

Analysis of Adsorption of Malachite Green Dye on Copper Nanoparticles Using Statistical Software

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ABSTRACT

The co-precipitation approach was used in this study to create copper nanoparticles, which were then characterized by FTIR, EDS, and SEM. The malachite green dye was then removed using these nanoparticles as an adsorbent. RSM based on CCD was used to optimize the adsorption experiment, predicting a 98.99% elimination of the dye under ideal circumstances. The best settings were confirmed by repeating the experiment six times, with a low %RSD and %Mean Error. Using ANOVA and Pareto analysis, the model's and each parameter's relevance was evaluated. The results of error analyses demonstrating that the data were most well-fit by the pseudo second-order kinetic model and the Langmuir isotherm model, which were k_2 ($\text{g mg}^{-1} \text{min}^{-1}$), q_e (g g^{-1}), and R_2 (0.30665225, 100.617112 and 0.9997) respectively. To sum up, the suggested technique provides an economical and environmentally responsible means of rehabilitating the ecosystem.

Keywords:

INTRODUCTION

A metallic-lustrous dye, malachite green (MG) is usually found in the form of green crystals. It is highly soluble in organic solvents and water. Globally, MG is used extensively as a biocide in the aquaculture industry. It shows amazing effectiveness in curing a wide range of illnesses brought on by fungus and protozoa infections (Khawaja, Zahir, Asghar, & Asghar, 2021) (Stammati et al., 2005).

Malachite green is widely used in distilleries and is also known to be an excellent antiseptic for external treatment on wounds and ulcers, but it is crucial to remember that ingesting it can be extremely poisonous, dangerous, and cancer-causing (Gupta, Mittal, Krishnan, & Gajbe, 2004).

As a basic dye, malachite green is often used to tint a variety of materials, primarily paper and leather goods. It's also used in the fading of jute, cotton, silk, and wool. Further, distilleries employ malachite green dye (Dahri, Kooh, & Lim, 2014).

One major source of contaminants in the environment, including water and soil, is effluent from industrial processes. Its negative effects harm not just human

existence but also the ecosystem. For this reason, processing industrial effluent before releasing it into the environment must be given top priority (Mokif, 2019).

Environmental protection is currently receiving more public attention, which has raised awareness of the possible harm that industrial waste residues laden with contaminants like dyes could do to the environment (Karthik, Saravanan, Bharathi, Dharanya, & Meiaraj, 2014).

Different physico-chemical methods are frequently used to remove colored pollutants from industrial wastewaters in order to address this issue. These methods include membrane filtration, photocatalysis, oxidation, flocculation, coagulation, electrochemical methods, and adsorption. By using these techniques, dyes and other contaminants are successfully removed from industrial effluent (Adebawale, Olu-Owolabi, & Chigbundu, 2014) (Atia, Donia, & Al-Amrani, 2009).

In particular, adsorption is thought to be one of the best methods for advanced wastewater treatment when it comes to dye removal. Adsorption is one of the tried and tested processes that produces consistently good outcomes when compared to other processes like coagulation, advanced oxidation, and membrane separation (Bhatnagar et al., 2014) (Ngo et al., 2015).

Silver nanoparticle (AgNP) toxicity is effectively reduced by using green synthesis methods. The kind of coating that is used on the AgNPs largely determines their level of toxicity.

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AgNP coating aids in particle stabilization and agglomeration prevention. Green produced AgNPs can be used in a variety of medical applications due to their biocompatible covering (Roy, Bulut, Some, Mandal, & Yilmaz, 2019).

In this investigation, the scientists created copper oxide nanoparticles, or CuO NPs, and assessed how well they eliminated dyes. It was looked into how the adsorption process was affected by a number of variables, including the dosage of the adsorbent, the starting dye concentration, pH, and the presence of inorganic salts. The study utilized Langmuir, Freundlich, and Tempkin isotherms to examine the dye adsorption process on CuO nanoparticles. The adsorption mechanism was also ascertained by using intra-particle diffusion kinetic models and pseudo-first and pseudo-second-order models (Naghizade Asl et al., 2016).

