

Isothermal, Kinetic and Thermodynamic Study of Rhodamine-B Adsorption using Activated Carbon Synthesize from Geranium Plant Waste

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Abstract:

This work reports the application of activated carbon prepared from geranium plant waste as cheap and effective adsorbent for the removal of Rhodamine B dye (RhB) from aqueous solution. Required characterization of the prepared activated carbon was performed using BET, XRD, and FTIR Spectroscopy techniques, confirming its porous structure and functional surface groups. Batch adsorption experiments were carried out as a function of pH, contact time, initial concentration of the adsorbate, adsorbent dosage and temperature. Removal efficiency was 95.00% in 60 min with 3.0 pH, 0.05gm as dose, 150 mg/L RB concentration and $42 \pm 0.2^\circ\text{C}$ temperatures. The equilibrium data best fitted with Freundlich model. Pseudo second order kinetic model fitted well to the adsorption of RhB on activated carbon prepared from geranium plant waste. The values of free energy change (ΔG°), enthalpy change (ΔH°), and entropy change (ΔS°) indicated the process to be spontaneous, feasible and endothermic in nature. Regeneration of adsorbent was carried out by base treatment.

Keywords: Geranium Plant; Activated carbon; Rhodamine B; Isotherm; Kinetic; Thermodynamic.

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1. INTRODUCTION

Last few decades were witnessed of extensive environmental pollution caused due to increased agricultural mechanization, industrial revolution and technological advancements. One of the most dangerous issues related to water pollution and hence threatening human beings and aquatic life as well is the various synthetic dyes [1]. Processes related to cosmetics, food, textile, paper, plastics, and leather industries are the main source of these dyes [2]. The industrial effluents from various dyes and pigment industries make the water resources highly polluted when they are discharged into it. As a result the various quality parameters of such water body is get negatively impacted [3, 4]. The presence of organic pollutants is one of the serious problems for aquatic systems. The dyes containing coloured effluent discharge into the water body is extremely hazardous for marine life. So, presently the waste water treatments for the dye and allied industries are the topics of high interest among the researchers all over the world.

Various known methods viz., chemical oxidation, photochemical and biological degradation, reverse osmosis, coagulation, flotation, electro chemical treatment, adsorption, etc., has been used for dye removal studies. Being cost effective, operationally feasible and economical wastewaters treatment method, adsorption is most advantageous and widely used method [5, 6]. Activated carbon (AC) has earned much attention as adsorbent material because of its

numerous distinct advantages. Activated carbon owing high surface area, resulting from its porous structure, provides ample binding sites for adsorbate molecules [7]. Because of their abundance, availability and low cost the numerous agricultural waste and by-products are potential sources of activated carbon. Many carbonaceous materials like coconut shell, coal, lignite and wood have been used to produce activated carbon [8].

In this study, activated carbon was produced from Geranium (*Pelargonium graveolens*) stems. Geranium (*Pelargonium graveolens*) is a large bushy shrub. The plant's leaves, flowers, and stems contain oil-producing trichomes, producing pleasant greenish oil. It is used in the food industry and medical field for its antimicrobial activity. It also has antifungal and antioxidant activity. The waste produced after the extraction of oil is used to prepare Activated Carbon.

2. MATERIALS AND METHODS

2.1 Materials: Rhodamine B (99.8% pure, molecular formula $\text{C}_{28}\text{H}_{31}\text{ClN}_2\text{O}_3$, molar mass 479.02 g/mol, and appear as reddish-violet powder) and other essential chemicals used during this work were AR grade reagents obtained from S. D. Fine Chemicals, India and used as received. All the experimental solutions were prepared in double distilled water. The pH of these solutions was set to the desired values with aqueous NaOH (0.1 N) and HCl (0.1 N) wherever necessary.

