Synthesis of Mg-doped CuO Nanoparticles for Efficient Removal of Congo Red Dye from Wastewater: Adsorption study

ABSTRACT

Water contamination from a variety of sources has made it increasingly difficult to contact clean drinking water. The release of effluents into water bodies is a serious environmental problem. This study presents the synthesis of magnesium-doped copper oxide (Mg-CuO) nanoparticles as a promising adsorbent for the efficient removal of Congo Red dye from aqueous solutions. Mg-CuO nanoparticles were synthesized via a facile and cost-effective co-precipitation method and characterized using various techniques. The adsorption capacity of Mg-CuO nanoparticles for Congo Red dye was systematically investigated, revealing outstanding adsorption efficiency. Equilibrium adsorption data were well-fitted to the Langmuir isotherm model, suggesting monolayer adsorption behavior, while the Freundlich Isotherm model described the adsorption behavior accurately. The influence of various experimental parameters, including initial dye concentration, pH, adsorbent dosage, and contact time, on the adsorption process was studied in detail. Optimal conditions (pH=6, concentration=50ppm, contact time=120minutes) for maximal adsorption efficiency were determined. Moreover, the thermodynamic analysis specified that the adsorption of Congo Red onto Mg-CuO nanoparticles was spontaneous and endothermic. The exceptional adsorption performance of Mg-CuO nanoparticles, attributed to the synergistic effect of magnesium doping and high surface area, highlights their potential as an eco-friendly and efficient adsorbent for the removal of Congo Red dye from wastewater. This research contributes to the advancement of sustainable materials for environmental remediation and underscores the importance of exploring nanomaterials for water purification applications.

Keywords: Nanotechnology, CuO nanoparticles, Adsorption, Coprecipitation method, Doping, equilibrium modeling.

1. INTRODUCTION

Water is essential for life, and access to clean water is a fundamental human right. It is also a vital resource for multiple sectors, including those that produce food, drugs, and electronics [1]. However, the world is facing a growing water crisis [2], as freshwater resources are declining due to environmental change [3], population growth, and pollution [4]. One of the biggest challenges is that only 3% of the Earth's water is freshwater, and most of this is locked up in glaciers and ice caps [5]. The most readily available source of freshwater is seawater [6], but it is too salty to drink or use for most industrial purposes. Water effluents are wastewater that is discharged from homes, businesses, and industries [7]. They can contain a variety of pollutants, including organic matter, nutrients, heavy metals, and pathogens [8]. Water effluents can have a negative impact on the environment and human health if they are not properly treated [7]. There are various ways to treat water effluents, including physical, chemical, and biological methods [9]. Physical methods, such as sedimentation and filtration, can remove suspended solids and other large particles from the water [10]. Chemical methods, such as coagulation and disinfection, can remove dissolved pollutants and kill harmful bacteria. Biological methods, such as activated sludge and trickling filters, can remove organic matter and nutrients from the water [11].

The type of treatment that is used for water effluents depends on the specific pollutants that are present in the wastewater [12]. For example, if the wastewater contains a lot of organic matter, then a biological treatment method would be most effective. If the wastewater contains a lot of heavy metals, then a chemical treatment method would be most effective. Biological treatment methods, such as activated sludge and trickling filters, are very effective at removing organic matter from water effluents. Organic matter is broken by bacteria in these systems, which produces carbon dioxide and water [13]. Nutrients, such as nitrogen and phosphorus, can be removed from water effluents using a variety of methods, including chemical precipitation, biological nutrient removal, and ion exchange. Heavy metals can be removed from water effluents using chemical precipitation, ion exchange, and adsorption. Pathogens [14], such as bacteria and viruses, can be removed from water effluents using disinfection [15]. Disinfection can be achieved using a variety of methods, including chlorination, ozonation, and ultraviolet radiation. The specific treatment methods that are used for water effluents will depend on the specific pollutants that are present in the wastewater, the desired removal efficiency, and the cost of the treatment process [16].

Nanotechnology is the manipulation of matter at the atomic and molecular level to create new materials and devices with unique properties [17]. It has the potential to revolutionize various industries, including water treatment [18]. Nanomaterials have a number of advantages over conventional materials for water treatment. They are very small in size, with a large surface area to volume [19]. This makes them very effective at adsorbing and removing contaminants from water. Nanomaterials can also be functionalized to target specific contaminants [20]. Nanotechnology is already being used in a variety of ways to treat wastewater. For example, nanoparticles can be used to remove heavy metals, organic pollutants, and bacteria from water. Nanotechnology can also be used to develop new water filters and membranes that are more efficient and durable than conventional filters and membranes. Nanotechnology is a promising new technology for water treatment. It has the potential to help us solve the global water crisis by providing us with new and innovative ways to clean and reuse wastewater [21].

