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RESEARCH PAPER

Activate CoMo/γ-Al_2O_3 Nano Composite and Use It to Remove Rhodamine B Dye from Aqueous Solution

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ABSTRACT

The (CoMo/γ-Al_2O_3) was used for the adsorption of Rhodamine B from aqeous solution . Spent CoMo/γ-Al_2O_3 hydrodesulfurization catalyst was regevenated using n-hexane to remove the soluble coke and the oxalic acid to remove the foulant elements and insoluble coke by calcinating it at under the temperature of 500 °C for 4 hr. And finally, Rhodamine B was separated from its aquoues solution via the adsorption batch method with varying concentrations of Rhodamine B. They are characterized by FTIR, XRD, FESEM techniques also (BET) techniques was used for mesure the surface area of the nano catalyst.The results shows that the best conditions for adsorption were (pH 2) at a (0.25 g/L) dose with in (40) minutes of contact time. The thermodynamic studies showed that the process is endothermic and spontaneous. The kinetic was described by pseudo-second-order. The equilibrium adsorption data fitted the Freundlich isotherm well.

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INTRODUCTION

Adsorption is the process by which atoms, molecules or ions of another substance accumulate on the surface of a substance [1], and is defined as the Adsorption is the separation process of guest molecules from the environment to the bulk or surface of the solid or liquid phase. The unique features of adsorption, such as low cost and ease of operation coupled with high efficiency, render it superior among other separation and purification techniques such as photoremediation, membrane technology, ion exchange, and electrochemical separation [2]. The adsorbent is the one that suffers adsorption on the surface, and the adsorbent is the surface on which the adsorption process takes place. In the case of the formation of a single molecular layer on the adsorption surface called uni molecular adsorption, and in the case of the formation of several layers on the adsorption surface called multi-layer adsorption [3] Dyes are usually large aromatic molecules, often with many rings linked together. An aromatic ring structure linked to a side chain in the dye molecule structure is necessary for resonance and hence for the transfer of color [4]. The resonance structures responsible for color are those that cause the shifting or appearance of absorption bands in the visible spectrum of light. In the synthesis of a dye, the correlation of chemical structure and color is achieved by a chromogen-chromophore-auxochrome combination. Three essential groups can be found in a dye molecule: the chromophore, auxochrome and matrix [5]. Thus, dyes are organic colorants that contain at least one unsaturated compound (chromophores) and one functional group (auxochromes). The chromophore present

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in the structure may be an aromatic structure containing benzene, naphthalene, or anthracene rings. [6–9]. . Rhodamine B (RhB) is among the toxic dyes due to the carcinogenic, neurotoxic effects and ability to cause several diseases for humans. [10]. The traditional wastewater treatment methods such as sedimentation, chemical flocculation and coagulation, filtration and aeration have some efficiency in the dye removal from the textile effluents. However, these techniques are associated with several disadvantages such as toxic by-products, energy consumption, smelly and require a large area for the treatments [11].

MATERIALS AND METHODS

Instruments:UV-Visible (Shimadzu, Japan 1700) was used to measure the concentration of rhodamine B dye.The pH meter(7110wtw, Germany) was used to mesure the pH of an aqueous solution. The temperature controlled using an isothermal water bath shaker (BS-11, Korea).All nano materials characterized using XRD (Shimadzu company (Japan) (XRD-6000). FTIR (Shimadzu (IR PRESTIGE 21) with KBr pellet technique. The effective range was from 4000 to 400cm^{-1}.SEM (Type Tescan), BET (Q-surf 9600 (USA)).All the chemicals were used without further purification.

Activation of CoMo/γ- Al_2O_3 catalyst

The spent CoMo/γ-Al_2O_3 catalyst with a shape cylindrical (2.5mm x 1.5mm). The catalyst contained residue of oil which was grind and sieve (size 75 Mm) to convert it from (A to B) as show in Fig. 1 and then washed by distilled water and dried in an oven maintained at 100˚C for 2 hours. Finally It is used the in three-step pre-treatment processes for processing and regeneration.

Extraction of soluble coke from CoMo/γ- Al_2O_3

(9g) of spent CoMo/γ- Al_2O_3 catalyst from step above were introduced into extraction apparatus(round bottom and condenser) and treated with n-hexane and refluxed at temperature of (75-85 ºC) for 6 hours with stirring fixed (200 rpm) in order to extract the soluble coke. Then it is filtered to remove the solvent and dried in an oven under (110 ºC) for 3 hours and then the organic matter recovered was weighed.

Leaching of foulant elements

(5g) of spent CoMo/γ- Al_2O_3 treated above with n-hexane were introduced into a soxhlet apparatus again with (50 mL) of aqueous oxalic acid (0.08 M) and extracted for (1h) at a temperature of (75-85 ºC), stirring was fixed at 200 rpm . The leached catalyst were filtered, washed with distilled water for several time, dried in oven at 150 ºC for 2 hours and then cooled down in the dessicator and finally weight.

Removal of insoluble coke from CoMo/γ- Al_2O_3

(0.3g) of all samples (the spent catalysts, the

(A) **(B)**

Fig. 1. Spent CoMo/γ- Al_2O_3.

ones treated with n-hexane only, and the one treated with oxalic acid and n-hexane) were placed in crucibles and introduced in a furnace where insoluble coke was burned at a temperature 500 ºC for (4 hours) . The oxidized samples were cooled down in furnace and transferred to a dessicator and then weight to estimate the amount of volatiles (carbon, moisture and sulfur compounds) removed during this step.