2.2 Preparation of Activated carbon: Geranium plant waste was collected from nearby agricultural fields of Otur. The collected material was thoroughly washed with tap water, followed by distilled water to make it free from any sort of impurities. This biomass was then dried under solar light and dried material was crushed using a mixer. The biomass powder was further oven-dried at 120 °C until a constant weight was achieved. The dried biomass powder was then effectively impregnated with phosphoric acid (H_3PO_4) as an activating agent and kept for 24 h at room temperature. After impregnation, the material was carbonized in a muffle furnace at 500 °C for 1 h. After cooling phosphoric acid treated material was repeatedly washed with distilled water until it became acid-free (neutral pH). The washed product was then dried and sieved through a 106 μm mesh to obtain powdered activated carbon (AC), which was subsequently used for adsorption studies.

3. RESULTS AND DISCUSSION

3.1 Characterization of Adsorbent:

Characterization of adsorbent was carried out using multipoint Brunaur - Emmett – Teller (BET), Fourier transform Infrared (FTIR) spectra, Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray Spectroscopy (EDS/EDX).

3.1.1 BET of AC adsorbent before adsorption:

Surface area of AC before adsorption was determined by nitrogen adsorption at temperature 77.35K using instrument NOVA Quantachrome TouchWin v1.24. Study assumes a cross-sectional area of nitrogen molecule is 16.2 $\text{\AA}^2/\text{mol}$. The surface area is calculated using BET-Multipoint method and is found to be 1484.78 m^2/g . Pore volume and pore diameter are calculated using BJH adsorption method and they are found to be 0.615 cc/g and 3.396 nm respectively.

3.1.2 FTIR study of AC adsorbent: The FTIR spectra of activated carbon recorded with PerkinElmer Spectrum Two instrument before and after adsorption of RhB is shown in Fig. 1 reveals the functional groups present on the surface of the material. Peaks in the spectrum of GAC before adsorption 3200–3600 cm^{-1} (–OH groups), 1580–1650 cm^{-1} (C=O stretching of carbonyl or carboxyl groups), 1100–1300 cm^{-1} (C–O stretching vibrations of alcohols, phenols, or ethers). Peak in the spectrum of AC after adsorption the changes in intensity of 3200–3600 cm^{-1} (–OH groups) indicating the involvement of these groups in adsorption through hydrogen bonding. The absorption band around 1580–1650 cm^{-1} is assigned to C=O stretching vibrations of carbonyl or carboxyl groups, a slight shift or reduction in transmittance after adsorption indicates interaction between these functional groups and the dye. Peaks situated in the region of 1100–1300 cm^{-1} correspond to C–O stretching vibrations of alcohols, phenols, or ether groups. The noticeable

changes in this region after adsorption further confirm the binding of dye molecules to oxygen-containing functional groups on the carbon surface. The comparison of spectra before and after adsorption confirms the successful adsorption of the dye over the AC / biocarbon surface, mainly through electrostatic interactions, hydrogen bonding, and surface complexation.

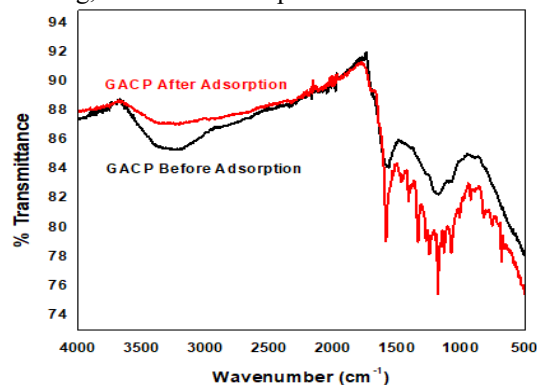


Figure 1: FTIR Spectra of Adsorbent (AC)

3.1.3 XRD study of AC adsorbent: Fig. 2 indicate XRD pattern of Adsorbent (AC). Broad peak centred at 25.58° corresponds to (002) plane of cellulosic carbon whereas presence of merged peak along with main peak indicates that graphite crystals is present in this material as well [9]. All these were the evidence of graphitization of activated carbon prepared from Geranium plant waste. The crystallite size was found to be 0.75nm for activated carbon estimated using Scherrer's equation [10].

