Research article

Gold-selenide quantum dots supported onto cesium ferrite nanocomposites for the efficient degradation of rhodamine B

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ABSTRACT

In this work, different weight percentage of gold-selenide quantum dots (AuSe QDs) (1.0, 2.5, 5.0 and 7.0 wt.%) were successfully synthesized and decorated on cesium ferrite nanocomposite (Cs2Fe2O4 NC). The as-prepared pure AuSe QDs, pure Cs2Fe2O4 NC, and x wt.% AuSe QDs/Cs2Fe2O4 NC photocatalysts were investigated using different characterization techniques such as nitrogen adsorption desorption isotherms (BET), X-ray diffraction patterns (XRD), transmission electron microscopy (TEM), and UV-vis absorption spectroscopy. The results show that AuSe QDs were uniformly distributed on Cs2Fe2O4 NCs surface as spherical dots with an average size of 1.0–8.0 nm. While the Cs2Fe2O4 NCs possess an average size between 10 to 35 nm. The photocatalytic performance of x wt.% AuSe QDs/Cs2Fe2O4 NCs were measured through the photodegradation of rhodamine B (RhB) dye as a model water pollutant, under a 150 W-Mercury lamp with a filter (JB400) as a simulated source of visible light. The results revealed that the % degradation of RhB increased from 50.0 %, 59.1 %, 76.4 %, and to 99.15 % within 150 min for the pure Cs2Fe2O4, 1.0, 2.5 and 5.0 wt.% AuSe QDs/Cs2Fe2O4 NC photocatalysts, respectively. The 5.0 wt.% AuSe/Cs2Fe2O4 NC sample showed highest photocatalytic activity. The effect of recycling also studied. High photocatalytic performance and superior stability confirmed that the prepared nanocomposites act as good photocatalysts.

1. Introduction

Nanotechnology is a rapidly growing field in which researchers deal with the synthesis and design of nanomaterials which are ranging from 1-100 nm [1, 2, 3, 4, 5]. The nanomaterials (NMs) have a remarkable differences in the catalytic, photocatalytic, biological and structural properties compared to the same materials in the bulk [6]. These differences in the nanomaterial's properties are due to differences in their size, shape and surface area with advancement in nanotechnology [7]. Quantum dots (QDs) [8, 9] have been attracted more attention among the researchers, may be due to their unique catalytic/photocatalytic capability in the chemical catalysis and the photocatalysis processes [10, 11, 12, 13]. The QDs have been extensively applied in the fields of environmental treatment/water treatment, chemical industry, sensors, solar cells, electronics, optoelectronics devices, cosmetics, drug delivery systems, and biological processes [10, 14, 15, 16, 17, 18]. There are many nanoparticles (NPs) such as CdSe, ZnSe, CuSe, AgSe, Se, Ag, Cu, Au, Zn, and other metallic QDs which are coupled with/or supported on the magnetic composites [19, 20]. This coupling can be tuned to get the desired electrical, optical, and photocatalytic/catalytic properties on the surfaces of nanocomposites (NCs) [21, 22, 23, 24, 25, 26, 27, 28, 29, 30]. Thus, there is a need to develop green, rapid, and reliable experimental protocols for the synthesis of QDs, NCs or NPs [31, 32]. Precipitation method as a significant synthetic approach has many benefits over other methods. Thus, attention has been focused on the preparation of NMs using this approach [33].