Adsorption process

The dye adsorption by the batch method to study completely different parameters like contact time (10-50) min the dose of adsorbate CoMo/γ-Al_2O_3 (0.5-0.25) g, pH (2-10), the concentration of dye (10-50) ppm temperature (20- 40°C). The samples were agitated a Shaker water bath for (40) min the samples were then filtered during a centrifuge for (15) min at (3500rpm) and then filtered and analyzed spectrophotometrically and calculated percentage of dye adsorption from the solution. The percentage dye adsorption (% adsorption) from the aqueous solution was determined according to the Eq. 1 [12].

$$\% \text{ adsorption} = \frac{(C0-Ce)}{C0} \text{ x100} \quad (1)$$

Where, C_o is the initial m concentrationm of dye solution (mg/L) and C_e is the final concentration (mg/L) after the adsorption.

$$Qe = \frac{(C0-Ce)X\ Vsol}{m} \quad (2)$$

Qe : Quantity of solute adsorbed each unit weight of adsorbent (gm/g). Ce : Equilibrium concentration of solute (gm/L). Vsol :Volume of solution (L). m: mass of adsorbent (g).

RESULTS AND DISCUSSION

FTIR spectrum of the CoMo/γ-A1$_2$O$_3$ was shown in Fig. 2, broad band around (3527 cm^{-1}) related to OH stretching band of γ-Al_2O_3 , another peak can be observed around 1647cm^{-1} which is assigned to OH bending vibrational mode.

The XRD analysis of CoMo/γ-Al_2O_3 was carried out to confirm the crystalline nature of the synthesized nanoparticles. The data of strongest three peaks for CoMo/γ-A1$_2$O$_3$ are shown in Fig. 3. The peaks position and intensities are in a good agreement with those reported in Join committee on power diffraction standards (JCPDS) file The XRD results of CoMo/γ-Al_2O_3 agree to the result in the refrence [13].

The (BET) equation for specific surface area was by the (Brunauer-Emmett-Teller). The results of surface area for the (CoMo/γ-Al_2O_3) nanoparticles are 78.86 m^2/g .The adsorbent average mean pore diameter and total pore volume were estimated

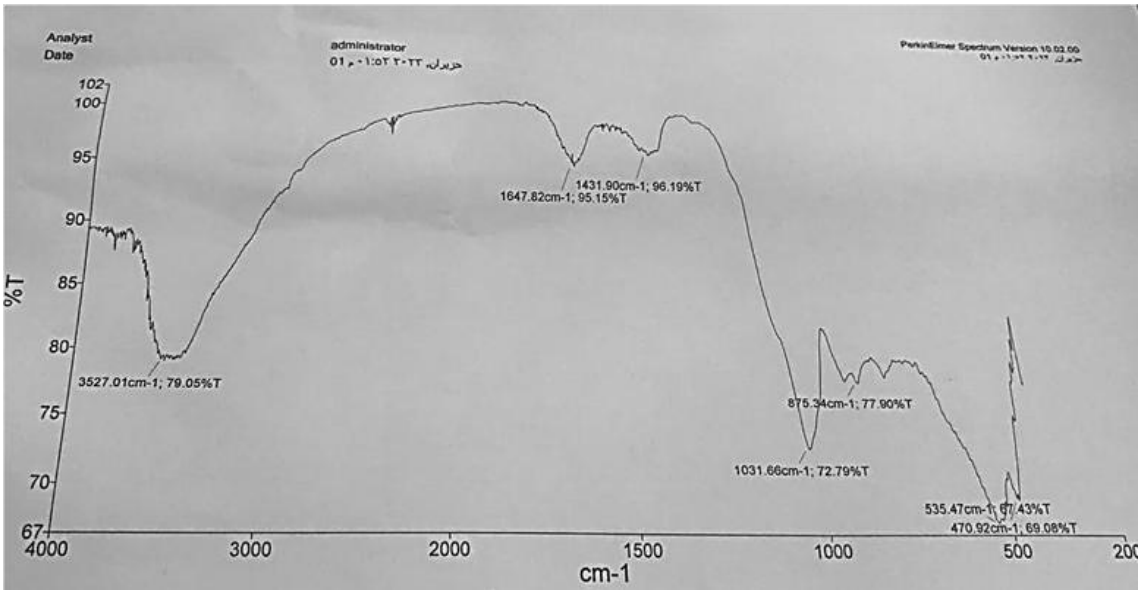

Fig. 2. FT IR spectrum of CoMo/γ-Al_2O_3.

to be 6.5036 nm and 0.2145 cm^3/g respectively. Thus, the presenceof mesoporous structures and high pore volume offers afavorable condition for the liquid phase adsorptive removal of dye [14].

FESEM was used for the morphological assessment of obtained CoMo/γ-Al_2O_3 -NPs as depicted in Fig. 4 [15]. As per the FESEM images, the nanoparticles predominantly appear to be spherical in shape with little shape variation. The high resolution images clearly show the tapering surface features where the nanoparticles distribution over the entire surface can be seen.

These extremely fascinating surface features allow nanoparticle to possess higher active sites compared to other morphologies. FESEM images revealed that average size of CoMo/γ-Al_2O_3 (16.44.21.79nm).

