

Evaluation of the use of loofa activated carbons as potential adsorbents for aqueous solutions containing dye

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Abstract

In this study, two activated carbons were prepared from loofa, loofa carbon-1 (LC1) and loofa carbon-2 (LC2). The surface structure of LC1 and LC2 were analyzed by scanning electronic microscopy (SEM). The ability of LC1 and LC2 to remove reactive orange (RO) dye from aqueous solutions by adsorption has been studied. A series of experiments were conducted to assess the effect of system variables such as initial pH, initial metal ion concentration and temperature. The results showed that the adsorption of dye was maximal at the lowest value of pH (pH = 1) and the most suitable sorption temperature was 25°C with maximum capacities of 38.3 and 50 mg/g for LC1 and LC2 respectively. Langmuir, Freundlich, Dubinin–Radushkevich (D–R), Temkin and Harkins–Jura isotherms were used to analyze the equilibrium data at different temperatures. The adsorption kinetics was found to follow a pseudo-first-order rate kinetic model. In addition, various thermodynamic parameters, such as standard Gibbs free energy (ΔG^0), standard enthalpy (ΔH^0), standard entropy (ΔS^0), and activation energy (E_a) have been calculated. The sorption process of reactive orange (RO) dye onto different activated carbons prepared from loofa was found to be spontaneous and exothermic process.

Keywords: Adsorption; Activated carbon; Reactive orange dye; Loofa; Equilibrium; Thermodynamics

1. Introduction

Dyes are synthetic aromatic water-soluble dispersible organic colorants, having potential application in various industries. The dyestuff usage has been increased day by day due to tremendous increase of industrialization and mans desire for color [1]. Reactive dyes represent

20–30% of the total dye market because they are used for cotton dyeing. On the other hand, reactive dyes typically have a relatively low fixation degree on the textile substrate, and the corresponding wastewaters may be highly colored [2,3]. Reactive dye wastewater has limited biodegradability in an aerobic environment. Many reactive azo dyes,

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constituting the largest dye groups decompose to potential carcinogenic aromatic amines under anaerobic conditions [4,5].

The discharge of colored wastes into receiving streams disturb the biological process. Further, dye effluents may contain chemicals which exhibit toxic effects toward microbial populations [6,7]. Therefore, environmental legislation has imposed severe limits on the concentrations of the discharged effluents from dyestuff manufacturing and textile industries. Treatment of dye based effluents is considered to be most challenging in the environmental organizations and the industries.

Various treatment methods viz., physical, physico-chemical, biological and chemical processes have been investigated for treating dye bearing effluents [8,9]. Adsorption is a physico-chemical wastewater treatment method, which has gained popularity in the wastewater treatment industry because of the high quality treated effluents are produced. In many cases, these treated effluents can be re-used in a range of processes as good quality water [10]. Activated carbon is the most widely used adsorbent with great success because of its high adsorption capacity. But its use is limited due to its high cost. This has led to search for cheaper substitutes. Several wastes and residues have been investigated for the adsorption of dyes with varying success [11–13]. However, new economical, easily available and highly effective adsorbents are still needed.

An Egyptian loofa is a cylindrical agriculture product with a fibrous sponge like interior. The dried fibrous part of the loofa fruit, used as a washing sponge or as a filter.

In this work, two activated carbons, LC1 and LC2, were prepared from Egyptian loofa plant and investigated as adsorbents for reactive dyes removal from aqueous solution. Various parameters affecting adsorption onto the prepared activated carbons such as pH, initial dye concentration, contact time and temperature were

studied and discussed. Furthermore, the work is aimed to evaluate the isotherm and kinetics as well as the diffusion parameters for the adsorption of Reactive Orange (RO) dye onto LC1 and LC2.

2. Materials and methods

2.1. Preparation of activated carbons

Loofa was obtained from the local market in Egypt, washed with distilled water and oven dried at 105°C. Loofa carbon 1 (LC1) was prepared by soaking a part of the dried loofa in a mixture of H₃PO₄ (20%) and ZnCl₂ solution (50%) in 1:1 ratio for 24 h. The solution was decanted and the adsorbent was dried at 105°C and then transferred to a sealed stainless steel cylinder. The mixture was activated in a muffle furnace at 500°C for a period of 1 h. The produced carbon was washed with diluted Na₂CO₃ solution, then with distilled water. The washed material was dried in a hot air oven at 100°C for 3 h, grinded and sieved to a constant particle size. This activated carbon was named as LC1.

