



Adsorption kinetics and thermodynamics of hazardous dye Tropaeoline 000 unto Aeroxide Alu C (Nano alumina): a non-carbon adsorbent

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ABSTRACT

The present paper is aimed to investigate and develop adsorption methods for colour removal from wastewater using waste material a non-carbon adsorbent Nano alumina (NA). The unique properties of Nano materials have promisingly exhibited in solving many environmental issues. The high surface area and unique adsorption capacity of NA were successfully utilized in removing water-soluble azo dye, Tropaeoline 000 from wastewater. Equilibrium isotherms for the adsorption of the dye were measured experimentally. Results were analysed by the Freundlich and Langmuir equation at different temperatures and determined characteristic parameters for each adsorption isotherm. Specific rate constants of the processes were calculated by kinetic measurements and pseudo-second-order adsorption kinetics was observed in each case. Thermodynamic parameters like free energy (ΔG), enthalpy (ΔH) and entropy (ΔS) of the systems were calculated by using Langmuir constant. The adsorption process followed pseudo-second-order model.

Keywords: Tropaeoline 000; Adsorption; Kinetics; Nano alumina; Thermodynamics

1. Introduction

The presence of dyes in effluents is a major concern due to their adverse effects to many forms of life. The discharge of dyes in the environment is a matter of concern for both toxicological and esthetical reasons [1]. Industries such as textile, leather, paper, plastics, etc. use dyes in order to colour their products and also consume substantial volumes of water. As a result, they generate a considerable amount of coloured wastewater [2]. A very small amount of dye

in water is highly visible. Further, discharging even a small amount of dye into water can affect aquatic life and food webs due to the carcinogenic and mutagenic effects of synthetic dyes [3]. It is recognized that public perception of water quality is greatly influenced by the colour. The colour is the first contaminant to be recognized in wastewater. The presence of even very small amounts of dyes in water less than 1 ppm for some dyes is highly visible and undesirable [4,5]. Azo dyes are difficult to biodegrade due to their complex aromatic structures, which provide them physico-chemical, thermal and optical stability [6], thus bringing some difficulties for the treatment of these dyes.

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Tropaeoline 000 is a well-known monoazo dye, which is widely used for colouring wool, cotton and silk as well as to synthetics like polyesters, acrylic and rayon. They are also used in paints, inks, plastics and leather. It is now well established that a prolong intake of Tropaeoline 000 can cause tumors, allergy and also eye, skin, respiratory and digestive tract irritation [7] since it possesses exceptionally good solubility in water. In recent years, many methods including coagulation and flocculation [8], reverse osmosis [9], chemical oxidation [10] biological treatments [11], photodegradation [12] and adsorption [13] have been developed for treating dye-containing wastewater. Among various treatment technologies, adsorption technique is quite popular due to its simplicity and high efficiency. Adsorption is a well-known equilibrium separation process and an effective method for water decontamination applications [14–17]. Adsorption has been found to be superior to other techniques for water reuse in terms of initial cost, flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. Adsorption also does not result in the formation of harmful substances as well as the availability of a wide range of adsorbents. Various adsorbents have been tested and used for the removal of dyes [18,19] from polluted water e.g. husk [20], saw dust [21], de-oiled mustard [22–24], wheat husk [25], rice husk [26], bentonite [27,28], rice hull ash [29], leaf [30], flyash [31], chitosan [32] and unsaturated polyester resin [33]. Gupta et al. and Mittal et al. [34–43] have also utilized various adsorbents for the removal of dyes from wastewater [44]. In the present paper, Nano alumina (NA) was employed for the removal of Tropaeoline 000 and used as an effective adsorbent in the wastewater treatment.

2. Materials and methods

Tropaeoline 000 (Fig. 1) sodium 4-[(2E)-2-(oxonaphthalen-1-ylidene)hydrazine] benzene sulpho-nate; (molecular formula $C_{16}H_9N_4Na_3O_9S_2$, molecular weight 350.33 gmol^{-1}) was obtained from Merck, and 0.01 mol dm^{-3} stock solution was prepared in double-distilled water. To prepare various solutions at desired concentrations from the stock solution, double-distilled water was used for necessary dilutions. All reagents used in the present work were of analytical grade.

