

# Removal of Basic Blue 41 dyes using *Persea americana*-activated carbon prepared by phosphoric acid action

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**Abstract** Adsorption study of Basic Blue 41 dye onto activated carbon from *Persea americana* nuts with phosphoric acid activation was achieved. The effect of operating parameters, the effect of pH (2–12), adsorbent amount (5–30 mg/50 mL), dye concentration (25–125 mg/L), contact time (0–200 min) and temperature (298–323 K), on the adsorption capacity was examined. The experimental isotherm data were analyzed using Langmuir and Freundlich models, which showed that the best fit was achieved by the Langmuir model with the maximum monolayer adsorption capacity at 625 mg/g. The adsorption kinetic process followed pseudo-second-order kinetics. Thermodynamic evaluation showed that the process was endothermic ( $\Delta H^0 = 144.60$  kJ/mol) and spontaneous ( $\Delta G^0$  varied from to  $-11.64$  to  $-19.50$  kJ/mol), while the positive value of entropy ( $\Delta S^0 = 524.3$  J/mol K) revealed increased randomness at the adsorbent–adsorbate interface. It was found to be a very efficient adsorbent and a promising alternative for dye removal from aqueous solutions.

**Keywords** Removal of dye · *Persea americana*-activated carbon · Surface area · Adsorption · Kinetics and thermodynamic studies

## Introduction

The textile industry plays a part in the economy of several countries around the world. However, effluents from textile and dyeing have a low biological oxygen demand and strong chemical oxygen demand. Disposal of this colored water into receiving water can be toxic to aquatic life and cause food chain contamination, resulting in deleterious health effect even in very low concentrations. Moreover, most of these dyes can cause allergy, dermatitis, skin irritation and also provoke cancer and mutation in humans [1, 2]. Dyes are usually highly visible, very difficult to biodegrade, and extremely difficult to eliminate in natural aquatic environments [3, 4].

To improve the effluent quality, the addition of physical and/or chemical treatments comprising adsorption [5–9], photocatalytic [10, 11] or electrochemical methods [5] and reverse osmosis [12] are necessary. Adsorption is the most simple and known for the treatment of effluents containing dyes using the new low-cost and environmentally friendly adsorbents in the carbon-based or not activated means [13–18].

The potential properties of activated carbon as adsorbents are due to their highly developed porosity, favorable pore size distribution, large surface area, and high degree of surface reactivity [19]. Chemical activation and physical activation are two methods for the preparation of activated carbon. Chemical activation uses chemical agents for the preparation of activated carbon in a single step method, while physical activation involves carbonization of

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carbonaceous materials followed by activation of the resulting substrate in the presence of dioxide carbon or steam as activating agents [20]. It is recognized that the carbon yields of chemical activation are higher than the physical one. The most common precursors used for the production of activated carbon are organic materials that are rich in carbon.

Several studies to find low-cost carbonaceous materials have been reported. These materials include Jerusalem artichoke [21], waste rice hulls [22], homemade cocoa shell [23], waste tea [24], coir pith [25], orange peels [26], jute sticks [27], walnut [28], palm oil shell [29], Acacia mangium wood [30] and waste tires [31].

In the present study, we examine the feasibility of using activated carbon prepared using *Persea americana* as adsorbent for the removal of Basic Blue 41 dyes from aqueous solutions. The effect of different parameters including solution pH, adsorbent dosage, dye concentration, temperature and contact time were studied to optimize the adsorption process. The isotherm and kinetic and thermodynamic parameters were examined to analyze the experimental data.

## Materials and methods

The *Persea americana* nuts were collected, washed with distilled water and dried at ambient temperature for several days. The unmodified *Persea americana* nuts were abbreviated as PAN. The carbonization of PAN was carried out using an appropriate weight of PAN and 25 mL concentrated phosphoric acid with a mass ratio (1:4). A glass beaker of 100 mL was heated to 500 °C for 1 h producing a black carbonaceous residue. The solid material was neutralized with KOH solution until a neutral pH was obtained. The resulting carbonized *Persea americana* nut (C-PAN) was filtered and washed intensively with water. The C-PAN was then dried at 100 °C for 2 h and kept in desiccators for further use.