Determination of contact time

Add 30 ml of dye solution (10 ppm) to 0.25 g of CoMo/γ-Al_2O_3 to adsorb (RhB) dye. The temperature and pH were kept constant at 25 oC

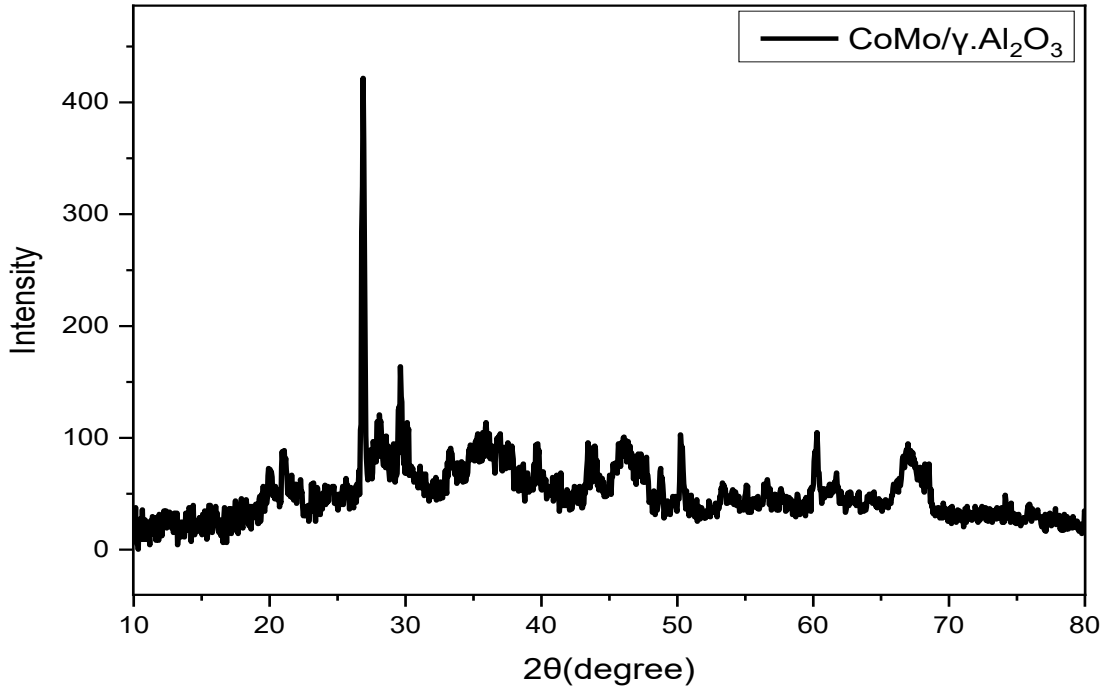

Fig. 3. XRD spectrum of CoMo/γ-Al_2O_3 nanoparticles.

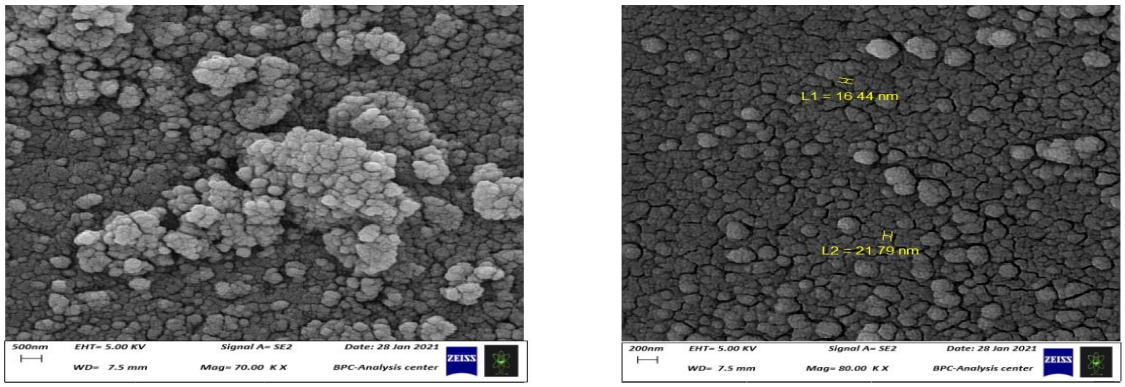

Fig. 4. FESEM images of CoMo/γ-Al_2O_3 nanoparticles.

during the adsorption studies.Fig. 5 displayed the data. The removal rises until the equilibrium time (40 min), at which point the greatest amount of (RhB) dye adsorption takes place. As time passes, the amount of adsorbent grows, creating a large number of active places on the adsorption surface where molecules can move. The action of the dye adsorption increasing quickly is what distinguishes the dye from the solution and to the surface. Fast initial contact with the catalyst causes rapid adsorption; however, as contact time increased, the amount of active sites gradually dropped and the driving force weakened because the adsorption process was slow and it took a long time to reach adsorption equilibrium [16].

Effect of adsorbent weight

The weight of (CoMo/γ-Al_2O_3) had an impact on the amount of Rhodamine B dye that could be absorbed. Weights ranging from (0.5 to 0.25gm) were used. The temperature and pH in the adsorption experiments were kept constant at 2 and 25 oC. As demonstrated in Fig. 6, 0.25 gm is the best removal level.