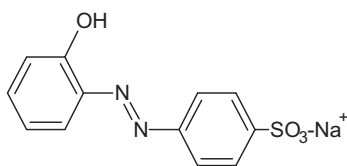


Fig. 1. Structure of monoazo dye Tropaeoline 000.

Adsorbent NA was procured from Evonik Industries, India, sold under the trade name Aeroxide Alu C. Particle size is 13 (nm) and Specific surface area is $100 \pm 15 \text{ (m}^2/\text{g)}$. All pH metric measurements were carried out with a decibel DB 1011 digital pH meter, fitted with a glass electrode. Absorbance measurements were recorded on a Systronics spectrophotometer (166) over the wavelength range of 340–990 nm.

2.1. Adsorption studies

Adsorption kinetics was performed by the batch technique at 30, 40 and 50°C temperatures. The experiments were carried out in a series of 100 mL graduated conical flask containing 30 mL of solution of each concentration and a fixed amount of adsorbent. The initial pH of the solution was adjusted with 0.1 N HCl or NaOH solutions by using a pH meter. Adsorption was achieved by adding a known amount of each adsorbent into the dye solution of known concentration and pH, and the conical flask was agitated intermittently. The data obtained in the batch mode studies were used to calculate the equilibrium dye adsorption amount at $30 \pm 1^\circ\text{C}$. The residual amount of Tropaeoline 000 in each flask was investigated using ultraviolet/visible spectrophotometer (systronics spectrophotometer (166) at λ_{max} of 484 nm. The amount of dye adsorbed per unit adsorbent (mg dye per g adsorbent) was calculated according to a mass balance on the dye concentration using Eq. (1):

$$q_e = (C_0 - C_e) \times V/W \quad (1)$$

where C_0 and C_e are initial and equilibrium adsorbate concentrations (mg/L), respectively. V is the volume of solution (L) and W is the mass of adsorbent (g).

All the experiments were triplicate and the average results were taken. Control experiments, performed without the addition of adsorbent, confirmed that the sorption of dye on the walls of conical flask was negligible.

2.2. Adsorption isotherms

The Freundlich and Langmuir isotherms were applied in the experimental equilibrium data for Tropaeoline 000 adsorption at different temperatures. The isotherm constant was determined by using linear regression analysis.

2.3. Adsorption kinetics

Adsorption kinetics studies were carried out in a similar manner to the adsorption isotherms, but the concentration of dye during adsorption was analysed at regular time intervals.

3. Results and discussion

3.1. Adsorbent characterization

Powder X-ray diffraction (XRD) measurements were performed on diffractometer system XPERT-PRO. X-ray powder diffractometer used a graphite monochromatic Cu K α radiation ($k=1.5406 \text{ \AA}$). XRD pattern at 2θ ranges between 10° and 70° was used to phase characterization. XRD pattern (Fig. 2) exhibits sharp diffraction peak at $2\theta=10^\circ$, which indicate that particles are crystalline in nature.

3.2. Effect of amount of adsorbent

To optimize the adsorbent dose for the removal of Tropaeoline 000 from its aqueous solutions, adsorption was carried out with different adsorbent dosages at different temperatures. The dose of adsorbent varied from 0.66 to 4.66 g/L for NA at fixed pH 8.5, temperature and adsorbate concentration ($7.0 \times 10^{-5} \text{ mol dm}^{-3}$). It is apparent from Fig. 3 that at all the temperatures, adsorption increases with increase in the amount of adsorbent from 0.66 to 4.66 g/L. The initial rise in adsorption with adsorbent dose is probably due to a stronger driving force and larger surface area. Hence, optimum dose for efficient adsorption of Tropaeoline 000 is 0.266 g/L onto NA.

3.3. Effect of pH

The pH is one the most important factors controlling the adsorption of dye on to adsorbent. To determine the optimum pH conditions for the adsorption of Tropaeoline 000 over NA, the effect of pH was observed over the entire pH range (2.5–12.0). The studies were conducted at a fixed concentration of adsorbate $7 \times 10^{-5} \text{ mol dm}^{-3}$. The results obtained are presented in Fig. 4, which describes maximum adsorption around 89.2–80.5% for NA, respectively, at pH 8.5. Hence, all the succeeding investigations were performed at pH 8.5 for NA.

