



Ascorbic Acid Mediated Ecofriendly Synthesis of Copper Nanoparticles for Statistically Optimized Adsorptive Removal of Reactive Navy Blue 19 Dye

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ABSTRACT

This study focuses on the green synthesis of copper nanoparticles (CuNPs) using L-ascorbic acid as a reducing and stabilizing agent. The synthesized nanoparticles were characterized by Fourier Transform Infrared Spectroscopy (FTIR), Energy Dispersive X-ray Spectroscopy (EDS), and Scanning Electron Microscopy (SEM). The point of zero charge (pH_p , z_c) was determined to assess the surface neutrality of the nanoparticles. These CuNPs were applied as efficient adsorbents for the removal of Reactive Navy Blue (RNB) dye from aqueous solutions. The adsorption process was optimized using Response Surface Methodology (RSM) based on a four-factor, five-level Central Composite Design (CCD). Under optimized conditions, a maximum dye removal efficiency of 99.79% was achieved. Experimental validation of the model, replicated six times, yielded a relative standard deviation (%RSD) of 0.0652 and a mean error of 0.1441%, confirming the model's accuracy. Analysis of variance (ANOVA) demonstrated the statistical significance of both the overall model and individual parameters. Error analysis revealed that the Freundlich isotherm and pseudo-second-order kinetic models best described the adsorption process. Thermodynamic studies confirmed the spontaneous and feasible nature of dye adsorption onto CuNPs. The results suggest that this green and cost-effective approach offers a promising solution for the treatment of dye-contaminated industrial wastewater.

Keywords: Green synthesis, Copper nanoparticles, Reactive Navy Blue Dye, Adsorption and Responsive Surface Methodology

INTRODUCTION

Reactive Blue 19 (RB19) is an anionic anthraquinone dye containing a β -sulfatoethylsulfonyl group, widely used in the textile industry as a colouring agent. However, it is classified as a toxic and carcinogenic organic pollutant [1]. Industrial effluents often contain chemicals and synthetic dyes which may cause harmful effects on living organisms. The disposal of this wastage into rivers and lakes show the opposite effect to increase the photosynthetic activity and completely disturbed the aquatic life as well as turbidity level high due to this penetration level of sunlight may be decreased [2,3].

Synthetic dyes are highly soluble in water that's why this is a critical task to remove it from water. Therefore, treating wastewater before its release into the environment is essential. There are several physicochemical and biological methods are used at the industrial level for dye removal, including advanced oxidation processes, membrane separation, chemical precipitation, adsorption, and electrochemical degradation [4-5]. Among these, adsorption stands out as a cost-effective and efficient technique, particularly due to its simplicity, reusability of adsorbents, and minimal chemical requirements [6-8].

The use of nanomaterials as adsorbents has gained significant attention due to their high surface area, tunable surface properties, and enhanced adsorption capacity [9-11]. At the industrial scale, rapid and efficient dye removal is critical. Nanoparticle-based adsorption offers a promising solution because of its speed, efficiency, and scalability. The structural and chemical properties of nanomaterial-based adsorbents make them reusable, easy to handle, and highly effective in capturing dye molecules from wastewater [12-13].

Recent advancements have focused on designing novel, eco-friendly nano adsorbents to improve adsorption efficiency. Among these, copper nanoparticles (CuNPs) have shown great potential due to their excellent catalytic, optical, electrical, and antimicrobial properties [14-19]. Additionally, CuNPs are economically favourable, offering high yields with low synthesis time compared to other metal nanoparticles [20-21].

Green synthesis approaches, particularly using ascorbic acid as a reducing agent, offer an environmentally benign route for CuNPs production. Ascorbic acid, with a hydrogen potential of +0.08 V, effectively reduces Cu^{2+} ions due to the higher standard reduction potential of copper. The free hydrogen radicals released by ascorbic acid initiate reduction and oxidation reactions, leading to the formation of pure CuNPs [22-28].

