



Cetyltrimethylammonium Bromide@CoFe₂O₄ Nanocomposite for Photocatalytic

Reduction of Cr(VI) Employing a Simple Colorimetry Assay

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Abstract

The present study involved the synthesis of a nanocomposite material composed of cobalt ferrite (CoFe₂O₄) modified with cetyltrimethylammonium bromide (CTAB). The preparation method employed a chemical reduction approach. The structural characterization of the CTAB@CoFe₂O₄ nanocomposite was carried out using techniques such as transmission electron microscopy (TEM), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). The XRD analysis confirmed the single-phase cubic crystal structure of the CTAB@CoFe₂O₄ nanocomposite. Additionally, the FTIR spectra provided evidence of the presence of CTAB functional groups within the CoFe₂O₄ matrix. The zeta potential measurements revealed a positive surface charge of +17.3 mV for the CTAB@CoFe₂O₄ nanocomposite. The application of this nanocomposite material was studied for the reduction of chromium (VI) ions (Cr(VI)) from aqueous solutions using a colorimetric method based on 1,5-diphenylcarbazide dye (DPC). The effects of various parameters, including pH, nanocomposite dosage, and contact time, were investigated to assess their influence on the photoreduction efficiency of the CoFe₂O₄/CTAB nanocomposite. Remarkably, at a nanocomposite dosage of 3 mg/mL, 99% reduction of Cr(VI) was achieved at pH3 with a contact time of 150 minutes. These findings indicate the potential of the synthesized CoFe₂O₄/CTAB nanocomposite as an efficient material for the elimination of Cr(VI) ions from contaminated water sources. One notable distinction of this method is that it goes beyond solely removing Cr(VI) from water, it also converts it into a non-toxic form.

Keywords: Photocatalytic reduction; CTAB@CoFe₂O₄; Cr(VI); Diphenyl carbazide; Contaminated water

1. Introduction

Clean drinking water is a significant challenge, particularly in developing countries where industrial growth has resulted in the generation of increasing amounts of wastewater without adequate management and purification systems (Zedan et al., 2022). These wastewater streams often contain a variety of contaminants that pose environmental hazards and potential harm to both human and animal health (Elbadawy et al., 2023). Among the numerous wastewater contaminants, Cr(VI), is of particular concern due to its mutagenic and carcinogenic properties (Ngo et al., 2018). Cr(VI) is known to be extremely toxic, even in small doses, highly oxidizing in nature, and long-term exposure causes reproductive toxicity (Costa, 1997). Cr(VI) can enter the environment through various industrial processes, including painting, furnace lining, tanning, dyeing, photography, and steel manufacturing (Ngo et al., 2018). When consumed in high doses, Cr(VI) can cause severe acute reactions such as gastrointestinal disorders, hemorrhagic diathesis, and convulsions, which may ultimately lead to cardiovascular shock and even death (Mushtaq et al., 2022). To address this environmental concern, scientists have proposed numerous wastewater treatment methods with different chemical and physicochemical properties. Some of the traditional methods include ion-exchange, chemical precipitation, coagulation, membrane processes, reduction, and adsorption (Ahmed et al., 2022). These traditional wastewater treatment methods have certain limitations and may not provide complete removal of Cr(VI), necessitating the development of new and improved approaches. One such promising method is heterogeneous photocatalytic reduction processes, which offer advantages such as cost-effectiveness, high catalytic performance, and the absence of secondary byproduct pollutants (Zare et al., 2021). In recent studies, CoFe₂O₄ nanoparticles (NPs) have emerged as a notable heterogeneous catalyst for wastewater treatment (Al-Sherbini, 2019; Uzunoğlu et al., 2019) owing to their unique magnetic properties and chemical stability. The utilization of CoFe₂O₄ NPs in heterogeneous photocatalytic reduction processes holds promise for more efficient and effective removal of Cr(VI) from wastewater, thus addressing the environmental and health concerns associated with this compound. Additionally, the CoFe₂O₄ NPs catalyst can be simply extracted from the treated

