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Removal of Congo Red Dye Using *Ficus benghalensis*-Assisted Copper Oxide Nanoparticles

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Authors' contributions

This work was carried out in collaboration among all authors. Author WH did the conceptualization, investigation, supervision of the manuscript. Authors TS and AY did the synthesis and characterization of the manuscript. Authors ZY and MK did the adsorptive analysis of the manuscript. Authors WH, FUN and NA did the review, editing and authors WH, TS, FUN and AY wrote the protocol and wrote the first draft of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Novel water purification technologies are emerging to satisfy the requirement of clean drinking water for human life. The development of long-term water treatment systems is a serious concern. In this regard, nanotechnology holds enormous promise for improving the effectiveness and

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efficiency of polluted water remediation. The goal of this work was to create a simple and effective water purification system that used cost-effective, durable, and environmentally friendly novel materials. Green nanoparticle synthesis has emerged as a result of recent advancements in environmentally friendly technologies. The use of plant extract to synthesize nanoparticles is thought to be an incredible green strategy.

In the present study, leaf extract of *Ficus benghalensis* was chosen to synthesize the low cost and novel copper oxide (CuO) nanoparticles. The aim was to investigate the ability of these novel nano particles in removing Congo red dye from water. Various parameters including contact time, adsorbent dosage, initial dye concentration and pH of dye solution that affect the adsorption were optimized by carrying out batch adsorption experiments. The mechanism of pseudo-first-order, pseudo-second-order, Langmuir equilibrium isotherm, and Freundlich isotherm was investigated using kinetic and equilibrium experimental data. These models gave an insight into the adsorption kinetics and adsorption mechanism. The pH effect revealed that Congo red dye being an acidic dye showed good adsorption at acidic pH. The findings of this work show that copper oxide nanoparticles are promising adsorbents for the removal of Congo red dye from aqueous based system.

Keywords: Nanotechnology; green synthesis; copper oxide nanoparticles; Ficus benghalensis; Congo red; adsorption; kinetics; isotherm.

1. INTRODUCTION

Water is the most important component of all life on Earth, as well as a source of human civilization's evolution. Water is regarded as a universal liquid because it dissolves a wide range of compounds. Water contamination is a serious issue and challenge for the world in the twenty-first century. Water resources are polluted by a variety of sources, including commercial, residential areas, agricultural and industrial activities etc [1]. Population expansion, more strict health-based laws, and economic development have all raised focus on good quality water [2]. Dyes are one of the principal contaminants that harm the marine systems among the different organic pollutants found in wastewater. Dye containing wastewater effluents pose human health and environmental hazards [3]. Removal of dyes has received a lot of attention in recent decades before being released into the natural streams. Membrane filtration, coagulation, electrochemical procedures, biological treatment, chemical oxidation and adsorption strategies have all been employed to remove dyes [4]. The process of adsorption is the most common of these approaches because it is a cost-effective, simple-to-use method for removing practically all kinds of pollutants. As a result, this method is frequently used to remove not only pigments and dyes from waste water and sewage, but also other contaminants like heavy metals [5].

Numerous novel adsorbents, including zeolites/clay and their composites, bio-adsorbents, agricultural solid waste, commercial by-products and their composites, and various materials have been synthesized and modified for removal of dyes from wastewater over the last few decades [6]. A huge variety of nano adsorbents are being used for contaminated water purification. Nano-adsorbents are nanoparticles derived from inorganic or organic origins with a high adsorption affinity. They are particularly successful at separating a wide variety of pollutants of varied molecular sizes due to their high porosity, good specific surface area, and active surface. The nano-adsorbents act effectively and quickly, with no hazardous substances released [7]. Copper oxide nanoparticles display the characteristics of precious metals like gold, silver and platinum because of non-expensive and catalytic characteristics [8]. Due to its numerous potential applications, researchers have become interested in the synthesis of copper oxide nanoparticles, a semi-conductive material with a variety of electrical, magnetic and optical properties [9,10]. Physicochemical processes can be used to synthesize these nanoparticles. These processes, however, are costly and have a number of drawbacks, such as the use of hazardous solvents, the surface structure's imperfection and the generation of toxic by-products [11]. In comparison to these synthetic processes, bio-inspired approaches are more cost-effective and limit the use of harmful chemicals, and also high pressure, temperatures and energy [12]. Recently, CuO nanoparticles have been prepared using *Wedelia urticifolia*, *Eucalyptus globoulus, Aloe barbadensis miller* *and Punica granatum* [9,13-15]. In this work, we have employed *Ficus benghalensis* leaf extract as a reducing agent for the production of copper oxide nanoparticles. Extensive research was conducted on the synthesis, characterization and application of copper oxide nanoparticles. We also investigated the adsorption chemistry and optimized the removal of Congo red dye using copper oxide nanoparticles. The adsorption process was elucidated through kinetics and isotherm analysis.