In this work, dyes were extracted from industrial wastewater by employing ascorbic acid to create copper nanoparticles. Thermodynamics, kinetics, and design of experiments techniques like CCD and RSM were used to measure the percentage of dye removal and explore the properties of the copper nanoparticles as an adsorbent. Through the use of FTIR, EDS, SEM, and XRD studies, the adsorbent was characterized.

MATERIALS AND METHOD

Chemicals

Analytical grade chemicals include reactive navy-blue dye, ammonium hydroxide (NH₄OH), L-ascorbic acid (vitamin C, purity of 98%), as well as copper sulphate (CuSO₄.5H₂O, 99.9% pure). There was no additional processing involved; only analytical grade chemicals were utilized. During the investigation, double-distilled water was used.

Synthesis of Copper Nanoparticles

A simple but ecologically sound method was used to synthesize the nanoparticles. Dissolving the copper salt in distilled water yielded a 50 mL solution of 0.1 molar Copper sulfate (CuSO₄.5H₂O). Furthermore, L-ascorbic acid solutions with a concentration of 1.0M were made using distilled water. In a beaker, the 50 mL CuSO₄.5H₂O solution was combined with the L-ascorbic acid solution. The end product was heated using an electrical or mechanical heating method for 30 minutes at 60 degrees Celsius while being constantly stirred.

Ascorbic acid has a couple of roles: it is a reducing agent and an antioxidant. Centrifugation was employed to separate the solution after adding 1.0 ml of NH₄OH. The precipitate that formed was then cleaned with ethanol. After that, the produced nanoparticles were dried for fifteen minutes in an oven. FTIR analysis revealed that, astonishingly, no discernible product deformation was seen, even after a 30-week period. This approach yielded a yield percentage of 70%.

Characterization of Adsorbent Materials

Several analytical methods were used to determine the properties of the adsorbent materials, especially the elemental composition of Cu NPs. The size and the structure of the nanoparticles were assessed using JSM6380A model Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS) in Karachi, Pakistan. In Karachi, Pakistan, Fourier-transform infrared spectroscopy (FTIR) was performed with a SHIMADZU model. Using Cu K α radiation, Japan's JDX-3532 was employed for X-ray diffraction (XRD) investigation. covering 2 θ values from 5 to 70 degrees.

Central Composite Design (CCD)

The optimal quadratic response surface model operating parameters are typically found using the central composite design (CCD). Increasing precision by adjusting the central points and experimental error has a direct impact on CCD accuracy. Equation 1 is used to get the RSM value.

$$\alpha = \sqrt[4]{2f} \quad (1)$$

The CCD experimental samples are calculated using equation no. 2:

$$N = 2f + 2f + N_o \quad (2)$$

The number of factors in the relationship above is indicated by f , and the factorial and central points are represented by $2f$ and N_o , respectively. This study found that CCD is made up of four distinct elements that were used in conjunction with five tiers and six central points. The CCD analysis involved 30 consecutive runs. Time ranges in this assay are 2 to 10 minutes, pH ranges are 3.0 to 9.0, adsorbent amounts range from 5 to 25 mg, and dye concentrations range from 5 to 25 mg/L. these distant components,

indicated by "A," "B," "C," and "D," respectively. The Beer-Lambert law most likely would have altered Equation No. 3, which is utilized to determine the proportion of the corresponding dye removed with the aid of CCD.

Response Surface Methodology (RSM)

Response surface methodology (RSM) is a set of statistical and mathematical techniques used to firmly establish a functional relationship between a desired response and the variables influencing it. (Khuri, 1996).

It is crucial to comprehend the behavior of the data in order to generate statistical predictions for a dataset. This is especially helpful when there are several factors influencing the relevant responses. To have the optimum system performance, The goal is to concurrently maximize each of these variables' levels. (Hanrahan & Lu, 2006).

Dyes Solution Synthesis

All of the parameters (pH, dye quantity, sonication time, and dye solution concentration) in the Malachite Green dye stock solution were adjusted in accordance with CCD. Make a note of the absorbance at 627 nm using a UV spectrophotometer both before and after adsorption. These readings give us the dye removal percentage.

