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A Novel Coal-Associated Soil as an Effective Adsorbent for Reactive Blue Dye Removal

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ABSTRACT

The project aims to remove reactive blue dye from the effluent of textile industries by utilizing coal-associated soil as an adsorbent, as it possesses effective physical properties and distinguishing characteristics. In comparison to other separation techniques, the adsorption method is the most effective, cost-effective, and straightforward. A batch adsorption investigation was carried out to examine the various adsorption-influencing factors, including solution pH, adsorbent dosage, contact time, temperature, and dye concentration. Contact time of 30 min, an adsorbent dosage of 10g.100 mL^{-1}, a solution pH of 7, a temperature of 30°C, and an initial dye concentration of 100 $mg.L^{-1}$ were found to be optimal for dye adsorption. Using two distinct kinetic models, the evaluation of kinetic studies revealed that the pseudo-second-order provided the greatest fit, with a higher R^2 value than the pseudo-first-order. The thermodynamic parameters Gibbs free energy (ΔG°), entropy (ΔS°), and enthalpy (ΔH°) indicated that the current adsorption system was exothermic and spontaneous. Further study of the adsorption isotherm revealed that the Langmuir isotherm model provided the best fit, with an R^2 value of 0.977%.

INTRODUCTION

Water contamination or pollution results from the discharge of industrial effluents and the continuous, excessive use of fertilizers and pesticides in agricultural fields (Mahvi et al. 2011, Malakootian et al. 2013). The degradation of the water quality that results in water contamination or pollution is brought on by the discharge of industrial effluents and the continued excessive use of fertilizers and pesticides in agricultural areas (Sharifpour et al. 2018, Javid et al. 2020). Freshwater demand is rising rapidly as it is heavily contaminated by the textile, leather, paper, and cosmetic industries (Muthulingam et al. 2018). Every year, a wide range of synthetic dyes are manufactured and used in industry (Malar et al. 2023). The dyeing process loses 50% of its reactive colors to effluents (Amin & Blackburn 2015). Reactive dyes are pigments with reactive groups that can make covalent connections between the dye's carbon atom and the OH-, NH-, or SH groups in fiber (Zhao et al. 2022). They are assemblages of hues that establish covalent bonds with fibers and become components of the fiber itself. When they engage chemically with the molecules of the fiber, covalent bonds are formed.

One example of a reactive dye is reactive blue 4. With the molecular formula $C_{23}H_{12}C_{12}N_6Na_2O_0S_2$, reactive blue 4 has an anthraquinone molecular structure (Epoloto et al. 2005). The molecular weight of reactive blue 4 is 637.43. The dye is dark blue and also comes in powder form. This dye is often used to color cotton, nylon, and other textiles. Also, reactive dyes directly contaminate the ecosystem and cause different carcinogenic or mutagenesis diseases in living beings (Berradi et al. 2019). Hence, removing reactive dyes from and other effluents before they enter natural water bodies is environmentally significant. As reactive dyes prevent light trapping for photosynthesis, plants are also harmed (Bae et al. 2006).

Textile manufacturing produces the most wastewater in the environment, which has intense color, highly variable pH, high chemical oxygen demand, and toxicity. Reactive dye dyes cotton, wool, and polyamide filters in textiles. Most reactive dyes do not bind to textiles (Lellis et al. 2019). Reactive dyes are non-degradable in aerobic biological treatment systems, leaving persistent colors in effluents. Many biological and chemical methods have been developed to remove dyes from aqueous-colored effluents and lessen their ecological impact (Adane et al. 2021). But traditional methods cannot purify textile industry wastewater of colored effluents.

Adsorption is a surface phenomenon that is the result of complex interactions between the three constituents: the adsorbent, the adsorbate, and the wastewater, which may be effluent, a synthetic solution, or water (Seenuvasan et al. 2021). The separation is based on the selective adsorption of pollutants by an adsorbent, i.e., thermodynamic and/or kinetic selectivity, as a result of particular interactions between the surface of the adsorbent material and the pollutants adsorbed. The affinity between the adsorbent and the adsorbate is the primary interaction factor affecting adsorption in this ternary system (Rapo & Tonk 2021). The carbon in coal comprises mesoporous components that are effective for adsorbing dye molecules from industrial or aqueous effluents, and these oxides have active sites. Hence, the soil associated with coal also contains significant amounts of carbon in addition to the mineral oxides (Baocheng et al. 2008, Tarkwa et al. 2019, Astuti et al. 2019).

In this study, the removal of reactive blue dye is expected using the coal-associated soil as an effective adsorbent. The batch adsorption study was performed with optimized parameters, including pH, temperature, contact time, adsorbent dosage, and dye concentration. The adsorption mechanism was validated through isotherm and kinetic studies.

MATERIALS AND METHODS

Preparation of Adsorbent

Coal-associated soil was collected from the Mannargudi Coalfield, Tamil Nadu, India. The soil was washed with water and then dried at room temperature to remove any dust particles. To get the ideal adsorbent for the dye removal, the dried soil samples were sieved through a mesh sieve. Using FTIR (Perkin Elmer spectrum RX 1) and SEM (Zeiss FESEM SIGMA VP03-04 MODEL) spectroscopy, coal-associated soil was qualitatively analyzed both before and after adsorption. The characteristics of the collected coal-associated soil were estimated as follows: pH: 2.8, organic matter content: 29.90%, silica content: 32.13%, alumina content: 1.12%, and iron oxide content: 1.97%.

Preparation of Adsorbate

Reactive Blue 4 dye ($C_{23}H_{14}C_{l2}N_6O_8S_2$, molecular weight is 637.43) was purchased from Sigma Aldrich Pvt. Ltd. Adding 1g of reactive blue dye to 1L of water made the Reactive Blue 4 dye standard solution. To get different concentrations of dye solution, ranging from 10 mg.L^{-1} to 100 mg.L^{-1}, the standard solution was diluted with distilled water. Reactive blue dye was discovered to have a maximum wavelength (max) of 580 nm (Dutta et al. 2021). Reactive blue dye was analyzed colorimetrically to quantify the concentration of the dye in the solution both before and after adsorption.