Dye-based color repels have several advantages over pigment-based color repels [22]. Due to their high solubility in solvents, dye-based repels rarely do not require additional dispersion steps [23]. Additionally, they have superior transmittance, purer color, and sharper absorption bands than pigments due to their lower particle masses. The textile dyeing industry produces a significant amount of colored wastewater, which is a major source of water pollution [24]. More than 15% of dyes are lost during the textile manufacturing process, contributing to the colorful wastewater streams. Textile wastewater is one of the most severely polluted fluids that need to be treated due to its unique properties. It contains a variety of chemicals, including dyes and inorganic compounds. These chemicals can pose major risks to the environment and human health if not properly handled. Organic dyes are particularly harmful because they are often toxic and carcinogenic [25]. They can endanger marine life, microorganisms, and human health. Textile wastewater treatment is a complex and challenging process. There are a variety of methods that can be used, but they are all expensive and require specialized expertise [26].

Adsorption is a process that occurs when a substance (adsorbate) accumulates on the surface of another substance (adsorbent) [27]. This process is driven by surface forces, such as van der Waals forces or chemical bonding. Adsorption is a versatile and effective process for removing contaminants from wastewater [28]. It is relatively inexpensive, easy to use, and environmentally friendly. Adsorption methods can be classified into two types: physical adsorption and chemical adsorption. In physical adsorption, the adsorbate is held to the adsorbent by weak van der Waals forces. This type of adsorption is reversible, meaning that the adsorbate can be released from the adsorbent by changing the conditions, such as temperature or pressure. In chemical adsorption, the adsorbate is held to the adsorbent by strong chemical bonds. This type of adsorption is irreversible, meaning that the adsorbate cannot be released from the adsorbent without breaking the chemical bonds [29]. Adsorption is a promising technology for wastewater treatment. It has the potential to remove a wide range of contaminants from wastewater, including heavy metals, organic pollutants, and bacteria [30].

Dye adsorption is a process of removal of dye molecules from wastewater using a solid adsorbent [31]. This process is popular because it is simple, flexible, and easy to operate. It is also relatively inexpensive, insensitive to harmful contaminants, and does not produce any hazardous compounds. Ideal adsorbents for dye removal are able to function under a variety of wastewater parameters, are cost-effective, able to remove a variety of contaminants, high adsorption capacity, highly selective for different concentrations, large surface area, durable and reusable. A variety of adsorbents can be used to treat wastewater containing dyes, including bio-sorbents, carbon-based nano-adsorbents [32], and polymer-based adsorbents. Activated carbon-based adsorbents [33] are widely studied in this field due to their excellent chemical strength, low density, structural flexibility, and suitability for field-scale applications. Natural and synthetic clays are also frequently used as adsorbents for dye removal from aqueous solutions due to their widespread availability, low cost, high effectivity [34].

Congo red is a water-soluble azo dye that has been used for a variety of industrial and commercial purposes [35], including textile dyeing, papermaking, and leather tanning. However, Congo red is also a known carcinogen and toxic pollutant [36] and its release into the environment can have serious adverse consequences. Congo red can enter the environment through a variety of pathways, including wastewater discharges from industrial and commercial facilities, runoff from agricultural fields fertilized with sewage sludge, and leaching from landfills. Once in the environment, Congo red is highly persistent and can bioaccumulate in aquatic organisms [37]. Exposure to Congo red can cause a variety of health problems in humans and animals, including cancer, liver damage, kidney damage, and neurological disorders. Congo red is also a known endocrine disruptor, and exposure can interfere with hormone function and reproduction.

The co-precipitation method is a simple, inexpensive technique for producing metal-doped nanoparticles [38]. It involves mixing atomic particles to create compounds with perfect stoichiometry at low temperatures. This method can be used to create drug delivery systems by encapsulating drugs in nanoparticles, or to remove contaminants from water or air by precipitating them out of solution. Co-precipitation is commonly used in industrial applications due to its simplicity and effectiveness in controlling the morphology of the nanoparticles [39].

CuO NPs have a variety of unique properties [40], including high surface area, high reactivity, and excellent electrical and thermal conductivity. These properties make CuO NPs promising candidates for a wide range of applications, including water purification, energy storage, and catalysis. In water purification, CuO NPs are used to remove a variety of contaminants from water, including heavy metals, dyes, and organic pollutants [41]. CuO NPs are also used to disinfect water by killing bacteria and other microorganisms. In energy storage, CuO NPs are used to develop lithium-ion batteries and supercapacitors [42] with high energy density and power density. CuO NPs are also used towards development of solar cells and other photovoltaic devices with high efficiency. In catalysis, CuO NPs can be used to catalyze a variety of chemical reactions, including the hydrogenation of unsaturated hydrocarbons, the oxidation of carbon monoxide, and the reduction of nitrogen oxides. CuO NPs can be synthesized using a variety of methods, including chemical precipitation, sol-gel synthesis, and hydrothermal synthesis. The synthesis method used will affect the size, shape, and crystallinity of the CuO NPs [43].