In this study, CuNPs synthesized via a green method using ascorbic acid were employed for the adsorptive removal of RB19 dye from industrial effluents. The synthesized nanoparticles were characterized by FTIR, SEM, EDS, and XRD. Furthermore, adsorption behaviour was investigated using thermodynamic and kinetic studies, and the process was optimized through Central Composite Design (CCD) and Response Surface Methodology (RSM).

MATERIALS & METHODS

Chemicals

Analytical grade chemicals were used, including copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 99.9%), L-ascorbic acid (98%), ammonium hydroxide (NH_4OH), and Reactive Navy Blue (RNB) dye. Double-distilled water was used throughout.

Synthesis Of Copper Nanoparticles

Copper nanoparticles were synthesized using a simple and green method. A 0.1 M solution of copper

sulfate was prepared by dissolving $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in distilled water (50 mL). Separately, a 1.0 M solution of L-ascorbic acid was prepared. The copper solution was mixed with the ascorbic acid solution in a beaker and stirred continuously at 60°C for 30 minutes. Ascorbic acid served as both a reducing and stabilizing agent. After adding 1.0 mL of NH_4OH , the solution was centrifuged, and the resulting precipitate was washed with ethanol. The nanoparticles were dried in an oven at 60°C for 15 minutes. FTIR analysis confirmed that no significant product deformation occurred over 30 weeks. The yield of copper nanoparticles obtained through this method was approximately 70 percentage.

Characterization

The synthesized nanoparticles were characterized using various techniques. Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS; JSM6380A, Karachi, Pakistan) was used to analyse surface morphology and elemental composition. Fourier Transform Infrared Spectroscopy (FTIR; SHIMADZU, Karachi, Pakistan) identified functional groups. X-ray Diffraction (XRD; JDX-3532, Japan) was conducted using Cu $\text{K}\alpha$ radiation over a 2θ range of $5-70^\circ$ to determine crystallographic structure. The point of zero charge (pH_p , z_c) was determined by Kosmulski's method by mixing 0.025 g of nanoparticles with 50 mL of 1 mM NaCl solutions at varying pH (2–12), adjusted with 0.1 M NaOH and HCl. Final pH values were measured after 24 and 48 hours, and the pH_p , z_c was identified at the intersection of the resulting curves.

Experimental Design (CCD & RSM)

A four-factor, five-level Central Composite Design (CCD) was applied with 30 runs to optimize dye removal. Variables included contact time (2–10 min),

pH (3–9), adsorbent dose (5–25 mg), and dye concentration (5–25 mg/L). Response Surface Methodology (RSM) was used to model and interpret interactions. Full experimental details are available in our previous study [29].

Adsorption Study

A 500 mg/L stock solution of RNB dye was prepared and diluted as required. After adding CuNPs to the dye solution, the mixtures were ultrasonicated and centrifuged. Dye removal (%) and adsorption capacity (q_e) were calculated using standard equations. Further details are provided in our previous paper. [29].

Adsorption Isotherms

Isotherm studies were conducted at 303–313 K to evaluate adsorbent–adsorbate interactions. Data were fitted to Langmuir, Freundlich, Dubinin–Radushkevich, and Temkin models to determine adsorption type, surface characteristics, and energy effects [30–35].

Kinetic Study of Adsorption

Kinetics were studied by adding 0.0214 g of adsorbent to 20 mL of RNB solution (32.2094 mg/L) at pH 5.5. Absorbance was recorded before adsorption and after 2, 4, 6, and 8 min of sonication. Data were evaluated using pseudo-first-order, pseudo-second-order, intraparticle diffusion, and Elovich models to assess adsorption rate and mechanism [35–39].

RESULTS

Characterization

SEM scanning electronic microscope, EDS energy dispersive Xray spectroscopy, FTIR Fourier transform

infrared spectroscopy, XRD Xray diffraction techniques used to characterize the nano particles.

SEM scanning electronic microscope

SEM analysis (Fig. 1) revealed well-dispersed spherical CuNPs with particle sizes ranging from 80-150 nm. The uniform morphology and nanoscale size provide a larger surface area, which plays a crucial role in enhancing adsorption efficiency [30,40].



