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Application of Microgel-Based Copper Oxide Nanocomposites in Catalytic Reduction of Congo Red

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Abstract: ***Congo Red (CR) is a synthetic azo dye widely used in the textile industry because of its strong coloring properties. Despite its industrial importance, it offers serious environmental challenges due to poor biodegradability and potential toxicity. The dye can persist in aquatic ecosystems, posing risks to living organisms. This persistence and toxicity highlight the need for safer alternatives and better management practices. In the present research work, the catalytic performance of a previously reported microgel-based copper oxide (M@CuO) nanocomposite was evaluated for the reduction of Congo Red dye in aqueous medium using sodium borohydride ($NaBH_4$) as a reducing agent. The nanocomposite, consisting of CuO nanoparticles uniformly embedded within a quater polymeric microgel matrix (based on N,N-dimethylacrylamide, styrene, N-hydroxymethylacrylamide, and methacrylic acid as monomers), provides a high surface area and improved dispersion of active catalytic sites. Further, the kinetics of the reduction reactions have been studied using ultraviolet–visible spectroscopy. M@CuO nanocomposite with $NaBH_4$ showed better catalytic efficiency, reaching 92.11% reduction of CR dye with the corresponding apparent rate constant of 11.9×10^{-2} min^{-1}.***

Keywords: Microgel, Nanocomposite, Cango red, Catalytic reduction.

I. INTRODUCTION

The continuous expansion of textile, paper, leather, and dye-processing industries has resulted in the large-scale release of synthetic dyes into natural water bodies. Among various industrial colorants, Congo Red (CR) is a widely utilized anionic azo dye due to its high tinting strength, structural stability, and strong affinity toward cellulose-based fibers [1]. However, the presence of azo (–N=N–) linkages and multiple aromatic rings in its molecular framework makes it chemically stable and resistant to biodegradation under natural environmental conditions. Because of its persistence, CR can remain in aquatic systems for extended periods, reducing light penetration and thereby disturbing photosynthetic activity in aquatic flora [2]. Moreover, several studies have reported its toxic, mutagenic, and potentially carcinogenic effects on living organisms, raising serious environmental and public health concerns [3]. The discharge of dye-laden effluents without adequate treatment may therefore contribute to ecological imbalance and long-term contamination of freshwater resources. These concerns highlight the urgent need for the development of sustainable and highly efficient wastewater treatment technologies [4].

A wide range of physicochemical and biological approaches has been explored for dye removal, including adsorption, membrane separation, coagulation–flocculation, biological degradation, photocatalysis, and advanced oxidation processes [5]. Although adsorption techniques are simple and effective, they often generate secondary waste and require additional regeneration steps. Membrane technologies provide excellent separation efficiency but suffer from fouling issues and relatively high operational costs [6]. Advanced oxidation processes can achieve near-complete mineralization of organic pollutants; however, they typically demand high energy input or expensive reagents. In recent years, catalytic reduction using nanostructured materials has emerged as an attractive alternative because of its operational simplicity, rapid reaction rates, and high degradation efficiency under mild experimental conditions [7, 8]. Among various nanocatalysts, copper oxide (CuO) nanoparticles have received considerable attention due to their low cost, natural abundance, semiconductor properties, and appreciable catalytic performance. Compared to noble metal nanoparticles such as gold or platinum, CuO offers a more economically viable and environmentally benign option for wastewater remediation [9]. Nevertheless, pristine CuO nanoparticles tend to aggregate in aqueous environments owing to their high surface energy, which reduces their effective surface area and limits catalytic activity.

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To address this challenge, polymer-based microgels have been investigated as stabilizing and supporting matrices for metal and metal oxide nanoparticles [10]. Microgels are three-dimensional, crosslinked polymer networks that can absorb significant amounts of water and exhibit reversible swelling behavior. Their porous architecture provides a confined nanoscale environment that prevents nanoparticle agglomeration, enhances dispersion, and improves accessibility of active catalytic sites [11]. In previous study, Microgel-stabilised CuO nanocomposite was synthesized through an in situ strategy and thoroughly characterized using spectroscopic and microscopic techniques. The results confirmed uniform incorporation of CuO nanoparticles within the polymeric matrix, indicating improved structural stability and potential catalytic applicability. The nanocomposite was successfully applied for the catalytic reduction of Methylene Blue dye and p-nitrophenol, demonstrating its effectiveness in degrading both organic pollutants; however, its broader efficiency toward other dyes had not yet been systematically examined [12].