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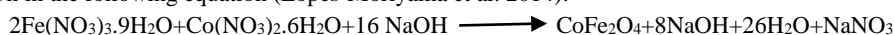
wastewater sample by centrifugation for recycling purposes. However, CoFe₂O₄ NPs are n-type semiconductor, with small optical band gaps (2.0 eV) making them active under visible light treatment (Sonu et al., 2019). For applying CoFe₂O₄ NPs for the photoreduction of Cr(VI) anions, their surface should be positive. To enhance the photocatalytic efficiency, the surface of CoFe₂O₄ NPs was modified using a cationic surfactant called CTAB (Elfeky et al., 2017a). CTAB modification is ideal for achieving a positive surface charge on CoFe₂O₄ NPs, which is necessary for the efficient reduction of Cr(VI) anions. To monitor the concentration of Cr(VI) during the photocatalytic reduction process, a simple and sensitive colorimetric method based on DPC (diphenyl carbazide) was employed (Gardner & Comber, 2002). This method allows for the accurate and specific detection of Cr(VI) in a mineral acid solution. Experiments were performed under different conditions to evaluate the photocatalytic reduction efficiency of Cr(VI) using the CTAB modified CoFe₂O₄ NPs as the catalyst. The advantages of using CTAB@CoFe₂O₄ NC for the photocatalytic reduction of Cr(VI) are evident in its high efficiency, stability, reusability, versatility, and eco-friendly nature by harnessing the synergistic effects of organic-inorganic hybrid materials (Sonu et al. 2019). Thus, this approach represents a promising strategy for addressing water pollution challenges associated with toxic heavy metals like Cr(VI), paving the way towards a cleaner and healthier environment.

2. Experimental

All chemicals and reagents were obtained from Sigma-Aldrich (USA) and used without further purification. Cobalt nitrate hexahydrate (Co(NO₃)₂·6H₂O) and iron(III) nitrate monohydrate (Fe(NO₃)₃·9H₂O) were used as metal ion sources. CTAB (Cetyltrimethylammonium bromide, C₁₉H₄₂BrN) was used as a cationic surfactant. Sodium hydroxide (NaOH) was used as a basic co-precipitating agent. Double-distilled deionized water was used throughout the preparations and experiments. Potassium dichromate (K₂Cr₂O₇), nitric acid, sulfuric acid, and 1,5-diphenylcarbazide were purchased from Fluka Co., Switzerland Pure Chemical Corp., and were of analytical reagent grade. A standard stock solution of Cr(VI) 1g/L was prepared for different dilutions of the photoreduction experiments.

2.1. Synthesis of CTAB/CoFe₂O₄ Nanoparticles

In this study, CoFe₂O₄ nanoparticles (NPs) were synthesized according to the method described in (Das et al., 2020), (Haneef et al., 2021) applying a series of steps. Firstly, a 0.1 M solution of Co(NO₃)₂·6H₂O was prepared in 50 ml of water. The solution was then stirred for 30 minutes at room temperature (RT). Simultaneously, a 0.2 M solution of Fe(NO₃)₃·9H₂O was prepared in another 50 ml of water, which was also stirred for 30 minutes at RT. The solutions of Co(NO₃)₂·6H₂O and Fe(NO₃)₃·9H₂O were then mixed together and further stirred for an additional 30 minutes at RT. Next, a 50 ml solution of 0.3 M CTAB (cetyltrimethylammonium bromide) in deionized (DI) water was added to the previous mixture then heated to 80°C and stirred vigorously for 1 hour. Following this, NaOH was abruptly added to the solution (Hosny, Hazem, and Moalla 2022), raising the temperature to 90°C and stirring for another 3 hours. This resulted in the formation of a black precipitate. The precipitate was then washed several times with DI water until a neutral pH was achieved. Finally, the precipitate was dried at 120°C for 12 hours. To remove any remaining moisture content, the dried precipitate was annealed at 200°C for 6 hours. Overall, this synthesis method involved the mixing and stirring of Co(NO₃)₂·6H₂O and Fe(NO₃)₃·9H₂O solutions, addition of CTAB, precipitation with NaOH, washing, and drying, followed by annealing to obtain CTAB/CoFe₂O₄ NC. The general mechanism of CoFe₂O₄ formation, including thermal decomposition of the precursors, Co(NO₃)₂·6H₂O and Fe(NO₃)₃·9H₂O, is shown in the following equation (Lopes-Moriyama et al. 2014).