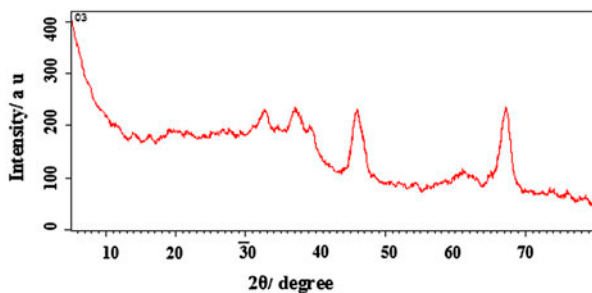


Fig. 2. X-ray diffractogram of nano-alumina.

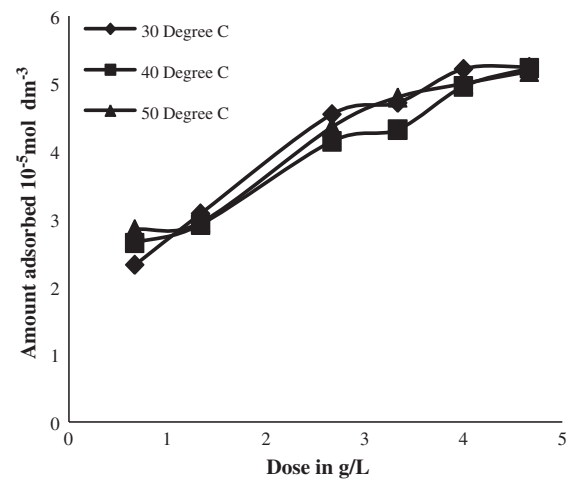


Fig. 3. Effect of amount of adsorbent for the removal of Tropaeoline 000 by NA at pH 8.5 and different temperatures.

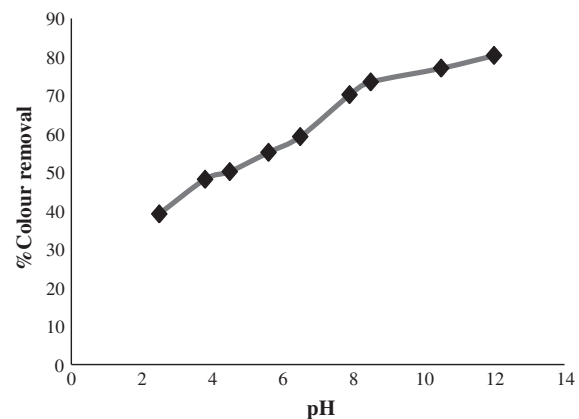


Fig. 4. Plot of pH vs.% colour removal for Tropaeoline 000 on to NA.

4. Adsorption isotherms

The adsorption equilibrium models often provide insight into the sorption mechanism, surface properties and affinity of adsorbent. The most commonly used equilibrium models are Langmuir and Freundlich isotherms [45].

Isotherms and their linear forms of Freundlich and Langmuir plot.

Isotherms	Linear forms	Plot
Freundlich	$\log q_e = \log k_f + 1/n \log C_e$	$\ln q_e$ vs. $\ln C_e$
Langmuir		
Type 1	$1/q_e = 1/q_o + 1/bq_o C_e$	$1/q_e$ vs. $1/C_e$
Type 2	$C_e/q_e = C_e/q_o + 1/bq_o$	C_e/q_e vs. C_e
Type 3	$q_e/C_e = bq_o - bq_e$	q_e/C_e vs. q_e

4.1. Freundlich model

Freundlich model is an empirical equation based on sorption on a heterogeneous surfaces or surfaces supporting sites of varied affinities. It is assumed that the stronger binding sites are occupied first and that the binding strength decreases with the increasing degree of site occupation [46]. The well-known logarithmic form of Freundlich isotherm is given by Eq. (2):

$$\log q_e = \log k_f + 1/n \log C_e \tag{2}$$

where K_F and n are Freundlich constants with n giving an indication of how favourable the adsorption process is and K_F (mg/g (L/mg)^{1/n}) is the adsorption capacity of the adsorbent. The empirical Freundlich model (Fig. 5) also showed a fairly good fit to the experimental equilibrium data at all temperatures studied ($R^2 > 0.97$). The magnitude of n gives a measure of favourability of adsorption. The values of n between 1 and 10 (i.e. $1/n$ less than 1) represents a favourable sorption. For the present study, the value of n presented the same trend representing a beneficial sorption.

