at 665 nm and 592 nm correspond to MB and CV dyes. Before illumination, adsorption/desorption experiment was taken under dark condition for 60 min to ensure the equilibrium of the solution. The maximum absorbance intensity peak of MB (665nm) and CV (592 nm) dyes are steadily decreased in 120 min irradiation time by occurrence of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS NCs. In addition, no other new peak was not appear under degradation of both dyes due to absence of photocatalyst there is a negligible amount of MB and CV dyes under visible light illumination. As can be seen from Fig. 7 (a &b), the photocatalytic degradation rate for 120 min of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS nanocatalyst for MB and CV dyes are tabulated in Table 1. The MgFe$_2$O$_4$/ZnS nanocatalyst has superior dye degradation efficiency than that of MgFe$_2$O$_4$ and ZnS NPs from the observed result. This may create owing to photo-excited $e^-$ in CB of MgFe$_2$O$_4$ migrate to the ZnS owing to MgFe$_2$O$_4$ has more negative than that of ZnS and the photo-induced h$^+$ on ZnS transfer to MgFe$_2$O$_4$ due to the different VB edge potentials [9]. Besides, $e^-$ in the CB of MgFe$_2$O$_4$ reacts with molecular oxygen adsorbed above the surface of MgFe$_2$O$_4$ to produce superoxide anions (O$_2^-$) [21, 22]. Meanwhile, photo-excited holes in VB of ZnS can simply response with surface-bound hydroxyl groups (H$_2$O) to generate a ’OH radicals [21]. Therefore, superoxide anions and OH radical's are plays a major role for degradation of MB and CV dyes. The photocatalytic activities of MgFe$_2$O$_4$, ZnS and MgFe$_2$O$_4$/ZnS nanocatalyst for degradation of MB and CV dyes obeys the pseudo–first–order reaction kinetics according to Langmuir–Hinshelwood model and its expression is as follows:
Table 1
Table content of rate constant, regression coefficient (R^2) values and photocatalytic degradation efficiency of MgFe_2O_4, ZnS and MgFe_2O_4/ZnS NCs

| Organic dyes | Prepared Catalysts | Rate constant (k) (s^{-1}) | Regression coefficient (R^2) values | Photocatalytic Degradation Efficiency |
|--------------|---------------------|----------------------------|-------------------------------------|---------------------------------------|
| MB           | MgFe_2O_4           | 10.64 \times 10^{-3}       | 0.88710                             | 76%                                   |
|              | ZnS                 | 12.75 \times 10^{-3}       | 0.95002                             | 80%                                   |
|              | MgFe_2O_4/ZnS NCs   | 28.60 \times 10^{-3}       | 0.75107                             | 98%                                   |
| CV           | MgFe_2O_4           | 11.09 \times 10^{-3}       | 0.89873                             | 78%                                   |
|              | ZnS                 | 12.96 \times 10^{-3}       | 0.95844                             | 82%                                   |
|              | MgFe_2O_4/ZnS NCs   | 18.97 \times 10^{-3}       | 0.91584                             | 91%                                   |

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

Where, \(C_0\) & \(C_t\) are the initial dye concentration and concentration of dyes at the irradiation time \(t\). Figure 7 (c) and 7 (d) exhibits the variation of \(\text{ln} \left( \frac{C_t}{C_0} \right)\) with time [22]. From the obtained linear slope, the regression coefficient (R^2) and rate constant (k) values of MgFe_2O_4, ZnS and MgFe_2O_4/ZnS nanocatalyst were estimated and tabulated those values in Table 1.

4. Conclusion

In conclusion, the novel MgFe_2O_4/ZnS heterojunction nanocatalysts have been developed for the first time by two step process. The MgFe_2O_4/ZnS nanocatalysts act as superior photocatalytic activity compare to MgFe_2O_4, ZnS towards MB and CV dyes during visible-light illumination. This may happen due to formation of n-MgFe_2O_4 and n-ZnS heterojunction nanocatalyst exhibit efficient charge separation and effectively reduces e^- - h^+ pair’s recombination, which is more helpful to degrading the organic dyes. Therefore, observed result concluded that the present study implying the possibility of its being used in industrial wastewater treatment in the future.

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**Figures**
Figure 1

The PXRD patterns of the as-prepared samples: (a) MgFe2O4, (b) ZnS, and (c) MgFe2O4/ZnS NCs
Figure 2

FTIR spectra of (a) MgFe2O4 and (b) ZnS, (c) MgFe2O4/ZnS NCs
Figure 3

(a) UV-Vis spectra (b) Tauc's plots of MgFe2O4, ZnS and MgFe2O4/ZnS NCs
Figure 4

The PL emission spectra of MgFe2O4, ZnS and MgFe2O4/ZnS NCs
Figure 5

(a&b) FE-SEM images and (c) EDAX spectra of MgFe2O4/ZnS NCs

Figure 6

(a) MB Dye and (b) CV Dye absorption spectra over time.
change in absorbance of (a) MB and (b) CV solution with time under UV light illumination in the presence of MgFe2O4/ZnS nanocatalyst.

Figure 7

Photodegradation behaviour of (a) MB and (b) CV with catalysts of MgFe2O4, ZnS and MgFe2O4/ZnS NCs under visible-light illumination. The apparent (c) MB and (d) CV degradation rate of ln(C/C0) against Time with catalyst of MgFe2O4, ZnS and MgFe2O4/ZnS NCs.