The CTAB@CoFe₂O₄ formation involved the adsorption of CTAB onto the nanoparticle surfaces, forming a protective layer around the nanoparticles, stabilizing them and preventing agglomeration. The CTAB-capping to CoFe₂O₄ nanoparticles is formed through a combination of electrostatic interactions, hydrogen bonding, and hydrophobic interactions between the CTAB molecules and nanoparticle surfaces (Vadivel et al. 2016).

2.2. Photoreduction Experiment

A photoreduction experiment was conducted by illuminating 100 mL of the Cr(IV) aqueous solution (10⁻³ M/L) with a halogen lamp (50 W/m²), for a duration of 150 minutes. Three different catalyst amounts (1, 3, and 5 mg/mL) were used to investigate the catalytic photoreduction efficiency. Control experiments were done without light irradiation in the presence of the photocatalyst CTAB/CoFe₂O₄ NC or in the presence of light irradiation without the photocatalyst. All experiments were carried out under magnetic stirring (300 RPM).

2.3. Cr(VI) Monitoring

The concentration of Cr(VI) at 10⁻³ M/L was monitored using a colorimetric method with DPC reagent as described by (Lace et al. 2019) with some modifications. During the photoreduction experiment, 100 µL of Cr(VI) was transferred to a glass vial containing 1 mL of 0.2 M sulfuric acid and 1 mL of DPC (0.5% w/v). The mixture was shaken and left for 5 minutes before measuring the absorbance at 543 nm against a reagent blank every 15 min.

2.4. Nanocomposites Dose Effect

The photoreduction of Cr(VI) ions was carried out using CTAB/CoFe₂O₄ NC catalyst at three different concentrations (1, 2, and 3 mg/mL) to determine the optimal dosage. The experiments were conducted under both dark and artificial light conditions.

2.5. Effect of pH

The effect of pH on the photocatalytic reduction activity of the CTAB/CoFe₂O₄ was investigated at three different pH values (3, 5, and 7). HCl and NaOH (0.1 N) were used to adjust the pH of the Cr(VI) samples. During the photoreduction experiment, 100 mL of 10⁻³ M/L Cr(VI) were mixed with three different amount of catalyst powder (1, 3, and 5 mg/mL). The photoreaction was carried out over the course of 150 minutes with stirring.

2.6. Recycling of Cobalt Ferrite

After the photocatalytic reduction of Cr(VI) was achieved, the photocatalyst CTAB/CoFe₂O₄ NPs, were recovered from samples by centrifugation, washed with DI water, dried then reused for further photoreduction experiments to investigate the economic impact of the photocatalytic approach (Bayuo, Abukari, and Pelig-Ba 2020).

2.7. Instrumentation

The Zeta potential measurement was done by Malvern Nano ZS. UV-visible absorption spectra were recorded using a double-beam spectrophotometer (JENWAY 6800, Germany). The absorbance was measured within the scan range (200-900 nm). The pH-meter used for this study (HANNA instruments, Germany) was calibrated at pH 4 and 7 with appropriate buffer solutions from Merck, USA. FT-IR spectroscopy analysis was performed using a Bruker spectrometer (Vertex 80, 4000-400 cm⁻¹, USA). X-ray diffraction measurements were carried out using an X'Pert-Pro powder diffractometer from PANalytical, Netherlands. Finally, the shape and size of the prepared nanocomposite were determined using transmission electron microscopy (TEM) with a JEOL JEM-1400 (acceleration voltage of 40 to 120kV from Germany. SEM and EDX analysis were performed using Quattro S (Thermo Scientific USA), Field Emission Environmental Scanning Electron Microscope (FE ESEM).