Batch Adsorption Studies

Adsorption experiments were carried out in batches for various adsorption-influencing parameters such as temperature, solution pH (2 to 10), initial reactive blue dye concentration (50 to 250 mg.L^{-1}), contact time (10 to 50 min), and adsorbent dosage (6 to 14 g.100 mL^{-1}). The batch adsorption experiments for each parameter study were carried out by varying the respective parameters while keeping the other parameters constant.

To 100 mL of working solution, a suitable amount of adsorbent was added and agitated in a rotary shaker at 100 rpm to ensure efficient adsorption. At a specified time interval, the samples were collected and centrifuged at 2000 rpm for 10 min to distinguish the solution and the adsorbent. The supernatant was collected, and the percentage removal of reactive blue dye from the aqueous solution was calculated by measuring absorbance at 580 nm with a colorimeter. Using Eqn. (1), the percentage removal of reactive blue dye was calculated.

$$\% \, Removal = \frac{C_i - C_f}{C_i} \times 100 \qquad \ldots(1)$$

Where C_i and C_f are the initial and final concentrations of dye (mg/L).

Isotherm and Kinetics Studies

The isothermal study aids in identifying the interface mechanism between coal-associated soil and reactive blue dye. At various adsorbate concentrations (50-250 mg.L^{-1}), adsorption isotherm investigations were conducted. The optimal amount of absorbent was added to 100 mL of the working solution and kept in the rotary shaker for the allotted period. The samples were taken after the specified time interval and centrifuged at 2000 rpm for 10 min, and the absorbance was measured at 580 nm. The equilibrium adsorption capacity of the coal-associated soil was calculated by Eqn. (2).

$$q_e = \frac{(C_i - C_e)V}{m} \qquad \ldots(2)$$

where q_e is the equilibrium adsorption capacity (mg/g), C_i and C_e are the initial and final concentrations of dye (mg.L^{-1}), V is the volume of reactive dye solution (L), and m is the weight of adsorbent used (g). Langmuir and Freundlich's isotherms were performed with the equilibrium data using MATLAB R2016a software.

The rate of adsorption was elucidated using the adsorption kinetics study. By stirring the flasks containing 100 mL of various dye solution concentrations (ranging from 50 mg.L^{-1} to 250 mg.L^{-1}) and the necessary amount of adsorbent, kinetic studies were carried out. The samples were collected every 5 min and centrifuged. Eqn. (3) was used to calculate the amount of reactive blue dye that has been removed from coal-associated soil over specific time intervals.

$$q_t = \frac{(C_i - C_t)V}{m} \quad \ldots(3)$$

Where q_t is the equilibrium adsorption capacity (mg/g), C_i is the initial concentration of dye (mg/L), C_t is the concentration of dye at different time intervals ($mg.L^{-1}$), V is the volume of reactive dye solution (L), and m is the weight of adsorbent used (g). Similar to the isotherm studies, the kinetic parameters were found using MATLAB R2016a software.

Thermodynamic Study

The experimental study investigated the thermodynamic aspects of the adsorption process between a reactive blue dye and coal-associated soil. The experiments were conducted at various temperatures ranging from 30°C to 50°C. A 250 mL Erlenmeyer flask was used, containing 100 mL of the dye solution and 10 g of soil. The thermodynamic parameters, including Gibbs free energy ($\Delta G°$, $KJ.mol^{-1}$), enthalpy change ($\Delta H°$, KJ/mol), and entropy change ($\Delta S°$, J/mol), were determined using Eqns. (4) and (5):

$$\Delta G° = -RT \ln K_d \quad \ldots(4)$$

$$K_d = \frac{q_e}{C_e} \quad \ldots(5)$$

Where K_d is the equilibrium constant, q_e is the equilibrium adsorption capacity (mg/g), C_e is the equilibrium concentration ($mg.L^{-1}$), and R is the gas constant ($8.314\ J\ mol^{-1}\ K^{-1}$).

RESULTS AND DISCUSSION

Characterization Studies of the Adsorbent

SEM analysis: SEM analysis was used to examine the adsorbent's porous characteristics and surface structure. The SEM analysis of coal-associated soil before and after the dye adsorption is shown in Fig. 1(a & b). Before adsorption, a rough, porous structure with higher voids was observed (Fig. 1(a)), and the enhanced surface area with irregular and linked pores, which are crucial for the effective adsorption of reactive blue dye molecules, was also exhibited. Following the adsorption of the dye, the formation of the agglomeration was evident in Fig. 1(b). As a result, the agglomerates and disappeared pores with a smooth surface support the dye's adsorption to coal-associated soil.

FTIR analysis: FTIR analysis was performed before and after adsorption of the dye and is shown in Fig. 2. A high signal at 2954.52 cm^{-1} indicates that alkanes stretch the C-H bond before adsorption (Malar et al. 2019). The presence of additional functional groups, such as aromatic ring stretching, aliphatic, and aromatic phosphate compounds, is indicated by the absorption peaks at 1620.40 cm^{-1}, 1011.97 cm^{-1}, and 927.84 cm^{-1}. The silicone group and the primary amine (C-N) broadening are related to the strong peak at 1054.21 cm^{-1}. The FTIR examination of coal-associated soil suggests that alcohol and amine groups may form covalent connections prior to adsorption, forming a matrix structure that would make it simpler to remove the coal.