Adsorption of Dyes onto Copper Nanoparticles

In this work, the efficacy of Cu nanoparticles in removing dye was examined by batch adsorption studies. With the use of Design Expert Software, the trials were created utilizing the Response Surface Methodology. We examined the interplay between a number of variables, such as the initial concentration of Malachite Green (MG) dye, contact time, pH, and adsorbent dose. For these factors, the experimental ranges were 0.05-0.01g, 10-50 mg/L, 3-9 pH, and 2-10 minutes, in that order.

Thirty tests in total were carried out according to the experiment design. for each MG dye independently at a particular wavelength. Following the addition of the appropriate quantity of adsorbent to the dye solution, the beakers were placed in the sonicator containing the dye solution (adsorbate). After the solutions were centrifuged, the differences in absorbance between the pre- and post-adsorption phases were used to analyze the remaining dye quotient. in a double beam spectrophotometer at 627 nm in wavelength. The efficiency of removal is calculated by equation no. 3

$$\% \text{Removal Malachite Green dye} = \frac{C_o - C_f}{C_o} \quad (3)$$

Where, respectively, C_o and C_f stand for the dye concentration before adsorption and the dye concentration after adsorption

Equation no. 4 used to determine the adsorbent's adsorption capacity:

$$q_e = (C_o - C_e)V/m \quad (4)$$

where, V and m represent the volume of liquid adsorbate and the mass of adsorbent respectively.

Adsorption Isotherm Models

Adsorbates are transferred onto adsorbents during the adsorption process, which mostly takes place at the adsorbent material's surface (Zhuang, Chen, Liu, & Wang, 2020) (Ahmad, Ali, Khan, & Rehman, 2020) (Ghazi et al., 2018).

Various isotherm models are employed to assess the relationships among the adsorbent and adsorbate, with the temperature ranges studied falling between 303 and 313 K.

When modeling adsorption data, a number of widely used isotherm models are employed. The Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich isotherm models are among these (Langmuir, 1916) (Freundlich, 1906; Temkin, 1940) (Dubinin, 1947)

An important non-linear curve that illustrates the adsorption process at constant pH and temperature is the modeled adsorption isotherm. The practical application and design of adsorption systems rely heavily on the mathematical correlation that is derived from modeling studies. An alternative mathematical method for forecasting the general adsorption behavior is to use isotherm models to do a linear analysis of isotherm data (Foo & Hameed, 2010).

Kinetic Study of Adsorption

The pace at which a solute is eliminated during the adsorption process, or adsorption kinetics, determines how long the adsorbate remains at the solid-solution interface. The optimal kinetic rate equation was found by fitting the data using linear regression. By looking at the coefficients of determination following the linear transformation of the equations, the kinetic rate equations and the experimental

data can also be verified using the linear least-squares approach. (Ho, 2006).

To comprehend the adsorption kinetics and rate-limiting phase, a number of adsorption kinetic models have been developed. These include the elovich model, intra-particle diffusion, and pseudo-first and second-order rate models (Djeribi & Hamdaoui, 2008).

20 milliliters of MG solution (15.0385 mg/L) were mixed with 0.045 g of adsorbent to perform the adsorption of the kinetic research. The solution's pH was kept at 4.3 for one, two, or three minutes of sonication.

RESULT AND DISCUSSION

Characterization

The nanoparticles were characterized using XRD (The X-ray diffraction), FTIR (Fourier transform infrared spectroscopy), EDS (energy dispersive X-ray spectroscopy), and SEM (Scanning Electronic Microscope).

Using scanning electron microscopy (SEM), high-resolution surface images were obtained. The image of the synthesized copper nanoparticles is shown in Figure 1.

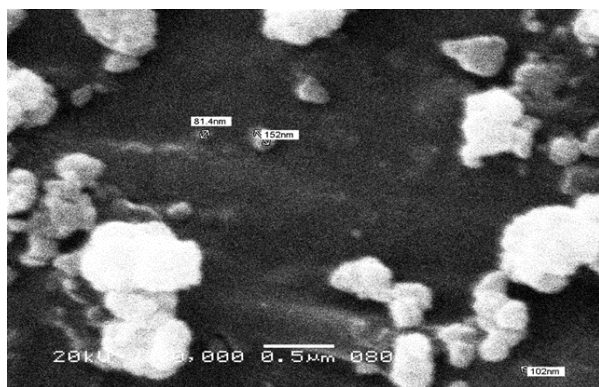


Figure 1. SEM image of copper NPs.