2. EXPERIMENTAL DETAILS

2.1 Material Synthesis

Green synthesis method was adopted to synthesize CuO nanoparticles using *Ficus benghalensis* leaf extract as a reducing agent. The *Ficus benghalenasis* leaf powder was prepared using dried leaves of *Ficus benghalensis*. First the *Ficus benghalensis* leaf extract was prepared by shaking 10 g leaf powder in 100 ml distilled water. The solution was sonicated for two hours at room temperature. The sonicated solution was filtered to get *Ficus benghalensis* leaf extract. 40 ml of *Ficus benghalensis* leaf extract was then heated using magnetic stirrer at 60-70 ^oC. 1 g copper nitrate trihydrate was added to the heated extract. The hue of the solution changed to bluish green tone very immediately, suggesting the production of copper oxide nanoparticles. The solution was further heated to 60-70 ^oC till the formation of thick paste. The paste was then calcinated in muffle furnace at 330 ^oC for 30 minutes. Finally black colored CuO nanoparticles were obtained [16].

2.2 Batch Adsorption

The batch adsorption approach was adopted to investigate the adsorption of Congo red dye onto CuO nanoparticles. In each batch adsorption study, 40mL of a known concentration of Congo red dye solution and a known quantity of nanoparticles were introduced in 50 ml shaking tube. The samples were agitated using orbital shaker at 300 rpm for specific time interval. After shaking for specific time interval, the samples were taken, centrifuged for 10 min at 3800 rpm, and the dye solution was evaluated for remaining concentration of Congo red dye at 498 nm (λ_{max} of Congo red dye) using UV-Visible spectrophotometer.

The percentage removal of dye and amount of dye adsorbed were determined by using equation (1) and equation (2) respectively [17].

$$\%\, age\ Removal\ = \frac{(C_o - C_e)}{C_o} \times 100 \qquad (1)$$

$$q_e\ = (C_o\ - C_e) \times \frac{V}{m} \qquad (2)$$

Here,

C_o (mg/L) = Initial dye concentration
C_e (mg/L) = Final dye concentration
V (L) = Dye solution volume in liter
m (g) = Mass of adsorbent

3. RESULTS AND DISCUSSION

3.1 Characterization of Copper oxide Nanoparticles

3.1.1 UV-visible spectroscopy

Fig. 1. illustrates the UV-Visible spectrum of *Ficus benghalensis* assisted CuO nanoparticles. There are two peaks of maximum absorption. One prominent peak is obtained around 250 nm, while at 337 nm a weak peak is obtained. These peaks indicate the synthesis of CuO nanoparticles using *Ficus benghalensis*. There was a noticeable color change, transitioning from blue to bluish green during the synthesis of CuO nanoparticles. This alteration in color can be attributed to the surface plasmon resonance of copper oxide nanoparticles [18].

3.1.2 Fourier transform infrared spectroscopy

The functional groups presented in *Ficus benghalensis* leaf extract and *Ficus benghalensis* assisted copper oxide nanoparticles were identified using FTIR. Fig. 2 a) shows the FTIR spectrum of *Ficus benghalensis* leaf extract. Its FTIR spectrum shows prominent peaks at 3414,1600 and 1414 cm^{-1}. The broad peak at 3414 cm^{-1} corresponds to stretching vibration of O-H bond. The peak observed at 1600 cm^{-1} corresponds to stretching vibration of C=O bond. Additionally, the peaks obtained in the vicinity of 1414 cm^{-1} can be attributed to the C-H vibration [19,20]. In Fig. 2 b), FTIR spectrum of *Ficus benghalensis* assisted CuO nanoparticles is shown. The broad peak at 3100 cm^{-1} is indicative of O-H stretching vibrations. Another peak at 1537 cm^{-1} corresponds to C=O stretching vibrations. The presence of peaks in the range of 400 to 700 cm^{-1} is characteristic of Cu-O vibrations, confirming the formation of CuO nanoparticles.