3. Results and Discussion

3.1. Characterization of CTAB/CoFe₂O₄ NC

The FTIR spectra of CTAB and CTAB/CoFe₂O₄ NC are presented in Fig. (1). CTAB exhibits two intense peaks at 2800–3000 cm⁻¹, which correspond to the symmetric (2848 cm⁻¹) and asymmetric (2918 cm⁻¹) stretching vibrations of methyl and methylene groups (Elfeky et al., 2017). The weak band at 3445 cm⁻¹ arises from the stretching vibration of hydroxyl groups. The bands in the region of 1480 cm⁻¹ and 1672 cm⁻¹ are attributed to the stretching vibration of N⁺CH₃. The bands at 720 and 960 cm⁻¹ could be related to -CH₂- rocking and C-N⁺ stretching vibration respectively (Li et al. 2016). In addition, C-N bands appear in the range 1000-1350 cm⁻¹ (INFRARED SPECTROSCOPY n.d.). In the FTIR spectrum of CTAB/CoFe₂O₄ NC, the observed peaks in the range of 655-580 cm⁻¹ correspond to the tetrahedral metal ion-oxygen complexes stretching), while the octahedral metal ion-oxygen complexes appear at 420-460 cm⁻¹ (Raut et al., 2014). The broadband at 3440 cm⁻¹ is likely due to the electrostatic interaction between CoFe₂O₄ surface hydroxyl groups and the ammonium moiety in CTAB. The presence of CTAB peaks at 2800–3000 cm⁻¹ region along with the shift and broadening of CTAB bands at 1480 cm⁻¹, 1672 cm⁻¹ and 960 cm⁻¹ (due to hydrogen bonding between CTAB and CoFe₂O₄) in CTAB/CoFe₂O₄ NC FTIR spectrum confirms the formation of CTAB/CoFe₂O₄ NC.

The zeta potential shown in Fig. (2) revealed that CTAB/CoFe₂O₄ NC has a positive surface charge of +17.3 mV indicating its good stability (Bhattacharjee, 2016). In contrast naked CoFe₂O₄ NPs have a negative zeta potential value (-13.46 mV) (Betal et al., 2015). Thus, capping CoFe₂O₄ NPs with cationic surfactant CTAB converted the surface charge to positive. The positive surface charge on CoFe₂O₄ nanoparticles is crucial for the efficient reduction of Cr(VI) anions due to several reasons, including, electrostatic attraction, enhanced reactivity, and efficient electron transfer during the reduction of Cr(VI) to Cr(III), leading to improved efficiency of the photo-reduction process and making it an ideal choice for wastewater treatment applications. Based on the XRD diffraction pattern (Fig. 3), it was confirmed that the annealed CTAB/CoFe₂O₄ NC has a single-phase cubic crystal structure belonging to the Fd-3m space group. The observed planes (220), (311), (400), (422), (511), and (440) at 2 θ values of 30.2, 35.5, 43.1, 53.5, 57.1, and 62.8° respectively, matched well with the standard JCPDS data reference (card no: 22-1086) (Vadivel et al. 2016). The average crystallite size of cobalt ferrite was calculated to be 19.3 nm applying Scherrer's approximation (Wu et al. 2019).

The SEM images (Fig. 4 a & b) and TEM images in Fig. (4c) reveal that the as-prepared CTAB/CoFe₂O₄ NC consists of aggregated sphere-like nanoparticles due to the surfactant effect (Thomas and Alexander 2018). The diameters of the nanoparticles range between 15 and 20±5 nm. Additionally, the nanoparticles exhibit a good cubic crystalline structure as displayed in Fig. (4-c) which is consistent with the XRD results mentioned earlier.

The existence of Co, Fe, and CTAB elements in CTAB@CoFe₂O₄ NC was confirmed by EDX elemental analysis (Fig. 5). Fig. (5-a) demonstrates that the sample components (Co, Fe, O, C, and N) in area (1) of Fig (5-b) are completely matches the anticipated CTAB@CoFe₂O₄ NC structure (Heydari et al., 2019; Jiang et al., 2022). The spectrum also illustrates that the sum of Co/Fe ratio is around 0.5, which is consistent with the formula CoFe₂O₄ (Heydari et al., 2019).