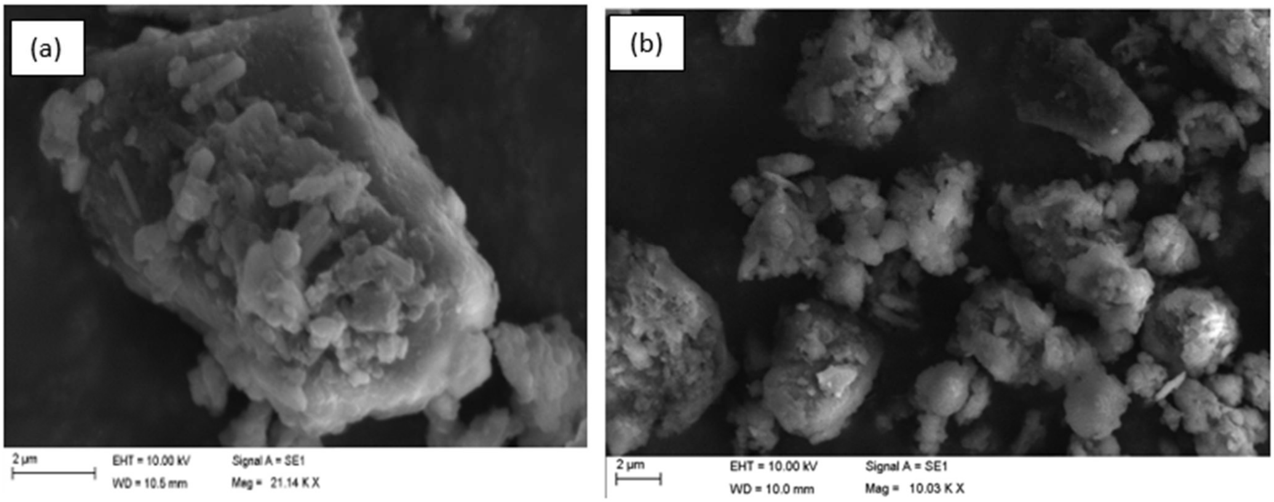

Fig. 1: Electron Micrographs of coal-associated soil (a) before and (b) after adsorption.

Effect of pH

In a batch adsorption experiment, the pH of a dye solution was varied from 2.0 to 10.0 while keeping other parameters constant. The results, shown in Fig. 3, demonstrate that the percentage removal of the dye increased gradually as the pH shifted from acidic to neutral (El-Nemr et al. 2020). Equilibrium adsorption occurred at neutral pH due to the dye's neutral nature. However, beyond neutral pH, the percentage removal decreased. The highest removal of the dye was observed at pH 7.0 (neutral).

Effect of Contact Time

In a batch study, the effect of contact time on the removal of reactive blue dye using coal-associated soil as the adsorbent was investigated. The dye concentration was 100 mg.L^{-1}, the pH was maintained at 7, and the adsorbent dosage was 10 g. The sample was agitated in an orbital shaker at 100 rpm, and samples were collected at 10-minute intervals. Fig. 4 revealed that the removal of reactive blue dye increased from 10 min to 30 min. The initial adsorption rate was high, likely due to the availability of more active sites. However, once equilibrium was reached, no further adsorption occurred (Zhang 2023). The highest percentage removal of the dye onto coal-associated soil was observed at a contact time of 30 mins.

Effect of Adsorbent Dosage

In the batch adsorption experiment, the impact of adsorbent dosage on the removal of reactive blue dye was investigated while keeping other parameters constant. The dosage of the adsorbent was varied from 6 g to 14 g, and as demonstrated in Fig. 5, lower adsorbent dosages resulted in lower percentage removal of the dye. This can be attributed to the limited availability of active sites on the surface of the coal-associated soil. However, as the adsorbent dosage increased, the percentage removal of the dye also increased due to the greater number of active sites provided by the soil. Beyond an adsorbent dosage of 10 g, there was no significant increase in dye removal due to factors such as low driving force, saturation of active sites, and insufficient dye presence in the solution. Therefore, the optimum adsorbent dosage for the removal of reactive blue dye was determined to be 10g.100mL^{-1}.

Effect of Initial Dye Concentration

In a batch adsorption experiment, the impact of initial dye concentration on the adsorption of reactive blue dye onto coal-associated soil was examined. The experiment involved different reactive blue concentrations ranging from 50 mg.L^{-1} to 250 mg.L^{-1} (Fig. 6). It was observed that as the dye concentration increased, the adsorption of reactive

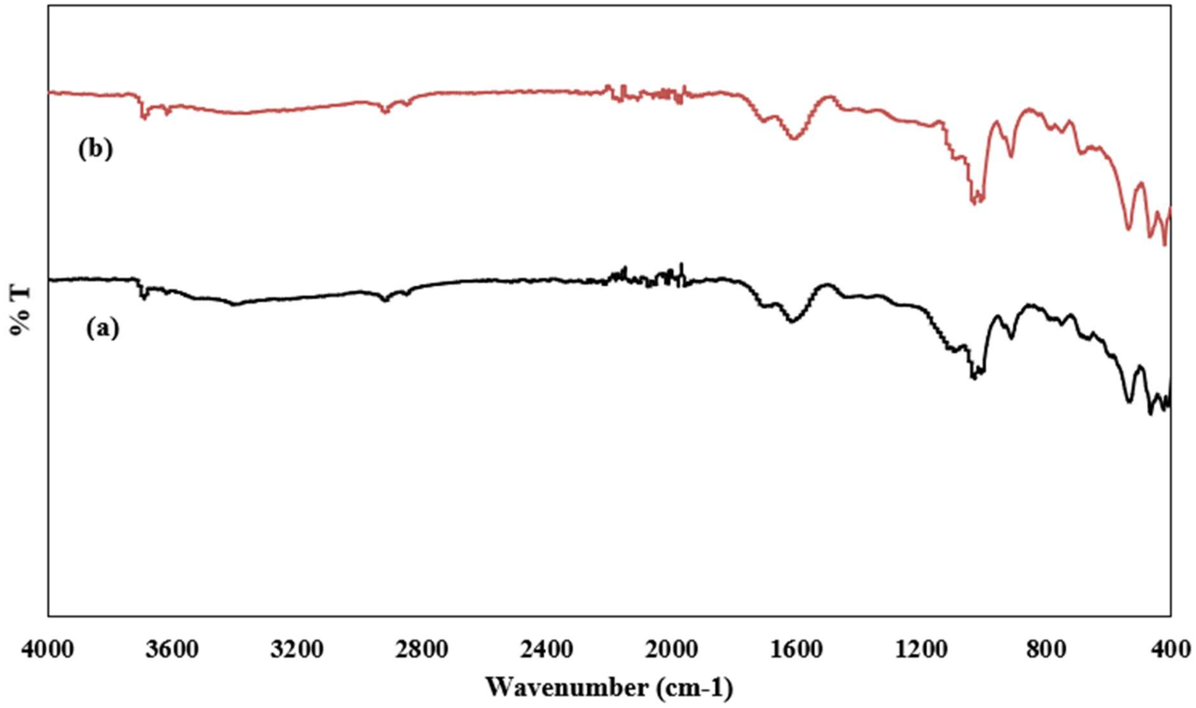

Fig. 2. FT-IR Spectrum of coal-associated soil (a) before and (b) after adsorption.

