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ADSORPTIVE REMOVAL OF BRILLIANT GREEN DYE USING COPPER OXIDE NANOPARTICLES: ISOTHERM, KINETIC AND THERMODYNAMIC APPROACH

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ABSTRACT

Synthetic dyes and their effluents are the primary sources of water pollution. Thus, it is essential that these dyes be eliminated prior to their release into the aquatic environment. The primary aim of the present investigation was to synthesise copper oxide nanoparticles as an adsorbent for the elimination of Brilliant Green dye from aqueous media. Fourier Transform Infra-Red, Scanning Electron Microscopy, Energy Dispersive X-ray, and Powder X-Ray Diffraction techniques were employed to characterise the synthesised adsorbent. Batch mode studies were conducted by changing the weight of adsorbent, concentration of the dye, contact period, agitation speed, temperature and pH. A maximum dye removal rate of 74.8% was obtained at 120 minutes. The batch experimental data were correlated to Temkin, Freundlich, Dubinin-Raduskevich, Langmuir and Javonoic isotherms. Parameters obtained from thermodynamic studies like ΔG^o, $\Delta H^{o,}$ and ΔS^o were determined. Thermodynamic studies revealed the spontaneity of the process and kinetic data fitted well with the pseudo-second-order kinetic model. The reusability of the adsorbent was analysed by desorption studies. The present study concludes that synthesised copper oxide nanoparticles have the potential to serve as highly efficient adsorbent for adsorbing dyes from aqueous solutions.

Keywords: Dyes, Copper Oxide Nanoparticles, Adsorption, Dye Removal, Brilliant Green, Isotherms.
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INTRODUCTION

Rapid industrial growth and advances in technology have led to water contamination, which has become a serious environmental problem. Aquatic ecosystems are contaminated by harmful chemicals from industrial effluents at levels higher than those recommended by the World Health Organisation (WHO). These pollutants include heavy metals, organic compounds and synthetic chemicals, which pose severe threats to aquatic ecosystems and human health. Currently, more than 100,000 distinct synthetic dyes are extensively employed in various industries. Effluents from industries such as textiles, tanneries, paint, polymer, paper, etc., contain synthetic dyes.[1] These industries discharge large amounts of colour effluents with high concentrations, which are typically more hazardous and difficult to decompose using conventional techniques.[2] As a dermatological agent and a supplement to feed chickens, brilliant green dye serves to inhibit the growth of parasites and fungi.[3] Furthermore, it is widely utilised in textile and printing dyeing. However, in humans, BG causes gastrointestinal tract irritation, leading to diarrhoea and vomiting.[4] It also causes cough and breathing difficulty due to irritation of the respiratory tract. When it comes in contact with the skin, causes redness and pain. Brilliant Green, when heated, undergoes decomposition to form sulphur oxides, carbon oxides and nitrogen oxides, which are hazardous to the environment.[5] It is important to eliminate these dyes before they are dumped into water bodies. Numerous methods are available for treating wastewater containing dyes released by factories. Adsorption is an essential and cost-effective method of treating dye effluents.[6] Adsorption techniques have emerged as effective methods for removing contaminants from water, offering a promising solution for controlling water pollution. These techniques involve the adhesion of pollutants to the surface of adsorbent materials, allowing their efficient removal from water sources. Many adsorbents used in adsorption treatments include biosorbents, zeolites, and cellulose-based wastes. The utilization of nano-adsorbents for wastewater treatment is an innovative and simple process. The use of metal oxide nanoparticles as adsorbents has drawn substantial interest in the biological, physical, chemical, and engineering fields because of their large surface area.[7] Recently, research

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has focused on metal oxide sorbents such as iron oxide, aluminium oxide, titanium oxide, manganese oxide, and zirconium oxide for effluent treatment.[8,9] However, from the literature survey, it was found that very few studies have been reported on copper oxide nanoparticles as adsorbents. The objective of the current work is to synthesise copper oxide nanoparticles using the co-precipitation technique and use them to remove brilliant green dye from aqueous media. The significance of clean water and sanitation is particularly addressed by Sustainable Development Goal (SDG) 6 of the UN, which highlights the need to minimise the discharge of harmful chemicals into water bodies, prohibit dumping, and reduce pollution.