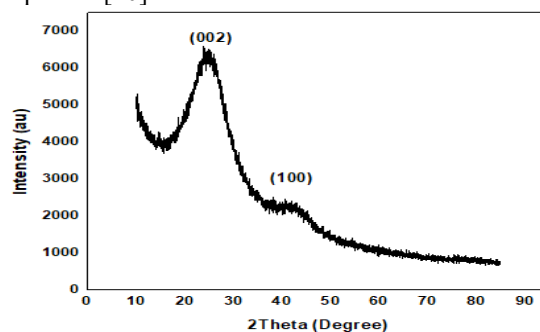


Figure 2: XRD Patterns of Adsorbent (GAC)

3.1.4 SEM study of AC adsorbent: The surface morphology and fundamental physical properties of the adsorbent material was studied with scanning electron microscopy (SEM) as a primary source. Fig. 3(a) indicates the image of AC before adsorption with presence of more rough and porous surface of adsorbent and is responsible for the adsorption of dye molecules into these pores. Fig. 3(b) shows the image of AC after adsorption having relatively smooth surface due to adsorption of dye molecules [11].

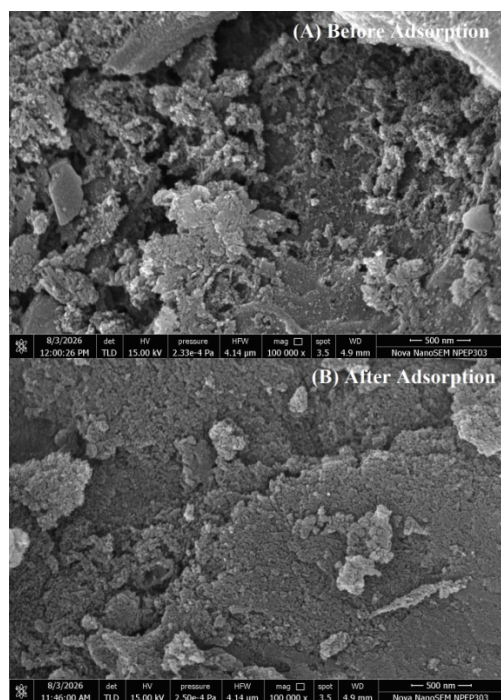


Figure 3: SEM of Adsorbent (GAC) Before (A) and After (B) Adsorption

3.1.5 EDS study of AC adsorbent: Fig. 4 indicates the EDS / EDX analysis of adsorbent / AC before (a) and after (b) adsorption study. The EDS / EDX spectrum of adsorbent before adsorption (a) and after adsorption (b) indicates that the intensities of the peaks corresponding to Nitrogen, Carbon and Oxygen elements has increased may be due to adsorption of dyes molecules. The additional peaks correspond to elements Al, Au, Si etc. are due to conducting medium used for the recording of EDS.

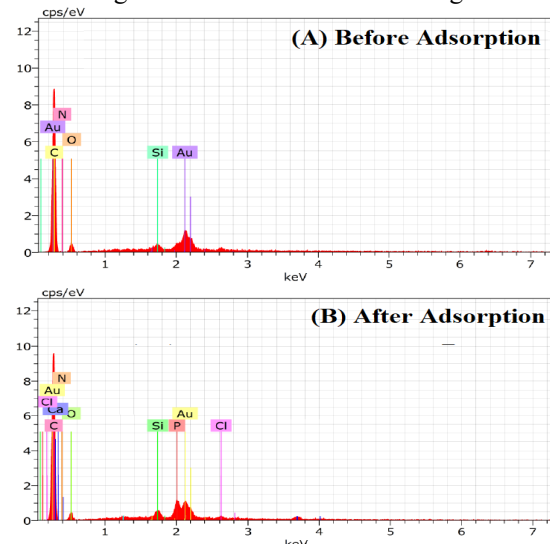


Figure 4: EDS of adsorbent / AC before (a) and after (b) adsorption

3.2 Adsorption study experiments: A series of experiments were performed to study the adsorption of RhB using AC adsorbent. The study was carried out with reference to various operational parameters like pH of dye solution, concentration of dye solution, adsorbent dose,

contact time and temperature. The outcomes of the study are discussed in the following sub-sections.