Doping is a method of improving the properties of materials by introducing impurities into their crystal lattice [44]. Doping is widely used in a variety of industries, including catalysis, electronics, and biomedicine. Metal-doped nanoparticles are particularly useful for these applications because their properties can be tailored to meet specific needs. Copper-doped iron oxide nanoparticles have been shown to be effective in removing organic dyes from wastewater [45].

The aim this research is to synthesize Mg-doped CuO nanoparticles by coprecipitation method. Different parameters of pH, Concentration and contact time will be applied to check the maximum removal efficiency of Red Congo Dye in wastewater. Equilibrium modeling will be applied to ensure the excellent results.

2. METHODOLOGY

2.1 Material Synthesis

In the co-precipitation method, aqueous solutions of copper and magnesium salts are mixed together in the presence of a base, such as sodium hydroxide. This results in the formation of a precipitate of Mg-CuO nanoparticles. The precipitate is then filtered, washed, and dried to obtain the desired Mg-CuO nanoparticles. In the present research, copper nitrate trihydrate ($Cu(NO_3)_2{\cdot}3H_2O$) and magnesium nitrate hexahydrate ($Mg(NO_3)_2{\cdot}6H_2O$) was dissolved in deionized water to acquire two separate solutions. Then magnesium nitrate solution was added to the copper nitrate solution under vigorous stirring. Sodium hydroxide (NaOH) solution was added to the mixed solution until the pH reaches 10. Stirring was continued for 30 minutes to ensure complete precipitation. The precipitate was filtered and washed thoroughly with deionized water. The precipitates were dried in an oven at 100°C for 2 hours [46].

2.2 BATCH ADSORPTION

To prepare a stock solution of Congo Red dye, 1g of the dye was dissolved in distilled water and the volume was made up to 1000ml in a measuring flask. Different ppm solutions, including 10ppm, 20ppm, 30ppm, 40ppm, and 50ppm, were prepared from the stock solution using the dilution formula ($C_1V_1=C_2V_2$). For Mg-doped CuO nanoparticles, the pH was adjusted using 0.1 M NaOH and 0.1 M HCl. Adsorption tests were carried out to determine the removal efficiency of Mg-doped Copper oxide nanoparticles. Different parameters of dosage amount, concentration, pH, and contact time were investigated with all adsorbent doses of 0.05g.

Adsorption tests were performed by taking 50ml of working solution with specific pH in 250ml conical flasks. All the conical flasks with samples were placed in an orbital shaker for 90 min at the speed of 110rpm. The dose rate effect was investigated with all adsorbent doses of 0.05g, 0.07g, 0.1g, 0.2g, 0.3g, and 0.5g. For Mg-doped CuO nanoparticles adsorption, the pH effect was observed at pH 2. The effect of the initial dye concentration was investigated at 10ppm, 25ppm, 50ppm, 75ppm, 100ppm, and 150ppm. Contact time was varied at the interval of 5, 10, 15, 20, 30, 45, 60, and 90 minutes. Temperature was varied at 30, 35, 40, 45, 50, and 60 ˚C

3. RESULTS AND DISCUSSION

3.1 CHARACTERIZATION OF NANOPARTICLES

3.1.1 Uv- Visible Spectroscopy

Ultraviolet-visible (UV-vis) spectroscopy is a versatile technique that can be used to characterize the optical properties of materials, including Mg-doped CuO nanoparticles. UV-vis spectroscopy measures the absorption of light by a material at different wavelengths. The absorption spectrum of a material are used to identify the different electronic transitions that occur within the material. Mg-doped CuO nanoparticles have a characteristic absorption peak in the UV-vis spectrum at around 320 nm [47]. This peak is attributed to the electronic transition from the valence band to the conduction band of CuO. The position and intensity of this peak can be affected by the doping concentration of Mg. For example, studies have shown that the absorption peak shifts to a higher wavelength and the intensity of the peak decreases as the Mg doping concentration increases. UV-vis spectroscopy can also be used to determine the band gap energy of Mg-doped CuO nanoparticles. The band gap energy is the energy difference between the valence band and the conduction band of a material. It is an important parameter for determining the electrical conductivity and optical properties of a material. To determine the band gap energy of Mg-doped CuO nanoparticles, the Tauc plot of the absorption spectrum can be used. The Tauc plot is a plot of the square root of the absorption coefficient versus the photon energy. The band gap energy can be determined by extrapolating the linear portion of the Tauc plot to the x-axis. Studies have shown that the band gap energy of Mg-doped CuO nanoparticles decreases as the Mg doping concentration increases. This is because the Mg dopant introduces new energy levels into the band gap of CuO. Overall, UV-vis spectroscopy is a powerful tool for characterizing the optical properties of Mg-doped CuO nanoparticles. The information obtained from UV-vis spectroscopy can be used to optimize the synthesis and processing of Mg-doped CuO nanoparticles for specific applications.