Loofa carbon 2 (LC2) was prepared by soaking loofa biomass in HNO₃ solution (30%) for 24 h, then decant and dry in oven at 105°C and carbonized following the method mentioned above. The produced activated carbon from this process was named as LC2.

2.2. Preparation of adsorbate

Reactive Orange dye (RO), a product of ISMA dye company of Egypt, has a molecular formula C₃₀H₁₆N₃Na₂O₈S₂. The dye was used as adsorbate without any purification. The color of the dye is stable within the pH range of the study. The concentration of the dye was determined using UV–VIS spectrophotometer (Milton Roy, spectronic 21D) at wavelength of 410 nm. Adsorbed amount of dye was calculated from a standard calibration curve.

2.3. Batch adsorption experiments

Adsorption experiments of RO dye onto LC1 and LC2 were conducted using 100 mL of adsorbate solution of a known concentration, pH and weight of adsorbent. The mixture was agitated in a temperature controlled shaker at agitation speed of 150 rpm. Samples (1 mL) were withdrawn at different time intervals (0–180 min), centrifuged and analyzed for remaining dye concentration. Initial pH of the solution was adjusted with dilute 0.1 M NaOH and 0.1 M HCl solutions. The percentage removal of dye was calculated using the following relationship:

$$\% \text{ Removal} = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

where C_0 and C_t (mg/L) are the initial dye concentration and concentration at time t , respectively.

Equilibrium studies were conducted by contacting 100 mL of RO dye solution of different initial dye concentration (20–100 mg/L) with 0.25 g of adsorbent and adjusted to pH 1.0. After equilibrium, the solution was separated and analyzed. Adsorption equilibrium experiments were performed at different temperatures (25–60°C). The equilibrium adsorption capacity was calculated from the relation

$$q_e = \frac{(C_0 - C_e)V}{w} \quad (2)$$

where q_e (mg/g) is the equilibrium adsorption capacity, C_e is the dye concentration at equilibrium,

V (L) is the volume of solution and w (g) is the weight of adsorbent.

3. Results and discussion

3.1. Characterization of adsorbents

Physico-chemical characterization of LC1 and LC2 are given in Table 1. Bulk density, porosity, moisture content and ash content were determined for both activated carbons. Scanning electron microscope analysis was carried out, for structural characteristics of different types of activated carbons (Fig. 1). Scanning electron microscope photograph of LC1 and LC2 revealed surface texture and porosity. This photomicrograph shows fibrous structure of both activated carbons, which may due to inherent fiber structure in the original raw material.

3.2. Optimum adsorption conditions

In order to determine the optimum operational parameters governing adsorption process, the

Table 1
Characteristic of activated carbons

Property	LC1	LC2
Bulk density (g/mL)	0.63	0.33
Porosity (%)	38.46	64.20
Moisture content (%)	3.85	5.52
Ash content (%)	4	6

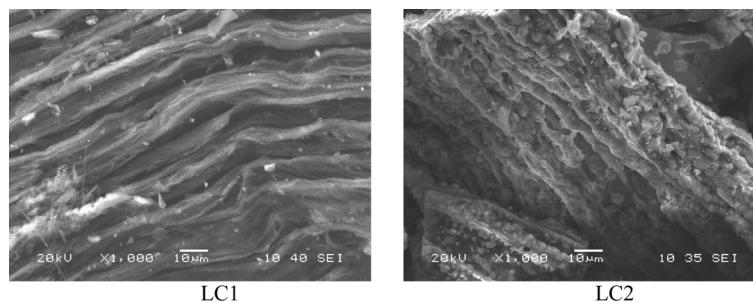


Fig. 1. Scanning electron micrograph of loofa activated carbons.

effect of pH, initial dye concentration at different temperatures and contact time were investigated.

The effect of pH on the adsorption of RO was studied over pH range of 1–9 at 25°C for 2 h. The initial dye concentration was 20 mg/L. The maximum percentage removal of RO was at pH 1 then decreased with further increase in pH (Fig. 2). This may be due to the nature of the binding sites in LC1 and LC2 surfaces. Therefore, the remaining adsorption experiments were carried out using this optimum pH value.