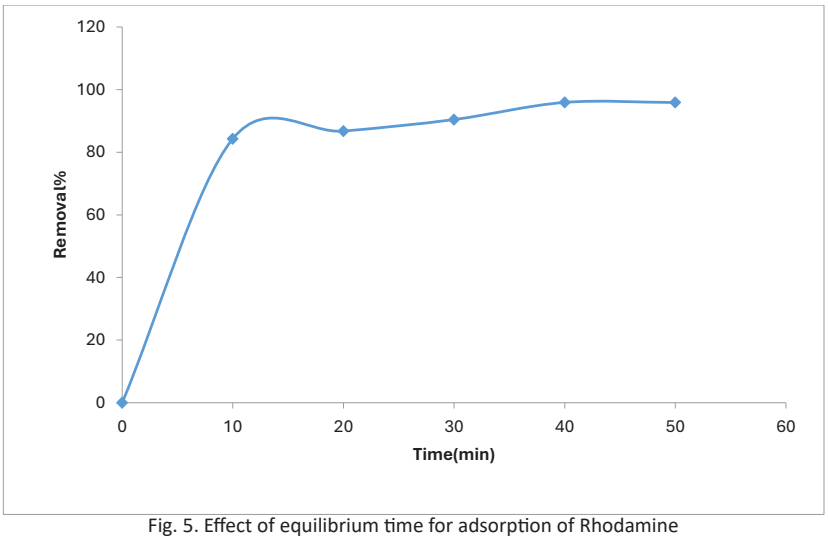

Fig. 5. Effect of equilibrium time for adsorption of Rhodamine

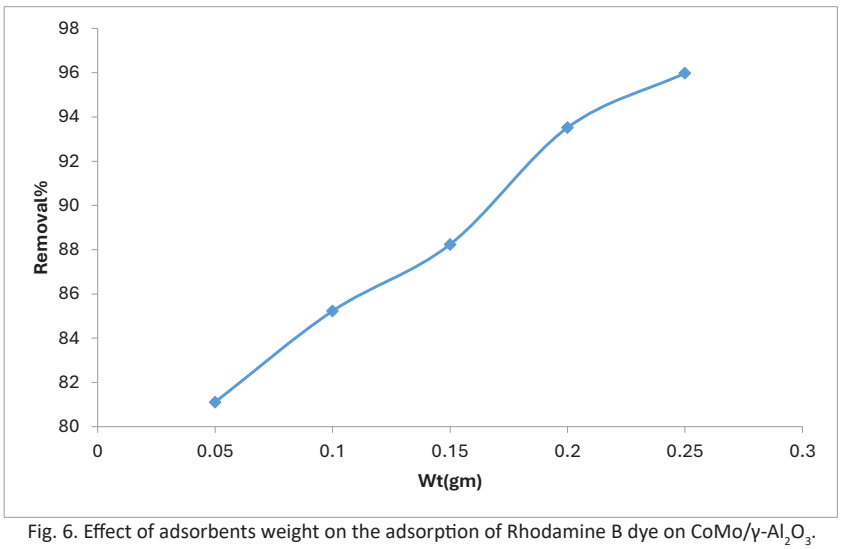

Fig. 6. Effect of adsorbents weight on the adsorption of Rhodamine B dye on CoMo/γ-Al_2O_3.

Effect of pH

Five samples with the same concentration (10 ppm) were mixed individually with (0.25)gm of the CoMo/γ-Al_2O_3 catalyst to determine the effect of pH at constant (conc. Temp. and time). The best elimination occurs when the pH is 2, as illustrated in Fig. 7.

Adsorption Kinetics

Kinetics of Both pseudo first and second order applied to the adsorption data [17].

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

$$t / qt = 1/ k_2 q_e^2 + (1/ q_e) t \quad (4)$$

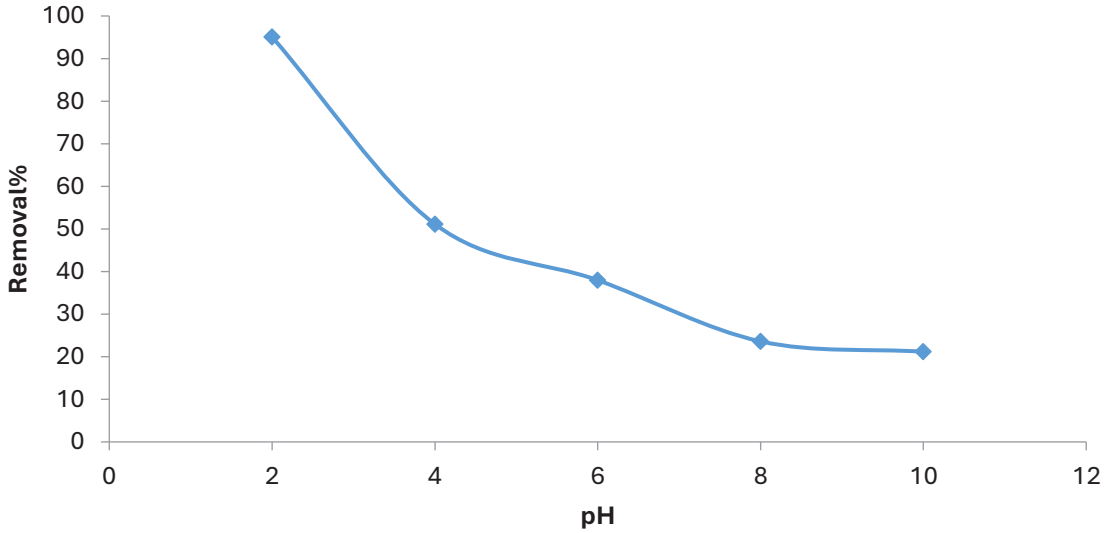

Fig. 7. Effect of pHon the adsorption of Rhodamine B dye on CoMo/γ-Al_2O_3.