Synthesis and coupling of metallic NPs with metallic ferrite materials [2, 39] are emerging as an alternative and efficient method for modifying their catalytic/photocatalytic, optical, and surface properties. Metallic ferrite materials have the following general formula MFe2O4, where M sites are occupied by the bivalent cations such as Mg2+, Co2+, Zn2+, Mn2+, and Ca2+ or by the monovalent cations such as K+, Cu+, and Rb+; while Fe ions are trivalent (Fe3+) [34, 35]. The metallic ferrite materials have remarkable electrical and magnetic properties as well as
their non-toxicity, cost-efficiency, and high stability, which can be used in numerous applications such as transformers, high-frequency devices, and quality filters. Recently, there are numerous techniques that used for removing dyes from wastewater such as chemical oxidation [36, 37], biological treatment [38, 39], microwave catalysis [40, 41], adsorption [42, 43, 44, 45, 46, 47], and photochemical catalytic degradation [48, 49]. Each of these techniques has inherent advantages, and disadvantages. Photodegradation of the organic pollutants by various photocatalysts have reached good degradation efficiencies via tuning of catalyst structures, manipulation of particle sizes and introduction of metal dopants, but the photocatalysts still require further efforts toward high photocatalytic activity, selectivity, stability and durability for degradation of the chemical organic pollutants.

In this work, we attempt to open a new door in the photocatalytic degradation field by introducing synthesized AuSe QDs decorated Cs₂Fe₂O₄ NC as novel photocatalysts for RhB degradation. The photocatalytic activity of catalysts is directly associated to the surface plasmon resonance phenomena (SPR), synergistic effect, and the band gap. By controlling the particle sizes, the electrons and holes are confined leading to an increase in the band gap, so this electron-holes confinement is referred to as quantum confinement. The QDs represent the same way as an individual atom. In fact, the band gap of nanoparticles increases as the particle sizes decrease. Thus, we tried to overcome this problem by immobilization AuSe QDs onto Cs₂Fe₂O₄ NCs. Due to the synergistic effect, and the overlapping of Fermi level of AuSe QDs and magnetic Cs₂Fe₂O₄ NCs, the separation of photogenerated holes and electrons will be significantly enhanced. Furthermore, several techniques have been used to investigate the chemical, optical and surface plasmon resonance and photocatalytic properties of the prepared photocatalysts using XRD, TEM, UV-visible spectroscopy, and BET.

2. Experimental

2.1. Materials

Sodium borohydride (NaBH₄, >98%), iron (III) nitrate (Fe(NO₃)$_3$, 9H₂O, > 99%), and cesium nitrate (CsNO₃, >99%) were obtained from Sigma Aldrich, while Selenium chloride and Gold (III) chloride hydrate were obtained from Merk.

2.2. Synthesis of Cs₂Fe₂O₄ composite

Firstly, 10 mmol of CsNO₃ and 20 mmol of Fe(NO₃)$_3$. 9H₂O were added to 50 ml of distilled water under vigorously stirring followed by addition of 50 ml of 2.0 M NaOH solution under constant stirring for 30 min. In the next step, the resultant mixture was concentrated on a water bath at 80 °C with continuous stirring until the formation of brown gel. Then, the brown gel was washed three times, centrifuged and dried at 80°C overnight. Finally, the resultant brown powder was calcined at 400°C for 4 h [10, 35, 50].

2.3. Fabrication of x wt.% AuSe QDs/Cs₂Fe₂O₄ NCs

Firstly, AuSe QDs were prepared as previously reported literature with some modification [51]. 5.0 mmol of gold chloride and 5.0 mmol of selenium chloride were dissolved in distilled-H₂O (40 mL) and transferred into 250 mL round bottom flask. Then NaBH₄ solution was added dropwise to the above solution with constant stirring for 30 min. Finally, the formed precipitate was centrifuged at 4000 rpm and washed with anhydrous ethanol and distilled water, then dried at 80 °C. To fabrication of x wt. % AuSe QDs/Cs₂Fe₂O₄ NCs, an appropriate amount of AuSe QDs (previously prepared) were dispersed in solution (50 ml) containing 10 mmol of CsNO₃ and 20 mmol of Fe(NO₃)$_3$. 9H₂O and sonicated for 30 min. After that, 50 ml of 2.0 M NaOH were added to the previous solution under vigorous stirring, then the resultant hydrogel was moved into an autoclave and heated at 150 °C for 4 h. The formed precipitate was centrifuged at 4000 rpm and washed with distilled water, then dried in an oven at 80 °C. The x wt.% AuSe QDs/Cs₂Fe₂O₄ NCs with various weight percentage of AuSe QDs (1.0, 2.5, 5.0 and 7.0 wt. %) were prepared and marked as 1-ACFO, 2.5-ACFO, 5-ACFO, and 7-ACFO, respectively.