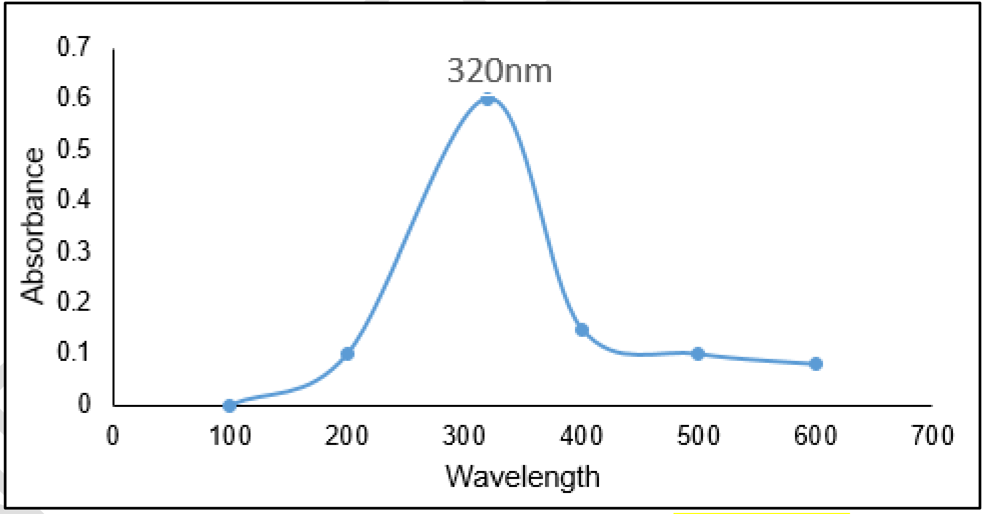

Fig. 1 UV- Vis Spectrum of Mg doped CuO Nanoparticles

3.1.2 Scanning Electron Microscopy

Scanning electron microscopy (SEM) images of Mg-doped CuO nanoparticles can reveal a variety of morphological features, depending on the synthesis method and doping concentration. For example, SEM images of Mg-doped CuO nanoparticles prepared by the hydrothermal method have shown that the nanoparticles can have a variety of shapes, including spherical, rod-shaped, and flower-like. The size of the nanoparticles can also vary depending on the synthesis conditions, but is typically in the range of 10-100 nanometers. SEM images can also be used to assess the dispersion of Mg-doped CuO nanoparticles in a matrix. For example, SEM images of Mg-doped CuO nanoparticles dispersed in a polymer matrix have shown that the nanoparticles can be evenly dispersed throughout the matrix. This is important for ensuring that the nanoparticles can effectively perform their desired function, such as enhancing the electrical conductivity of the polymer. Overall, SEM images can be a valuable tool for characterizing the morphology and dispersion of Mg-doped CuO nanoparticles. This information can be used to optimize the synthesis and processing of these nanoparticles for specific applications [46].