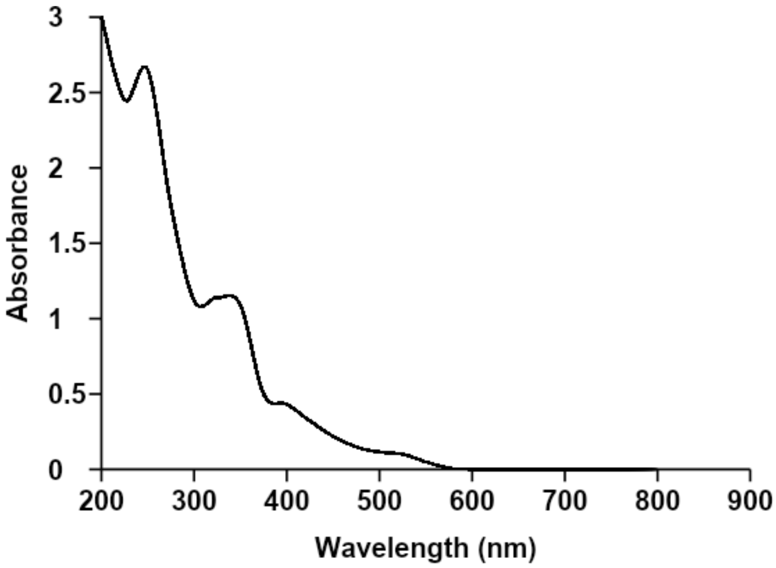
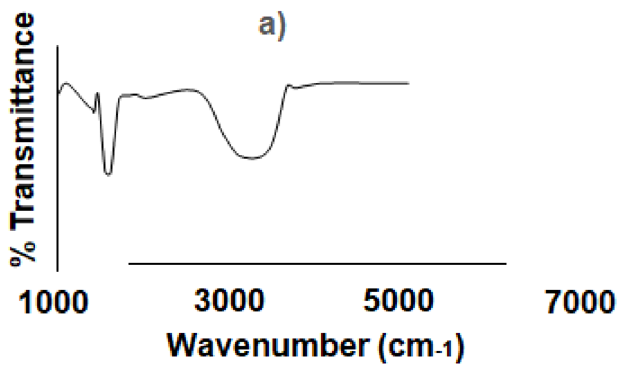


Fig. 1. UV-Visible spectrum of CuO nanoparticles synthesized using *Ficus benghalensis* leaf extract



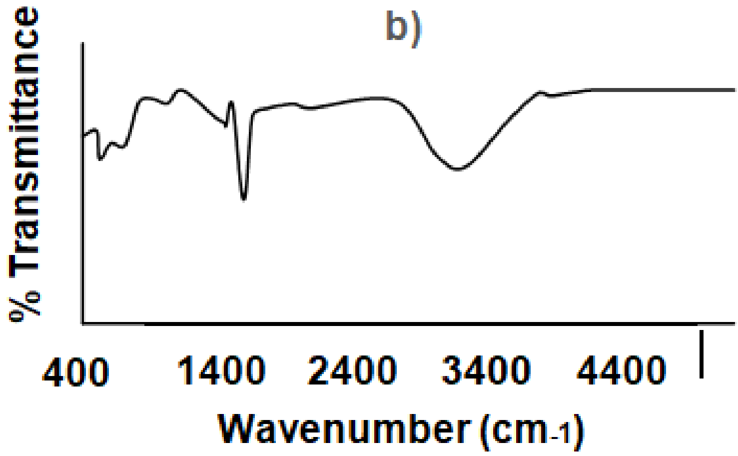


Fig. 2. a) FTIR spectrum of *Ficus benghalensis* b) FTIR spectrum of *Ficus benghalensis* assisted CuO nanoparticles

3.1.3 Scanning electron microscopy

SEM was used to assess the morphological characteristics of the produced CuO NPs. Fig. 3 depicts the SEM image of synthesized CuO nanoparticles indicating the porous nature of CuO nanoparticles and the pores are in the nanometer range. This is in agreement with literature [21,22].