Figure 1: SEM image of copper Nanoparticles

EDS energy dispersive X-Ray spectroscopy

EDS analysis (Fig. 2) confirmed the elemental composition of the synthesized nanoparticles, showing copper as the major component. The presence of strong carbon and oxygen signals is attributed to the green synthesis process, where plant-derived biomolecules interact with Cu precursor ions to form CuNPs [30,40-41].

X ray Diffraction (XRD)

The XRD pattern of CuNPs at room temperature (Fig. 3) exhibited distinct peaks at 2θ values of 43° , 50° , and 74° , corresponding to the (111), (200), and (220) planes of cubic copper crystals. The calculated crystallite size ranged between 12–24 nm, confirming the crystalline and cubic nature of the nanoparticles [26].

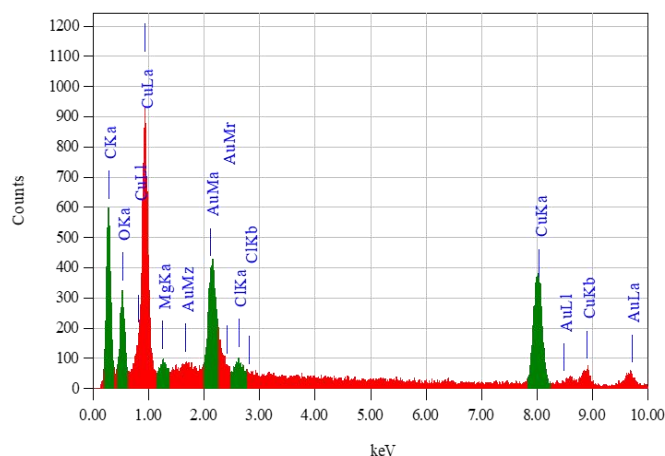


Figure 2: EDS spectra of copper Nanoparticles

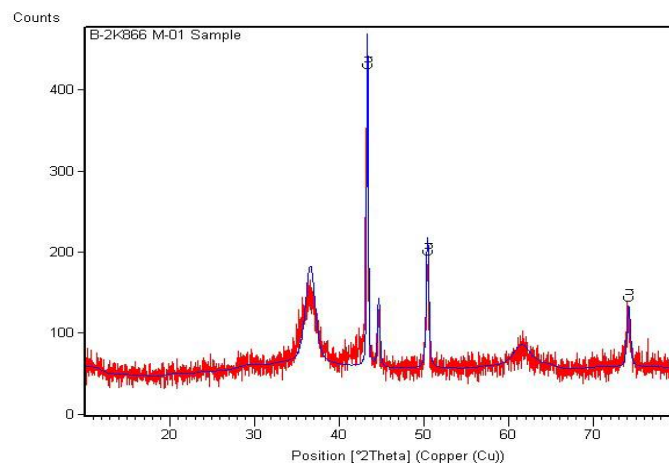


Figure 3: XRD spectra of copper Nanoparticles

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis (Fig. 4) revealed several characteristic absorption bands confirming the functional groups involved in CuNP formation. Peaks observed at 611 and 756 cm^{-1} may correspond to Cu–O vibrations of Cu_2O . The absorption band at 1120 cm^{-1} is attributed to NO_3^- stretching and bending [29]. A distinct band at 1386 cm^{-1} indicates the presence of C=O groups [43]. Peaks at 1625 cm^{-1} and 2372 cm^{-1} are associated with water molecule bending, while the bands at 2854 and 2926 cm^{-1} correspond to C–H stretching modes of hydrocarbon chains [42].

broad peak at 3419 cm^{-1} , due to O–H stretching, confirms the attachment of hydroxyl groups on the nanoparticle surface, which is consistent with green synthesis stabilization [30,44].