The characterization of C-PAN was achieved by FT-IR spectroscopy and X-ray powder diffraction measurements. FT-IR spectra (4000–450 cm<sup>-1</sup> range) were recorded with a Nicolet 5700 FT-IR spectrometer on samples prepared as KBr pellets. The polycrystalline sample of each adsorbent was lightly ground in an agate mortar and pestle and filled into 0.5 mm borosilicate capillary prior to being mounted and aligned on an Empyrean PANalytical powder diffractometer using Cu K<sub>α</sub> radiation ( $\lambda = 1.54056 \text{ \AA}$ ). Three repeated measurements were obtained at room temperature in the  $10^\circ < 2\theta < 60^\circ$  range with a step size of 0.01°. Scanning electronic microscopy (SEM) images were obtained with HITACHI-S4100 equipment operated at 20 kV.

Adsorption–desorption isotherms of nitrogen at  $-196 \text{ }^\circ\text{C}$  were measured with an automatic adsorption instrument (NOVA-1000 Gas Sorption analyzer) to determine the surface areas and total pore volumes. The BET surface area and the total pore volumes of the obtained *Persea americana*-activated carbon were found to be 1593 and 1.053 cm<sup>3</sup>/g, respectively.

Deionized water was used throughout the experiments for solution preparation. The adsorption studies for evaluation of the C-PAN adsorbent for the removal of the Blue Basic 41 dye from aqueous solutions were carried out in triplicate using the batch contact adsorption method. Basic Blue 41 dye used in this study, abbreviated as BB41, was purchased from Sigma-Aldrich. The chemical structure of BB41 is shown in Fig. 1.

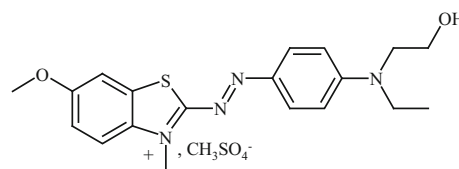
For the adsorption experiments, fixed amounts of adsorbents (5–30 mg) were placed in a 100 mL glass Erlenmeyer flask containing 50 mL of dye solution at various concentrations (25–125 mg/L), which were stirred for a suitable time (5–200 min) from 293 to 313 K. The pH of the dye solutions ranging from 2 to 10 was adjusted by 0.1 M HCl or 0.1 M NaOH to investigate the effect of pH on the adsorption processes. Subsequently, to separate the adsorbents from the aqueous solutions, the samples were centrifuged at 3600 rpm for 10 min, and aliquots of 1–10 mL of the supernatant were taken. At a predetermined time, the residual dye concentration in the reaction mixture was analyzed by centrifuging the reaction mixture and then measuring the absorbance by UV–visible spectroscopy of the supernatant at the maximum absorbance wavelength of the sample at 606 nm.

The amount of equilibrium adsorption  $q_e$  (mg/g) was calculated using the formula:

$$q_e = \frac{C_0 - C_e}{W} V, \quad (1)$$

where  $C_e$  (mg/L) is the liquid concentration of the dye at equilibrium,  $C_0$  (mg/L) the initial concentration of the dye in solution,  $V$  the volume of the solution (L) and  $W$  the mass of the dye biosorbent (g). The BB41 removal percentage (%) can be calculated as follows:

$$\% \text{Removal} = \frac{C_0 - C_e}{C_0} \times 100, \quad (2)$$



**Fig. 1** The chemical structure of Basic Blue 41

where  $C_0$ (mg/L) is the initial dye concentration and  $C_e$  (mg/L) is the concentration of the dye at equilibrium.