2.4. Characterization techniques

The crystal composition and chemical structure of pure AuSe QDs and AuSe QDs/Cs₂Fe₂O₄ NCs were confirmed by XRD patterns using PW150 (Philips) with Cu (Kα)-XR-radiation source (λ = 0.1541 nm) at 45 mA and 40 KV. The particle morphology and size of the AuSe QDs/Cs₂Fe₂O₄ NCs, were investigated through TEM-(Jeol-Jem-2100). Nitrogen adsorption/desorption isotherms were used to measure the specific surface area of these prepared composites which was determined at 77 K on a volumetric instrument (Micromeritics ASAP 2020, USA). The optical properties of the as-synthesized photocatalysts were measured using UV-Visible spectrometry.

2.5. Photocatalytic degradation process

The photocatalytic experiments carried out under a 150 W-mercury lamp with a filter (JB400) as a simulated visible-light source. The reaction mixture contained 0.1 g of the prepared photocatalysts in 40 ml RhB (10 ppm) and was placed in a quartz pipe in the middle of the reactor and irradiated by a Hg Lamp (150 W). The reaction performed in dark for 30 min to determine the adsorption rate on surface of AuSe QDs/Cs₂Fe₂O₄ NC photocatalysts, after that it irradiated for a definite time until the complete degradation of the RhB. Finally, the remained dye concentration measured using UV-Visible spectrometry.

3. Result and discussion

3.1. XRD analysis

The crystalline phases of AuSe QDs, Cs₂Fe₂O₄ and 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC were analyzed by XRD spectrum. Figure 1(a) shows that the diffraction peaks at 2θ equal 38° (111), 42° (200), 64° (220), and 78° (311) were observed for the AuSe QDs in accordance with Beta-structure (β-AuSe (JCPDS card 00-020-0458) [32,52] and very small two peaks of the α-AuSe as well appeared. This confirms the formation of crystalline β-AuSe QDs. No other peaks that ascribed to any impurity are detected. Also, XRD patterns for pure Cs₂Fe₂O₄ and 5.0 wt.% AuSe QDs/Cs₂Fe₂O₄ NCs were displayed in Figure 1(b and c). The patterns of Bragg reflection of the Cs₂Fe₂O₄ NC observed at 2θ angle equal 18.30, 28.42, 34.77, 39.49, 42.92, 50.77, 51.7, 56.68, 60.49, 66.98, 67.7, 73.82, 78.8, 84.52, 86.8, and 94.09 respectively.

To fabrication of x wt.% AuSe QDs/Cs₂Fe₂O₄ NCs, an appropriate amount of AuSe QDs (previously prepared) were dispersed in solution (50 ml) containing 10 mmol of CsNO₃ and 20 mmol of Fe(NO₃)$_3$. 9H₂O and sonicated for 30 min. After that, 50 ml of 2.0 M NaOH were added to the previous solution under vigorous stirring, then the resultant hydrogel was moved into an autoclave and heated at 150 °C for 4 h. The formed precipitate was centrifuged at 4000 rpm and washed with distilled water, then dried in an oven at 80 °C. The x wt.% AuSe QDs/Cs₂Fe₂O₄ NCs with various weight percentage of AuSe QDs (1.0, 2.5, 5.0 and 7.0 wt. %) were prepared and marked as 1-ACFO, 2.5-ACFO, 5-ACFO, and 7-ACFO, respectively.