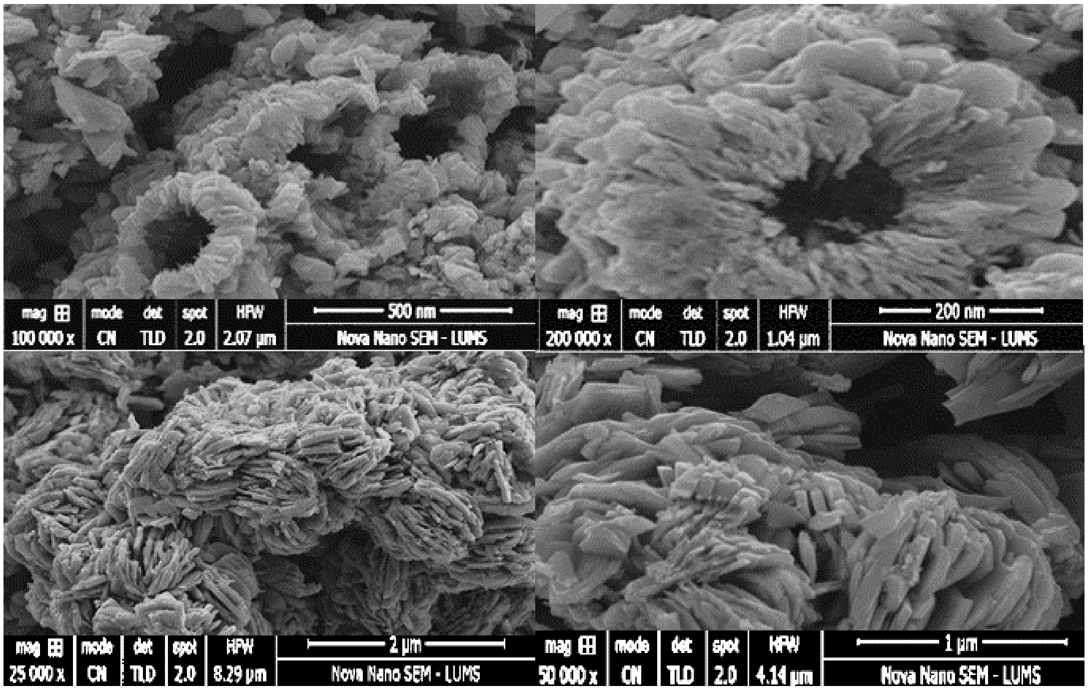

Fig. 2 SEM images of Mg-doped CuO Nanoparticles

3.2 Effect of pH

The pH of the solution has a significant effect on the adsorption of dye onto Mg-doped CuO nanoparticles. This is because the pH of the solution affects the surface charge of the Mg-doped CuO nanoparticles and the ionization state of the dye molecules. At low pH, the surface of the Mg-doped CuO nanoparticles is positively charged, while the dye molecules are neutral or negatively charged. This electrostatic attraction between the positively charged Mg-doped CuO nanoparticles and negatively charged dye molecules enhances the adsorption process. At high pH, the surface of the Mg-doped CuO nanoparticles becomes negatively charged, while the dye molecules become negatively charged or neutral. This electrostatic repulsion between the negatively charged Mg-doped CuO nanoparticles and the negatively charged dye molecules inhibits the adsorption process. Therefore, the optimal pH for the adsorption of dye onto Mg-doped CuO nanoparticles is usually in the acidic range. However, the optimal pH may vary depending on the specific dye molecule being adsorbed. A study on the adsorption of Congo red dye onto Mg-doped CuO nanoparticles found that the maximum adsorption capacity was achieved at a pH of 6. Overall, the pH of the solution has a significant effect on the adsorption of dye onto Mg-doped CuO nanoparticles. It is important to optimize the pH of the solution to achieve maximum adsorption efficiency.

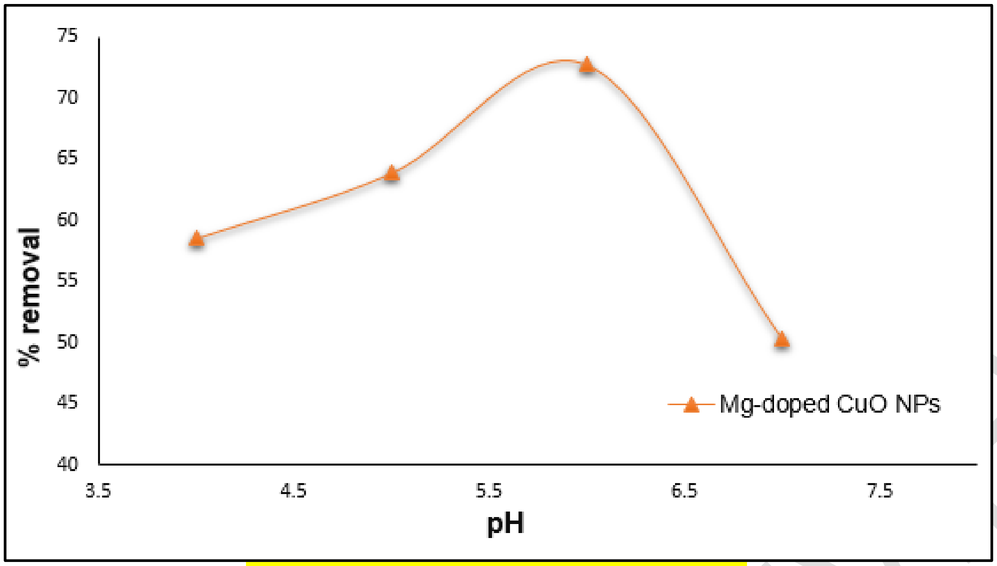

Fig. 3 Effect of pH on adsorption of Dye

Conditions of experiment

Adsorbents = Mg-doped CuO nanoparticles

pH for Dye = 6

concentration = 50ppm

Adsorbent quantity = 0.05g, 0.1g, 0.15g, 0.2g, and 0.25g.