The effect of initial concentration of RO on the percentage removal by LC1 and LC2 at different temperatures is shown in Fig. 3. It is evident that at low initial dye concentrations the percentage removal increases for both LC1 and LC2 at all temperatures. By increasing the initial dye concentration the percentage removal decreased, although the actual amount of dye adsorbed per unit mass of activated carbon increased with the increase in initial concentration. This increase is due to the decrease in resistance to the uptake of solute from dye solution. Furthermore, adsorption decreased with an increase in temperature, indicating that the adsorption of RO by LC1 and LC2 is an exothermic process in nature.

The effect of contact time on adsorption of RO on the LC1 and LC2 was studied and the results are presented in Fig. 4. Adsorption takes

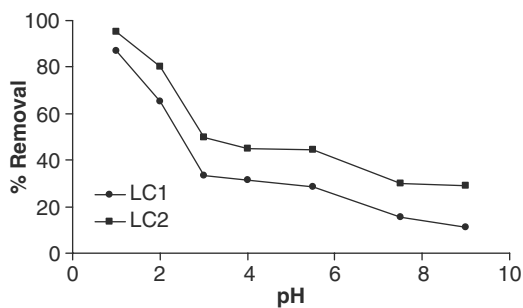


Fig. 2. Effect of pH on % removal of reactive orange dye for different activated carbons (contact time = 2 h, dose of activated carbon = 2.5 g/L).

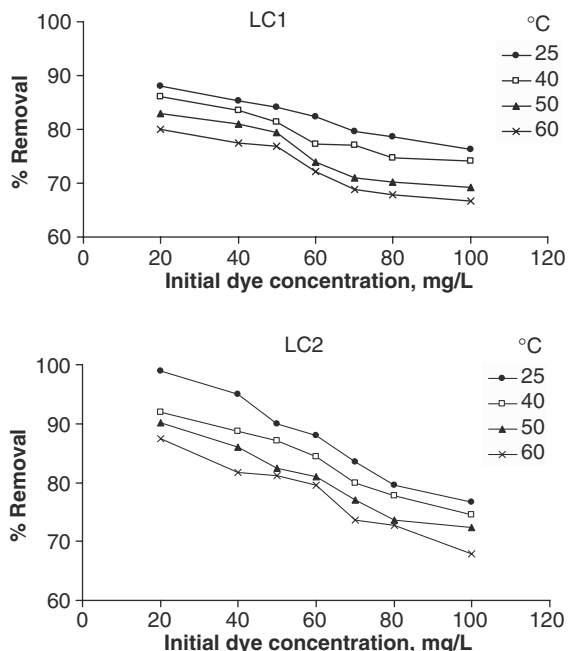


Fig. 3. Effect of initial dye concentration on adsorption of LC1 and LC2 at different temperatures (pH = 1, dose of activated carbon = 2.5 g/L).

place very fast for the first 60 min, where most adsorption occurred. A slight increase occurs during the next 60 min, then equilibrium attains. Further increase in contact time did not showed any increase in adsorption efficiency.

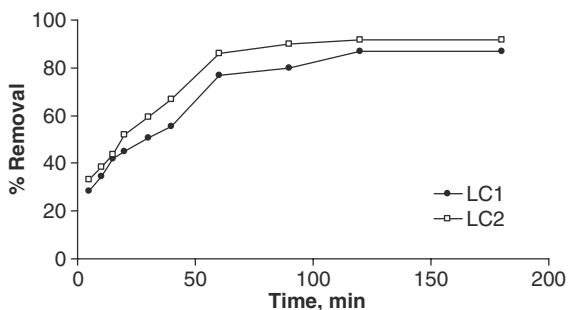


Fig. 4. Effect of agitating time in the percentage removal of RO by different activated carbons (pH = 1, temp. = 25°C, dye concentration = 20 mg/L, dose of activated carbon = 2.5 g/L).

3.3. Equilibrium adsorption isotherms

Various adsorption isotherms have been studied to describe the equilibrium nature of adsorption. The experimental data were fitted to the Langmuir, Freundlich, Temkin, D–R and Harkins–Jura isotherm equations and the constant parameters of the isotherm equations were calculated.

The Langmuir equation has been applied for monolayer sorption onto a surface of a finite number of identical sites [14]. The linearized form of Langmuir isotherm is presented by the following equation:

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \quad (3)$$

where C_e is the equilibrium concentration (mg/L), q_e the adsorption capacity in equilibrium (mg/g), K_L the Langmuir adsorption constant (L/mg), and Q_m is the theoretical maximum adsorption capacity (mg/g). Fig. 5 shows a linear plot of C_e/q_e vs. C_e for the removal of RO over LC1 and LC2 at different temperatures. The values of Q_m and K_L constants and the correlation coefficients for Langmuir isotherm are presented in Table 2. The value of Q_m decreases with increasing temperature confirming that the adsorption process is favored at lower temperatures.