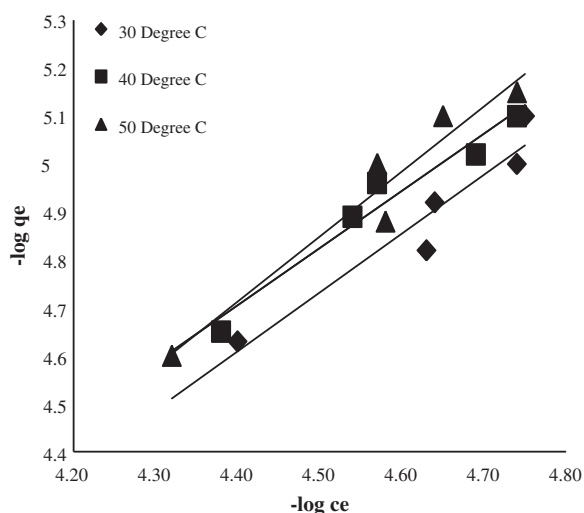


Fig. 5. Freundlich adsorption isotherms of Tropaeoline 000 for NA at pH 8.5 and different temperatures.

4.2. Langmuir model

The Langmuir isotherm assumes monolayer adsorption on a uniform surface with a finite number of adsorption sites. Once a site is filled, no further sorption can take place at that site. As such the surface will eventually reach a saturation point where the maximum adsorption of the surface will be achieved. The linear form of the Langmuir isotherm model is described as Eq. (3):

$$1/q_e = 1/Q^0 + 1/bQ^0 C_e \tag{3}$$

Table 1

Langmuir, Freundlich and Temkin constants for the adsorption of Tropaeoline 000 over NA at pH 8.5 and different temperatures

Temperature (°C)	b (mol g ⁻¹)	Q^0 (L mol ⁻¹)	bQ^0	R^2	%RSD [#]
<i>Langmuir Type 1</i>					
30	1.63	0.070	0.11	0.999	0.78
40	0.931	0.046	0.05	0.962	1.05
50	0.910	0.040	0.44	0.923	1.25
<i>Langmuir Type 2</i>					
30	2.27	2.21	0.928	0.988	0.69
40	2.15	1.80	0.837	0.933	0.99
50	2.08	1.88	0.864	0.983	1.10
<i>Langmuir Type 3</i>					
30	1.01	0.45	0.418	0.949	0.89
40	1.61	0.933	0.579	0.797	1.10
50	1.16	0.541	0.514	0.964	1.45
<i>Freundlich isotherms for Tropaeoline 000 over NA</i>					
Temperature (°C)	K_F	n	R^2	%RSD [#]	
30	6.02	0.816	0.920	0.99	
40	3.45	0.839	0.956	1.123	
50	18.15	0.736	0.934	1.56	
<i>Temkin isotherms for Tropaeoline 000 over NA</i>					
Temperature (°C)	B (J mol ⁻¹)	A (L g ⁻¹)	b	R^2	
30	24.21	9.40	933.70	0.988	

where q_e is the amount adsorbed (mol/g), C_e is the equilibrium concentration of the adsorbate (mol/L), and Q^0 and b are the Langmuir constants related to maximum adsorption capacity and energy of adsorption relatively. When $1/q_e$ is plotted against $1/C_e$, a straight line with slope $1/bQ^0$ is obtained which shows that the adsorption of Tropaeoline 000 follows Langmuir isotherm. Langmuir constants are calculated and values of these constants at different temperatures are given in (Table 1). It was observed that the Langmuir isotherms could be linearized to at least three different types. Type 1 Langmuir was the most commonly used linear expression to study the relation between the concentration of solute in the liquid phase and in the solid phase at equilibrium conditions (Fig. 6). Type 2 and Type 3 Langmuir expressions were also used to explain the equilibria phenomena of Tropaeoline 000 adsorption process [47].