time = 0 second to 1440 minutes

3.3 Effect of Contact Time

The contact time between the Mg-doped CuO nanoparticles and the dye solution has a significant effect on the adsorption of dye. This is because the contact time determines how long the dye molecules have to interact with the Mg-doped CuO nanoparticles and diffuse to the adsorption sites. At short contact time, there is less time for the dye molecules to interact with the Mg-doped CuO nanoparticles and diffuse to the adsorption sites. As a result, the adsorption efficiency is low. At long contact time, there is more time for the dye molecules to interact with the Mg-doped CuO nanoparticles and diffuse to the adsorption sites. As a result of more contact time, the adsorption efficiency increases. However, there is a limit to the increase in adsorption efficiency with increasing contact time. This is because all of the active adsorption sites on the surface of the Mg-doped CuO nanoparticles will be saturated at a certain contact time. Any additional contact time will not result in any further adsorption of dye molecules. Therefore, the optimal contact time for the adsorption of dye onto Mg-doped CuO nanoparticles is generally at a moderate level. However, the optimal contact time may vary depending on the specific dye molecule being adsorbed. A study on the adsorption of Congo red dye onto Mg-doped CuO nanoparticles found that the adsorption efficiency increased with increasing contact time, up to a point of saturation.

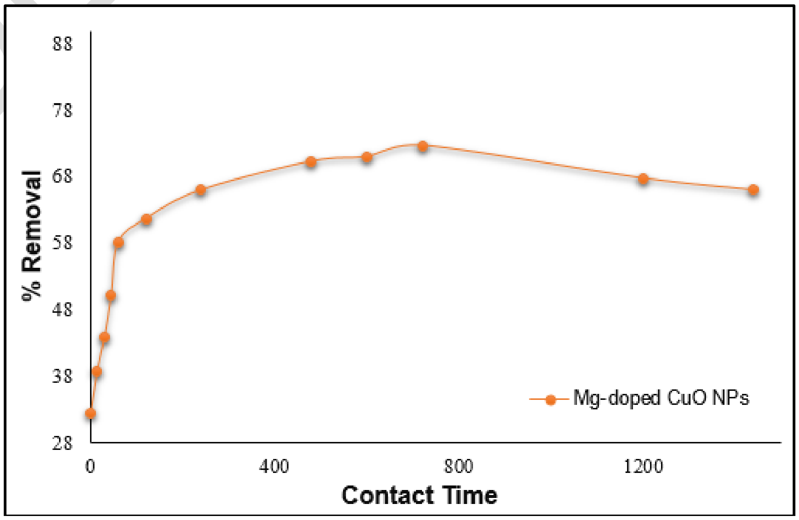

Fig. 4 Effect of Contact Time on adsorption of Dye

3.4 Effect of Concentration

The initial concentration of the dye solution has a significant effect on the adsorption of dye onto Mg-doped CuO nanoparticles. This is because the initial concentration of the dye solution affects the number of dye molecules available for adsorption. At low initial dye concentration, there are fewer dye molecules available for adsorption. As a result, the adsorption capacity of the Mg-doped CuO nanoparticles is high. At high initial dye concentration, there are more dye molecules available for adsorption. However, the adsorption capacity of the Mg-doped CuO nanoparticles is limited. As a result, the adsorption efficiency decreases. This is because all of the active adsorption sites on the surface of the Mg-doped CuO nanoparticles will be saturated at a high initial dye concentration. Any additional dye molecules will not be able to adsorb onto the Mg-doped CuO nanoparticles. Therefore, the optimal initial dye concentration for the adsorption of dye onto Mg-doped CuO nanoparticles is generally at a low level. However, the optimal initial dye concentration may vary depending on the specific dye molecule being adsorbed. A study on the adsorption of Congo red dye onto Mg-doped CuO nanoparticles found that the adsorption capacity decreased with increasing initial dye concentration.