The optical absorption spectrum of CTAB@CoFe₂O₄ NC demonstrates a broad splitted absorption peak in the λ = 600–700 nm range (Fig. 6-a). The splitted band between 600-700 nm is characteristic to the tetrahedral Co(II) stereochemistry, indicating the presence of numerous defects in the material, which may contribute to its photocatalytic enhancement (Wang et al. 2021). In this arrangement, each metal ion is surrounded by four ligands in a tetrahedral configuration, forming a close-packed structure with a total of eight metal ions in the unit cell. The cobalt ion (Co²⁺) and two iron ions (Fe³⁺) are located at

the center of a tetrahedron, the four oxygen ions (O^{2-}) act as ligands surrounding each metal ion, forming the vertices of the tetrahedron. The bond angles between the metal ion and the ligands are approximately 109.5 degrees, characteristic of a tetrahedral arrangement (Steven S. Zumdahl 2018).

Using the Tauc plot, generated from the UV-vis DRS spectrum, the band gap energy of the CTAB@CoFe₂O₄ NC was evaluated (**Fig. 6-b, c**). The band gap energy can be found at the x-intercept of the extrapolated linear fits (Tatarchuk et al., 2017). The illustrated bandgap has two values: 2.7 eV (**Fig. 6-b**) and 1.7 eV (**Fig. 6-c**), which most likely correlate to the direct and indirect band gaps, respectively (Duru, 2022). It was reported that a semiconductor having a band gap of 2.7 eV has the ability to absorb solar spectrum blue-violet light with a wavelength of less than 475 nm, making it a promising option for photocatalysis (Liu et al., 2020). However, indirect band gap semiconductors are preferable for longer charge carrier lifetimes (Hoang & Gao, 2016). Thus, the presence of both direct and indirect bandgaps can contribute to the overall absorption properties of the semiconductor across a broader range of the solar spectrum.

3.2. Photocatalytic Reduction of Cr(VI)

Various conditions control the photoreduction of Cr (VI), especially the amount of catalyst, the pH of the solution, and the contact time. Furthermore, CTAB modification changes the surface charge of CoFe₂O₄ NPs to positive, enabling efficient Cr(VI) reduction through electrostatic interactions.

In all experiments, there was always a dark control (DC) experiment using the catalyst and a light control (LC) in which light applied without any catalyst.

3.2.1. Effect of Catalyst Concentration

The influence of the catalyst amount is a very essential parameter. Results in **Fig. (7)** indicated that as the catalyst concentration increases, the reduction efficiency also increases. This is because a higher concentration of catalyst provides more active binding sites for the reactants, leading to faster and more efficient reactions. The reduction efficiency as a function of catalyst concentration was carried out applying 3 different catalyst amounts (1, 3, and 5mg/mL) as a function of time (0-150 min). The photoreduction efficiency is calculated according to Eq (1) (Elbadawy, El-Dissouky et al. 2023).

$$\begin{aligned} \text{Photoreduction \%} & \quad (1) \\ & = \frac{C_0 - C_t}{C_0} \times 100 \end{aligned}$$

where C_0 and C_t are the initial and final concentration (after time t) of Cr (VI) in the solution. The effect of CTAB@CoFe₂O₄ NC dose on the photoreduction of Cr(VI) is shown in **Fig. (7)**.

It was noticed that the photoreduction rate increases with increasing the catalyst concentration and the contact time. The Cr(VI) reduction increased from 45% to 78 % when increasing the CTAB@CoFe₂O₄ NC from 1 to 3 mg/mL. Similar effect was reported in (Thomas & Alexander, 2018) when they found that the photodegradation rate of Cr(VI) enhanced with the increase in CoFe₂O₄ catalyst amount due to more surface and photocatalytic active sites. However, 3mg/mL catalyst dose can be considered as an optimal concentration beyond which further increases to 5 mg/mL may not provide significant improvements (85%). The photocatalysis operational cost is also directly affected by the catalyst concentration, making it important to strike a balance between efficiency and cost.