## Results and discussion

### Characterization of C-PAN adsorbent

To investigate the surface characteristics of the C-PAN adsorbent, FT-IR and XRD spectra were recorded. As shown in the FT-IR spectrum in Fig. 2, the frequencies of the absorption bands of C-PAN are 876, 1074, 1149, 1563, 2916 and 3415  $\text{cm}^{-1}$ . The absorption band at 3415  $\text{cm}^{-1}$  is attributed to the hydroxyl group (O–H) vibration [32]. The bands at 2916, 1563 and 1381  $\text{cm}^{-1}$  correspond, respectively, to unsymmetrical aliphatic C–H stretching, C=C stretching of aromatic rings and aromatic C=C stretching vibration. The band at 1149  $\text{cm}^{-1}$  is ascribed to C–O stretching in alcohol or ether or the hydroxyl group [33, 34]. The bands at 1074 and 876  $\text{cm}^{-1}$  could result from ionized linkage of P+ O– in acid phosphate esters, to symmetrical vibration in a chain of P–O–P and to P–C phosphorus-containing compound [35, 36]. These functional groups are due to the presence of  $\text{H}_3\text{PO}_4$  acid as an activation agent in the preparation of C-PAN.

Figure 3 shows an X-ray powder diffraction pattern of C-PAN. An amorphous peak with the equivalent Bragg angle at  $2\theta = 24.6$  was recorded, together with other peaks recorded at  $2\theta = 17.5^\circ$ ,  $31.2^\circ$  and  $47.5^\circ$ . The surface morphology of the C-PAN adsorbent was examined. Figure 4 shows the SEM image indicating that the surface is relatively smooth and contains many pores. The SEM of C-PAN show very distinguished dark spots, which can be taken as a sign for effective adsorption of dye molecules in the cavities and pores of this adsorbent.