The Freundlich adsorption isotherm can be expressed in the linear form [15] as

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

where K_F (L/mg) and n are isotherm constants indicate the capacity and intensity of the adsorption, respectively. Fig. 6 shows the linear plot of $\log q_e$ vs. $\log C_e$ at different temperatures. Table 2 shows the Freundlich adsorption isotherm constants and correlation coefficients. The value of $1/n$ was found to lie between zero and one, indicating that RO dye is favorably adsorbed by LC1 and LC2 adsorbents.

The linear form of Dubinin and Radushkevich isotherm equation can be expressed as [16]

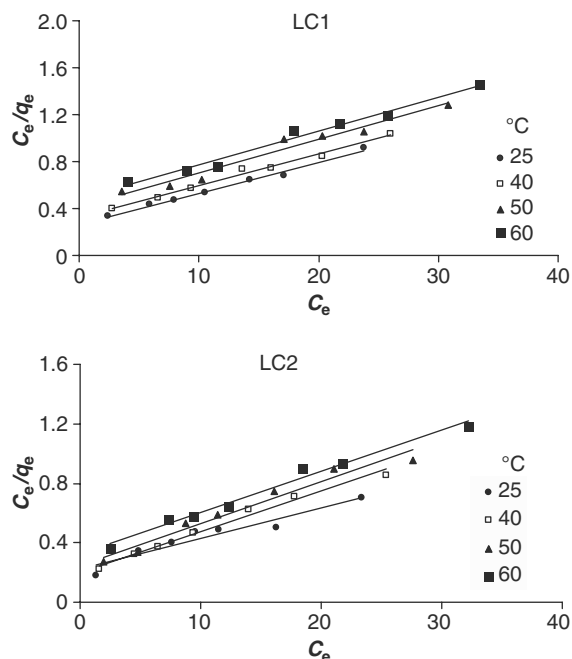


Fig. 5. Langmuir model for adsorption of RO dye by LC1 and LC2 at different temperatures (pH = 1, contact time = 180 min, dose of activated carbon = 2.5 g/L).

$$\ln q_e = \ln Q_s - B\varepsilon^2 \quad (5)$$

where Q_s is the theoretical monolayer saturation capacity (mg/g), B is the Dubinin–Radushkevich model constant (mol^2/kJ^2), ε is the Polanyi potential and is equal to

$$\varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right) \quad (6)$$

The mean energy of sorption, E (kJ/mol), is related to B as [17]

$$E = \frac{1}{\sqrt{2B}} \quad (7)$$

$\ln q_e$ vs. ε^2 was plotted at different temperatures for both LC1 and LC2 as shown in Fig. 7. The calculated D–R constants and mean free energy for adsorption are shown in Table 2. The mean adsorption energy was found to be in the

Table 2

Constant parameters and correlation coefficients calculated for various adsorption models at temperatures for LC1 and LC2

Isotherm equation	LC1				LC2			
	25°C	40°C	50°C	60°C	25°C	40°C	50°C	60°C
<i>Langmuir</i>								
Q_m (mg/g)	38.31	34	32	28.5	50	36.76	35.46	30.0
K_L (L/mg)	0.098	0.090	0.076	0.072	0.133	0.129	0.114	0.102
R^2	0.9898	0.9897	0.9739	0.9795	0.961	0.984	0.964	0.985
<i>Freundlich</i>								
$1/n$	0.586	0.580	0.724	0.802	0.497	0.526	0.544	0.645
K_F (L/g)	4.634	4.142	2.243	1.480	6.677	5.498	4.487	2.940
R^2	0.969	0.976	0.973	0.962	0.992	0.989	0.978	0.991
<i>D-R</i>								
Q_s (mg/g)	22.265	21.328	20.104	17.257	23.058	21.328	19.956	18.539
$B * 10^6$	1.60	1.86	3.40	4.02	6.26	8.11	1.042	1.603
E (kJ/mol)	5.59	5.18	3.83	3.53	2.83	2.48	6.93	5.58
R^2	0.885	0.891	0.918	0.918	0.839	0.843	0.827	0.824
<i>Tempkin</i>								
b_T	8.750	8.333	9.073	7.951	7.958	7.803	7.492	8.122
K_T (L/mg)	0.892	0.781	0.450	0.392	1.599	1.255	1.046	0.610
R^2	0.987	0.989	0.991	0.985	0.976	0.991	0.974	0.969
<i>Harkins–Jura</i>								
A	55.556	52.91	27.25	16.92	72.99	57.47	47.85	29.94
$B_2 * 10^3$	0.41	0.478	1.842	4.852	0.232	0.383	0.579	1.493
R^2	0.797	0.805	0.744	0.727	0.843	0.776	0.784	0.783

range of 2.48–6.93 kJ/mol, which is in the range of physical adsorption reactions.