The spherical-shaped copper nanoparticles (Cu NPs) are shown in Figure 1 along with their size range. The typical particle size falls between 82 and 152 nm. The size of the nanoparticles affects the adsorption effectiveness.

EDS energy dispersive X-ray spectroscopy:

Utilizing Energy-Dispersive X-ray Spectroscopy (EDS), the nanoparticles' composition was examined. The proportion of the total amount of each element to the composition of the nanoparticles is shown in Figure 2. The primary constituent of the nanoparticles is determined to be copper based on the EDS spectra. Because of the green

synthesis method, where in carbon and oxygen form bonds with the copper nanoparticles, there is a higher presence of carbon spectra.

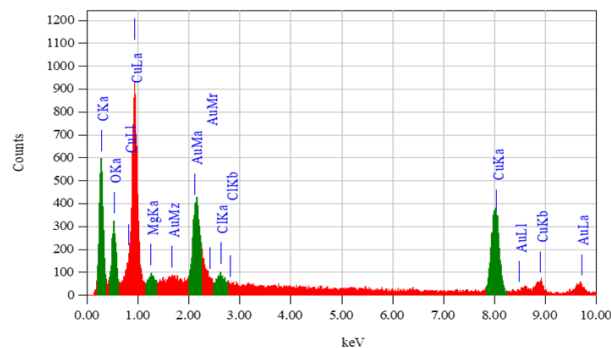


Figure 2. EDS spectra of Copper NPs.

X ray diffraction (XRD): The X-ray diffraction (XRD) pattern of room-temperature copper nanoparticles (Cu NPs) is presented in Figure 3. Different peaks in the XRD pattern indicate the presence of impurities from the green synthesis technique, including carbon. These peaks relate to copper. Copper nanoparticles are present because of the peaks at 2 theta angles of 43°, 50°, and 74°, which correlate to the crystallographic planes [470], [220], and [140], respectively. Additionally, this graphical representation implies that the unit cell has a cubic structure with a size range of 12 to 24 nm (Salavati-Niasari, Davar, & Mir, 2008).

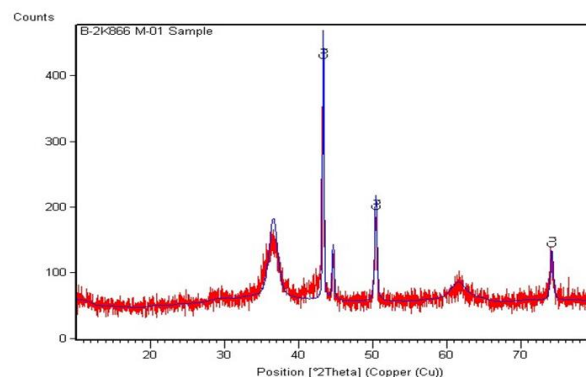


Figure 3. XRD spectra of copper NPs.

Fourier transform Infrared spectroscopy (FTIR): Based on their vibrations, the various molecules in the sample are distinguished by the FTIR spectrum shown in Figure 4.

Figure 4 shows several peaks at different wavelengths. Significantly, many small peak sizes were seen at 611 cm⁻¹ and 756 cm⁻¹, which may indicate the existence of Cu₂O. The stretching and bending of NO₃ was revealed by the

absorption peak at 1120 cm^{-1} . Moreover, the presence of C=O was suggested by the band at 1386 cm^{-1} . The stretching of O-H and the bending of H-O-H were represented by the peaks at 1625 cm^{-1} and 3419 cm^{-1} , respectively (Kayani, Umer, Riaz, & Naseem, 2015).