3.2 The Potential of Copper Oxide Nanoparticles in Congo Red Dye Adsorption

The ability of CuO nanoparticles was determined for the Congo red dye adsorption from water. The influence of various variables including time of contact, nanoparticles dosage, initial dye concentration and dye solution pH were examined on the percentage removal of Congo red dye. The obtained results are discussed in the next following sections.

3.2.1 Effect of contact time

The effect of contact time on the removal efficiency of Congo red dye from water was studied using copper oxide nanoparticles. The study was carried out using fixed nanoparticles dosage (0.05 g) and fixed initial dye concentration (100 mg/L). Analysis of the data revealed that as the duration increased up to 30 minutes, the efficiency of removing Congo red dye exhibited a rise as shown in Fig. 4. Concurrently, the adsorption capacity also experienced an upward trend, increasing from 34 mg/g to 58 mg/g. Notably, beyond 30 minutes, the % removal efficiency began to decrease. This decline can be attributed to the initial stages, where a large number of unoccupied porous sites on CuO nanoparticles were available. As a result, adsorption was happening at a faster rate. However, with the passage of time, the majority of the sites used for adsorption were taken up by dye molecules, which caused the rate of adsorption to be slow [23]. Therefore, after careful evaluation, it was determined that the equilibrium point was reached at 30 minutes of contact time, where the adsorption efficiency of CuO nanoparticles was observed to be at its maximum. This is in agreement with the literature [24-26]. Consequently, for subsequent batch studies, the equilibrium contact time was established to be 30 minutes.

3.2.2 Effect of adsorbent dosage

The amount of adsorbent plays a crucial role in determining its capacity to adsorb a specific quantity of adsorbate under operational conditions. Assessing the impact of adsorbent dosage provides insights into the dye’s ability to be adsorbed using the minimum amount of adsorbent, thus enabling cost-effective evaluation of its potential [27].

The impact of adsorbent dosage on the elimination of Congo red dye was investigated within the range of 0.01 to 0.08 g using fixed dye concentration (100 mg/L) and fixed contact time (30 minutes). The analysis of adsorption outcomes as indicated in Fig. 5 revealed that the removal efficiency for Congo red dye ranged from 38 to 74 % as the adsorbent dosage increased from 0.01 to 0.05 g. When the adsorbent dosage was further increased to 0.08 g, there was decrease in Congo red dye removal as shown in Fig. 5. This can be attributed to the fact that at low adsorbent dosage, the active sites are greater in number. With the increase in adsorbent dosage, particle aggregation occurs resulting in less percentage removal [28].