EXPERIMENTAL

Materials

Copper sulphate pentahydrate and sodium hydroxide were bought from Merck Life Science Pvt.Ltd, Mumbai. Brilliant Green dye was obtained Hi Media Laboratory Pvt Ltd., Mumbai.

Synthesis of Copper Oxide Nanoparticles

Copper sulphate solution was prepared and agitated at 500 rpm using a magnetic stirrer. While maintaining vigorous agitation, Sodium hydroxide solution (0.5M) was gradually added in a dropwise manner. Stirring was continued at 50-60°C for 1 hour to ensure completion of precipitation. After filtration, the precipitate was cleaned with distilled water and kept in an oven at 100°C for six hours to dry.[10,11]

Characterisation of Adsorbent

Morphological studies of the adsorbent were examined by the SEM technique (ZEISS). Elemental analysis was performed using an EDX (Oxford Instruments). The dye concentration was measured using a MAPADA-UV (1100) spectrophotometer. An FT-IR spectrometer (Jasco-5300) was used to obtain the IR spectra. PXRD analysis was performed using Malvern Panaltical diffractometer.

Adsorption Studies

A standard dye solution of concentration 10 mg/L was used for batch studies. Batch experiments were performed in a Pyrex bottle, in which 100 ml of the dye solution containing 0.05 g of copper oxide nanoparticles was shaken in an orbital shaker at 250 rpm. A spectrophotometer was used to measure the concentration of the dye before (C_0) and after adsorption (C_e). The % decolourisation of the dye was obtained by the eqn.-1:[12]

$$\text{Percentage Removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \qquad (1)$$

The experiment was repeated at different contact time until equilibrium was attained and batch mode studies were executed out by differing the contact period(10-120 minutes), initial dye concentration (5.0,7.5, 10,12.5,15 mg/L), adsorbent weight (0.1,0.3,0.5,0.7 and 0.9 g/L), temperature (303,308 and 313K), pH(4,7,10) and agitation speed(50,100,150,200,250rpm) to study the impact of these variables on adsorption.

RESULTS AND DISCUSSION

Fourier Transform Infra-Red Analysis

The IR spectra of copper oxide nanoparticles before adsorption exhibited peaks appeared at 517.02 cm^{-1} and 617.67 $cm^{-1,}$ which are attributed to Cu-O vibrational stretching frequency (Fig.-1).[13,14]. The elongation and distortion of the vibrating Cu-O bond are exhibited by a band at 848.54 cm^{-1}. $Cu^{2+}O^{2-}$ stretching vibration is shown by the peak at 1460.99 cm^{-1}.[15] The bands at 1644.81 cm^{-1} and 3458.85 cm^{-1} are caused by bending and stretching O-H vibrations due to moisture absorbed from the air.[16] The FT-IR spectra of the adsorbent after adsorption revealed many modifications, such as the development of new bands, variations in intensity, and displacement in the location of the peaks. (Fig.-2). These changes suggest that involvement of functional groups in adsorption process.[17]

SEM Analysis

The SEM image of the adsorbent before adsorption indicates that it was composed of irregularities in shape, had good homogeneity and was uniform in size (Fig.-3). After the adsorption of the dye, the structure became saturated with dye molecules[18] (Fig.-4).