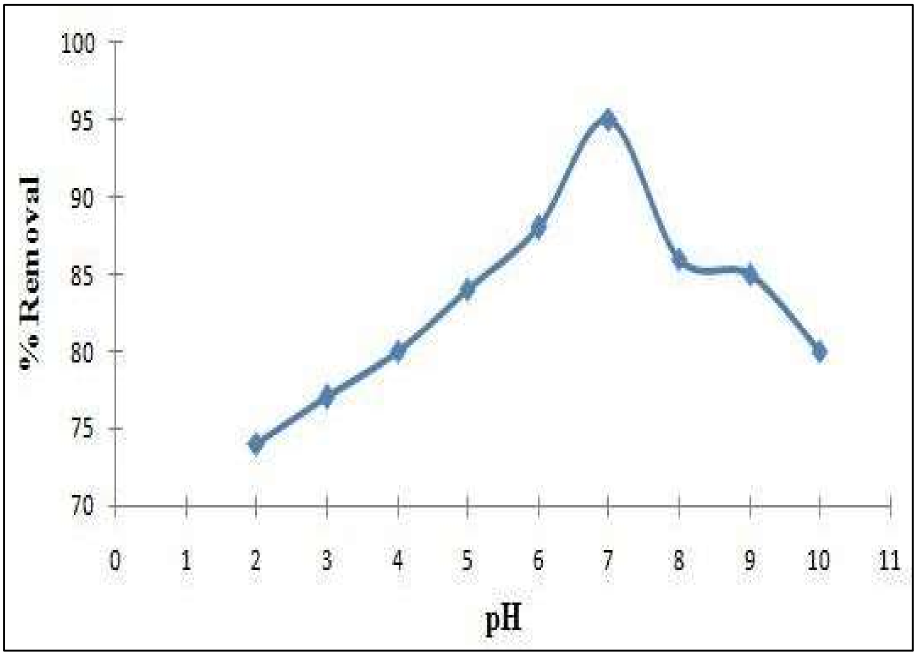

Fig. 3: Effect of pH on adsorption.

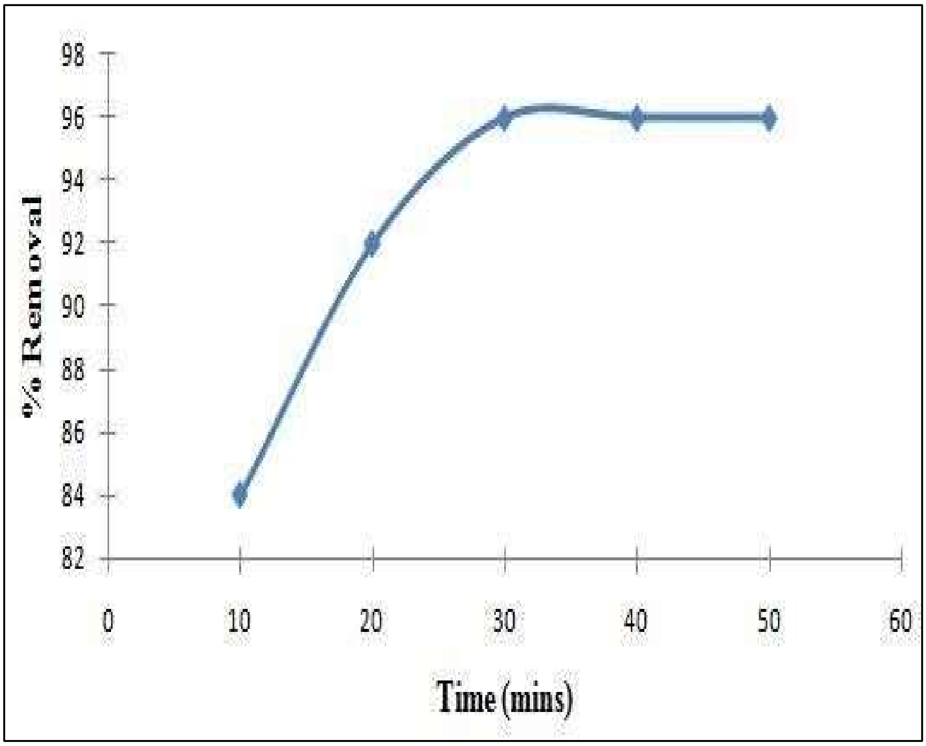

Fig. 4: Effect of contact time in adsorption.

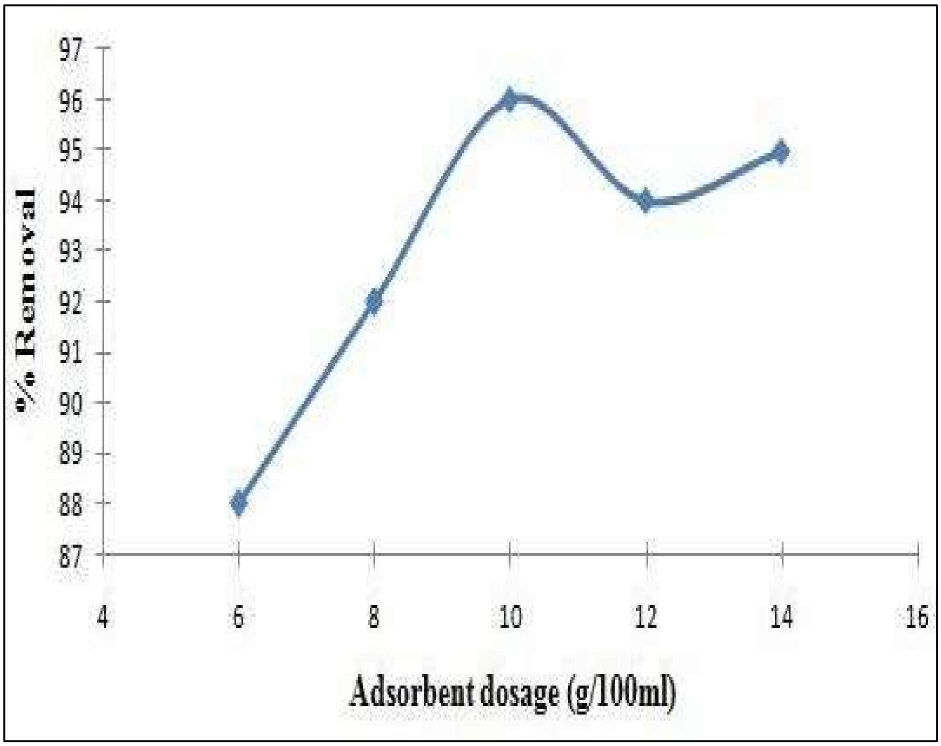

Fig. 5: Effect of Adsorbent dosage in adsorption.