The Tempkin isotherm has generally been applied in the following form [18]:

$$q = \frac{RT}{b_T} \ln(K_T C_e) \quad (8)$$

where b_T is Tempkin constant related to heat of sorption (J/mol), K_T the Tempkin isotherm constant (L/mg), R the gas constant (J/mol K), T the absolute temperature. K_T is the equilibrium binding constant corresponding to the maximum binding energy and constant b_T is related to heat of adsorption. A plot of q_e vs. $\ln C_e$ (Fig. 8) enables the determination of isotherm constants

K_T and b_T . The values of parameters are given in Table 2.

The Harkins–Jura adsorption isotherm can be expressed [19] as

$$\frac{1}{q_e^2} = \left(\frac{B_2}{A} \right) - \left(\frac{1}{A} \right) \log C_e \quad (9)$$

where B_2 is the isotherm constant. $1/q_e^2$ was plotted vs. C_e (Fig. 9). This isotherm explains the multilayer adsorption by the existence of a heterogeneous pore distribution. The isotherm constants are summarized in Table 2. Analysis of R^2 values (Table 2) showed that Tempkin and Langmuir equations have better coefficients than the other models describing the adsorption

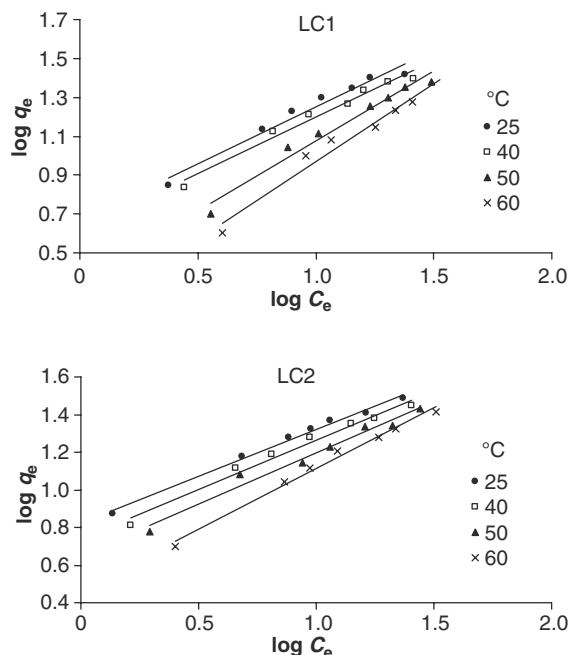


Fig. 6. Freundlich isotherm for adsorption of RO by LC1 and LC2 at different temperatures (pH = 1, contact time = 180 min, dose of activated carbon = 2.5 g/L).

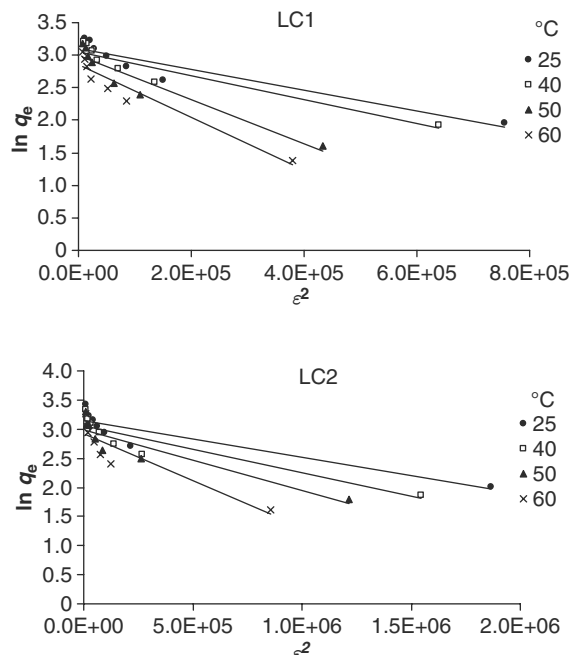


Fig. 7. Dubinin–Reduskevich isotherm for adsorption of RO by LC1 and LC2 at different temperatures (pH = 1, contact time = 180 min, dose of activated carbon = 2.5 g/L).

of RO by LC1, while Freundlich and Tempkin isotherm equations better fit the data for RO–LC2 system. Whereas, D–R equation represents the poorest fit of experimental data than the other isotherm equations in all investigated temperatures.