3.2.2. Effect of pH

Solution pH plays a crucial role in the photoreduction of metal ions, such as Cr(VI). It affects the surface charge of the adsorbent and the presence of functional groups, which in turn influence the adsorption and reduction processes. The results in **Fig. (8)** indicate that decreasing the pH significantly increases the photoreduction rate. This could be attributed to the interaction between the negatively charged Cr(VI) ions and the protonated (highly positive) surface of the catalyst at lower pH levels. The stronger attraction between the Cr(VI) ions and the catalyst surface leads to enhanced adsorption, promoting photocatalytic reduction efficiency. At pH 3 with 3 and 5 mg/mL catalyst amount, the photoreduction efficiency reaches 99% after 90 minutes approaching the complete reduction of Cr(VI). This result was confirmed by ionic chromatography analysis of the Cr(VI) concentrations before and after photocatalytic degradation by CTAB/CoFe₂O₄ NC photocatalyst (**Fig. S1 (a) and (b)** respectively, supplementary file). The figure illustrates the detection of the Cr(VI) in water samples after 5.03 minutes, which is consistent with the literature (Kaur & Malik, 2009). It also confirms that the remaining concentration Cr(VI) after photocatalytic degradation applying CTAB/CoFe₂O₄ NC and DPC for 150 minutes was found to be 1%.

Indeed, increasing the pH of the solution can lead to a reduction in the protonation of the catalyst surface and subsequently decrease the electrostatic attraction between Cr(VI) ions and the CTAB@CoFe₂O₄ NC catalyst. This in turn can result in a decrease in the photocatalytic reduction efficiency of Cr(VI). Thus the photocatalytic reduction of Cr(VI) decreased from 99% at pH 3 to 85% at pH 5 and 70% at pH 7. These results agree with the findings of (Bortot Coelho et al., 2020) for the photocatalytic reduction of Cr(VI) using Ce-ZrO₂ under visible light. These findings highlight the importance of pH control in optimizing the photocatalytic reduction process of Cr(VI).

3.2.3. Catalyst Recycling

The reuse of CTAB@CoFe₂O₄ NC was investigated in this study for photocatalytic reduction of Cr(VI) for four successive cycles. The photoreduction rate percentage was calculated according to Eq (2) (Emara et al., 2021).

$$R_r = \frac{C_r}{C_0} \times 100 \quad (2)$$

Where R represents the photoreduction ratio %, r is the cycle number, C₀ is the photoreduction efficiency in the first cycle, and C_r is the photoreduction efficiency at the second, third or fourth cycle. **Fig. (9)** displays the photoreduction ratio percentage of Cr(VI) using CTAB/CoFe₂O₄ NC at pH3 for four successive cycles. The figure illustrates that the photoreduction ratio percentage of CTAB/CoFe₂O₄ NC does not change in the four cycles. This could be due to maintaining the reactivity of CoFe₂O₄ NPs by protection capping with CTAB. Another study also noticed that there is no change in the photocatalytic activity of the CoFe₂O₄ NPs when combined with polyaniline for the removal of anionic dyes over 3 successive cycles (Xiong et al., 2012).

Table (1) shows a collective summary of all results at different pH levels and different CTAB/CoFe₂O₄ NC photocatalyst dose after 150 minutes.

Factor Concentration	1 mg/ml Removal %	3 mg/ml Removal %	5 mg/ml Removal %
Dark control	5	5	5
Light control	10	10	10
pH 7	42	63	69
pH 5	45	78	84
pH 3	38	99	99

4. Conclusion:

The CTAB/CoFe₂O₄ NC photocatalyst presents a promising solution for addressing the challenge of Cr(VI) contamination in water. It was synthesized by coprecipitation in an alkaline medium. The FTIR analysis revealed that there is an electrostatic attraction as well as hydrogen bonding between the CoFe₂O₄ surface hydroxyl groups and the ammonium moiety in CTAB. XRD and TEM analyses confirmed the cubic crystallite shape and average size (20 nm) of the CTAB/CoFe₂O₄ NC. The photocatalytic reduction experiment showed that 3 mg/mL of CTAB/CoFe₂O₄ NC can completely reduce Cr(VI) from the sample under acidic pH3 conditions after 150 minutes reduction time. Additionally, CTAB/CoFe₂O₄ NC was successfully recycled without affecting the effectiveness of Cr(VI) reduction throughout the tested cycles. This method differs from others as it not only removes Cr(VI), but also changes its chemical composition. The significance of this method lies in the transformation of Cr(VI) into a non-toxic form, avoiding the health risks associated with Cr(VI) water contamination, making it highly beneficial.

Conflict of interest

The authors have no conflict of interest to declare.

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