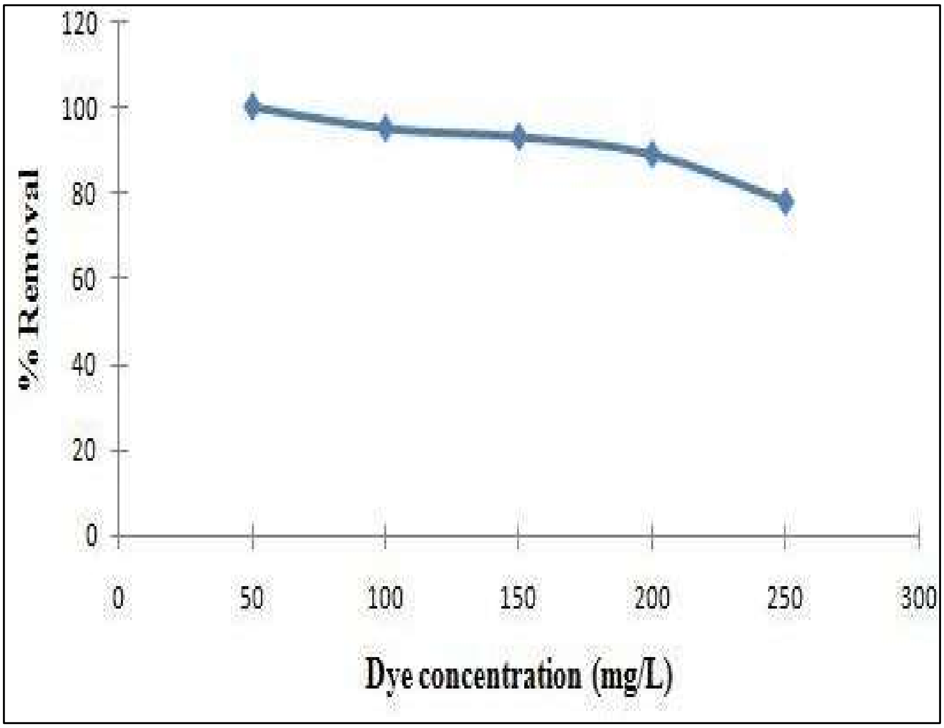

Fig. 6: Effect of Initial Dye Concentration in adsorption.

blue dye decreased. This decrease can be attributed to the limited availability of active sites on the soil's surface at higher dye concentrations, resulting in a reduced percentage removal of dye. Consequently, the optimal dye concentration for effective adsorption was determined to be 100 mg.L^{-1}.

Effect of Temperature

In a batch adsorption experiment, the impact of temperature on the removal of reactive blue dye using coal-associated soil as the adsorbent was investigated. The experiment involved three different temperatures (303K, 313K, 323K) while keeping other parameters constant. Fig. 7 depicts the effect of temperature on the dye removal. It was observed that as the temperature increased, the removal of reactive blue dye decreased. This can be attributed to the weakening of the attractive forces between the dye and the soil at higher temperatures. The results suggest that the adsorption process was exothermic, and the optimal temperature for efficient removal of reactive blue dye was determined to be 303K.

Adsorption Kinetics Studies

An adsorption kinetic experiment was conducted using coal-associated soil as the adsorbent to investigate the rate of reactive blue dye removal from an aqueous solution. The experiment involved varying the initial dye concentration (50 mg.L^{-1} to 250 mg.L^{-1}) and contact time (5 mins to 30 mins) while keeping the adsorbent dosage at 10 g and temperature at 303K.

Two adsorption kinetic models were used, and Table 1 presents the calculated rate constants (k_1, k_2), correlation coefficients (R^2), and adsorption capacities (q_e) for each model. The rate constant for pseudo-second-order kinetics decreased with increasing dye concentration, indicating competition for active sites on the soil's surface. Pseudo-

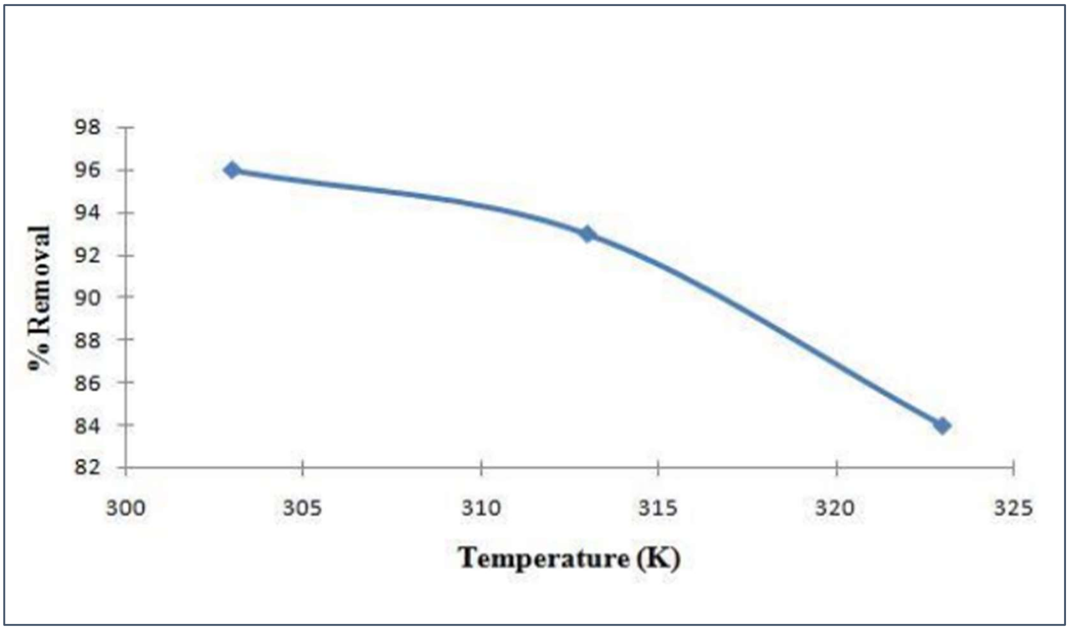

Fig. 7: Effect of temperature on adsorption.