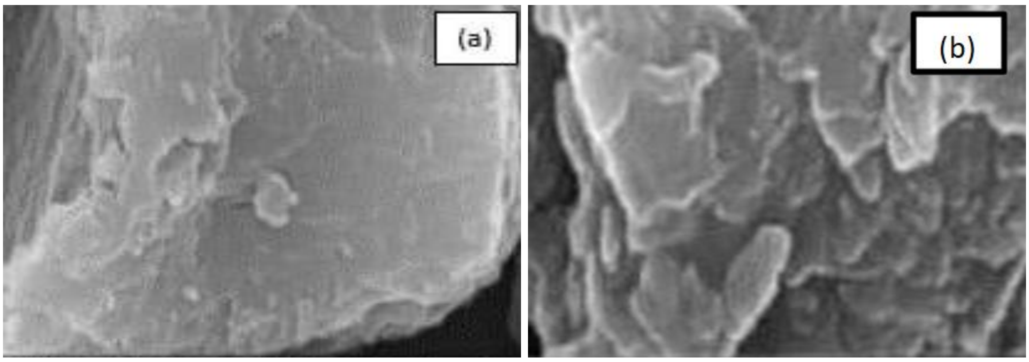


Fig. 3. SEM images under resolution (a) 40000 (b) 150000

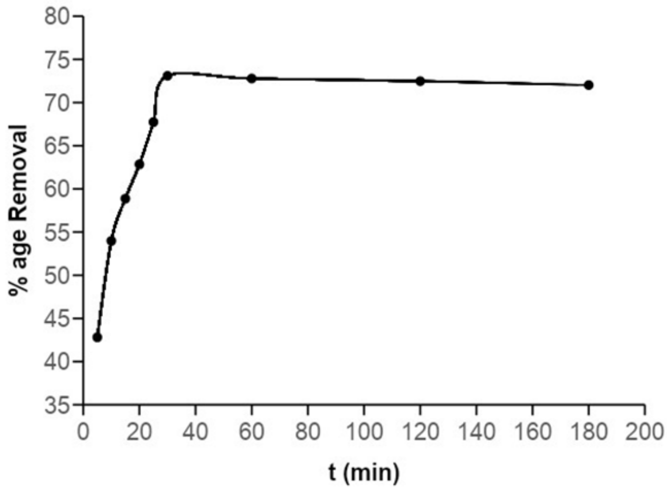


Fig. 4. Effect of contact time on percentage removal of Congo red dye

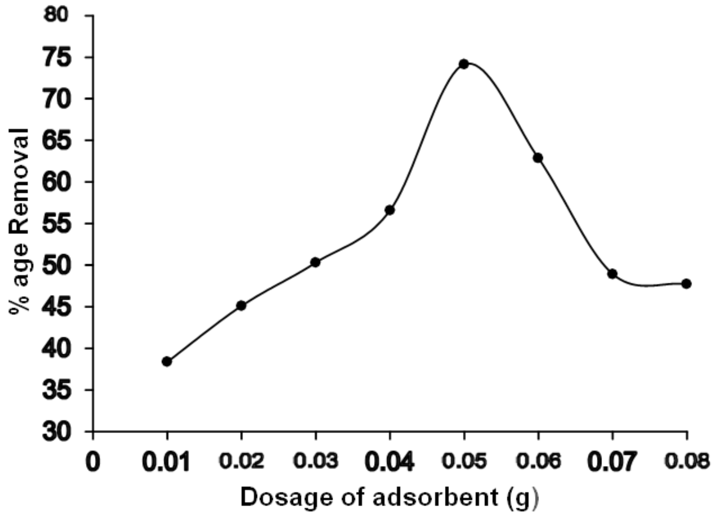


Fig. 5. Effect of CuO nanoparticles dosage on adsorption of Congo red dye

Literature survey indicated the similar trend for adsorbent dosage effect on adsorption of Congo red dye [29,30].

3.2.3 Effect of initial dye concentration

The influence of initial dye concentration on removal efficiency of Congo red dye is depicted in Fig. 6. The figure demonstrates a decrease in percentage removal of Congo red dye with an increase in the initial dye concentration. The percentage removal of Congo red dye was found to decrease from 87.5 to 71.1 % with the increase in initial dye concentration from 10 to 100 mg/L. The observed results can be explained by the presence of a limited number of active sites on the adsorbent. At a certain concentration, these sites become saturated, leading to a decrease in the percentage of removal [31].

3.2.4 Effect of pH of dye solution

The adsorption of the dye is influenced by the pH of the dye solution. The adsorption efficiency is pH dependent, as changes in pH affect both the ionization degree of the adsorbing molecules and the surface characteristics of the adsorbent [32]. The optimum pH for Congo red dye adsorption onto copper oxide nanoparticles was determined to be pH 2 as depicted in Fig. 7. A lower pH of dye solution appears to result in a higher percentage removal of Congo red dye owing to the attraction of anionic dye towards the positive charge of the adsorbent [33]. At high pH of dye solution, repulsion is operative between the surface of adsorbent which is negatively charged and Congo red dye as reported in literature [34]. Furthermore, dye anions and the hydroxide ions were in competition at high pH. Consequently, the percentage removal of Congo red dye was reduced by means of repulsion among the abundant hydroxide ions and anionic dye molecules [31].