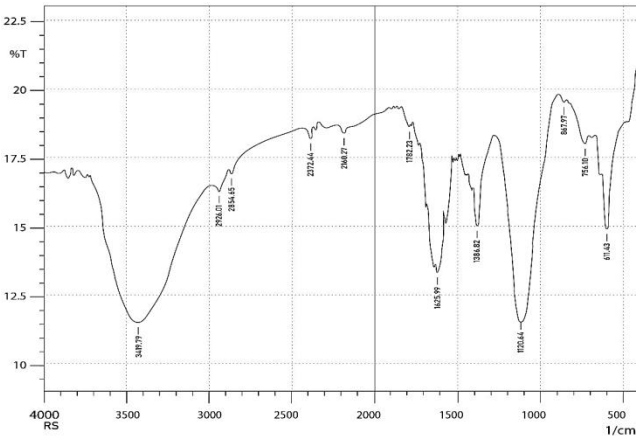


Figure 4: FTIR spectrum of the synthesized copper Nanoparticles

pH at zero point

Figure 5 shows the point of zero charge (pHPZC) at 8.4, indicating a slightly basic surface nature of CuNPs. Maximum adsorption was observed at pH 5.6, where the nanoparticle surface carried a net positive charge. This promoted strong electrostatic attraction between the positively charged CuNPs and the anionic dye molecules, thereby enhancing adsorption efficiency.

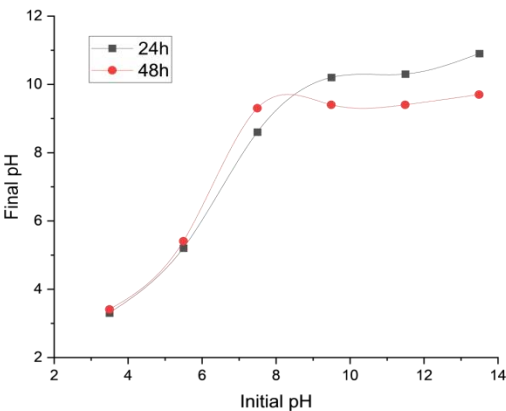


Figure 5: Graphical representation of pH at point of zero charge

A Central Composite Design (CCD) with four factors RNB concentration, adsorbent dose, contact time,

and pH was employed, including six centre points and a total of 30 runs. The results were analysed using response surface methodology (RSM) and ANOVA (Table 1 and 2) [30,32]. The model was statistically significant and provided a good fit to the experimental data. Among all variables, contact time had the strongest influence with the lowest p-value, followed by pH, adsorbent dose, and initial dye concentration. Interaction effects followed the order $BC > AC > AD > D^2$, while the significant lack-of-fit was attributed to non-significant model terms, which can be minimized by refinement [30].

Table 1. Central Composite Design for the adsorption of RNB by green copper

S.No.	Time (min)	pH	Amount (mg)	Conc. (ppm)	% Removal
1	8	7.5	40	40	32.6
2	6	6	10	30	80.97
3	4	7.5	20	40	45.4
4	6	3	30	30	92.8
5	4	4.5	20	20	47.05
6	4	7.5	20	20	52.76
7	4	4.5	40	40	82.27
8	8	4.5	20	40	85.2
9	6	6	30	30	86.5
10	8	4.5	20	20	97.2
11	6	6	30	30	79.74
12	6	6	30	30	80
13	4	7.5	40	20	13.3
14	6	9	30	30	64.9
15	10	6	30	30	99.6
16	6	6	30	30	79.75
17	4	4.5	20	40	62.17
18	8	4.5	40	40	85.5
19	6	6	30	10	76.45
20	6	6	30	50	66.9
21	6	6	30	30	79.77
22	6	6	30	30	79.9
23	4	7.5	40	40	56.07
24	8	4.5	40	20	87.4
25	2	6	30	30	47.78
26	8	7.5	40	20	47.78
27	6	6	50	30	90.94
28	4	4.5	40	20	99.8
29	8	7.5	20	20	66.66
30	8	7.5	20	40	50.65