Table 1: Kinetic fit for the adsorption of reactive blue dye onto coal-associated soil.

C_o [mg.L^{-1}]	Exp q_e [mg.g^{-1}]	Pseudo first order			Pseudo second order		
		Calc q_e [mg.g^{-1}]	k_1 (min^{-1})	R^2	Calc q_e [mg.g^{-1}]	k_2 (g.mg^{-1}.min^{-1})	R^2
50	0.50	0.183	-0.0035	0.966	0.523	1.002	0.999
100	0.98	1.203	-0.0054	0.874	1.526	0.044	0.777
150	1.46	1.877	-0.0052	0.932	1.976	0.052	0.941
200	1.86	2.181	-0.0052	0.890	2.227	0.075	0.988
250	2.20	3.346	-0.0063	0.949	2.597	0.073	0.998

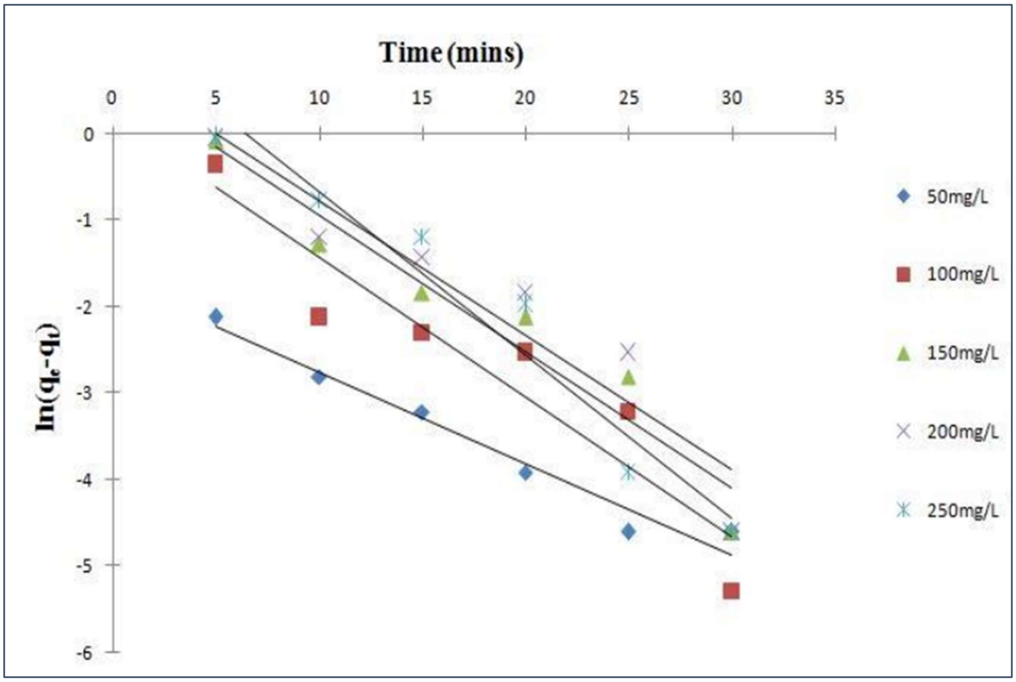

Fig. 8: Pseudo-first-order kinetic fit for the adsorption of reactive blue dye onto coal-associated soil.

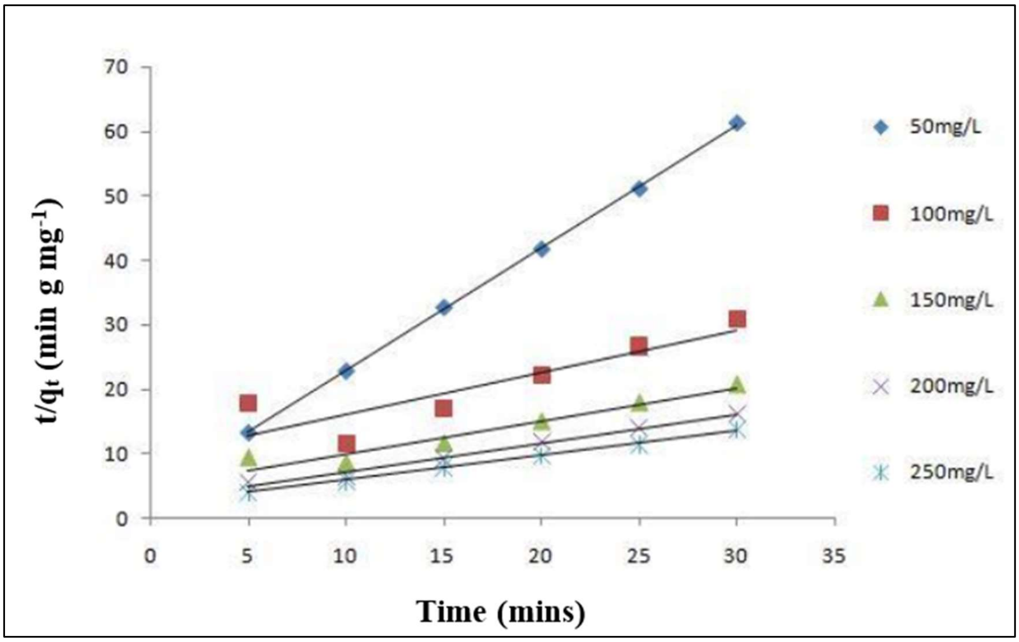

Fig. 9: Pseudo-second-order kinetic fit for the adsorption of reactive blue dye onto coal-associated soil.

first-order kinetics exhibited irregular values, as illustrated in Fig. 8. By comparing the experimental and calculated equilibrium adsorption capacities, it was determined that the pseudo-second-order kinetic model (Fig. 9) provided a better fit, supported by higher R^2 values. Hence, the pseudo-second-order model was considered the best fit for this adsorption process.