In the present work, we investigated the catalytic reduction of Congo Red using the previously developed microgel-based CuO nanocomposite in the presence of sodium borohydride ($NaBH_4$). Sodium borohydride acts as an electron donor, while the CuO nanocomposite facilitates electron transfer from BH_4^- ions to the dye molecules adsorbed on its surface [13]. This electron mediation accelerates the cleavage of azo bonds, resulting in rapid decolorization and breakdown of the dye structure [14]. The progress of the reaction was monitored using UV–Visible spectroscopy by recording the decrease in the characteristic absorption peak of Congo Red. Kinetic parameters were calculated to evaluate the catalytic efficiency and reaction rate constant. The findings of this study aim to demonstrate that microgel-stabilized CuO nanocomposites can serve as efficient, stable, and potentially reusable catalysts for the treatment of dye-contaminated wastewater.

II. EXPERIMENTAL SECTION

A. *Synthesis of microgel-based copper oxide (M@CuO) Nanocomposite*

The M@CuO nanocomposite was synthesized from a microgel and CuO nanoparticles through a hydrothermal method, as reported in the literature [12]. For the typical synthesis of the nanocomposite, first of all, the microgel was prepared via free-radical emulsion polymerization using N,N-dimethylacrylamide (DMA), styrene (St), N-hydroxymethylacrylamide (NHMA), and methacrylic acid (MAA) as monomers. The required amounts of DMA, St, NHMA, and MAA were dispersed in an aqueous medium containing a suitable surfactant. The reaction mixture was purged with nitrogen gas to remove dissolved oxygen and prevent unwanted side reactions. Afterward, a free-radical initiator was added to initiate polymerization. The reaction was carried out under continuous stirring at an elevated temperature for several hours until stable crosslinked polymeric microgel particles were formed. The obtained microgel was then washed and dried for further use. The CuO nanoparticles were synthesized separately by a precipitation method. Finally, the prepared microgel and CuO nanoparticles were dispersed in distilled water and transferred into an autoclave for hydrothermal treatment at an elevated temperature for a specific time. During this process, CuO nanoparticles were uniformly incorporated into the microgel network, leading to the formation of the M@CuO nanocomposite.

B. *Application*

The M@CuO nanocomposites were tested for their catalytic performance in the reduction of CR dye (purchased from SD Fine Chemicals Pvt. Ltd., India). The study of catalytic reduction was performed by using $NaBH_4$ (procured from Sisco Research Laboratories Pvt. Ltd., India) as a reducing agent. Further, the kinetics of the reductions were studied in the presence of the various grades of nanocomposites, while using suitable concentrations of the reducing agent at room temperature.

1) *Catalytic reduction of CR dye in the presence of various amounts of M@CuO nanocomposites*

The catalytic reduction of the organic pollutant CR dye was done in a cuvette. For this, the cuvette was charged with 2.7 mL of the stock solution (0.1 mg/mL) of the pollutant. To this solution, a specific amount of the M@CuO nanocomposite was added. After that, 0.2 mL of a solution of $NaBH_4$ (3 mg/mL) was added. While maintaining the amounts of all other substances intact, 8, 10, and 12 mg of the nanocomposite, separately, were added in three different tests. The characteristic absorption band (500 nm) of the ultraviolet-visible (Shimadzu UV- 2600) was used to record the absorption spectra of CR, solutions during their reduction of the organic pollutant, which was used to estimate the percentage of reduction using Equation 1, and the corresponding results are shown in Table 1. The corresponding UV-vis spectra were recorded and are shown in Figure 1.

$$\text{Dye removal } \% = [(A_0-A_t)/A_0]\times 100 \qquad \text{Equation 1}$$

Further, the pseudo first order kinetic study of the reactions was performed, and the corresponding apparent rate constant (k_{app}) values were determined by using Equation 2 [15].

$$\ln(A_t/A_0) = -k_{app}.t \qquad \text{Equation 2}$$

Where A_o and A_t represent the initial absorbance intensity and intensity at time t, respectively, and k_{app} is the apparent rate constant.