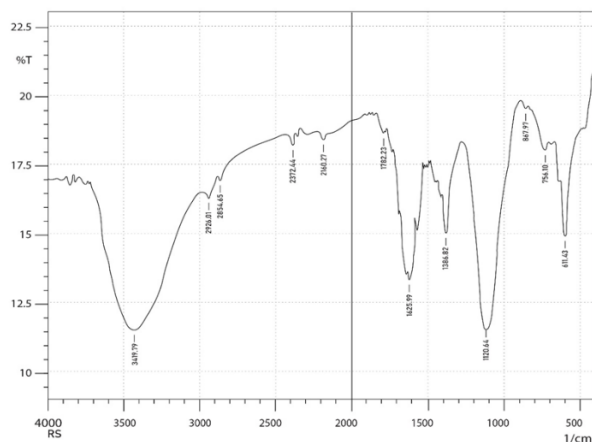


Figure 4. Show the FTIR spectrum.

The peaks at 1625 cm^{-1} and 2372 cm^{-1} are said to be caused by water molecules bending, according to published research. Furthermore, the existence of a carbonyl carbon is shown by the signal at 1782 cm^{-1} . The presence of C-H stretching modes from a hydrocarbon chain is suggested by the less intense bands located at 2854 cm^{-1} and 2926 cm^{-1} (Chowdhury, Beg, Khan, & Mina, 2013).

Analysis of Variance

An analysis of variance (ANOVA) was used to determine the model's relevance for the elimination of MG dye. It was observed that the model was significant as it represented by the P value of the model for nanoparticles. The analysis of variance (ANOVA) was performed to assess the significant and insignificant for the removal of MG dye. The model's P values support the findings of the analysis of variances, which indicated whether the model is significant or not. The parameters were assessed statistically in order to observe the individual significance or insignificance variables and their interactions of MG dye in Table 1. The statistical eminence of this model was assessed by the F test. The obtained P-value Table 1 ($P = 0.2424 > 0.05$) insignificant but. B^2 , C^2 are significant model terms. For the removal of MG dye. The considerable lack of fit is indicated by the F-value of 898.85 for the lack of fit. A high Lack of Fit F-value has a 0.01% probability of being caused by noise.

Tables 2 for the removal of MG dyes. Represent the estimation of the rate constant. The data shows that Cu NPs followed the kinetic models for both adsorbent and dye. Especially pseudo second order kinetics. Linear plots of t/q_t vs time were plotted correspond that desorption process follows pseudo second order kinetics. The q_e and q_t are the amount of the dye adsorbed on the adsorbent (mg/g) at equilibrium and t is the time in minutes K_2 is the adsorption of second order rate constant. Graphical representation has been shown in Figure 5.

Table 1. Show the ANOVA study.

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	7889.40	14	563.53	1.36	0.2484	not significant
A-Time	0.0551	1	0.0551	0.0001	0.9910	
B-pH	156.83	1	156.83	0.3774	0.5482	
C-Amount	170.72	1	170.72	0.4108	0.5312	
D-Concentration of dye	171.15	1	171.15	0.4118	0.5307	
AB	530.73	1	530.73	1.28	0.2762	
AC	189.41	1	189.41	0.4558	0.5099	
AD	476.00	1	476.00	1.15	0.3014	
BC	4.42	1	4.42	0.0106	0.9192	
BD	574.92	1	574.92	1.38	0.2578	
CD	109.25	1	109.25	0.2629	0.6156	
A ²	10.17	1	10.17	0.0245	0.8778	
B ²	3370.41	1	3370.41	8.11	0.0122	
C ²	1936.56	1	1936.56	4.66	0.0475	
D ²	219.85	1	219.85	0.5290	0.4782	
Residual	6233.47	15	415.56			
Lack of Fit	6230.00	10	623.00	898.85	< 0.0001	Significant
Pure Error	3.47	5	0.6931			
Cor Total	14122.87	29				

Table 2. Show the study of kinetic models.