3.2.1 Effect of pH: The effect of solution pH on the adsorption of Rhodamine B onto activated carbon was investigated over a pH range of 3–10 with constancy of rest of the parameters. The solution pH was set to desired value with 0.1M HCl or 0.1M NaOH. For each experiment, to 50 mL of 150ppm dye solution, 0.05 g of GAC was added at room temperature. As per the study the adsorption efficiency increased significantly under acidic conditions, reaching a maximum removal efficiency of 92.85% at pH 3.

The percentage removal of RhB dye as a function of pH is illustrated in Fig.5. It was observed that the removal efficiency decreased with increasing pH. As a result of more electrostatic interactions between the adsorbent and the adsorbate the percentage adsorption of dye is more under acidic pH [12].

Cationic and monomeric molecular form of RhB molecules at pH 3.0 supports to dye molecule to easily enter into the pore structure of geranium activated carbon. Above pH 3, the zwitter ionic form of RhB dye is exist and is responsible for the aggregation due to the attractive electrostatic interactions between the carboxyl and xanthene groups of the monomers. These aggregated large molecules cannot enter into pore structure of adsorbent hence at high pH percentage adsorption of dye decreases [13,14].

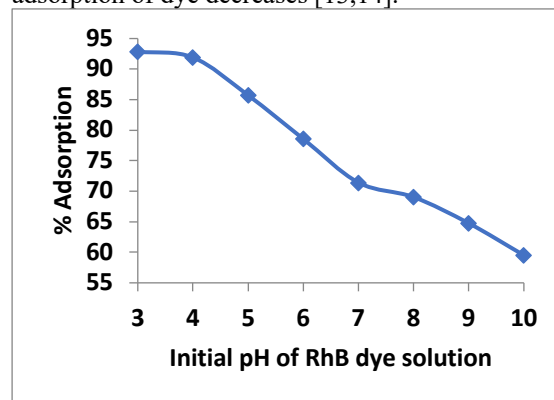


Figure 5: Effect of pH on adsorption of RhB

3.2.2 Effect of Initial Dye Concentration: The adsorption study was carried out with diverse initial concentrations of dye (150 to 200 ppm). Volume of 50 mL of each concentration is taken separately its pH is adjusted to 3 and 0.05 g of Geranium Activated Carbon (GAC) was added. These solutions were stirred for 1h at 500 rpm. Each solution was then centrifuged and the absorbance was measured at λ_{max} . It was observed from Fig.6 that with increase in the initial RB concentration the percentage removal decreased from 92.38 to 76.10, The percentage removal decreased with increase in concentration because at higher concentration the available sites on the adsorbent become limited [9].

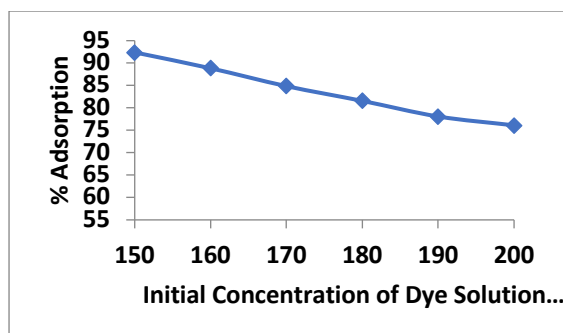


Figure 6: Effect of Initial RhB Dye Concentration on adsorption

3.2.3 Effect of Adsorbent Dose: Adsorbent dose is important parameter of adsorption study, because it helps in determination of capacity of adsorbent that how much amount of adsorbent could adsorb the maximum amount of initial dye concentration by differing the amount of adsorbent while keeping other factors constant like, temperature, initial dye concentration, pH constant. For this study, 50 mL of a 150 mg/L of RhB dye solution was taken. The dose of Geranium plant based activated carbon dose varied from 0.01 g to 0.05 g. These solutions were stirred for 1 hour. It was found that as the amount of adsorbent increases, the removal percentage also increases from 30.95% to 92.38% as shown in Fig. 7. The adsorption increases with increase in adsorbent concentration; this is due to the increase in surface area and availability of more adsorption site. [11, 15]