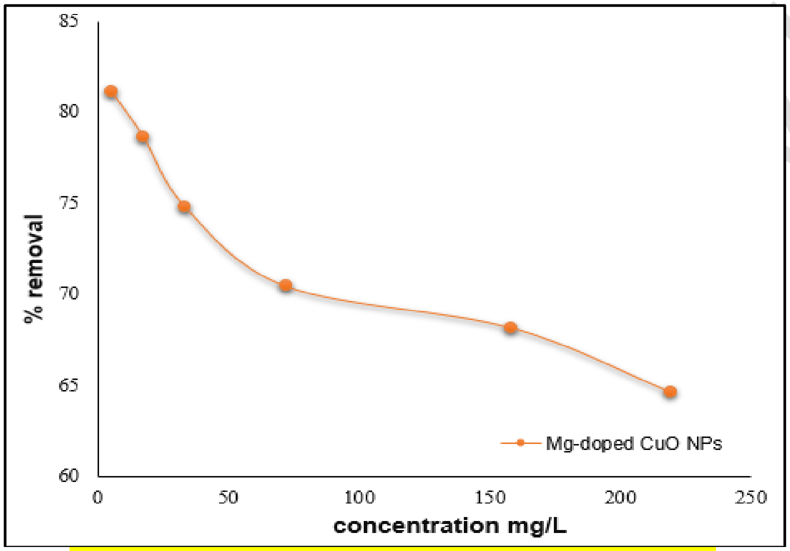

Fig. 5 Effect of Concentration on adsorption of Dye

4 EQUILLIBRIUM MODELS

4.1 Langmuir Adsorption Isotherm

The Langmuir isotherm model assumes that optimum adsorption is achieved when a single layer of solute molecules completely covers the adsorbent surface, and that there is no migration of adsorbate molecules across the surface plane. This model is based on the assumptions that the adsorbent surface is uniform and homogeneous. Each adsorption site can only accommodate one adsorbate molecule. Adsorption is reversible, meaning that both adsorption and desorption can occur simultaneously. There is no interaction between adsorbed molecules [48].

The Langmuir isotherm equation relates the concentration of adsorbate in the aqueous solution to the amount of adsorbate adsorbed on the adsorbent surface at equilibrium:

$$Ce/qe = 1/KLqm + Ce/qm$$

Where Ce is the equilibrium concentration of adsorbate in the aqueous solution (mg/L), qe is the amount of adsorbate adsorbed on the adsorbent surface at equilibrium (mg/g), qm is the maximum adsorption capacity of the adsorbent (mg/g), KL is the Langmuir adsorption constant (L/mg)

The Langmuir isotherm model is a useful tool for describing the adsorption of a wide variety of substances, including heavy metals, dyes, and organic pollutants. It is particularly useful for systems where monolayer adsorption is the dominant adsorption mechanism. Overall, the Langmuir isotherm model is a valuable tool for understanding and predicting adsorption behavior. However, it is important to note that the model is based on a number of assumptions, and it may not be applicable to all systems.

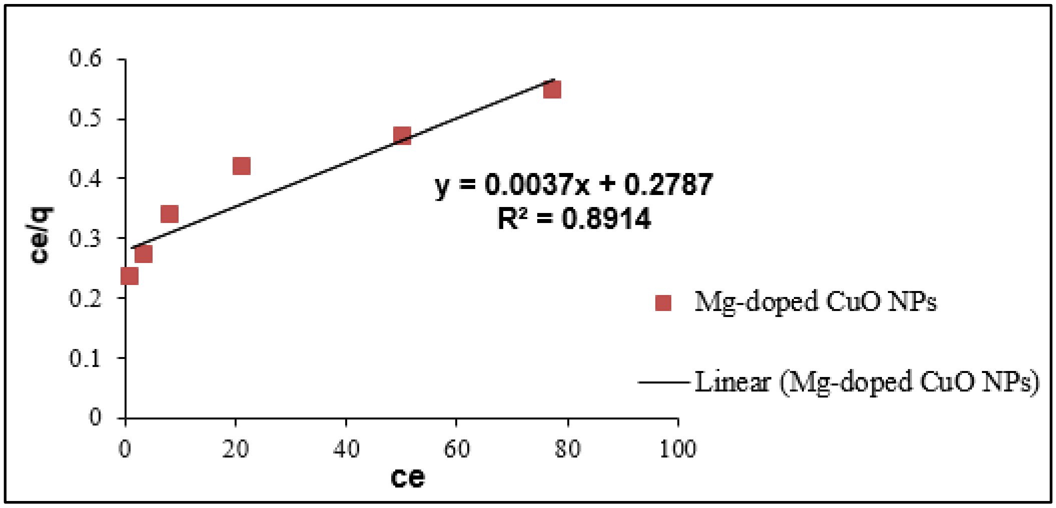

Fig. 6 Langmuir Isotherm

4.2 Freundlich Adsorption Isotherm

The Freundlich isotherm model is an empirical equation that describes the adsorption of solutes on the surface of sorbents. The model is based on the assumptions that the adsorbent surface is heterogeneous, with different adsorption sites having different affinities for the adsorbate. Multilayer adsorption can occur. There is no interaction between adsorbed molecules [49].