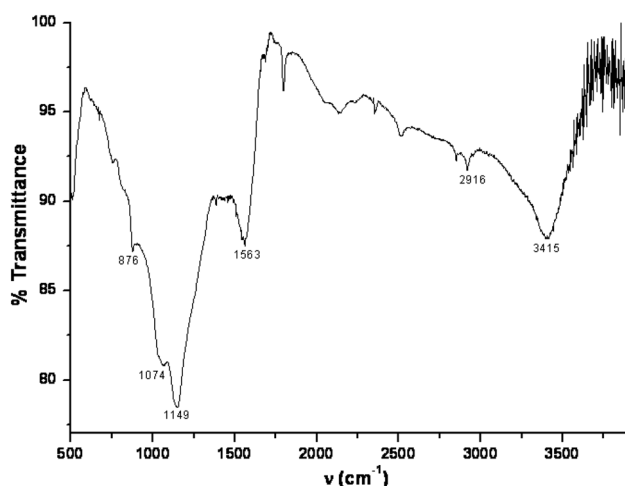


Fig. 2 FT-IR spectrum of C-PAN adsorbent

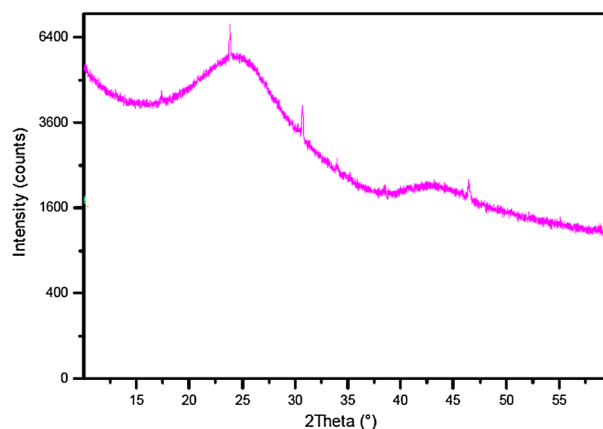


Fig. 3 XRD spectrum of C-PAN adsorbent

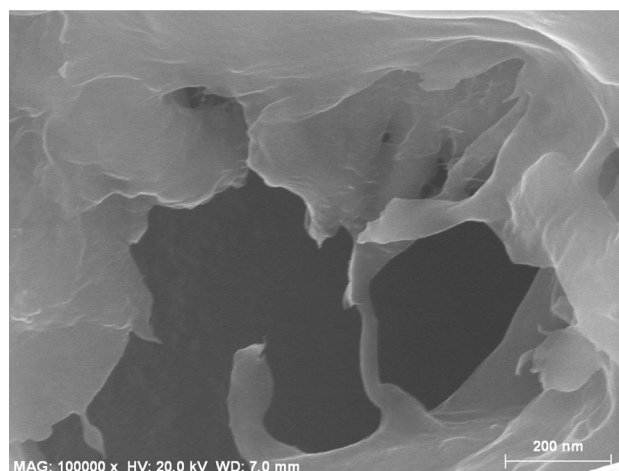
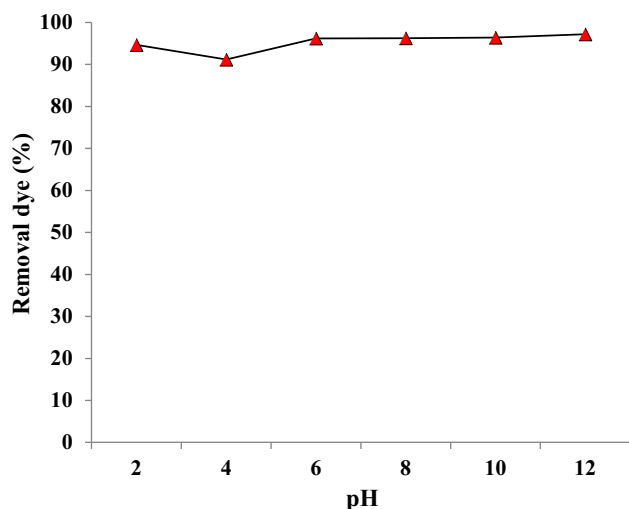


Fig. 4 SEM image of C-PAN adsorbent

### Effect of pH

To examine the pH effect of the initial aqueous dye solution, a concentration of BB41 dye at 100 mg/L and 20 mg of C-PAN adsorbent was used, keeping the temperature at 20 °C at different pH values in the range 2–12. Figure 5 shows the variation of dye removal vs. pH. In fact, the amount of dye adsorbed onto C-PAN was found to be constant for all pH values being studied. The experiments carried out at different pH values showed that there was no significant change in the percent removal of dye over the entire pH range. This indicates the strong affinity of the dye to C-PAN and that either  $\text{H}^+$  or  $\text{OH}^-$  ions could influence the dye adsorption capacity. The dye removal % uptake comprised between 92 and 97 %. If the adsorption would have occurred through an ion exchange mechanism, there should have been an influence on the dye adsorption while varying the pH. This observation and the high positive  $\Delta H^0$  value obtained indicate irreversible adsorption, probably

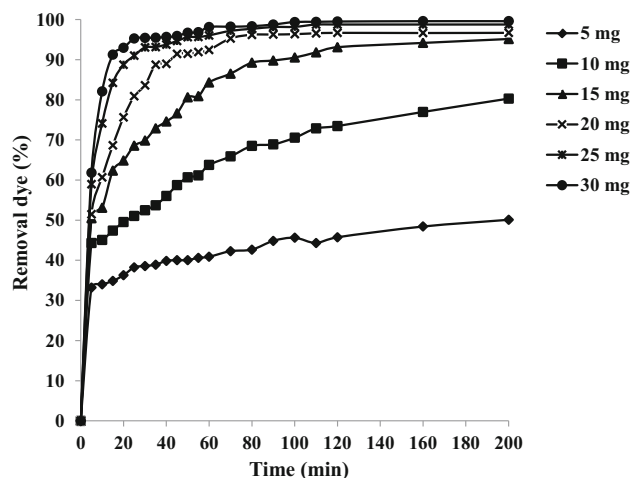


**Fig. 5** Effect of pH on the removal of BB41 from aqueous solution onto C-PAN. Initial dye concentration 100 mg/L; adsorbent amount 200 mg; agitation time 80 min; temperature 20 °C

due to nonpolar interactions [37]. Other studies for different dyes were also found to be independent of pH [37–39].