3.4. Adsorption kinetic study

The kinetics of adsorbate uptake is important for choosing optimum operating conditions for design purposes. In order to analyze the adsorption kinetics of RO on LC1 and LC2, the following kinetic equations have been studied.

3.4.1. Pseudo-first-order model

The pseudo-first-order equation is [20]

$$\frac{dq}{dt} = k_1(q_e - q_t) \quad (10)$$

where k_1 is first-order rate constant. Integrating the equation at initial conditions $q = 0$ at $t = 0$ and $q = q$ at $t = t$, the equation becomes

$$\log(q_e - q) = \log q - \frac{k_1}{2.303} t \quad (11)$$

Values of k_1 (min^{-1}) can be obtained from the slope of the plot of $\log(q_e - q)$ against t , as shown in Fig. 10. The first-order rate constants and the regression coefficient values are presented in Table 3. Mall et al. [21] has reported k_1 values of 0.0115 and 0.0219 min^{-1} for Orange-G and Methyl Violet dyes, respectively, onto bagasse fly ash. These values are close to the values obtained in the present study. It was observed from Fig. 10 that the pseudo-first order model fits well ($R^2 > 0.996$). Thus, the model can be applied for the entire data. A similar trend was previously observed by Namasivayam and Arasi

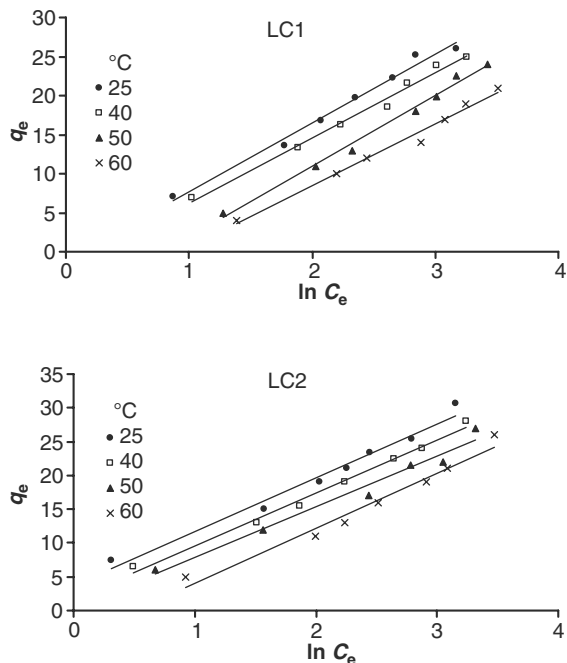


Fig. 8. Temkin isotherm for adsorption of RO by LC1 and LC2 at different temperatures (pH = 1, contact time = 180 min, dose of activated carbon = 2.5 g/L).

[22] for Congo red dye over waste red mud and Mittal and Gupta [23] for orlamar red BG and orlamar blue G dyes onto fomitopsis carnea. This shows that the first-order model can be applied in an appropriate way to describe the entire process of adsorption of RO dye onto LC1 and LC2 adsorbents.

3.4.2. Intra-particle diffusion study

The adsorption of RO dye onto LC1 and LC2 may be controlled via external film diffusion at earlier stages and later by the particle diffusion. The possibility of intra-particle diffusion resistance was identified by using the following intra-particle diffusion model as [24]

$$q_t = K_{\text{dif}} t^{1/2} + C \quad (12)$$

where K_{dif} is the intra-particle diffusion rate constant ($\text{mg}/(\text{g} \cdot \text{min}^{1/2})$), C is the intercept. The