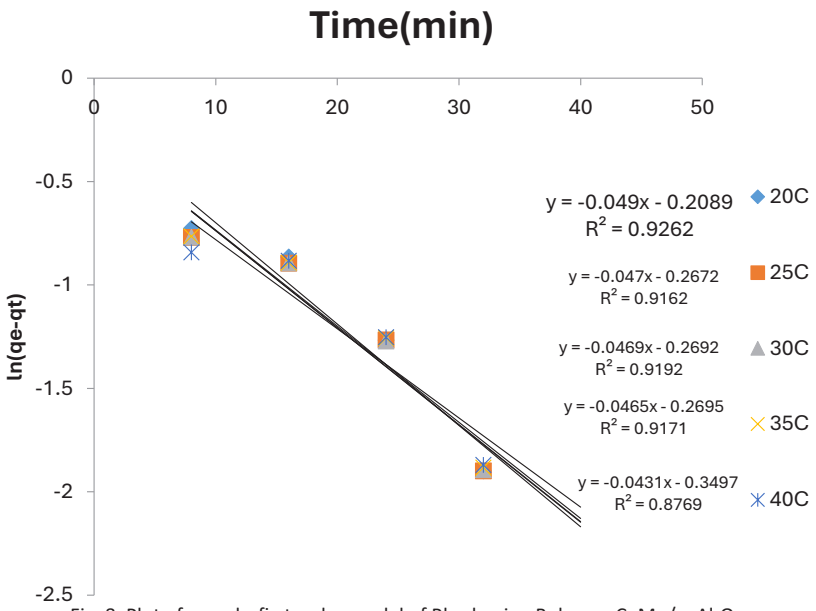

Fig. 8. Plot of pseudo-first order model of Rhodamine B dye on CoMo/γ- Al_2O_3.

Where q_e(mg/g) represents the amount of dye absorbed at equilibrium, q_t(mg/g) represents the amount of dye adsorbed at time (t) min, k1(min^{-1}) represents the pseudo-first order rate, and k_2(g/mg. min) represents the pseudo-second order rate constant. Figs. 8 and 9 show the data obtained from the slope of the plots of ln(q_e-q_t) vs. time for K_1 and t/q_t vs. time for K_2, respectively. The kinetic parameters for adsorption are shown in Table 1. The correlation coefficients are analyzed and explained in terms of second-order kinetics.

Adsorption isotherms

Adsorption isotherm is very impotant to identify mechanism of adsorption process. The adsorption of Rhodamine B dye using CoMo/γ-Al_2O_3 was determined at tempratures (20-25-30-35-40 ºC) and concentration (10-20-30-40-50 ppm) . Adsorption isotherms were plotted as shown in Figs. 10 and 11. This study was carried out by matching the equilibrium data to four different isotherms: Langmuir, Freundlich, Temkin,and Dubinin isotherms respectively.

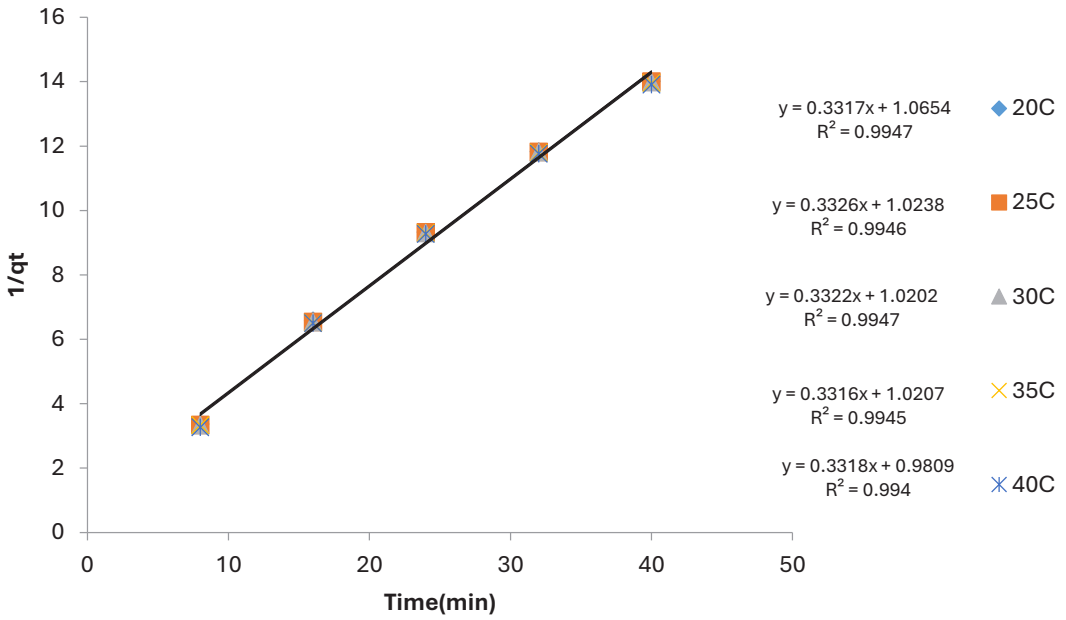

Fig. 9. Plot of pseudo-second order model of Rhodamine B dye on CoMo/γ-Al_2O_3

Table 1. Kinetics parameters for adsorption of of Rhodamine B dye on CoMo/γ-Al_2O_3.

C_o	T (ºC)	pseudo-first-order				pseudo-second –order		
		k_1 Min^{-1}	q_e (exp.)	q_e (calc.)	R^2	qe (calc.)	K_2 $g.mg^{-1}.min^{-1}$	R^2
10 ppm	20	0.049	2.8539	0.8114	0.9262	3.014	0.1032	0.9947
	25	0.047	2.8563	0.7655	0.9162	3.0066	0.1080	0.9946
	30	0.0469	2.8599	0.7639	0.9192	3.0102	0.1081	0.9947
	35	0.0465	2.8659	0.7637	0.9171	3.0156	0.1077	0.9945
	40	0.0431	2.8746	0.7078	0.8765	3.0138	0.1122	0.994

Langmuir isotherm

The Langmuir model supposed that intermolecular forces lowering rapidly with distance. It useful to predict the presence of monolayer coverage of the adsorbate on the outer surface of adsorbent. The Langmuir isotherm linear formula is given by the Eq. 5 [18].

$$\frac{Ce}{Qe} = \frac{1}{KL\ qmax} + \frac{Ce}{qmax} \quad (5)$$

(Ce) : is the equilibrium concentration of adsorbate(mg/L), qmax,Qe are the maximum adsorption capacity of the adsorbent corresponding to complete monolayer coverage