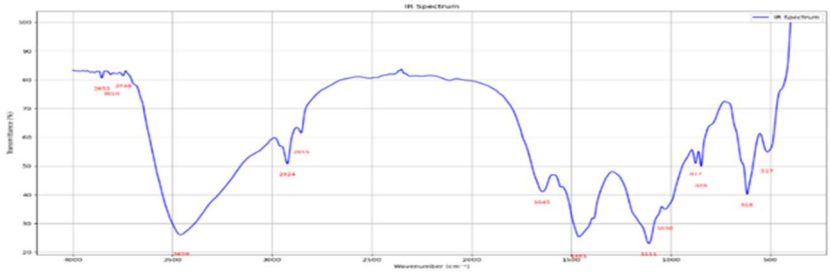

Fig.-1: FT-IR Spectra of Copper Oxide nanoparticles before adsorption

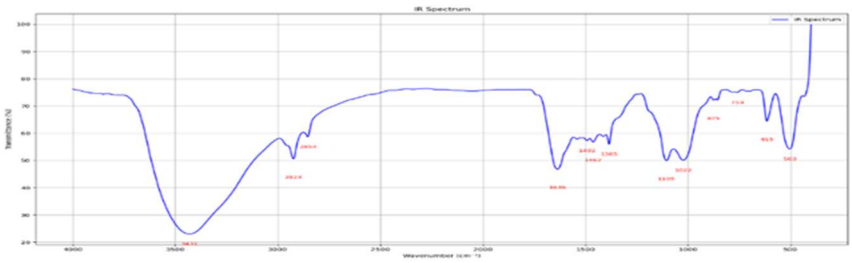

Fig.-2: FT-IR Spectra of Copper Oxide nanoparticles after adsorption

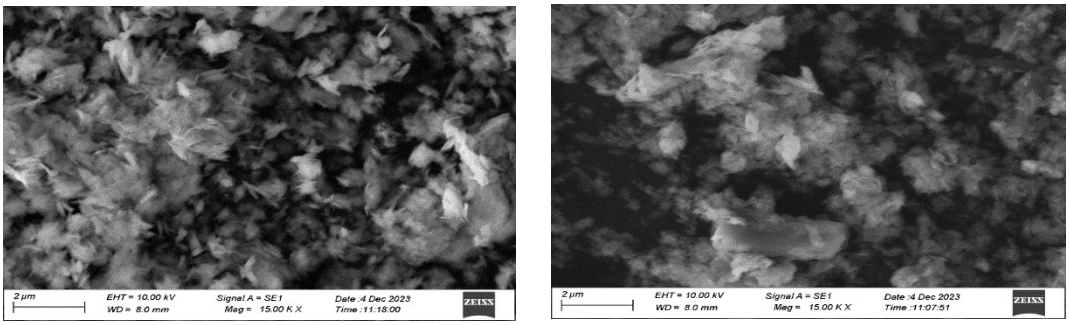

Fig.-3 and 4: SEM Image of Copper Oxide nanoparticles before adsorption and after adsorption

EDX Analysis

EDX spectra were analysed to assess the composition of the prepared nanomaterial. The EDX analysis (Fig.-5) revealed distinct peaks corresponding to copper (Cu) and oxygen (O), indicating that the synthesised nanomaterial consisted only of these elements without any impurities.[17]

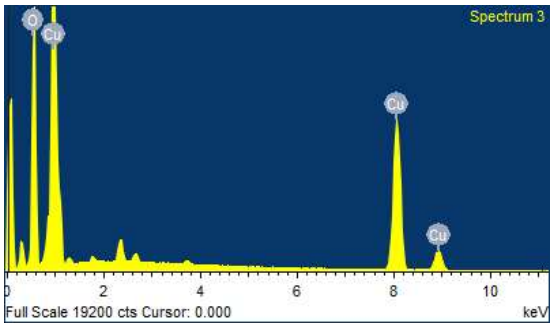

Fig.-5: EDX Spectrum of Copper Oxide Nanoparticles

PXRD Analysis

PXRD examination (Fig.-6) of the synthesized copper oxide nanoparticles revealed diffraction peaks at 32.9°, 36.1°, 39.1°, 49.4°, 53.8°, 58.7°, 61.9°, 66.8°, and 68.6°, which could be attributed to the (110),

(002), (200), (202), (020), (202), (113), (311), and (220) planes. This indicated that copper oxide nanoparticles were in the monoclinic phase (JCPDS 00-45-0937). The average crystallite size is found to be 30.25nm.[19]

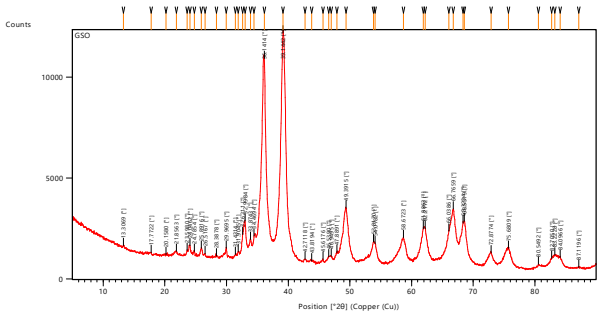

Fig.-6: PXRD Analysis of Copper Oxide Nanoparticles

Adsorption Procedure

Effect of Contact Time

Using an orbital shaker, the dye solution (10 mg/L) was shaken with copper oxide nanoparticles (0.5 g/L) at 30°C and 250 rpm. The absorbance was measured at 10-minute intervals, and % decolourisation was calculated. It was found that 74.8% of the dye was decolourised at 120 minutes. In the initial stage, owing to the abundance of adsorption sites, the rate of decolourisation was high and remained constant after equilibrium was reached **(**Fig.-7).