2) Catalytic reduction of CR dye by varying the amount of $NaBH_4$ in the presence of the M@CuO nanocomposite.

The effective change in amounts of $NaBH_4$ concentration, on the catalytic reduction of the organic pollutants, was studied while keeping the amount of M@CuO nanocomposite constant. For this, various volumes (0.2, 0.3, and 0.4 mL separately) of 3 mg/mL $NaBH_4$ solution were added to a cuvette containing 2.7 mL (0.1 mg/mL) of the organic pollutant stock solution. Then, 8 mg of the nanocomposite was added to it. The percentage of reduction of the organic pollutant was determined by using Equation 1. and the corresponding results are reported in Table 1. The corresponding UV-vis spectra were recorded and are shown in Figure 1.

Further, the pseudo-first-order kinetic study of the reactions was performed, and the k_{app} values were determined by using Equation 2 [16].

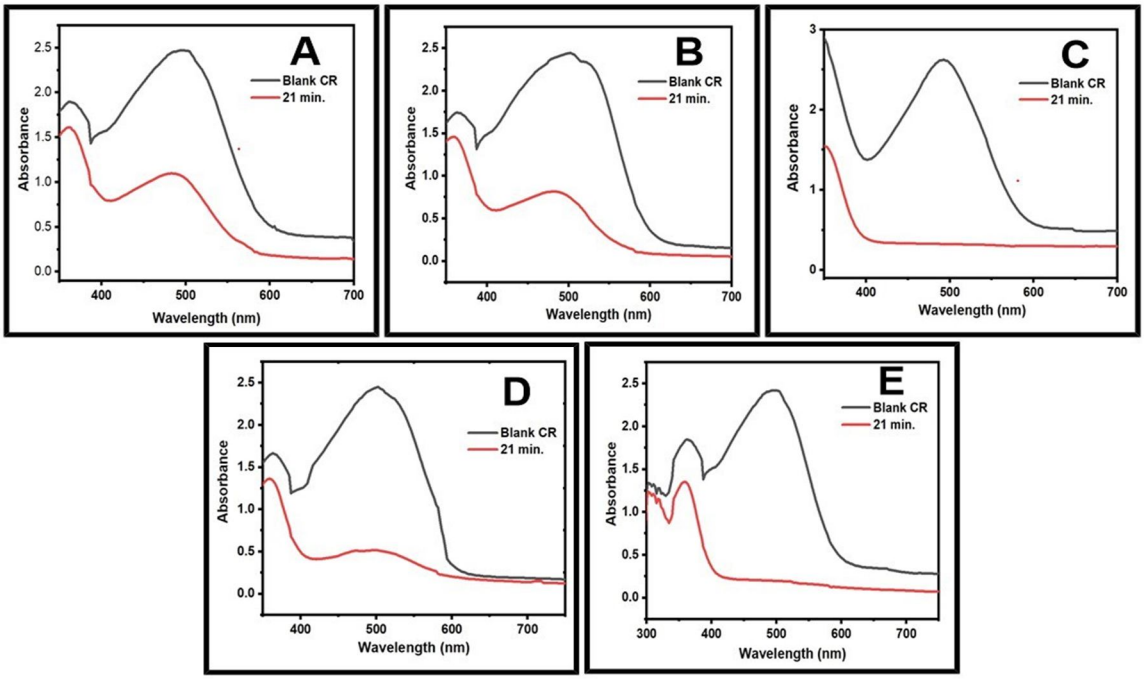

Figure 1. The UV-visible spectra of: CR dye in its catalytic reduction in the presence of (A) 8 mg (B) 10 mg, and (C) 12 mg of M@CuO nanocomposite while using 0.2 mL of $NaBH_4$; CR dye recorded in the presence of 8 mg of M@CuO nanocomposite while using (D) 0.3 mL and (E) 0.4 mL of $NaBH_4$.