Table 2. Analysis of Variance

Source	Sum Squares	of differences	Mean Square	F-value	p-value
Model	11708.22	14	836.3	7.09	0.0003
A-Time	4274.4	1	4274.4	36.24	< 0.0001
B-pH	1916.92	1	1916.92	16.25	0.0011
C-Amount	983.94	1	983.94	8.34	0.0113
D-Concentrati	76.79	1	76.79	0.6511	0.4323
AB	5.84	1	5.84	0.0495	0.8269
AC	821.83	1	821.83	6.97	0.0186
AD	656.26	1	656.26	5.56	0.0323
BC	1471.68	1	1471.68	12.48	0.003
BD	0.8327	1	0.8327	0.0071	0.9342
CD	172.86	1	172.86	1.47	0.2448
A ²	500.61	1	500.61	4.24	0.0572
B ²	243.93	1	243.93	2.07	0.1709
C ²	493.32	1	493.32	4.18	0.0588
D ²	625.63	1	625.63	5.3	0.036
Residual	1769.24	15	117.95		
Lack of Fit	1718.34	10	171.83	16.88	0.003
Pure Error	50.9	5	10.18		
Cor Total	13477.45	29			

Normal Probability Plots

Fig. 6 shows the normal probability plot of the present study. Statistical analysis indicates that all data points lie within the 95% confidence interval, with no points falling outside the range. This confirms that the residuals are normally distributed and supports the adequacy of the quadratic model as the best fit for this research [30, 33-34].

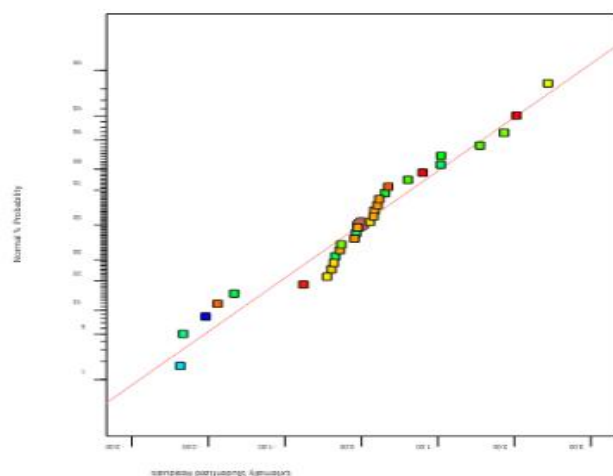


Figure 6: Normal Probability graph

Isotherm Analysis:

The interaction between adsorbent and adsorbate was studied using Langmuir, Freundlich, Temkin, and D-R isotherm models. The R^2 values in Table 3 show that the Langmuir isotherm provided the best fit, indicating monolayer adsorption on a homogeneous surface. The Temkin model also showed a reasonable fit, while the Freundlich and D-R models were the least suitable. Graphical representations are shown in Fig. 7a-7e [31, 46].

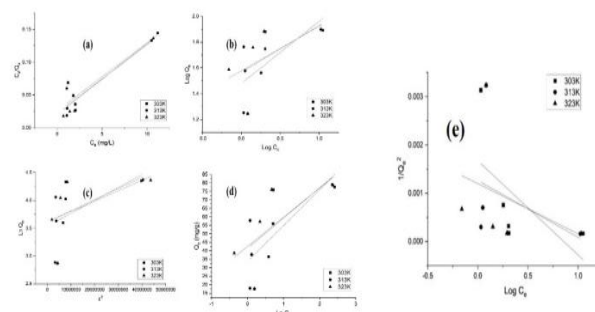


Figure 7: Isotherm representations of (a) Langmuir, (b) Freundlich, (c) D-R, (d) Temkin and (e) Harkins-Jura model respectively

Kinetic Analysis

The kinetics of RNB dye adsorption on Cu NPs were studied using Elovich, intraparticle diffusion, pseudo-first-order, pseudo-second-order, and Boyd's kinetic models. The values of the kinetic parameters are

presented in Table 4. Based on R^2 values, the intraparticle diffusion model showed the best fit, indicating that diffusion within the pores plays a major role in the adsorption process. The pseudo-second-order model also provided a good fit, suggesting chemisorption involvement. The Elovich model was the third most effective, while the Boyd and pseudo-first-order models were not suitable. Graphical representations of these kinetic models are shown in Fig. 8 [30].