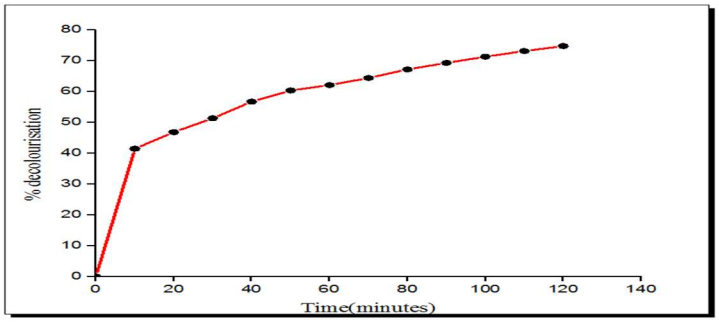

Fig.-7: Variation of Contact Time

Effect of Concentration of Dye

Different concentrations of Brilliant Green dye solutions (5–15 mg/L**)** were used, keeping the other variables constant. The adsorption data were tabulated, and a graph was drawn (Fig.-8). The % decolourisation was found to decrease as the concentration of dye was increased. This is because the initial dye concentration of dyes increases, but active spots remain the same and thus % removal is decreased.[20, 21]

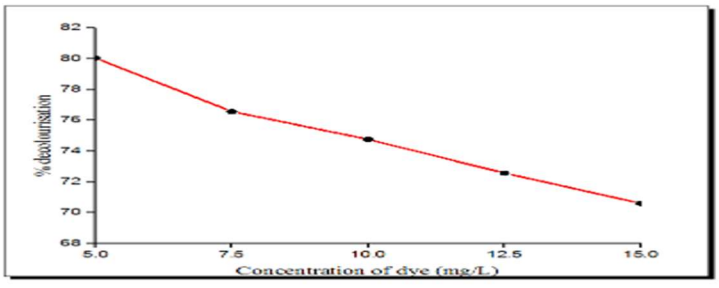

Fig.-8: Variation of Initial Dye Concentration

Effect of Amount of Adsorbent

The impact of dosage of copper oxide adsorbent on elimination of dye was examined by adjusting the adsorbent (0.1-0.9 g/L). The experimental results indicated that % decolourisation rises with an increase in the amount of adsorbent due to more number of active spots (Fig.-9).

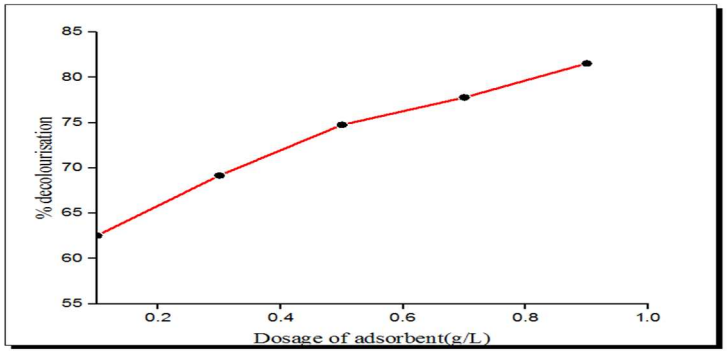

Fig.-9: Variation of Amount of Adsorbent

Effect of the Speed of Agitation

The impact of shaking speed on % decolourisation was investigated by changing the shaking speed to 50, 100, 150, 200, and 250 rpm while keeping other variables constant**.** The experimental data **s**howed that increasing the agitation speed increased the dye adsorption by decreasing the boundary layer thickness (Fig.-10).