3.3 Kinetic Models

Adsorption kinetic models reveal details on adsorption rate, the performance of adsorbents and the path of mass transfer. Understanding of adsorption kinetics is crucial for designing of adsorption systems.

Two adsorption kinetic models including pseudo first order model and pseudo second order model were employed to study the Congo red dye adsorption process onto CuO nanoparticles. The linear equation for pseudo first order kinetics is given below [35]:

$$In\,(q_e - q_t) = -K_1 t + ln q_e$$

Where q_e and q_t are adsorption capacity of CuO nanoparticles at equilibrium and time t respectively. The first order rate constant is described by K_1. For pseudo second order kinetics, the linear equation is given below [36]:

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{q_e^2 K_2}$$

Where K_2 represents the second order rate constant.

The rate constants (K_1 & K_2), calculated adsorption capacity at equilibrium (q_e cal) and experimental adsorption capacity at equilibrium (q_e exp) are described in Table 1. Based on the analysis of kinetic data shown in Fig. 8 a) and b), it was determined that the pseudo second order kinetic model provides a better fit compared to pseudo first order model. R^2 value of pseudo second order is found to be 0.9965. Moreover, the calculated value of q_e for pseudo second order model is closer to experimental value of q_e. This indicates that the pseudo second order model is appropriate to study the Congo red dye adsorption kinetics by CuO nanoparticles and Congo red dye is adsorbed onto the nanoparticles surface via chemical interaction [37].

3.4 Adsorption Isotherms

The adsorption isotherm is a crucial graphical representation that elucidates the mechanism by which a substance moves from an aquatic system to a solid phase under constant pH and temperature conditions [38]. Two well-known models including Freundlich and Langmuir are used in order to study the interaction between adsorbate and adsorbent [6]. Adsorption isotherm experiments were conducted in batch mode for Congo red dye using CuO nanoparticles. The batch experiment was done by introducing optimized adsorbent amount (0.05 g) to a set of shaking tubes containing 40 ml of Congo red dye solution ranging from 10 to 100 mg/L. The Langmuir model hypothesizes that adsorption occurs at distinct uniform sites within the adsorbent and has been effectively utilized in various monolayer adsorption studies. The linear equation for Langmuir isotherm is [39]:

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_L} + \frac{C_e}{Q_L}$$

Where C_e, q_e and Q_L represents the equilibrium concentration of dye (mg/L), amount of dye adsorbed per unit mass of adsorbent at equilibrium (mg/g) and maximum adsorption capacity (mg/g) respectively. K_L describes the Langmuir constant (L/mg). The slope and intercept of the graph between C_e/q_e and C_e can be used to calculate Q_L and K_L respectively. The Freundlich model takes into account a heterogeneous adsorption surface having unequal accessible sites with different adsorption energies [27]. For Freundlich adsorption isotherm, the linear equation is given below [39]:

$$ln q_e = \frac{1}{n} ln C_e + ln K_F$$

Where q_e and C_e describes the amount of dye adsorbed per unit mass of adsorbent at equilibrium (mg/g) and equilibrium concentration of adsorbate (mg/L) respectively. 1/n and K_F are Freundlich constants that represents the adsorption intensity and adsorption capacity of the system respectively. n and K_F can be calculated from the slope and intercept of the graph drawn between lnq_e and lnC_e. Based on the analysis of Fig. 9 a) and b) and Table 2, the R^2 value for Freundlich adsorption model was determined to be 0.99, which was higher compared to the Langmuir model. This suggests that the Freundlich adsorption model provides a better fit to the adsorption process and indicates the presence of a heterogeneous surface for the CuO nanoparticles [23, 40].