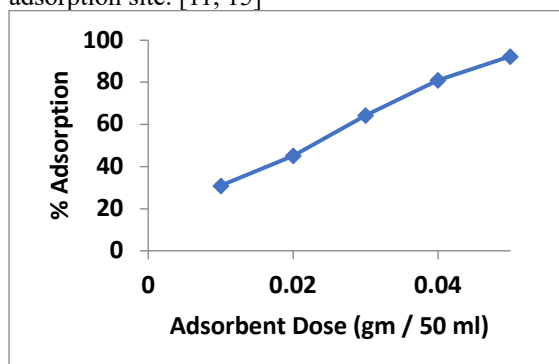


Figure 7: Effect of Adsorbent Dose on adsorption of RhB

3.2.4 Effect of Contact Time: The change in the concentration of the substance with time is called as contact time. The adsorption results at optimum contact time are shown in Fig. 8. For this study, 50 ml of 150 mg/L of RhB dye solution was taken, and 0.05 g of GAC was added. As observed from Figure, for a contact time of 5 min, the adsorption was 58.09%, while it increased with an increase in contact time of 60 min to about 92.09%. The optimum contact time in the Rhodamine B adsorption process using activated carbon of the geranium plant waste was 60 min with a percent adsorption value of 92.09%. The optimum contact duration depends on the balance between adsorption and desorption processes as well as the

available surface area of the adsorbent. An increase in contact time may encourage additional pore formation, which subsequently enhances the surface area available for adsorption [16].

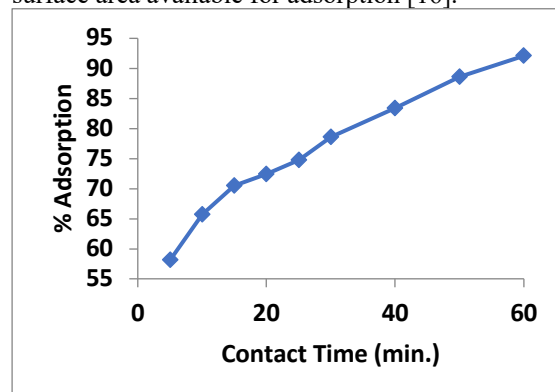


Figure 8: Effect of Contact Time on adsorption of RhB

3.2.5 Effect of Temperature: The effect of temperature of the solution must be considered while analysing the factors influencing the adsorption process. Study at three different temperatures (22, 32 and 42 ± 0.2°C) indicating that with the increasing temperature adsorption percentage increases from 90.95 to 95.00. Because as temperature increases viscosity decreases resulted into rapid mobility of RhB dye to interact with the active sites of activated carbon prepared from geranium waste. This observation indicates that adsorption process is endothermic and it favoured by increase in temperature [10].

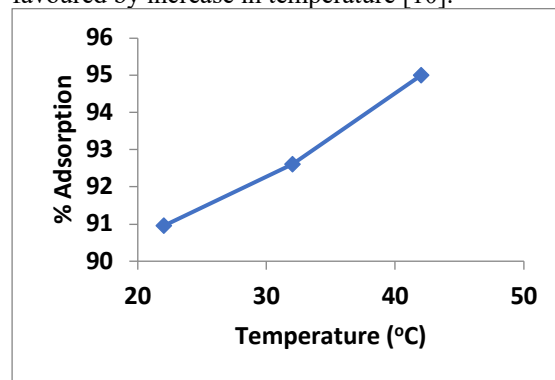


Figure 9: Effect of Temperature on adsorption of RhB

3.3 Adsorption Isotherms: The study of Langmuir and Freundlich adsorption isotherms are essential in assessing the adsorption efficiency of the adsorbent. In this work, we used these models to explain mechanism of RhB adsorption on GAC samples obtained.