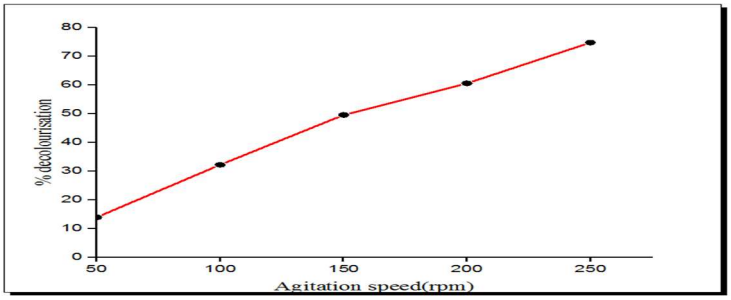

Fig.-10: Variation of Agitation Speed

Effect of pH

The change in the % of decolourisation of dye by the change in pH was evaluated at pH values of 4, 7, and 10, while maintaining the other variables constant. It was noticed that adsorption increased as the pH increased (Fig.-11). As the pH of the solution rises, the number of negatively charged sites increases and hence the adsorption of cationic dye is more.[22]

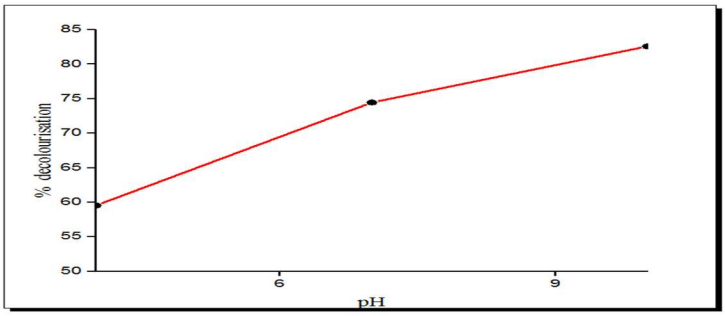

Fig.-11: Variation of pH

Effect of Temperature

A batch adsorption study was executed at 303 K, 308 K and 313 K to assess the impact of temperature on % decolourisation. The percentage decolourisation increased with temperature because of the increase in active adsorption spots and pore volume porosity (Fig.-12).[23, 24]

Desorption Study

The economic effectiveness of adsorption operations is largely dependent on the potential of the adsorbent material for reuse. The capacity of this method to target specific pollutants, coupled with its potential for

the regeneration and reuse of adsorbent materials, renders it a cost-effective and environmentally sustainable solution for water treatment.

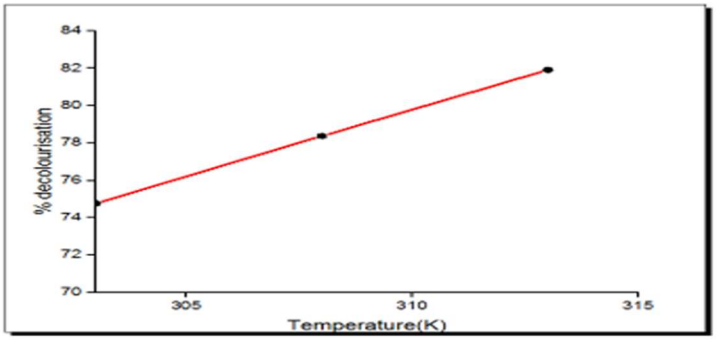

Fig.-12: Variation of Temperature

This approach aligns closely with Sustainable Development Goal 6 of the UN, which is to make sure that everyone has access to and can effectively handle water and sanitation. HCl (0.1N-0.5N) was used as the desorbing agent. The results (Fig.-13) revealed that % decolourisation increase with rise in concentration of HCl, which may be due to an increase in H+ concentration causes electrostatic repulsion resulting in increase of dye desorption.[25]

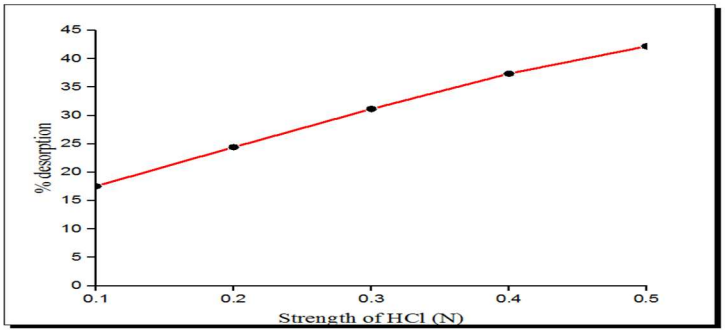

Fig.-13: Desorption Study

Adsorption Isotherm

Adsorption isotherms were utilised to evaluate the mechanism of process adsorption and the adsorbent's maximal adsorption capabilities.[26] It also helps to determine the affinity of the adsorbent.