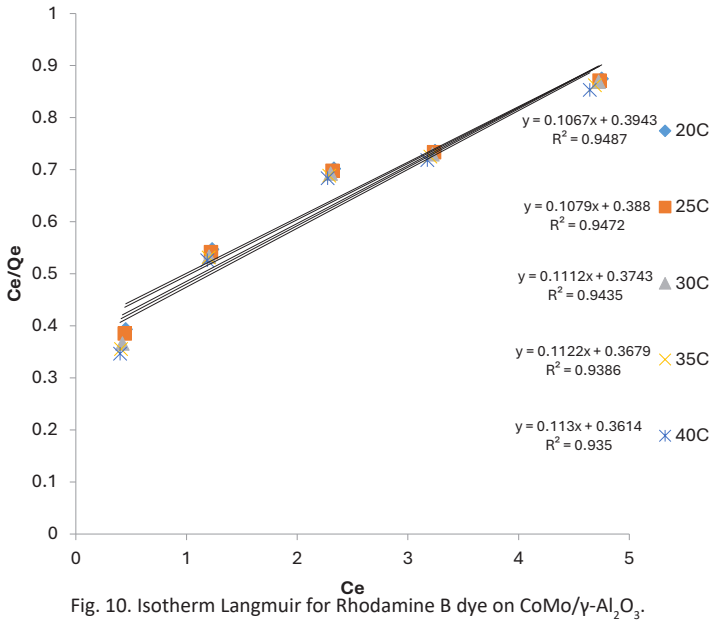

Fig. 10. Isotherm Langmuir for Rhodamine B dye on CoMo/γ-Al_2O_3.

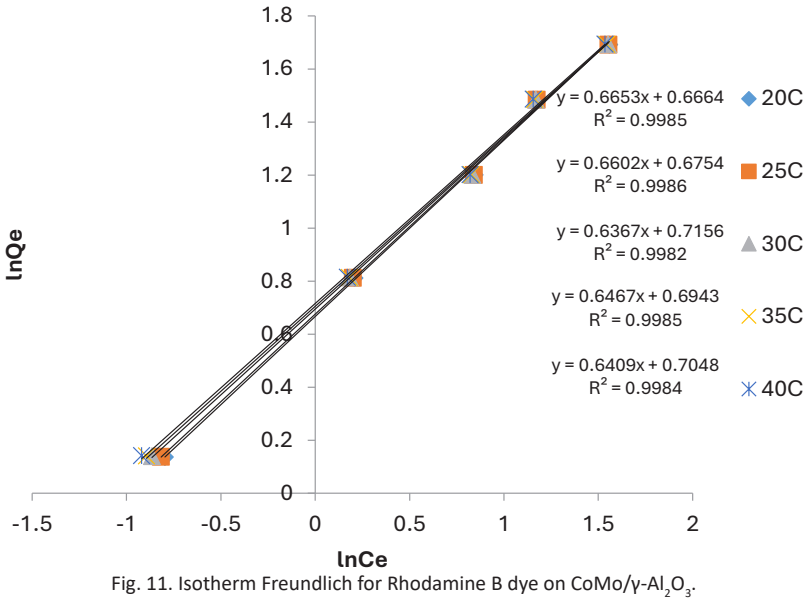

Fig. 11. Isotherm Freundlich for Rhodamine B dye on CoMo/γ-Al_2O_3.

of the surface (mg/g) and capacity atequilibrium (mg/g) and (K_L) is Langmuir constant (L/mg). Straight line with slope ($1/q_{max}$) and intercept of ($1/K_L\ q_{max}$) will obtained when we plot Ce/qe against Ce as shown in Fig. 10. .Adimensionless constant separation factor of Langmuir isotherm (RL) was also calculated using Eq. 6 :

$$R_L = 1/\ (1+KL\ C_o) \quad (6)$$

C_o is the initial concentration of (RhB)dye solution (mg/L) and K_L (L/mg) is the Langmuir adsorption constant .

Freundlich isotherm

The Freundlich isotherm assumes that the adsorption occurs on a heterogeneous medium through multilayer means and that the amount of adsorbate adsorbed increases infinitely with an increasein concentration. It is the most popular model for a single solute system. It is based on the equilibrium distribution of molecules of solute between the solid and aqueous phase. This model is expressed by[19]:

$$\ln Qe = \ln K_F + (1\ /\ n)\ \ln Ce \quad (7)$$

Qe : Weight of adsorbed material in mg / g
Ce : Concentration at equilibrium in gm / L
K_f : the Freundlich constant it is a measure of adsorption capacity

n : a constant value that expresses adsorption affinity and depends on the type of surface and the nature of the adsorbent and temperature.

When, plotting lnQe vs. lnCe, we get a linear relation with a slope of (1/n) and an intersept of $\ln K_f$ as shown in Fig. 11.The Freundlich constant (K_F) decreases with increasing the temperature and this indication for exothermic reaction. The values of n are larger than 1, which represents a favorable removal condictions.

Temkin isotherm

The Temkin isotherm model [20], has been developed on the concept of chemisorption. It assumes that the heat of adsorption of the molecules of the adsorbate linearly decreases with adsorbent layer coverage due to adsorbate-adsorbent interactions. The Temkin model can be expressed as:

$$Qe = B\ \ln KT + B\ \ln Ce \quad (8)$$

Qe: is the amount of adsorbate adsorbed at equilibrium(mg/g), B is related to the heat of adsorption (L/mg), K_T is the equilibrium binding constant (L/mg), C_e is the equilibrium concentration of adsorbate (mg/L). When, plotting Qe vs. LnCe, we get a linear relationship with a slope of (B) and an intersept of (BlnKT) as shown in Fig. 12.