3.3.1 Langmuir isotherm: The Langmuir isotherm (Equation 1) describes a single layer molecular adsorption process of adsorbate on the adsorbent surface for maximum adsorption capacity [17].

$$\frac{1}{q_e} = \frac{1}{q_{max}K_L C_e} + \frac{1}{q_{max}}$$

Equation 1

Where, q_e is the amount of adsorbed adsorbate molecule per gram of adsorbent (mg/g),
 $q_{\max}$ is the capacity of the adsorbent monolayer (mg/g),
 K_L is the Langmuir adsorbent constant,
 C_e is the adsorbate equilibrium concentration (mg/L)

This model assumes that all adsorption sites are energetically equivalent and that each site can accommodate only one adsorbate molecule, with no interactions occurring between neighbouring adsorbed species. As a result, the adsorption process reaches saturation once all available sites are occupied, leading to a constant adsorption capacity at equilibrium.

The values calculated with above graph are $R^2 = 0.8628$, $q_{\max} = 153.37$, $K_L = 0.7644$, $R_L = 0.0524$. As value of R_L is in between 0 to 1 the system is suitable for adsorption. Lower value of R_L also indicates high and appreciable adsorption of RhB over activated carbon.

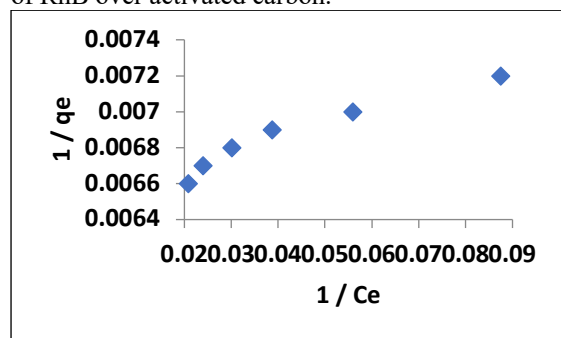


Figure 10: Langmuir Adsorption Isotherm

3.3.2 Freundlich isotherm: The Freundlich isotherm (Equation 2) represents a multiple layer physical adsorption process on a heterogeneous surface with the relatively weak interactions between the adsorbate and adsorbent.

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

Equation 2

K_f is Freundlich constant,

C_e is the concentration of adsorbate under equilibrium concentrations (mg/L),

q_e is the amount of adsorbate adsorbed per unit of adsorbent (mg/g),

n is the the degree of linearity between the adsorbate solution and adsorption process.

This empirical model assumes that adsorption energy decreases with increasing surface coverage due to the non-uniform distribution of active sites. Consequently, the Freundlich isotherm is particularly useful for describing adsorption behaviour over a wide range of concentrations without predicting a saturation limit.

The values calculated with the graph given below are $R^2 = 0.9506$, $K_F = 119.41$, $n = 16.69$, $1/n = 0.059$, from this as the value of n is greater than 1 or the value of $1/n$ is less than 1 indicates that the

RhB dye is favourably adsorbed on synthesized activated charcoal.

As the value of R^2 in case of Freundlich Adsorption Isotherm is more than that of Langmuir Adsorption Isotherm hence present case follows the Freundlich Adsorption Isotherm model.

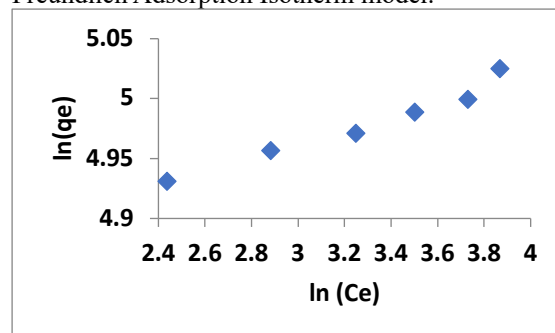


Figure 11: Freundlich Adsorption Isotherm

3.4 Adsorption Kinetics: In a batch adsorption process, kinetic studies provide valuable information regarding the optimum operating conditions, adsorption mechanism, and possible rate-controlling steps. To evaluate the adsorption kinetics, pseudo-first-order and pseudo-second-order kinetic models were applied to the experimental data [16]. For this study, 50 ml (50ppm) of RhB dye solution loaded with 0.05 g of adsorbent was used. The adsorption experiments were conducted with contact times ranging from 10 to 60 minutes. The kinetic parameters obtained from the pseudo-first-order Equation (3) and pseudo-second-order Equation (4) models.