Freundlich Isotherm

It is employed on systems with heterogeneous surfaces where adsorption often occurs.[27] It is represented by the following equation:

$$q_e = K_f C_e^{1/n} \quad (2)$$

The linearised form is represented by the following equation:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (3)$$

The graph of $\ln q_e$ versus $\ln C_e$ exhibited a linear relationship (Fig.-14), indicating that the adsorption process obeying Freundlich isotherm model.

Langmuir Isotherm

Langmuir's model describes the single layer distribution of the adsorbate, assuming similar adsorption sites.[28] The Langmuir equation is given by:

$$q_e = (Q_0 b C_e)/(1 + bC_e) \quad (4)$$

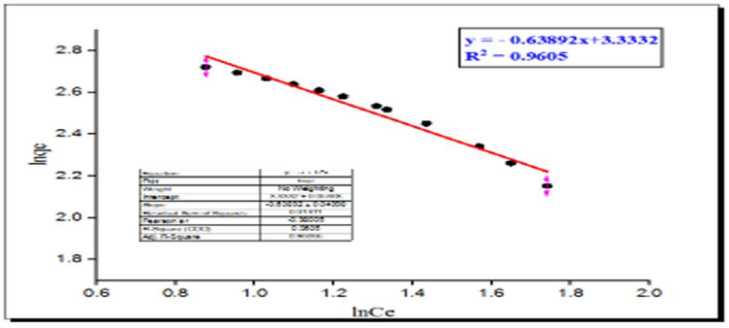

Fig.-14: Freundlich Isotherm

It is expressed in linear form as:

$$\frac{C_e}{Q_e} = \left(\frac{1}{Q_0 b}\right) + \left(\frac{C_e}{Q_0}\right) \qquad (5)$$

The linearity of graph of $\frac{C_e}{Q_e}$ Vs C_e confirms that experimental data is most fitting with the Langmuir isotherm (Fig.-15).

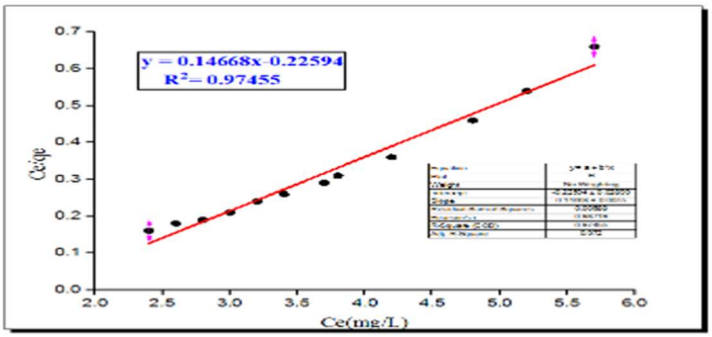

Fig.-15: Langmuir Isotherm

The separation factor R_L in the Langmuir isotherm can be represented as:[29,30]

$$R_L = \frac{1}{(1 + bC_i)} \qquad (6)$$

R_L value obtained is 0.1334 ($0 < R_L < 1$) which shows that the adsorption is a favourable process.

Temkin Isotherm

According to the Temkin isotherm, the interactions occurring between the adsorbate and adsorbent lead to a drop in the adsorption heat of each molecule with surface coverage.[31]

It is denoted by the following equation:

$$q_e = B_T \ln K_T + B_T \ln C_e \qquad (7)$$

Plot of q_e Vs $\ln C_e$ is linear which reveals that adsorption data was fitted with Temkin isotherm (Fig.-16).

Dubinin-Raduskevich Isotherm

It is employed to identify the adsorption mechanism. The linearised equation is given by the following equation:

$$\ln q_e = \ln q_D - 2B_D RT \ln\left(1 + \frac{1}{C_e}\right) \qquad (8)$$

Plot of $\ln q_e$ vs $RT \ln\left(1 + \frac{1}{C_e}\right)$ (Fig.-17) is linear, which shows that the experimental data obeys the Dubinin- Raduskevich isotherm.