![Figure 1. XRD patterns of (a) Pure AuSe QDs, (b) Pure Cs₂Fe₂O₄ NC and (c) 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC.](image-url)
The TEM images of the pure AuSe QDs and 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC were shown in Figure 2. The TEM images displayed that pure AuSe QDs appeared as dots on the surface of Cs₂Fe₂O₄ NC, which confirmed the existence of AuSe QDs on Cs₂Fe₂O₄ surface. The sizes of spherical Cs₂Fe₂O₄ nanoparticles were ranged between 10.32–35.08 nm, while the sizes of AuSe QDs were 1.18–8.69 nm. Also, the TEM images displayed that there is no clear accumulation of AuSe QDs on the surface of Cs₂Fe₂O₄ NC, as well as the AuSe QDs were well suspended in deionized water and were facile to disperse on the Cs₂Fe₂O₄ surface. The Cs₂Fe₂O₄ NC played a more important role in the dispersion process of the AuSe QDs on its surface due to their magnetic properties. The results confirm that the spherical Cs₂Fe₂O₄ structure did not change even after loading AuSe QDs on its surface as it displayed in XRD analysis. Based on the size distribution histograms of particles as shown in Figure 3, the average sizes of AuSe QDs were found to be 3.6 nm, while the average sizes of Cs₂Fe₂O₄ NC were equal 25.4 nm.

3.3. UV-visible spectroscopy

The energy band structure and optical absorption properties of the prepared AuSe QDs/Cs₂Fe₂O₄ NCs were investigated by UV/Vis diffuse reflectance spectra as shown in Figure 4. The figure revealed that there was a good absorption ability for visible light by both pure AuSe QDs and pure Cs₂Fe₂O₄ NC. Also, the absorbance curves of pure AuSe QDs and AuSe QDs/Cs₂Fe₂O₄ NCs have absorption curves reached to 743 nm. The prepared AuSe QDs/Cs₂Fe₂O₄ photocatalysts showed better optical absorption than pure AuSe QDs and pure Cs₂Fe₂O₄ NC, which in its turn improved the efficiency toward visible light. Figure 4 showed an obvious shifting with a wavelength from 462 nm to 565 nm in AuSe QDs/Cs₂Fe₂O₄ NC. These results revealed that the visible light absorption properties of the Cs₂Fe₂O₄ NC improved after loading of different AuSe QDs percentages. The enhanced absorption of x wt. % AuSe QDs/Cs₂Fe₂O₄ NCs can be attributed to the surface Plasmon resonance effect of AuSe QDs originating from the interaction of the magnetic and electric field of light on the surface of AuSe QDs/Cs₂Fe₂O₄ NCs. This SPR and synergistic effect of AuSe QDs and Cs₂Fe₂O₄ can be used to sensitize these photocatalysts to enhance the optical and photocatalytic properties, so it will enhance the visible light–photocatalytic performance of x wt.% AuSe QDs/Cs₂Fe₂O₄ NCs. The SPR band recommended that the prepared AuSe QDs supported on the Cs₂Fe₂O₄ surface were almost small dots shape, which were confirmed by XRD and TEM images. The addition of AuSe QDs on the Cs₂Fe₂O₄ led to increasing in the absorption intensity in the SPR band with shifted into a higher wavelength. From Figure 4, we can also observe that 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC reflected the strongest-SPR at a broad absorption peak centering at 545 nm, which was considered the largest absorption in the visible region (it’s broad from 400 nm to 740 nm). And one of the most important factors that affected on SPR band was particle size. Because of AuSe QDs have a smaller size, it is not hard to understand that the x % AuSe QDs/Cs₂Fe₂O₄ NCs displayed the largest visible absorption and the strongest SPR, compared with the pure Cs₂Fe₂O₄ NC.