Pseudo-first-order equation

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad \text{Equation (3)}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad \text{Equation (4)}$$

q_t is the amount adsorbed at time t , q_e is the equilibrium amount, t is time in min and k_1 and k_2 are the rate constants.

With equation 3 the values of various parameters obtained are $k = 0.1789 \text{ min}^{-1}$, $q_e = 835.12$ and $R^2 = 0.4936$. With equation 4 the values of various parameters obtained are $k = 0.0011 \text{ min}^{-1}$, $q_e = 147.05$ and $R^2 = 0.9927$.

The coefficient of determination (R^2) obtained from the pseudo-second-order kinetic model (0.9917), calculated from Figure (b), is significantly higher than that of the pseudo-first-order model (0.4936), obtained from Figure (a). Moreover, the calculated equilibrium adsorption capacity ($q_e = 138.14 \text{ mg/g}$) is in close agreement with the experimentally determined value ($q_e = 147.05 \text{ mg/g}$) for the pseudo-second-order model. These results indicate that the adsorption of RhB dye onto the AC adsorbent follows pseudo-second-order kinetics

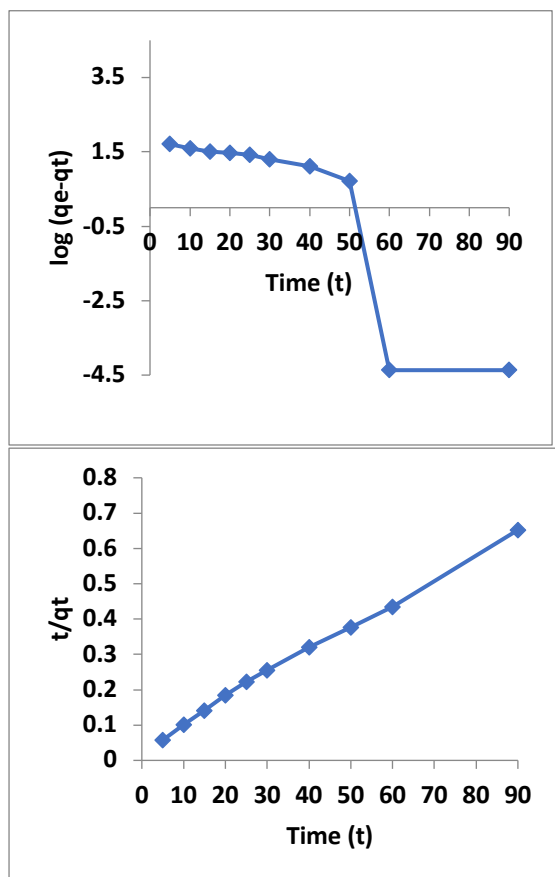


Figure 12: Kinetics Study

3.5 Thermodynamic Parameters: Van't Hoff equation is used to determine the various thermodynamic parameters of the adsorption study [16, 17]. These parameters are vital in deciding whether the adsorption process is spontaneous or non-spontaneous, endothermic or exothermic, feasible or not [18].

Van't Hoff equation

$$\Delta G^0 = -RT \ln K_0 \quad \text{Equation (5)}$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0$$

Equation (6)

ΔG^0 - Change in Gibbs free energy

ΔH^0 - Change in Enthalpy

ΔS^0 - Change in Entropy

K_0 - Equilibrium Constant

From the graph $\ln K$ vs $1/T$ (Fig.13) the values of ΔH^0 (24525.23 J/mol) and ΔS^0 (101.99 J/mol/K) are calculated and ΔG^0 (-7616.91 J/mol) value is obtained from Van't Hoff equation (Equation 5).