Table 1. The percentages of reduction of CR dye by $NaBH_4$ in the presence of the M@CuO nanocomposite.

S. No.	Pollutant	Amounts of the nanocomposite (mg)	Volume of $NaBH_4$ (3 mg/mL) used (mL)	Percentage of reduction (%)	Reduction time (min)
1	CR	8	0.2	59.34	21
		10	0.2	69.01	21
		12	0.2	87.5	21
		8	0.3	76.89	21
		8	0.4	92.11	21

III. RESULTS AND DISCUSSION

A. Kinetic study of CR in the presence of various amounts of M@CuO nanocomposites

The catalytic activity of the nanocomposite in reducing CR dye was studied by using different amounts of the nanocomposite while keeping the volume of $NaBH_4$ constant. The M@CuO nanocomposite quickly accepts hydride (H^-) ions produced by $NaBH_4$ and transfers them to Congo red dye. This process is known as electron transfer. In this redox reaction, CuO nanoparticles act as a mediator by transferring electrons from BH_4^- (electron donor) to the dye (electron acceptor). As a result, the catalyst converts the colored CR dye into its non-toxic, colorless reduced form. The catalytic performance of the reduction of CR was tested using 8 mg, 10 mg, and 12 mg of the nanocomposite with 0.2 mL of $NaBH_4$ separately. After 21 minutes, the reduction efficiencies were 59.34%, 69.01%, and 87.5%, respectively, in Table 1.

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Kinetic studies were performed using the pseudo-first-order reaction model to calculate the apparent rate constant, and the results are presented in **Table 2.** As the amount of nanocomposite increased, the amount of CuO nanoparticles also increased. Since the concentration of $NaBH_4$ remained constant, the higher amount of catalyst improved the reduction efficiency, increased the percentage of reduction, and the reaction rate [16, 17].

B. Kinetic study of CR dye by varying the amount of $NaBH_4$ in the presence of the M@CuO nanocomposite.

The catalytic reduction of CR dye was carried out using various $NaBH_4$ solutions, while keeping the amount of M@CuO nanocomposite. After 21 minutes, the reduction percentages were 59.34%, 76.89%, and 92.11% for 0.2 mL, 0.3 mL, and 0.4 mL of $NaBH_4$, in the presence of 8 mg of M@CuO nanocomposite, in **Table 1**. The increase in reduction percentage is due to the higher concentration of $NaBH_4$, which provides more reducing agents while the catalyst amount remains constant. The apparent rate constant values calculated at the same times are listed in **Table 2.** It was observed that the k_{app} value increased as the amount of $NaBH_4$ increased. The reduction reaction generally follows pseudo-first-order kinetics [16, 18].

Table 2. The corresponding apparent rate constant (k_{app}) in the presence of the M@CuO nanocomposite.

S. No.	Pollutant	Amounts of the nanocomposite (mg)	Volume of $NaBH_4$ (3 mg/mL) used (mL)	$k_{app} \times 10^{-2}$ (min^{-1})
1	CR	8	0.2	4.12
		10	0.2	5.58
		12	0.2	9.90
		8	0.3	7.41
		8	0.4	11.9

IV. CONCLUSION

This research paper demonstrates that the microgel-based copper oxide (M@CuO) nanocomposite is an efficient and robust catalyst for the reduction of cango red dye in aqueous media using sodium borohydride ($NaBH_4$). The polymeric microgel matrix effectively stabilizes the CuO nanoparticles, providing high surface area and enhanced dispersion of active sites, which facilitates rapid electron transfer from BH_4^- ions to CR molecules. Catalytic tests revealed that increasing either the amount of nanocomposite or the concentration of $NaBH_4$ significantly improves the cango red dye reduction efficiency, achieving up to 92.11% decolorization within 21 minutes, with a corresponding apparent rate constant of 11.9×10^{-2} min^{-1}. The M@CuO nanocomposite showed strong catalytic activity for the reduction of CR dye using $NaBH_4$. Increasing either the amount of nanocomposite or the concentration of $NaBH_4$ improved the reduction efficiency and reaction rate. The reaction followed pseudo-first-order kinetics, and the apparent rate constant increased with higher catalyst and reducing agent amounts. Overall, the M@CuO nanocomposite demonstrated good catalytic performance and potential for wastewater treatment applications.