Furthermore, the band gap energy of AuSe QDs/Cs₂Fe₂O₄ NC samples were calculated according to the Kubelka-Munc equation \( \alpha(h\nu) = A(h\nu - E_g)^{-\frac{1}{2}} \). Figure 5 displayed that were two regions demonstrating the two optical absorption edges \( E_{g1} \) and \( E_{g2} \) double energy gap, for AuSe QDs/Cs₂Fe₂O₄ NCs as described by many authors [54, 55, 56]. The presence of two energy gaps ascribed to the optical transitions from the valence sub-bands to the conduction band minimum, i.e., overlapping of Fermi levels of both AuSe QDs and Cs₂Fe₂O₄ nanoparticles. The band gap values showed decrease with increasing of AuSe QDs content on the Cs₂Fe₂O₄ surface as are seen in Fig. 5. The energy gap of pure AuSe QDs, pure
Cs$_2$Fe$_2$O$_4$, 1-ACFO, 2.5-ACFO and 5-ACFO was measured and showed in Table 1. It was observed that the values of energy band gap $E_{g2}$ in case of direct and indirect allowed transition decrease with increasing of AuSe QDs. However, values of energy band gap $E_{g1}$ in case of both transitions are close to each other and slightly changed with AuSe QDs contents. It was found that $E_{g1}$ of AuSe QDs/Cs$_2$Fe$_2$O$_4$ NCs ranged from 1.79 eV to 2.06 eV compared with $E_{g1}$ of the pure Cs$_2$Fe$_2$O$_4$ NC (3.2 eV) and pure AuSe QDs (2.13 eV). And the presence of a new band provided strong evidence that AuSe QDs have successfully embedded on the Cs$_2$Fe$_2$O$_4$ NC.

3.4. Textural measurement (BET)

Nitrogen adsorption desorption isotherms of the as-synthesized Cs$_2$Fe$_2$O$_4$ NC and x wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC samples were displayed in Figure 6, which exhibited type-IV isotherms according to IUPAC classification [57, 58, 59] and have inflection around $P/P_0$ (0.20–0.40) with narrow hysteresis loops. The specific surface area of the Cs$_2$Fe$_2$O$_4$ NC and x wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NCs was calculated from the isotherms. The x wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC samples exhibited a slightly smaller BET surface area than that of pure Cs$_2$Fe$_2$O$_4$ NC (18.70 m$^2$/g). For the sample of 1.0, 2.5, and 5.0 wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC, the BET surface area was equal to 15.43, 13.51 and 11.27 m$^2$/g, respectively. These results are in the same order of magnitude, inducting that loading of x wt.% AuSe QDs on the Cs$_2$Fe$_2$O$_4$ surface affected on the surface area. Meanwhile, there is a correlation between the AuSe QDs content and the surface area value, which was observed. It is clear from Figure 6, the adsorption capacity of x wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NCs was decreased with increasing of the AuSe QDs content, which could be related to the deposition of AuSe QDs on both the surface and pores of Cs$_2$Fe$_2$O$_4$ NC [60, 61, 62].

3.5. Photocatalytic activity

The photocatalytic performance of the prepared AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC catalysts was estimated by monitoring the photodegradation of organic RhB dyes under simulated visible light irradiation in an aqueous solution. The degradation of RhB versus reaction time displayed in Figure 7. The degradation ratios were gradually decreased with increasing of the reacting time, which indicated that the RhB has been significantly decomposed by x wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ photocatalysts. The photocatalytic activity of the pure Cs$_2$Fe$_2$O$_4$, 1.0, 2.5, 5.0 wt.% AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC samples for degradation of RhB dye was 50.0 %, 59.1 %, 76.4 %, and 99.15 % after 150 min under visible light, respectively. Enhancement of the degradation ratio indicated that the heterostructure AuSe QDs/Cs$_2$Fe$_2$O$_4$ NC efficiently increased, which induced the
separation of photogenerated holes and electrons after the addition of AuSe QDs on the Cs₂Fe₂O₄ surface.