4.3. Tempkin isotherm

Tempkin isotherm [48] contains a factor that explicitly takes into account the adsorbent–adsorbate interactions (Fig. 7). The heat of adsorption of all the molecules in the layer would decrease linearly with coverage due to adsorbent–adsorbate interactions. The adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy. The Tempkin isotherm is expressed as Eq. (4):

$$q_e = (RT/bT) \ln(AC_e) \quad (4)$$

where $RT/bT = B$ (J/mol), which is the Tempkin constant related to heat of sorption, whereas A (L/g) is the equilibrium binding constant corresponding to the maximum binding energy. R (8.314 J/mol K) is the universal gas constant and T (K) is the absolute solution temperature.

5. Adsorption thermodynamics

The thermodynamic parameters like ΔG° , enthalpy change ΔH° and entropy change ΔS° for the adsorption processes are calculated using the following Eqs. (5)–(7):

$$\Delta G^\circ = -RT \ln b \quad (5)$$

$$\Delta H^\circ = -R(T_2 T_1)/(T_2 - T_1) \ln(b_2/b_1) \quad (6)$$

$$\Delta S^\circ = (\Delta H^\circ - \Delta G^\circ)/T \quad (7)$$

where b , b_1 and b_2 are the equilibrium constants at different temperatures, which are gathered from the

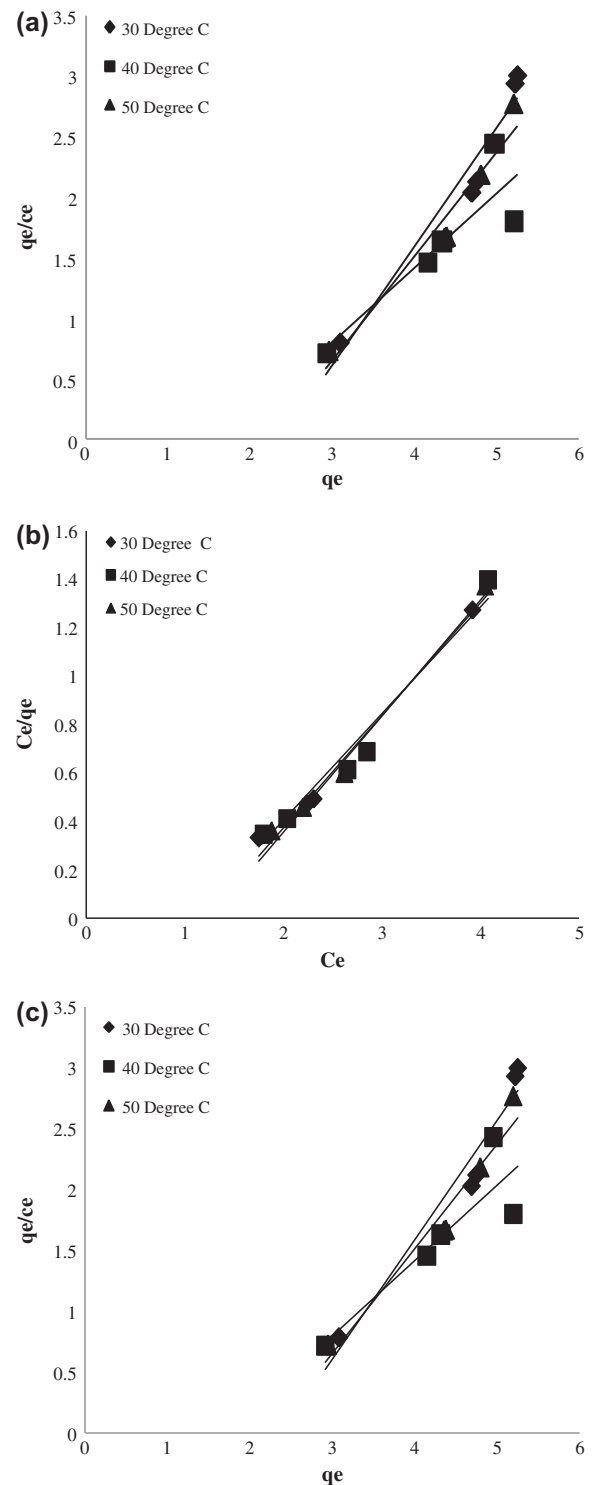


Fig. 6. (a) Type 1, (b) Type 2, (c) Type 3 and (d) Langmuir Adsorption Isotherms of Tropaeoline 000 for NA by using the linear method for the sorption of Tropaeoline 000 onto NA.