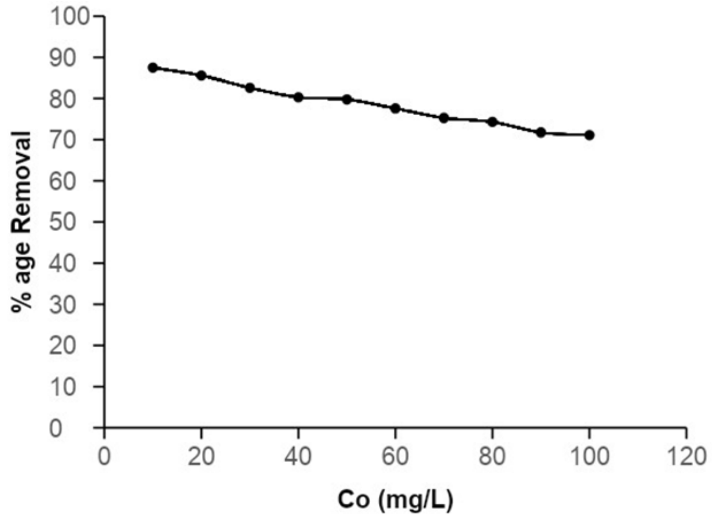


Fig. 6. Effect of initial dye concentration on percentage removal of Congo red dye

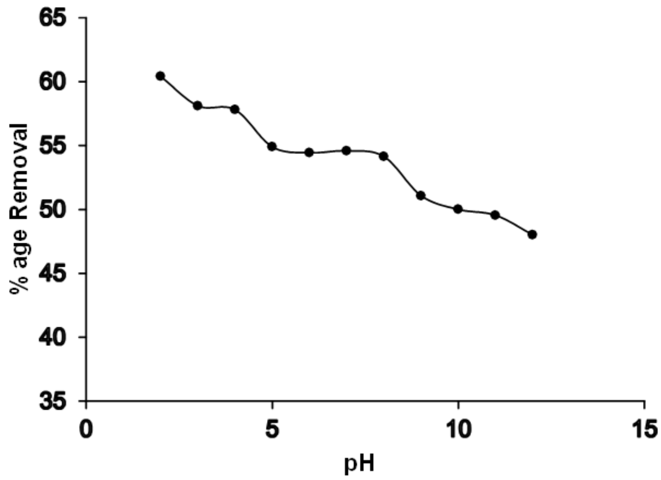
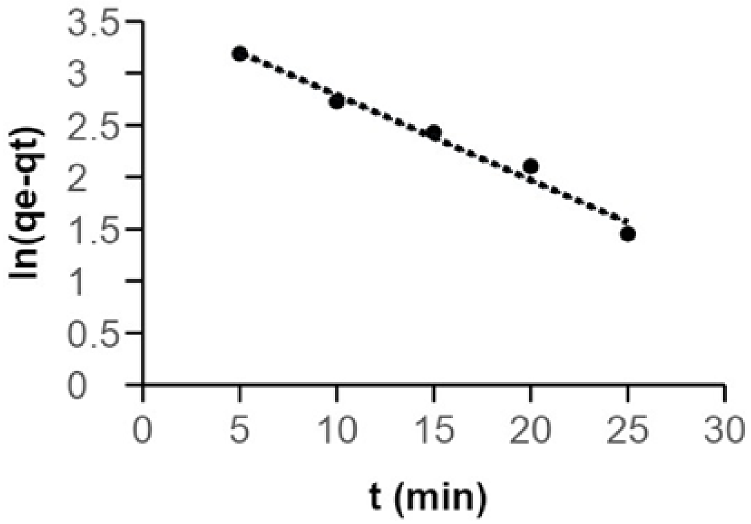


Fig. 7. Effect of pH of Congo red dye solution on percentage removal efficiency of dye



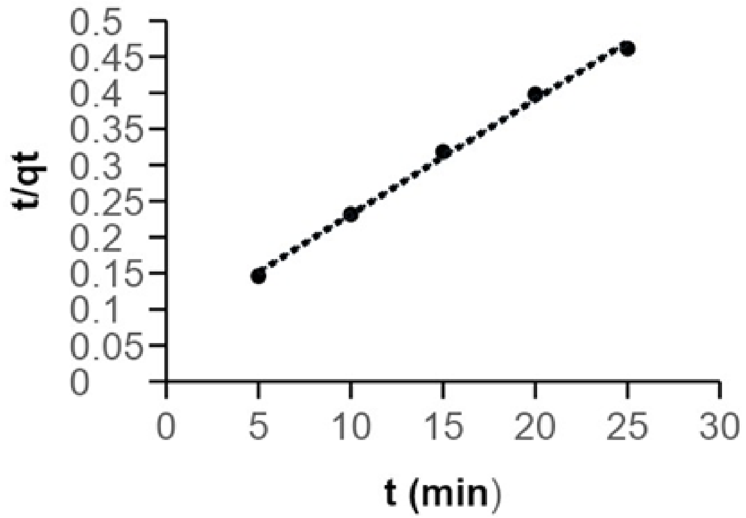


Fig. 8. a) Pseudo first order b) second order kinetic models for adsorption of Congo red dye onto CuO nanoparticles