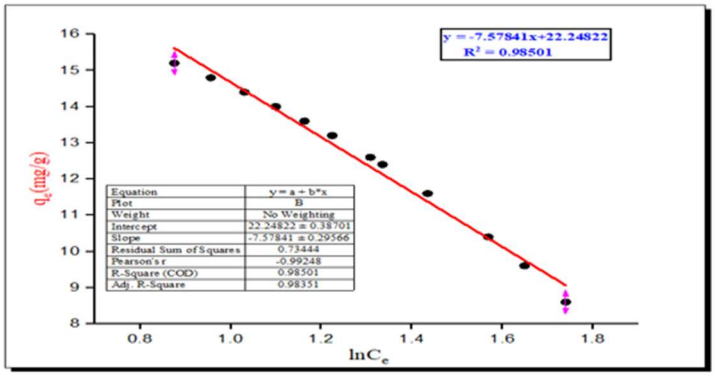

Fig.-16: Temkin Isotherm

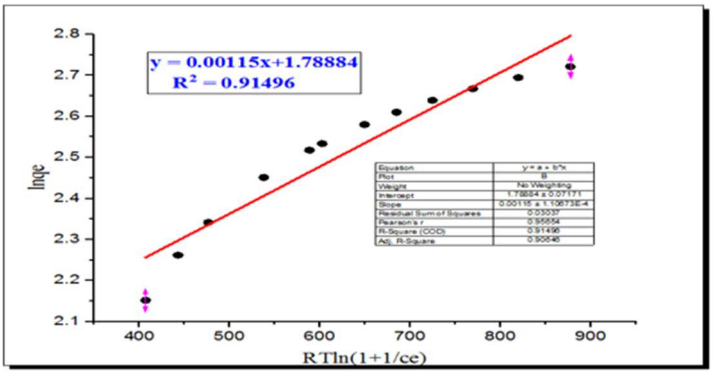

Fig.-17: Dubinin-Raduskevich Isotherm

Jovanovic Isotherm

The Jovanovic model elucidates the adsorption behaviour on heterogeneous surfaces. It is given as:

$$\ln q_e = \ln q_{\max} - K_J C_e \qquad (9)$$

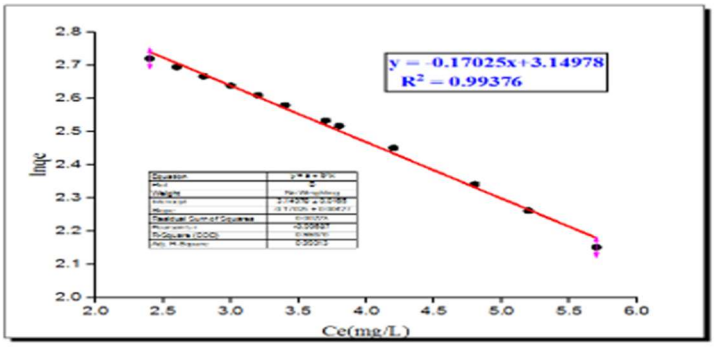

Fig.-18: Jovanovic Isotherm

The linearity of the graph of C_e Vs $\ln q_e$ confirmed that the experimental outcome was well correlated with the Jovanovic model (Fig.-18).

Kinetic Studies

Pseudo-first order model is represented by:[32]

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (10)$$

The experimental results are inconsistent with the pseudo-first-order kinetic model (Fig.-19).