The negative ΔG^0 value for present experimental conditions indicates that the

adsorption of RhB over prepared activated carbon (AC) material is feasible and spontaneous process. Also with increase in the solution temperature the feasibility of adsorption of RhB over prepared AC increases. The positive ΔH^0 value is an indication of endothermic nature of adsorption of RhB over prepared AC. The positive ΔS^0 value is an indication of increased randomness at solid-solution interfacial interactions [19, 20] and it also suggest that the adsorbent material has affinity for RhB dye molecules.

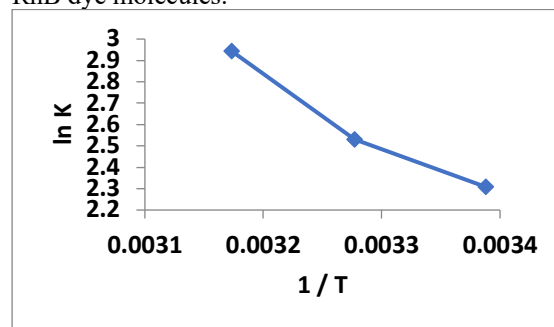


Figure 13: Van't Hoff plot for Thermodynamic Study

3.6 Comparison of Commercial AC and Prepared AC as adsorbent for RB dye: The Commercial AC and Prepared AC were tested in the removal of RhB dye from aqueous solution under identical and optimum operational parameters. It was found that Commercial AC and Prepared AC have percentage adsorption of about 95.72% and 98.54%, respectively.

3.7 Regeneration: After adsorption process dye-saturated adsorbents are generally dumped in landfill. So, to have an economical approach such dye-saturated adsorbent can be regenerated to have minimum adsorbent treatment cost as well as minimum cost of waste disposal [17]. In this study adsorption by geranium activated carbon depends on pH and a maximum adsorption or removal 95.00% was observed at pH 3. As mentioned previously, the removal percentage decreased at the pH increased. Therefore, desorption of RhB is also pH dependent and a proper selection of the eluent is required. An alkaline solution (NaOH) with a high pH should decrease the interactions between the RhB and the adsorbent which would facilitate desorption of RhB. A solution of 0.1N NaOH was used to remove the RhB from adsorbed material. Desorption of adsorbed RhB in the NaOH solution was found in the range of 81.25% to 78.11% in four cycles (Figure 14).

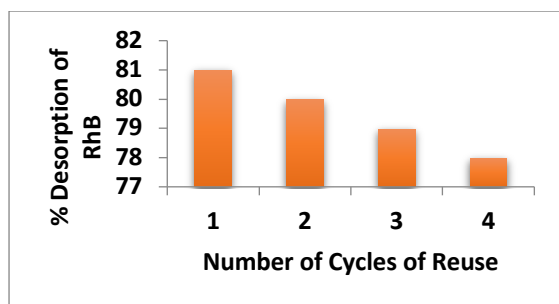


Figure 14: Desorption of RhB with NaOH during reuse

4. CONCLUSION

The activated carbon synthesized from the *Cuscuta Reflexa* plants waste material was used as effective adsorbent material for the removal of Rhodamine B dye from its aqueous solutions.

This investigation showed that the optimum operational parameters for the adsorption of Rhodamine B over activated carbon / charcoal prepared from *Cuscuta reflexa* are pH 3, dye concentration 50ppm, adsorbent dose 50mg/50ml, contact time 60min at room temperature. Also kinetic study reveals that this process follows pseudo-second- order kinetics model. Obtained thermodynamic parameters supports for the feasible and spontaneous nature of the adsorption process involved. The prepared AC has greater adsorption removal efficiency for RhB dye from aqueous solution than commercial AC were tested in the under identical and optimum operational parameters.

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6. CONFLICT OF INTEREST

The authors declare that they have no conflict of interests.

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