Models	Parameters	Values
First order kinetic model	$k_1(\text{min}^{-1})$	0.7041
	$q_e(\text{mg g}^{-1})$	3.8216
	R^2	0.6716
Second order kinetic model	$k_2(\text{g mg}^{-1} \text{min}^{-1})$	0.3066
	$q_e(\text{g g}^{-1})$	100.6171
	R^2	1
Intraparticle Diffusion	$K_{id}(\text{mg g}^{-1} \text{min}^{-1/2})$	2.5685
	$C(\text{mg g}^{-1})$	95.1526
	R^2	0.9952
Elovich	$a(\text{g g}^{-1} \text{min}^{-1})$	2.6681E+22
	$b(\text{mg g}^{-1})$	0.5939
	R^2	0.9795
Boyd's kinetic model	R_2	0.6028

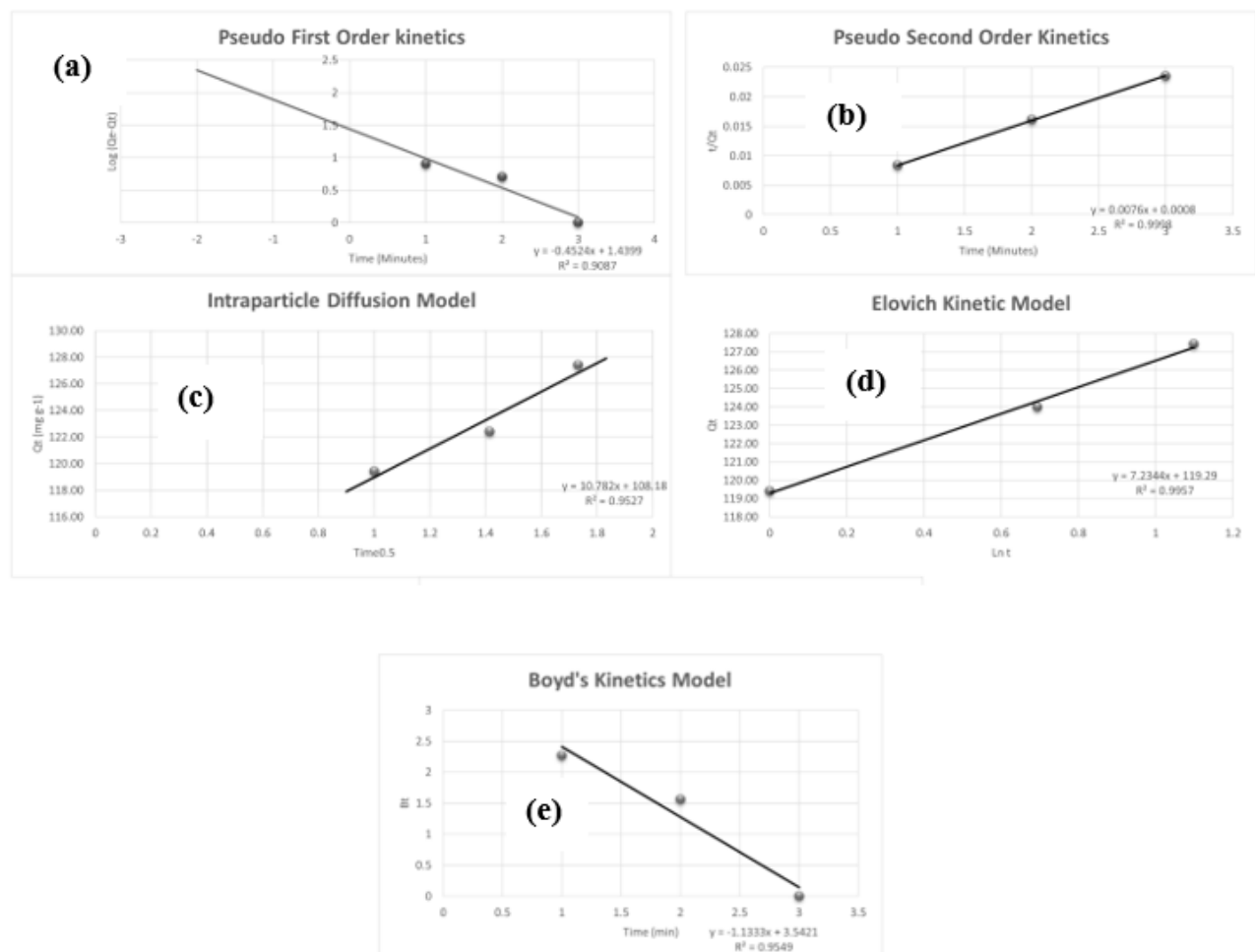


Figure 5. Shows kinetic models.