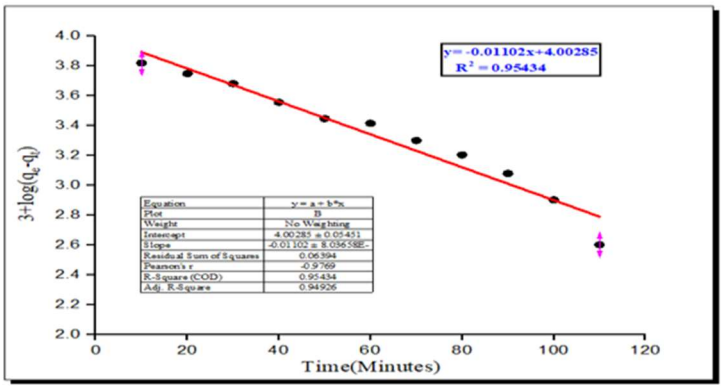

Fig.-19: Pseudo First Order Kinetic Model

Pseudo-Second order model is expressed by:[33]

$$\frac{t}{q_t} = \frac{1}{k_2}\left(q_e^{\,2}\right) + \frac{t}{q_e} \qquad (11)$$

The plot (Fig.-20) exhibits linearity, indicating that the adsorption adheres to the pseudo-second-order kinetic model.

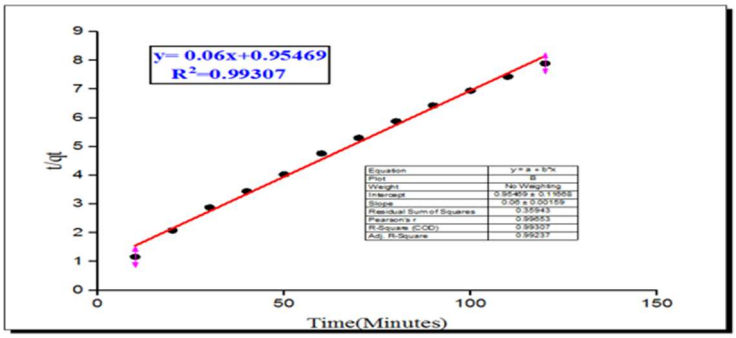

Fig.-20: Pseudo-Second Order Kinetic Model

Thermodynamic Investigation

ΔG^o, ΔH^o, and ΔS^o are essential parameters that provide information about the spontaneity of adsorption. ΔG^o is associated with K_L by the eqn.-12.[34]

$$\Delta G^o = -RT\ln K_L \qquad (12)$$

ΔG^o and ΔS^o is related by the eqn.-13.

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \qquad (13)$$

A Positive of ΔS^o (0.0124) and ΔH^o (+0.0004) values indicates the increase of disorderness and endothermic nature of the adsorption process. Negative ΔG^o (-3.7477) shows the feasibility of the adsorption (Fig.-21).

CONCLUSION

The present work is focused on the synthesis and characterization of copper oxide nanoparticles. The characterization employed various analytical techniques, including SEM, FTIR, EDX, and PXRD. Experimental findings from batch mode studies revealed that the percentage of dye elimination was enhanced with prolonged contact period, increased quantities of copper oxide nanoparticles and increased in pH, agitation speed, and temperature, while exhibiting an inverse relationship with the initial dye concentration. The experimental results were observed to be best fitted with Freundlich, Langmuir, Temkin, Dubinin-Raduskevich, and Jovanovic isotherms. Adsorption obeyed pseudo-second-order kinetics. Thermodynamic investigations confirmed the spontaneity of the adsorption process. The synthesized copper oxide nanoparticles exhibit considerable promise as effective adsorbents for dye elimination. The

present methodology not only reduces pollution but also enhances sustainable water management practices in response to increasing water scarcity, thereby supporting ss Sustainable Development Goals (SDG).

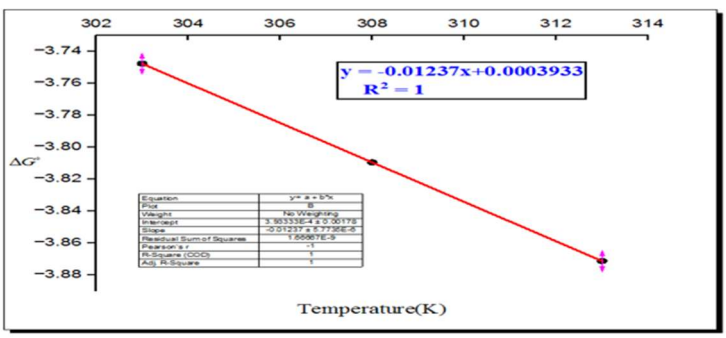

Fig.-21: Adsorption Thermodynamics for BG Removal by Copper Oxide Nanoparticles

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest in this paper.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids given below:

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