Adsorption Isotherm Studies

To gain insights into the adsorption mechanism between

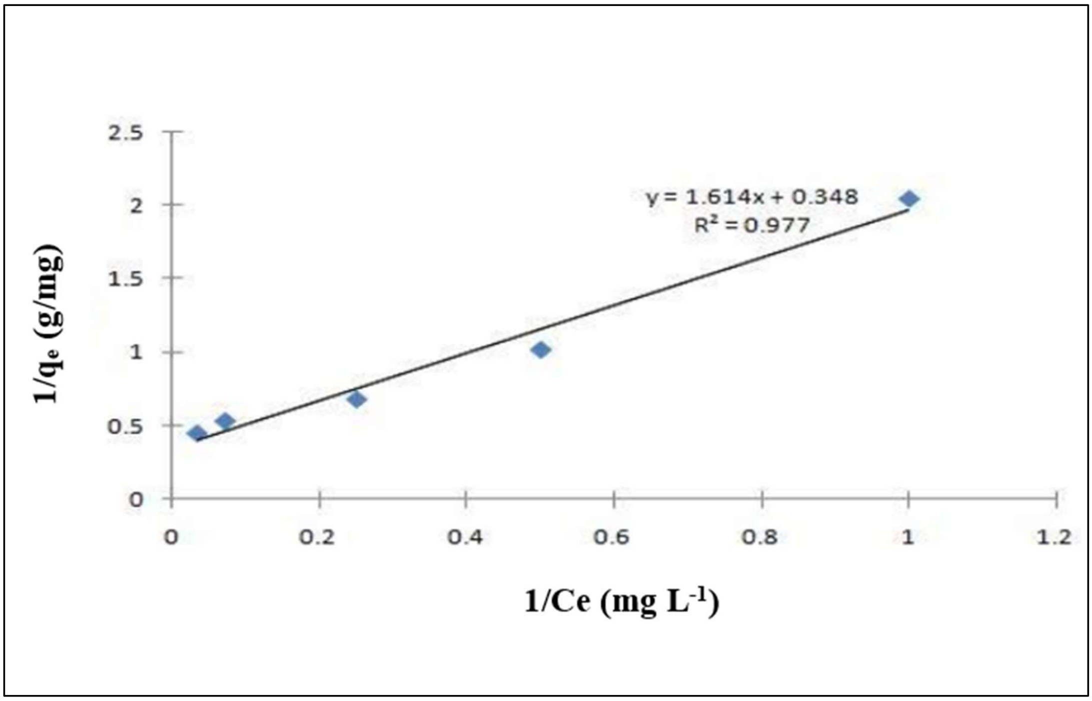

Fig. 10: Langmuir isotherm fit for the adsorption of reactive blue dye onto coal-associated soil.

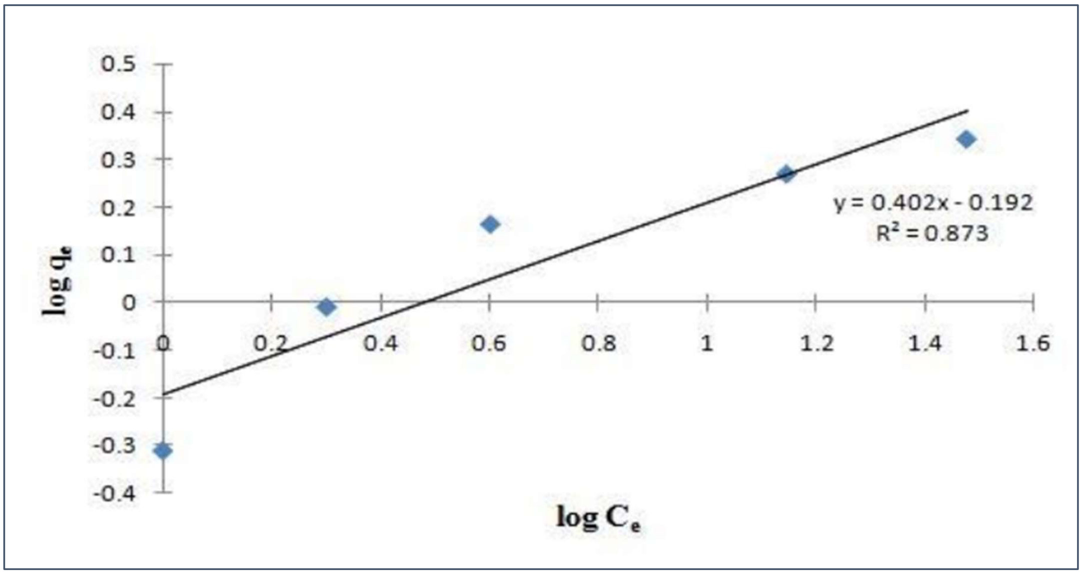

Fig. 11: Freundlich isotherm fit for the adsorption of reactive blue dye onto coal-associated soil.

reactive blue dye and coal-associated soil, an isothermal study was conducted. Adsorption isotherm analysis was performed at equilibrium using two different models: Langmuir and Freundlich adsorption isotherm models. These two-parameter models aimed to determine the maximum adsorption capacity of the coal-associated soil. The adsorption isotherm study results are depicted in Fig. 10 and Fig. 11, showcasing the Langmuir and Freundlich adsorption isotherm plots, respectively. By employing nonlinear equations of different isotherm models and utilizing the experimental data, adsorption isotherm parameters such as correlation coefficients (R^2) and maximum monolayer

Table 2: Isotherm fit for the adsorption of reactive blue dye onto coal-associated soil.

Adsorption isotherm models	Parameters	Values	R^2
Langmuir	q_{max} [$mg.g^{-1}$]	2.8735	0.977
	K_L [$L.mg^{-1}$]	0.2156	
Freundlich	K_F [$(mg.g^{-1})(L.mg^{-1})^{(1/n)}$]	0.8253	0.873
	n [$g.L^{-1}$]	2.4875	

adsorption capacity (q_{max}) were calculated and presented in Table 2.

The analysis revealed that the Langmuir isotherm model exhibited the best fit for the removal of reactive blue dye by coal-associated soil. This conclusion was supported by the higher correlation coefficient (R^2) value compared to the Freundlich isotherm model. Thus, the Langmuir adsorption isotherm model, describing the adsorption process as a monolayer and homogeneous in nature, was identified as the most suitable model for explaining the adsorption behavior of reactive blue dye onto coal-associated soil.