slopes of straight lines obtained in case of Langmuir adsorption isotherms at different temperatures, R

Table 2
Thermodynamic parameters for the uptake of Tropaeoline 000 over NA at pH 8.5

Adsorbent	$-\Delta G^\circ$ (kJ mol ⁻¹)			ΔH° (kJ mol ⁻¹)	ΔS° (JK ⁻¹ mol ⁻¹)
	30°C	40°C	50°C	30°C	30°C
NA	-12.3×10^3	-12.98×10^3	-13.12×10^3	68.60×10^3	22.69

Table 3
Values of rate constant for the uptake of Tropaeoline 000 over NA at pH 8.5 and different temperatures

Temperature (°C)	Nano alumina		
	K_{ad}	R^2	%RSD
30	341.4×10^{-3}	9.75	0.99
40	310.3×10^{-3}	9.35	1.02
50	424.8×10^{-3}	9.85	1.22

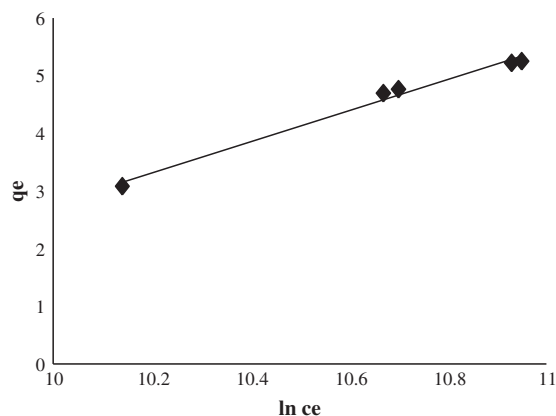


Fig. 7. Tempkin adsorption isotherms of Tropaeoline 000 for NA.

(8.314J/molK) is the universal gas constant and T (K) is the absolute solution temperature. ΔG° is obtained using Langmuir Type 1; the high negative value of ΔG° confirms the feasibility of the process and the spontaneous nature of sorption with a high preference for Tropaeoline 000 to adsorb onto NA (Table 2). The value of ΔH° was positive, indicating that the sorption reaction is endothermic [49].

6. Adsorption kinetics

The pseudo-first-order and second-order kinetic models were tested at different concentrations. In this study to determine which model is in good agreement with experiment q_e (adsorption capacity) value, thus suggesting which model the sorption system follows. The pseudo-first-order-model can be expressed [50] as in Eq. (8):

$$\log(q_e - q_t) = \log q_e - k_{ad} \times t/2.303 \quad (8)$$

where q_e and q_t (mg/g) are the mass adsorbed at the equilibrium (adsorptive capacity), and mass adsorbed at any time " t ", K_{ad} (min⁻¹) is the equilibrium constant of the pseudo-first-order adsorption (Fig. 8). The value of K_{ad} and q_e are determined, respectively, from the slope and intercept of the plot of $\text{Log}(q_e - q_t)$ vs. t . The k_{ad} values evaluated, for each system, from the respective Lagergren plot are presented in Table 3.