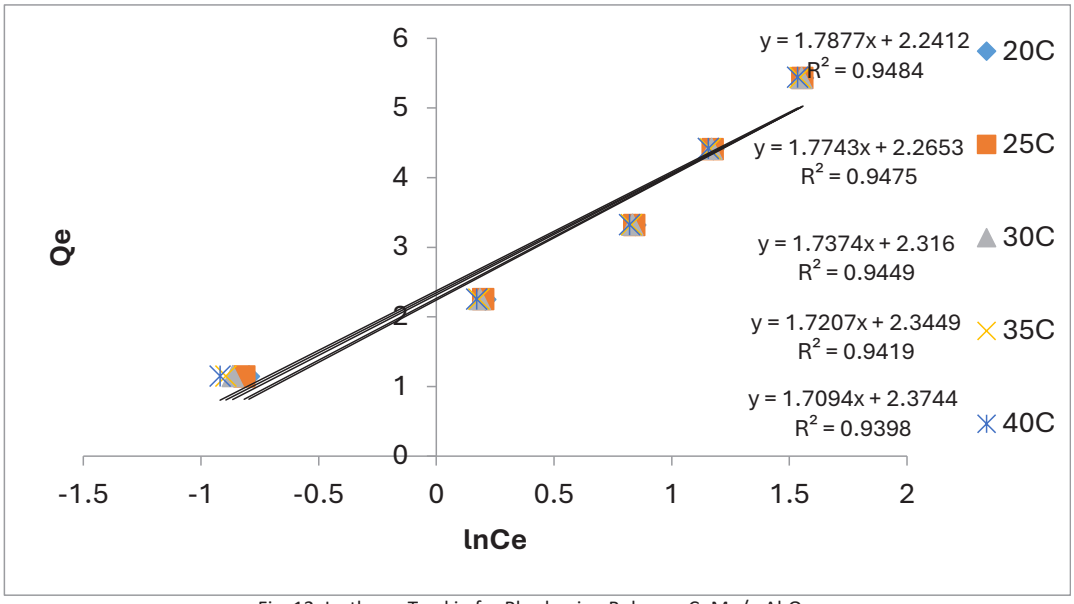

Fig. 12. Isotherm Temkin for Rhodamine B dye on CoMo/γ-Al_2O_3.

Dubinin-Radushkevich isotherm

The Dubinin-Radushkevich is expressed as :

$$\ln Qe = \ln q_{max} - \beta\,\varepsilon^2 \quad (9)$$

$$\boldsymbol{\varepsilon = RT\ln\left(1+\frac{1}{Ce}\right)} \quad (10)$$

where: Qe, is the amount of adsorbate adsorbed at equilibrium, q_{max}: is the maximum adsorption capacity. B, is the Dubinin-Radushkevich constant, R is the universal gas constant. T: is the absolute solution temperature, Ce: is the equilibrium concentration of adsorbate.

When, plotting lnQe vs. ε2, we get a linear relationship with a slope of (β) and an intersept of (lnqmax) as shown in Fig. 13.The adsorption energy E helps in determining the nature of adsorption. The adsorption is physical if E ranges from 1 to 8 kJ/mol.If the value of E ranged between 9 and16 kJ/mol, it is chemical adsorption.

$$E=\frac{1}{\sqrt{\beta}} \quad (11)$$

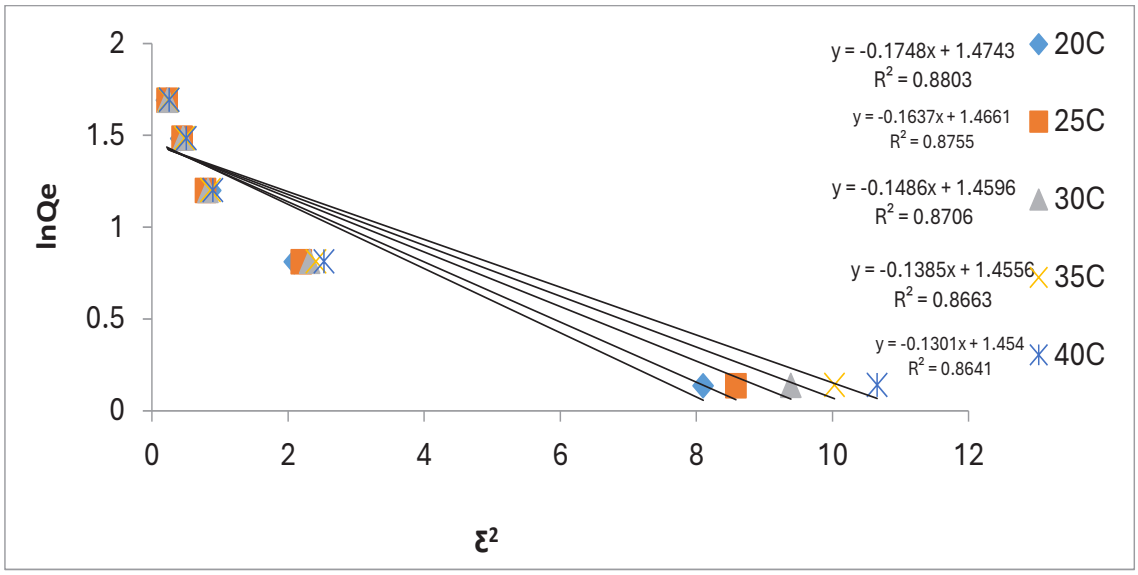

Fig. 13. Isotherm Dubinin for Rhodamine B dye on CoMo/γ- Al_2O_3.

Table 2. The calculated adsorption parameters of the four used isotherms.