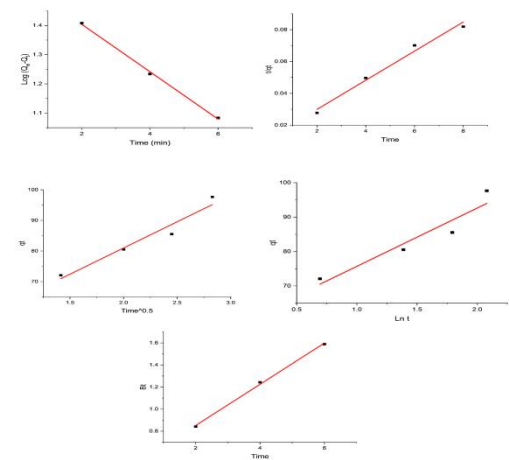


Figure 8: (a) pseudo first order, (b) pseudo second order, (c) intraparticle diffusion, (d) Elovich and (e) Boyd's kinetic model respectively.

CONCLUSION

In this study, *L-ascorbic acid* was successfully used to synthesize copper nanoparticles (Cu NPs) through a green and eco-friendly approach. The nanoparticles were well-dispersed with sizes ranging from 80–150 nm, as confirmed by SEM, while EDS verified a high copper content. The synthesized Cu NPs effectively adsorbed Reactive Navy Blue (RNB) dye, achieving 99.79% removal efficiency with an adsorption capacity of 49.57 mg/g under optimized conditions (pH 5.6, adsorbent dose 32.20 mg, dye concentration 32 mg/L, and contact time ~8 minutes).

Isotherm and kinetic studies indicated that the adsorption process followed the Langmuir isotherm, suggesting monolayer adsorption, while intraparticle diffusion and pseudo-second-order models best described the adsorption kinetics. Electrostatic attraction between the positively charged Cu NPs surface (pH < pH_{pzc}) and the anionic dye enhanced adsorption efficiency. The presence of salts, especially KCl, further improved adsorption.

Table 3: study of adsorption of RNB on Cu NPs by different isotherm model.

Isotherms	Parameters	RNB		
		303 K	313 k	323k
Langmuir	Q ^o (mg g ⁻¹)	96.51	95.57	93.06
	K _L (L mg ⁻¹)	0.4024	0.4758	0.5291
	R ²	0.8784	0.8154	0.8285
Freundlich	K _F (mg g ⁻¹) (L mg ⁻¹) ^{1/n}	30.11	36.73	37.76
	n	2.079	2.761	2.896
	R ²	0.4842	0.3138	0.3337
D-R	Q _s (mg g ⁻¹)	34.2	36.91	37.06
	B (mol ² kJ ⁻²) (X10 ⁻⁸)	-2.33	-2.01	-1.87
	E (kJ mol ⁻¹)	4.64	4.98	5.17
Temkin	R ²	0.3224	0.2654	0.2746
	A (L g ⁻¹)	4.939	10.161	11.85
	B	21.14	17.86	17.22
Harkins-Jura	R ²	0.5342	0.4389	0.4798
	A	513.2	849.2	961.9
	B	-0.8565	-1.072	-1.152
	R ²	0.3529	0.1407	0.1268

Table 4; show the kinetic model of this recent

Models	Parameters	Values
First order kinetic model	k ₁ (min ⁻¹)	0.5033
	Q _e (mg g ⁻¹)	105.6
	R ²	0.7909
Second order kinetic model	k ₂ (g mg ⁻¹ min ⁻¹)	0.0072
	Q _e (g g ⁻¹)	109.3
	R ²	0.9833
Intraparticle Diffusion	K _{id} (mg g ⁻¹ min ^{-1/2})	17.11
	C (mg g ⁻¹)	46.77
	R ²	0.9489
Elovich	a (g g ⁻¹ min ⁻¹)	0.5425
	b (mg g ⁻¹)	0.059
	R ²	0.9076
Boyd's kinetic model	R ²	0.1689

research:

Overall, this ecofriendly method demonstrates that Cu NPs are highly effective for the removal of textile

dyes and hold great promise for industrial wastewater treatment and environmental remediation.

Declaration of conflict of interest

The authors declare no conflict of interest, financial or otherwise. No artificial writing assistance was utilized in the production of this manuscript.

Consent for publication

All authors have read and approved the final version of the manuscript.

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