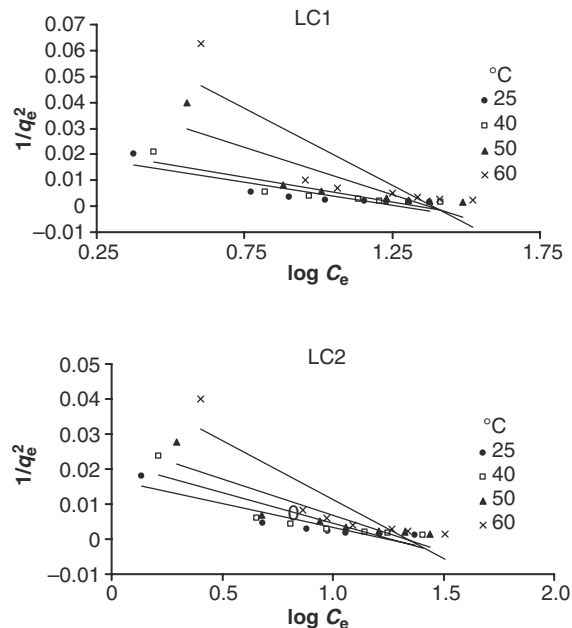


Fig. 9. Harkins–Jura isotherm for adsorption of RO by LC1 and LC2 at different temperatures (pH = 1, contact time = 180 min, dye concentration = 20 mg/L, dose of activated carbon = 2.5 g/L).

values of q_t correlated linearly with values of $t^{1/2}$ and the rate constant K_{dif} directly evaluated from the slope of regression line (Fig. 11). The value of C (Table 3) gives an idea about the thickness of boundary layer, the larger the intercept the greater the boundary layer effect [25].

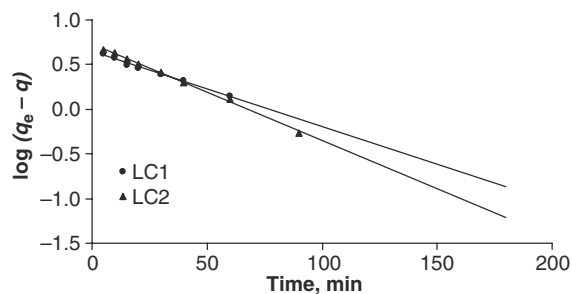


Fig. 10. Pseudo-first-order kinetics for RO adsorption onto LC1 and LC2 (pH = 1, temp. = 25°C, dye concentration = 20 mg/L, dose of activated carbon = 2.5 g/L).

Table 3
Kinetic parameters for the removal of RO dye by LC1 and LC2

Type of adsorbent	First-order kinetic model		Intra-particle diffusion model		
	k_1	R^2	K_{dif}	C	R^2
LC1	0.019	0.983	0.513	1.22	0.995
LC2	0.025	0.996	0.587	1.42	0.988

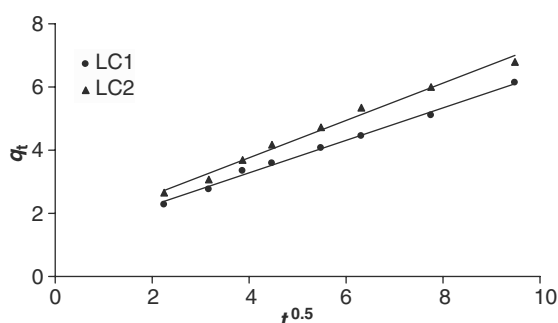


Fig. 11. Intraparticle diffusion plot for RO adsorption onto LC1 and LC2 (pH = 1, temp. = 25°C, dye concentration = 20 mg/L, dose of activated carbon = 2.5 g/L).

3.5. Thermodynamic study

Thermodynamic data such as adsorption free energy change, considering the equilibrium constant, K_0 , can be obtained from the following equation:

$$\Delta G^0 = -RT \ln K_0 \tag{13}$$

where ΔG^0 is the free energy change (kJ/mol); R the universal gas constant (8.314 J/mol K); K_0 the thermodynamic equilibrium constant and T the absolute temperature (K). Values of K_0 may be calculated from the relation $\ln \frac{q_e}{C_e}$ vs. q_e at different temperatures and extrapolating to zero [26]. The corresponding values of ΔG^0 are presented in Table 4. ΔG^0 values were negative at all investigated temperatures, indicating the spontaneous nature of adsorption of RO dye by LC1 and LC2. The values of ΔG^0 obtained were ranged from 2.01 to 4.7 kJ/mol, which is an indication of the physical adsorption nature of the process. The values of other thermal parameters such as enthalpy change (ΔH^0), and entropy change (ΔS^0), may be determined from the Van't Hoff equation [27]