	Langmuir				Freundlich		
$(C^0)T$	K_L	R^2	Q_{max}	R_L	R^2	1/n	K_F
20	0.2706	0.9487	9.372	0.2698	0.9985	0.6653	1.947
25	0.2781	0.9472	9.267	0.2644	0.9986	0.6602	1.9648
30	0.2971	0.9435	8.992	0.2518	0.9982	0.6367	2.0454
35	0.3049	0.9386	8.912	0.2469	0.9985	0.6467	2.0023
40	0.3126	0.935	8.849	0.2423	0.9984	0.6409	2.0234
	DKR				Temkin		
R^2	E	q_{max}	β	R^2	B	K_T	
0.8803	0.5912	4.3679	0.1748	0.9484	1.7877	2.2412	
0.8755	0.5721	4.3323	0.1637	0.9475	1.7743	2.2653	
0.8706	0.5451	4.3042	0.1486	0.9449	1.7374	2.316	
0.8663	0.5263	4.2913	0.1385	0.9419	1.7207	2.3449	
0.8641	0.5100	4.2802	0.1301	0.9398	1.7094	2.3744	

Thermodynamic studies

Thermodynamic parameters are used to observe the feasibility and the nature of adsorption process, adsorption studies of Rhodamine-B onto CoMo/γ-Al_2O_3 ctalyst have been conducted at five different temperature: 293, 298,303,308 and 313 K. Thermodynamics parameters such as the free energy change (ΔG), enthalpy change (ΔH) and entropy change (ΔS). (ΔG)were calculated from the Eq. 12 :

$$\Delta G^\circ = -RT \ln K \quad (12)$$

That's where, K : equilibrium constant ,the amount of heat associated with the adsorption(DH) was calculated by plotting InXm versus (1 / T) according to the Eq. 13.

$$\ln Xm = -\Delta H / R\,T + K \quad (13)$$

Using Eq. 14, the value of ΔS was calculated:

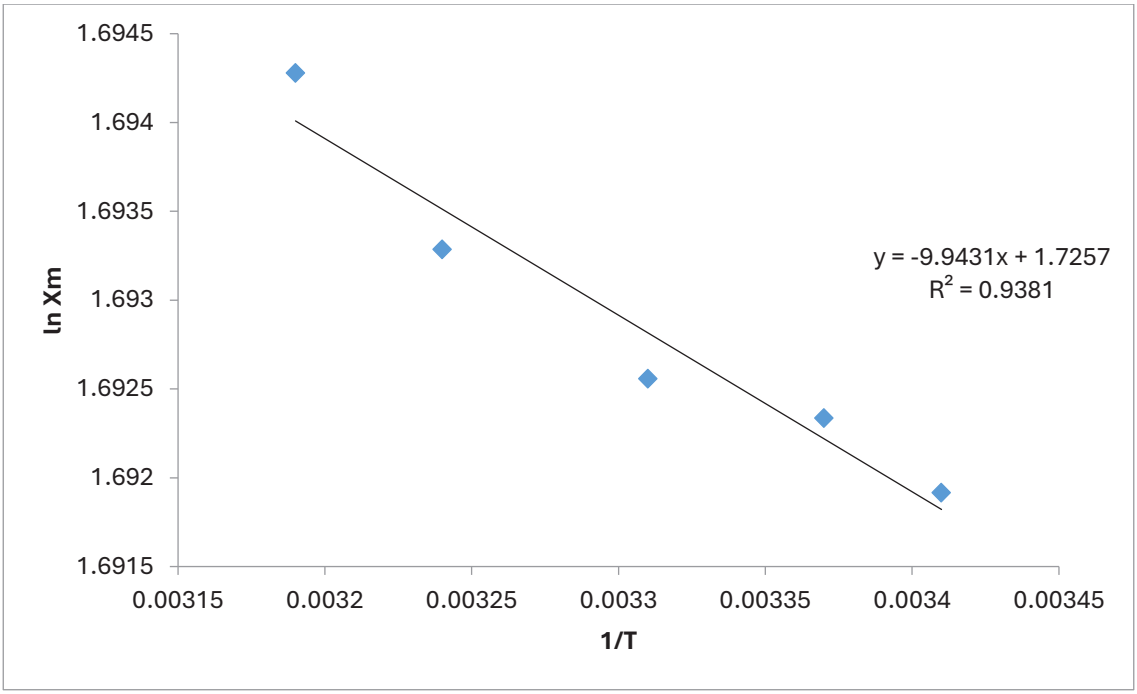

Table 3. Values of thermodynamic functions for adsorption Rhodamine B dye on CoMo/γ-Al_2O_3.

C_e (mg/L)	Thermodynamic function	20 °C	25 °C	30 °C	35 °C	40 °C
50ppm	ΔH kJ.mol^{-1}			0.0826		
	ΔG kJ.mol^{-1}	-0.3253	-0.3418	-0.3534	-0.3791	-0.4129
	ΔS KJ.mol^{-1} K^{-1}	1.392	1.424	1.438	1.499	1.583

$$\Delta G = \Delta H - T\Delta S \quad (14)$$

When, plotting lnXm vs.1/T, we get a linear relationship and the slope represent((- ΔH)/R) as shown in Fig. 14 [21].

CONCLUSION

The results of this study show that the removal of Rhodhamine B increase with decrease the pH. The maximum percentage removal at pH 2 . The proceses of removal Rhodamine B dye on CoMo/γ-Al_2O_3 is endothermic and spontaneous whis is determined by thermodynamic studies . The kinetic was described by pseudo-second-order. The equilibrium adsorption data fitted the Freundlich isotherm well.E was smaller than 8KJ/mol indicate that influence the physical force .

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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