Thermodynamic Studies

A thermodynamic study was conducted to gain insights into the adsorption behavior of reactive blue dye onto coal-associated soil, focusing on the randomness, spontaneity, and nature of the process. The thermodynamic parameters, including Gibbs free energy (ΔG°), change in entropy (ΔS°) and change in enthalpy (ΔH°), were calculated from the

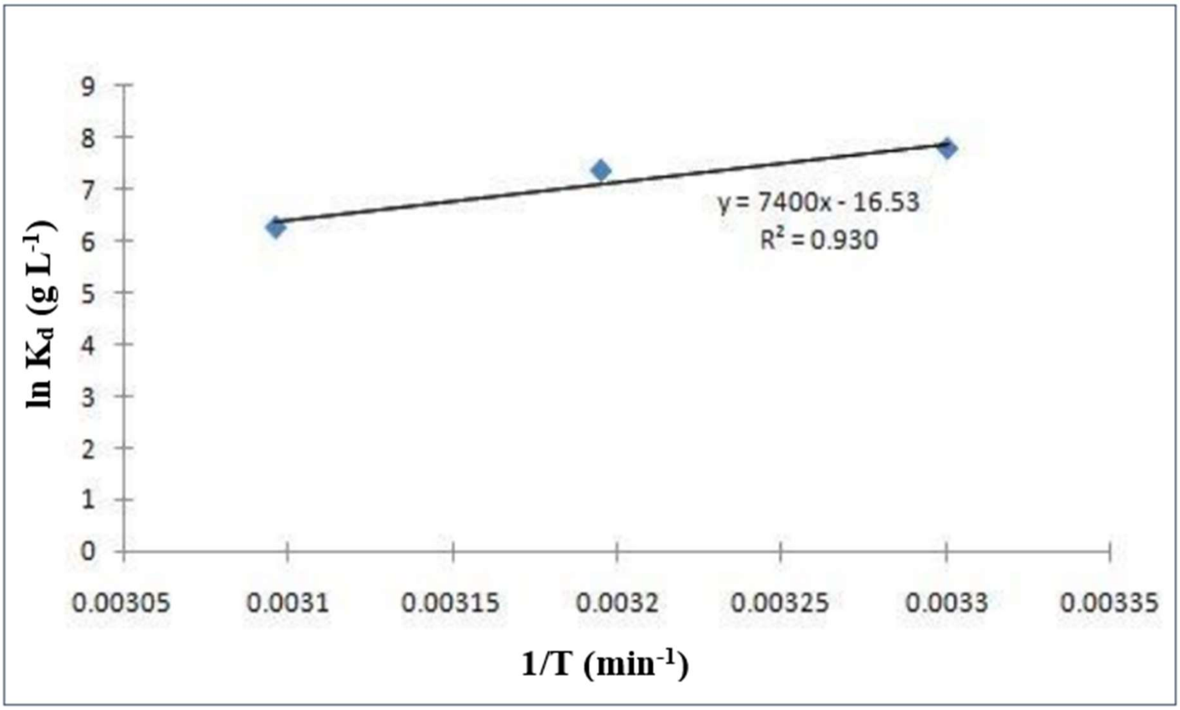

Fig. 12. Thermodynamic parameters for the adsorption of reactive blue dye onto coal-associated soil.

Table 3: Thermodynamic parameters for the adsorption of reactive blue dye onto coal-associated soil.

Concentration of dye [$mg.L^{-1}$]	Temperature [°**C**]	**ΔH**° [$KJ.mol^{-1}$]	**ΔS**° [$J.mol^{-1}$]	**ΔG**° [$KJ.mol^{-1}$]
100	30	-61.523	-137.43	-19.586
	40			-19.121
	50			-16.797

Table 4: Comparison of Various adsorbents used in Reactive Blue Dye Adsorption.

S.No.	Dye	Adsorbent	Kinetic Model Fitted	Isotherm Fitted	References
1.	RB 13	White Iraqi Kaolin clay	-	Langmuir	Baqir and Halbus (2014)
2.	RB	Natural and Modified Wheat Straw	Pseudo-second-order	Freundlich	Mousa and Taha (2015)
3.	RB 222	Industrial waste sludge	Second-order	Freundlich	Gunes and Kaygusuz (2013)
4.	RB 221	Chitosan Glycan	Pseudo-second-order	Langmuir	Chiu et al. (2018)
5.	RB 4	Lemon Peel Bead	Pseudo-second-order	Freundlich	Praipipat et al. (2022)
6.	RB 13	Fe_3O_4 loaded Chitin	Pseudo-second-order	Langmuir	Gautam et al. (2020)
7.	RB 4	Coal-associated soil	Pseudo-second-order	Langmuir	This Study

graph plotted between ln Kd and 1/T, as shown in Fig. 12. The results of the thermodynamic parameters are presented in Table 3. It was observed that the negative value of change in entropy ($\Delta S°$) indicated that the adsorption process was driven by enthalpy. Furthermore, the negative value of change in enthalpy ($\Delta H°$) suggested that the process was exothermic, while the negative value of Gibbs free energy ($\Delta G°$) indicated that the adsorption of reactive blue dye onto coal-associated soil was spontaneous and feasible. Table 4 shows the comparison of various literature that reported the adsorption of reactive blue dye using various adsorbents.

CONCLUSION

The coal-associated soil used in this study is readily available and cost-effective, making it a promising adsorbent for removing Reactive blue dye. Through batch adsorption experiments, various important parameters were optimized, including adsorbent dosage ($10 g.100mL^{-1}$), temperature (30°C), pH (7.0), contact time (30 mins), and initial dye concentration ($100 mg.L^{-1}$). The Langmuir isotherm model effectively described the adsorption of Reactive blue dye onto coal-associated soil, suggesting a monolayer adsorption mechanism. The kinetics of the process were best represented by the pseudo-second-order model, indicating a chemical nature of adsorption. Thermodynamic analysis confirmed that the adsorption process was exothermic, feasible, and spontaneous. Based on these findings, this study demonstrates the potential of coal-associated soil as a favorable and economically viable adsorbent for the removal of toxic dyes from wastewater.

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