The results indicated that the AuSe QDs content played a significant role in the enhancement of photocatalytic properties of AuSe QDs/Cs₂Fe₂O₄ photocatalysts, due to the AuSe QDs having the ability to trap electrons, so the real separation of electron-hole pairs occurred. This confirmed that there was overlapping between Fermi levels of both the AuSe QDs and Cs₂Fe₂O₄ NC which in turn led to decrease in the band gap with increasing the AuSe QDs content. On the other hand, the decrease in the pore diameter with increasing the AuSe QDs content led to promote photocatalytic activity due to the AuSe QDs diffuse through the entire pores and surface of AuSe QDs/Cs₂Fe₂O₄ photocatalyst, so, there will be more available active sites on the AuSe QDs/Cs₂Fe₂O₄ surface. This meant that the higher photocatalytic activity may be attributed to increasing the available active sites on the AuSe QDs/Cs₂Fe₂O₄ surface. The photocatalytic degradation of RhB dye by 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC sample was the highest. The existence of AuSe QDs on the Cs₂Fe₂O₄ NC made the excited electron easily transferred into the surface of AuSe QDs/Cs₂Fe₂O₄ catalyst. These surface electrons can be reacted with the oxygen molecules to produce O₂⁻/OH⁻ radicals, while the positive charges (the holes) can be reacted with the water molecules to produce the OH⁻ radicals. These radicals led to the degradation of RhB dye easily. The stability of the as-synthesized 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC was testified for four cycles under the same condition and using 0.1 g of the photocatalyst. The results revealed that the photodegradation of RhB dye reached to 98.25 % after four cycles as shown in Figure 8. This decrease could be due to the decreasing surface interaction between the reaction intermediates in the photodegradation process of RhB dye. This results obviously revealed the excellent stability of 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC as an excellent heterogenous photocatalyst as well. We can conclude by stating that the fabrication and high photocatalytic performance of x wt.% AuSe QDs/Cs₂Fe₂O₄ NC photocatalysts have been realized in this work.

4. Conclusion

This study described a green and efficient pathway for gold selenide quantum dots and cesium ferrite nanocomposite. In summary, novel AuSe QDs/Cs₂Fe₂O₄ NCs with the visible light response were successfully prepared. The heterogeneous x wt. % AuSe QDs/Cs₂Fe₂O₄ NCs were applied for photocatalytic degradation of RhB dye in the aqueous solution. The results displayed that the photocatalytic activity of AuSe QDs/Cs₂Fe₂O₄ NCs was improved after loading different mass ratios of AuSe QDs content (1.0, 3.0 and 5.0 wt.%). Among the synthesized photocatalysts, the 5.0 wt. % AuSe QDs/Cs₂Fe₂O₄ NC sample showed high photoactivity for the RhB degradation may be due to surface plasmon resonance (SPR), synergistic effect and its smallest energy gap. The coexistence of AuSe QDs and Cs₂Fe₂O₄ NC in the prepared photocatalysts confirmed by TEM images and XRD patterns. The photocatalytic activity of the pure Cs₂Fe₂O₄, 1.0, 2.5, 5.0 wt.% AuSe QDs/Cs₂Fe₂O₄ NC photocatalysts for degradation of RhB dye, was 50.0 %, 59.1 %, 76.4 %, and 99.15 % after 150 min under visible light, respectively, which confirms that the separation of photogenerated holes and electrons have been significantly enhanced, in turn to degradation of RhB dye easily. The results showed that AuSe QDs/Cs₂Fe₂O₄ NCs were stable and efficient photocatalysts, that act as promising photocatalysts for water environmental remediation.

Declarations

Author contribution statement

Fares T. Alshori Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.
Abdullah A. Alsawt: Contributed reagents, materials, analysis tools or data; Wrote the paper.
Reda S. Salama: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Data will be made available on request.

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The authors declare no conflict of interest.

Additional information
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