Table 1. Data for pseudo first and second order for adsorption of Congo red dye onto CuO nanoparticles

	Pseudo first order model			Pseudo second order model		
q_e (exp) (mg/g)	q_e (cal) (mg/g)	K_1 (min^{-1})	R^2	q_e (cal) (mg/g)	K_2 ($g\ mg^{-1}\ min^{-1}$)	R^2
58	36.8774	0.0818	0.9791	62.8930	0.0035	0.9965

Table 2. Freundlich model parameters for Congo red dye adsorption onto CuO nanoparticles

Freundlich constant (n)	Freundlich constant (K_F)	Coefficient of determination (R^2)
1.524855	6.5417	0.9951

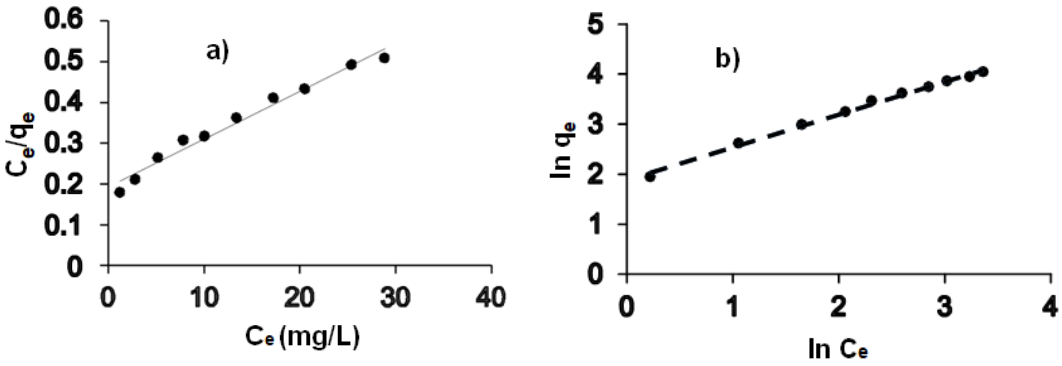


Fig. 9. a) Langmuir model b) Freundlich model for adsorption of Congo red Dye onto CuO nanoparticles

4. CONCLUSION

Effluents of textile industry contribute significantly to increased water contamination. These effluents contain a wide range of chemicals, including synthetic dyes, that are tough to breakdown using conventional approaches. A new field of science termed as nano science has thoroughly superseded the formerly utilized traditional technologies. Nanomaterials have the tendency to remove the organic matters especially dyes from contaminated water.

The biogenic fabrication of copper oxide nanoparticles was done using leaf extract of *Ficus benghalensis* without the usage of extra capping agents. The synthesized nanoparticles were characterized using UV-Visible spectroscopy, FTIR and SEM. The potential of the synthesized nanoparticles was investigated in the treatment of water contaminated with Congo red dye. The study of various parameters including contact time, adsorbent dosage, initial dye concentration and pH of dye solution have confirmed that copper oxide nanoparticles can be used as promising adsorbents in water purification protocols. For copper oxide nanoparticles, the equilibrium time was determined to be 30 minutes while the optimum adsorbent dosage was 0.05 g. The kinetic models reveal that the pseudo second order model is suitable to describe the adsorption kinetics. The analysis of the adsorption isotherm revealed that the Freundlich model provided a good fit to the adsorption data, indicating the occurrence of multi-layer adsorption of Congo red dye on the heterogeneous surface of CuO nanoparticles. Congo red being an acidic dye was found to show excellent adsorption at acidic pH i.e., pH 2. Hence, it is concluded that the green synthesized copper oxide nanoparticles are promising adsorbents in removing hazardous Congo dye from aqueous solution.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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