Table 3. Show the isotherm models analysis.

Isotherms	Parameters	Malachite Green		
		30 °C	35 °C	40 °C
Langmuir	Qo (mg g ⁻¹)	29.8608	30.9888	34.6936
	KL (L mg ⁻¹)	-5.4176	-11.4670	89.2146
	R ²	0.9999	0.99998	0.9999
Freundlich	KF (mg g ⁻¹)(L mg ⁻¹) ^{1/n}	43.4775	33.8194	26.3991
	N	5.8729	-32.3834	16.6836
	R ²	0.8676	0.8972	0.1666
D-R	Qs (mg g ⁻¹)	25.6607	33.9865	34.2498
	B (mol ² kJ ⁻²)	-2.23E-09	1.37E-09	-2.01E-10
	R ²	0.8845	0.56649	0.0867
Temkin	A (L g ⁻¹)	66319	1.3711E-17	2.4E+111
	B	1.2636	-0.8718	0.13372
	R ²	0.9169	0.5386	0.10527

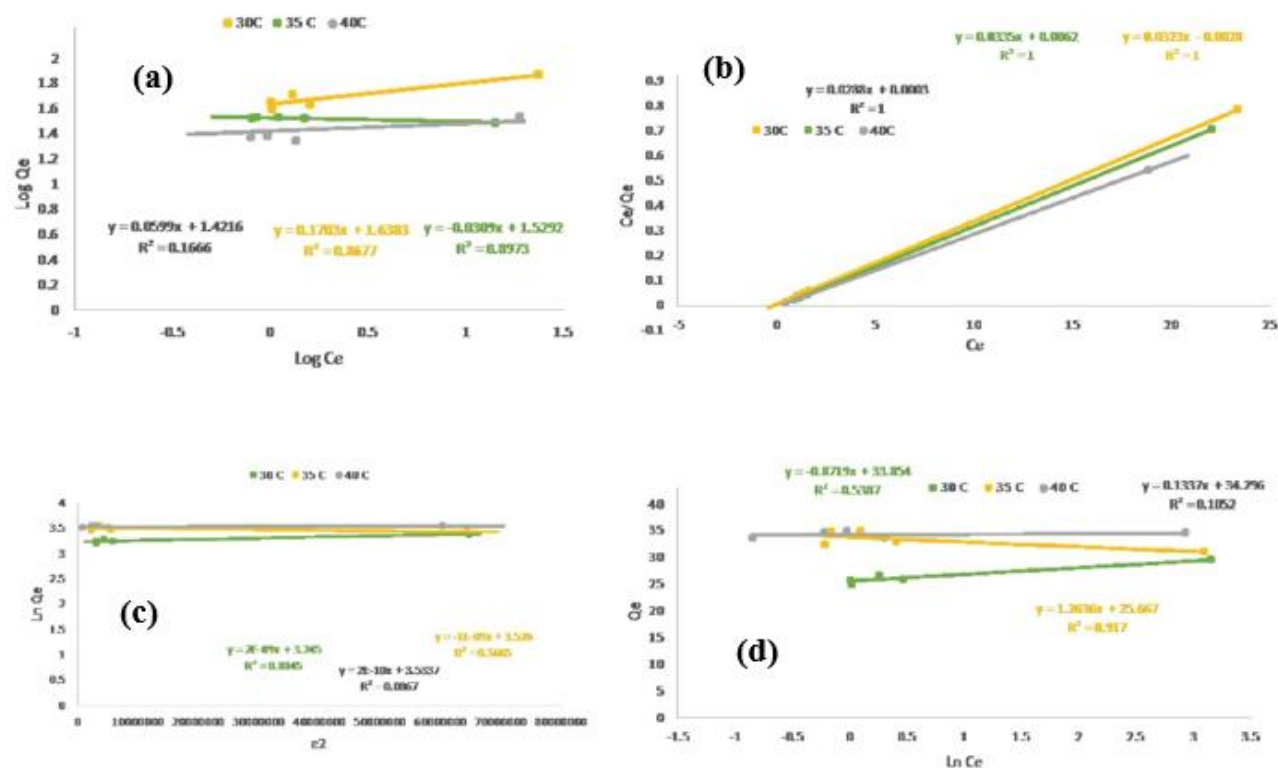


Figure 6. a, b, c and d shows frendlich , Langmiur, D-R and Temkin isotherm model respectively