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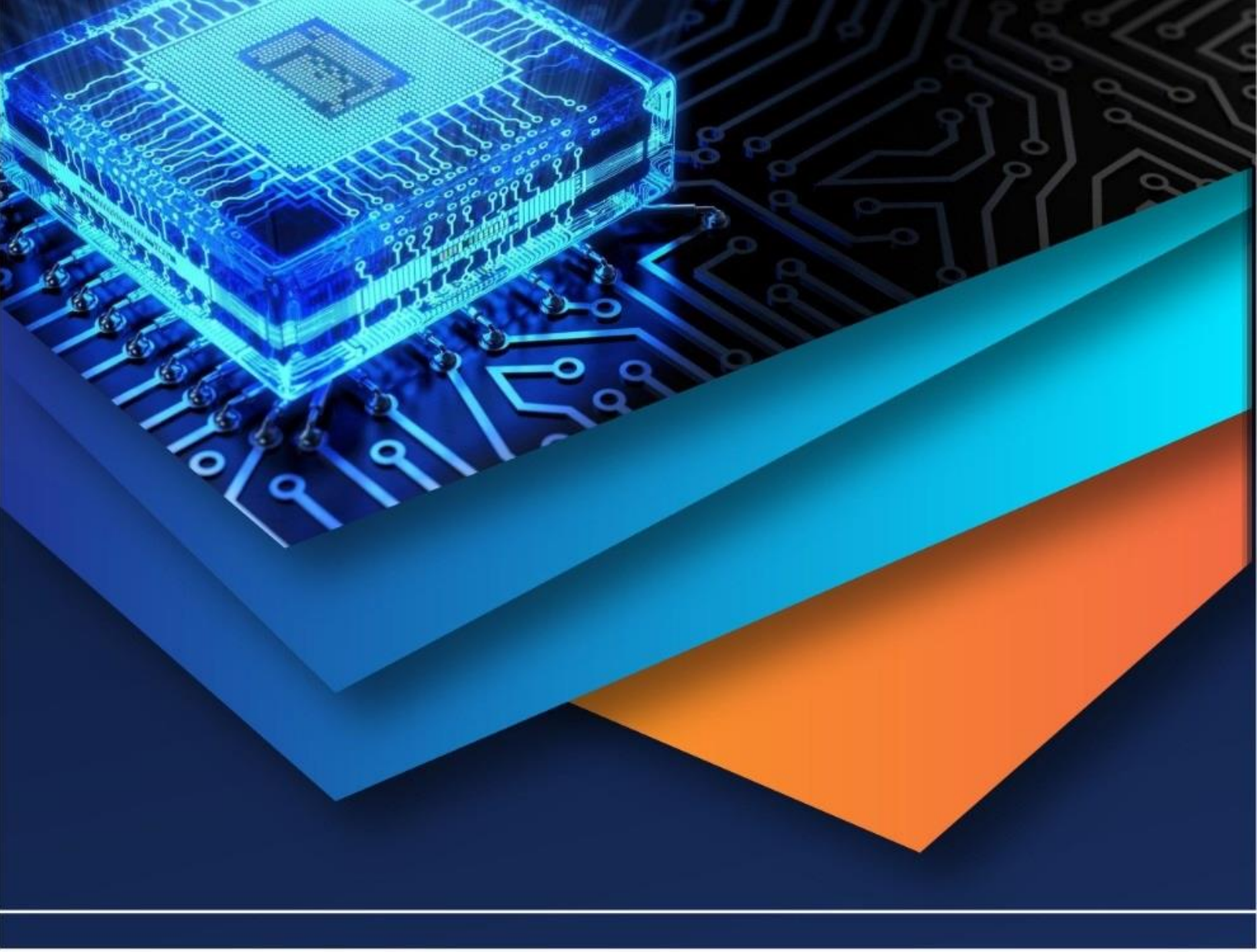

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