6.1. Intraparticle diffusion model

Intra-particle diffusion model used here refers to the theory proposed by Weber and Morris. The initial

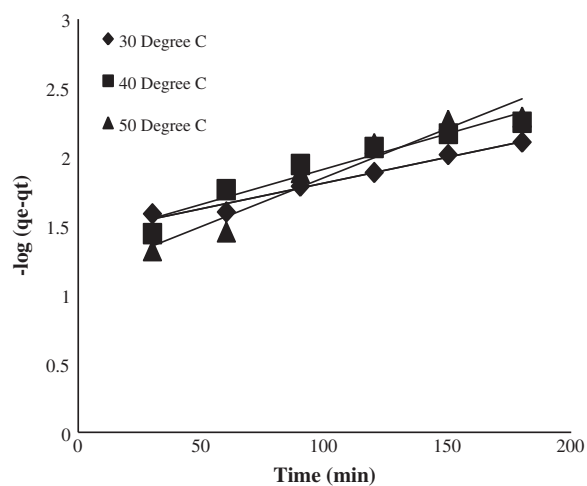


Fig. 8. Pseudo-first-order kinetic model plots plot for Tropaeoline 000 adsorption on NA at different temperature.

Table 4
Intraparticle diffusion coefficients and intercept values for Tropaeoline 000 adsorption on NA at different temperatures

Temperature (°C)	Nano alumina		
	K_d	C	R^2
30	3.45	1.26	0.969
40	3.44	1.05	0.994
50	3.03	1.38	0.994

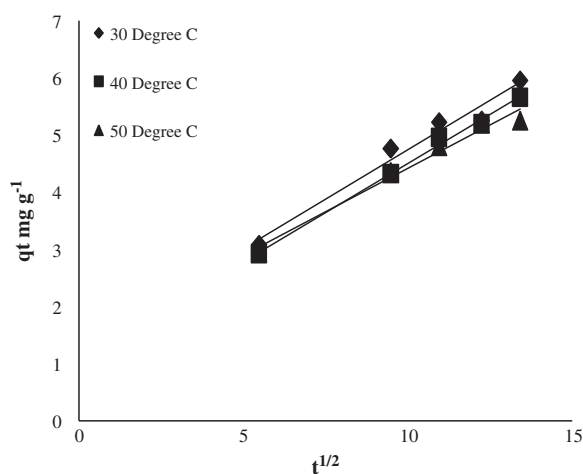


Fig. 9. Intraparticle diffusion plot for Tropaeoline 000 adsorption on to NA at pH 8.5 and different temperatures.

rate of intra-particle diffusion can be obtained by linearization of the curve according to Eq. (9):

$$q_t = K_{\text{dif}} t^{1/2} + C \quad (9)$$

where C (mg g^{-1}) is the intercept and K_{dif} is the intra-particle diffusion rate constant ($\text{mg g}^{-1} \text{min}^{-1/2}$). The values of q_t were found to be linearly correlated with values of $t^{1/2}$ and the rate constant K_{dif} directly evaluated from the slope of the regression line (Table 4). The R^2 values given in Table 4 are close to unity indicating the application of this model. The linear portions of the curves do not pass through the origin (Fig. 9) indicating that mechanism of Tropaeoline 000 removal onto NA is complex and both the surface adsorption as well as intra-particle diffusion contributes to the rate determining step [51].

7. Conclusion

The non-carbon adsorbent NA was successfully used for the adsorption and removal of organic dyes from aqueous solution. The present study offers major advantage of the NA which was used without any previous activation treatment which decreases adsorption costs. The nano-adsorbent is in nano-meter scale, so they have large specific surface area which is in favour of adsorption. The adsorption characteristics of Tropaeoline 000 onto NA were evaluated in terms of equilibrium, kinetic and thermodynamic parameters; and second-order equation has been successfully applied to predict dynamical behaviour for the adsorption of Tropaeoline 000 onto NA.

The linear plot between $1/C_e$ and $1/q_e$ for NA indicates the validity of Langmuir adsorption isotherm; the applicability of the Langmuir isotherm indicates good monolayer coverage of NA on the surface of the Tropaeoline 000. Decrease in b (Langmuir constant) values for Langmuir Types 1, 2 and 3 with the rise in temperature indicates weakening of adsorbate-adsorbent interactions at high temperature. It reveals that the adsorption affinity of NA decreases with the rise in temperature. Endothermic nature of the process was confirmed by obtaining positive values of ΔH° . The Langmuir 1, 2 and 3 plots show good correlation coefficients. The Freundlich model as observed in Fig. 3 was also found to be linear and the coefficient of correlation value (r^2) was also high. The Langmuir isotherm was found to be the best-fitting isotherm at all temperatures.

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