The Freundlich isotherm equation is as follows:

$\log(q_e) = 1/n \log(C_e) + \log(K_f)$

where: qe is the amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium (mg/g), Ce is the equilibrium concentration of adsorbate in the aqueous solution (mg/L), Kf is the Freundlich adsorption constant (L/mg), 1/n is the Freundlich heterogeneity factor. The Freundlich adsorption constant (Kf) is a measure of the adsorption capacity of the adsorbent. The Freundlich heterogeneity factor (1/n) provides information about the nature of the adsorption process. If 1/n is less than 1, then the adsorption process is favorable. If 1/n is greater than 1, then the adsorption process is unfavorable. Mg-doped CuO NPs have been shown to be effective adsorbents for the removal of heavy metals from aqueous solutions. The Freundlich isotherm model has been used to describe the adsorption of heavy metals onto Mg-doped CuO NPs. Studies have shown that the Freundlich isotherm model fits the experimental data well, indicating that multilayer adsorption is the dominant adsorption mechanism. Overall, the Freundlich isotherm model is a useful tool for describing the adsorption of heavy metals onto Mg-doped CuO NPs. The model suggests that multilayer adsorption is the dominant adsorption mechanism.

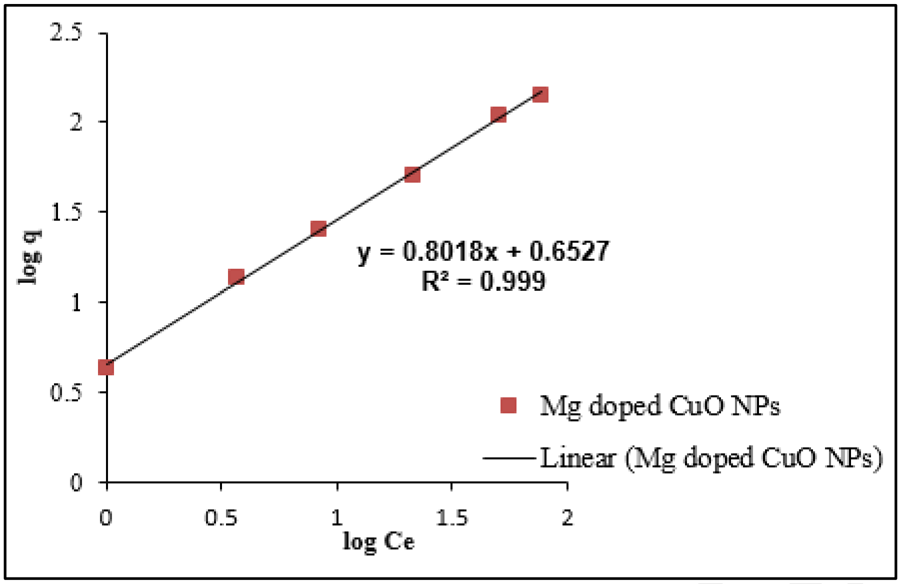

Fig. 7 Freundlich Isotherm

Table 1: Comparison of Models

Langmuir model				Freundlich model			
q_e (exp) (mg/g)	X_m (mg/g)	K_L (L/mg)	R^2	1/n	q_e (cal) (mg/g)	K_2 (g mg^{-1} min^{-1})	R^2
142	75.3	0.0132	0.891	0.801	147	0.426	0.999

4. CONCLUSION

Mg-CuO nanoparticles were synthesized via a facile and cost-effective co-precipitation method and demonstrated outstanding adsorption efficiency for Congo Red dye removal from aqueous solutions. The Langmuir isotherm model and Freundlich adsorption isotherm model are well-fitted the equilibrium adsorption data and adsorption behaviour. The maximum R^2 is close to 0.99 of Freundlich isotherm indicates that adsorption occurs with multilayer formation rather than monolayer formation. It revealed a more linear behaviour than the Langmuir model owing to its better R^2 values for congo red dye. This was determined by comparing the two models. The value of the correlation coefficient in the Freundlich model is quite close to one. Optimal conditions (pH=6, concentration=50ppm, contact time=120minutes) for maximal adsorption efficiency were determined. The thermodynamic analysis indicated that the adsorption of Congo Red onto Mg-CuO nanoparticles was spontaneous and endothermic. The exceptional adsorption performance of Mg-CuO nanoparticles, attributed to the synergistic effect of magnesium doping and high surface area, highlights their potential as an eco-friendly and efficient adsorbent for the removal of Congo Red dye from wastewater.

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