$$\ln K^0 = \frac{\Delta S^0}{R} + \frac{\Delta H^0}{RT} \tag{14}$$

ΔH^0 and ΔG^0 can be obtained from the slope and intercept of Van't Hoff plot of $\ln K^0$ vs. $1/T$. The data are presented in Fig. 12 and Table 4. The negative values of ΔH^0 further confirm the exothermic nature of the adsorption process, while the positive values of ΔS^0 confirming a physical adsorption nature. The values of activation energy, E_a , were calculated from the modified Arrhenius equation related to surface coverage, θ , as follows [26]:

$$S^* = (1 - \theta)e^{-\left(\frac{E_a}{RT}\right)} \tag{15}$$

Table 4
Thermodynamic parameters for the adsorption of RO onto LC1 and LC2

Temp.	LC1					LC2				
	ΔG^0 (kJ/mol)	ΔS^0 (J/mol K)	ΔH^0 (kJ/mol)	E_a (kJ/mol)	S^*	ΔG^0 (kJ/mol)	ΔS^0 (J/mol K)	ΔH^0 (kJ/mol)	E_a (kJ/mol)	S^*
25	-3.270					-4.700				
40	-2.920					-4.059				
50	-2.388	8.59	-14.2	-10.33	0.08	-3.743	13.65	-17.93	-7.93	0.27
60	-2.014					-3.105				

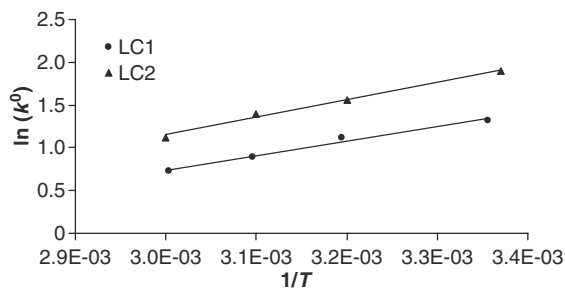


Fig. 12. Plot of Van't Hoff equation for adsorption of RO onto LC1 and LC2.

where S^* is the sticking probability, which is a function of adsorbate/adsorbent system under investigation, its value should be $0 < S^* < 1$ and is temperature dependent. θ is the surface coverage and can be calculated from Eq. (16).

$$\theta = \left(1 - \frac{C_e}{C_0} \right) \quad (16)$$

The activation energy and sticking probability were estimated from a plot of $\ln(1 - \theta)$ vs. $1/T$ (Fig. 13). The activation energy, E_a , calculated from the slope of the plot were found to be -10.3 kJ/mol, -7.9 kJ/mol for adsorption of RO onto LC1 and LC2 respectively. The negative values of E_a indicate the exothermic nature of the adsorption process. Furthermore, the low values of E_a and S^* confirm that the adsorption system is a diffusion controlled process. Interestingly,

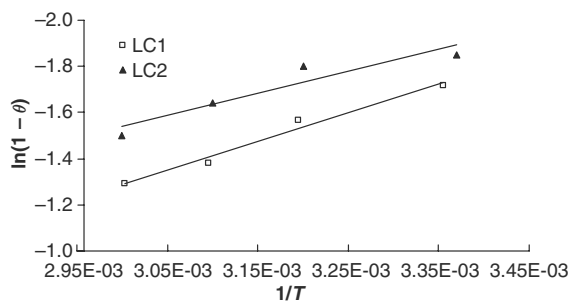


Fig. 13. Plot of $\ln(1 - \theta)$ vs. reciprocal temperature for adsorption of RO onto LC1 and LC2.

the values of activation energy obtained from Eq. (15) are in the same order of the values of activation energy obtained from D–R isotherm.

4. Conclusions

The present investigation shows that the two activated carbons produced from loofa, LC1 and LC2, are effective for the removal of reactive orange (RO) dye from aqueous solutions. Removal of RO is pH dependant and the maximum removal was attained at pH 1.0. It was also a function of adsorbate concentration and temperature of the solution. Adsorption equilibrium data follows Tempkin and Langmuir isotherm equations with LC1 while the equilibrium data was better fitted using Freundlich and Tempkin isotherms. The maximum adsorption capacity was 38.3 and 50 mg/g for LC1 and LC2, respectively. Adsorption kinetics followed pseudo-first-order rate equation. The activation energy of adsorption of RO dye over LC1 and LC2 were calculated as -10.33 and -7.93 kJ/mol for LC1 and LC2, respectively, indicating that the adsorption process is an exothermic with a physical nature. The results would be useful for the design of wastewater treatment plants for the removal of dye.

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