Isotherm model study:

The adsorption of (MG) dye on the nanoparticles was studied using isotherms. Temkin, Freundlich, Langmuir, and D-R

isotherm models were used to study the adsorption equilibrium at various temperatures.

According to the experimental data, the MG-Cu system closely followed the Langmuir and Freundlich model at 30 and 35

degrees Celsius. The R^2 value of Langmuir and the model also confirmed its applicability, which was observed to be (0.99998762, 0.99998797) and (0.86766939, 0.89725415) respectively.

In Table 3 represent the Freundlich model was well followed by [MG]-Cu at different temperatures. The observed R^2 value was found to be (0.86766939, 0.89725415) respectively. In Table 3 represent the D-R model was well followed by [MG]-Cu at [30° C -35° C]. The R^2 value which was observed to be (0.8845, 0.5664) respectively. Figure 6 shows graphical representation of isotherm models.

Normal probability:

Normal probability plot of the current studies were set and presented in Figure 5 show that all the points for the removal of MG dyes respectively are well within 95% confidence level. The symmetrical arrangement of the data points around the central line, which depicts the linear positive relationship, and the absence of any points outside the bounds demonstrate the model's relevance. Since no outlier was found outside the data's 95% confidence level, this figure demonstrates how well the CCD utilized in the design suited the research.

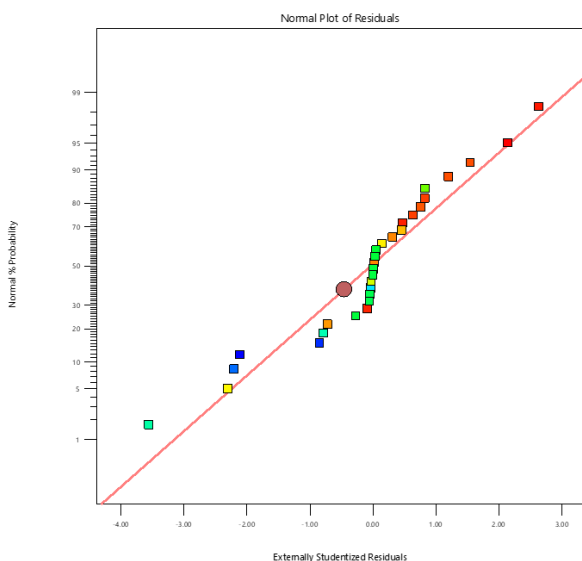


Figure 5. Show normal probability graph.

CONCLUSION

Using a green approach, the study successfully produced Cu NPs, and then used SEM, FTIR, and EDS to examine their shape, structure, and chemical studies. The size of the NPs varied from 82 to 152 nm. Following their synthesis, these NPs were employed as an adsorbent in an ultrasonication-assisted adsorption procedure to remove MG dye. Four parameters were used in the experiment with the help of CCD: time, pH, amount of adsorbent, and dye concentration. It was discovered that 2.4 minutes, pH 4.3, 0.045 g of adsorbent, and a dye concentration

of 15.0385 mg/L were the ideal operating parameters for MG removal, yielding a 98.99% removal of dyes solution. The results of an ANOVA study indicated that the variables pH, adsorbent quantity, and duration were important. Freundlich, Langmuir, D-R, and Tempkin isotherm models were used to study the adsorption equilibrium, and pseudo second order kinetics were used to remove the dye. Additionally, thermodynamic characteristics for the MG dye adsorption onto the Cu NPs were assessed. Overall, the findings point to the viability of the suggested green technique for eliminating MG dyes from aqueous solutions.

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