#### The effect of C-PAN adsorbent dosage

The effect of C-PAN dosage on adsorption was studied at 20 °C with 100 mg/L dye solution. The adsorbent dosage ranged from 5 to 30 mg. The plots of dye removal (%) versus time at different adsorbent dosages are shown in Fig. 6. The results follow the expected pattern, in which the removal (%) of BB41 increased with increase in C-PAN adsorbent dosage. This might be due to an increase in the surface active sites in C-PAN samples. The adsorption

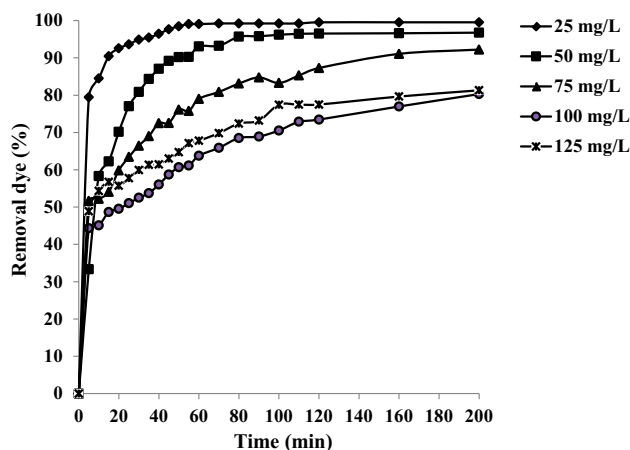


**Fig. 6** Effect of C-PAN adsorbent dosage and contact time on the removal of BB41 from aqueous solution. Initial dye concentration 100 mg/L; temperature 20 °C

equilibrium was achieved after 120 min of stirring the dye solution with the appropriate amount of C-PAN adsorbent. At this time, the removal of BB41 (%) increased from 45.7 to 99 % for C-PAN adsorbent dosage of 5 mg to 30 mg. However, the amount of BB41 adsorbed onto C-PAN,  $q$  (mg/g) was found to decrease from 457.46 to 165.83 mg/g upon increasing the adsorbent dosage. This behavior could be due to the high number of unsaturated sorption sites during the adsorption process.

#### Effect of BB41 concentration

The initial adsorbate concentration provides an important driving force to overcome all mass transfer resistances of dye between the aqueous and solid phases. The effect of initial concentration of dye on the adsorption was studied at 20 °C with 30 mg of C-PAN adsorbent. The adsorbate concentration ranged from 25 to 125 mg/L. The plots of dye removal (%) vs. time at different initial BB41 concentrations are shown in Fig. 7. The removal dye (%) shows a decreasing trend as the initial dye concentration of the dye is increased. At lower concentrations, all adsorbate ions present in the medium could interact with the binding sites, resulting in higher dye removal (%). At higher concentrations, the dye removal (%) shows a decreasing behavior because of the saturation of the adsorption sites. Similarly, the adsorption equilibrium was achieved after 120 min of stirring the appropriate dye solution with the C-PAN adsorbent. At this time, the removal of BB41 (%) decreased from 99 to 77.5 % for the initial BB41 concentration of 25 to 125 mg/L. However, the amount of BB41 adsorbed onto C-PAN,  $q_e$  (mg/g) was found to increase from 124.41 to 484.35 mg/g upon increasing the initial dye concentration



**Fig. 7** Effect of initial dye concentration and contact time on the removal of BB41 from aqueous solution. C-PAN 30 mg/L; temperature 20 °C



### Adsorption kinetics

To study the sorption process of BB41 onto C-PAN adsorbent, the data obtained from kinetic adsorption experiments were simulated with pseudo-first-order and pseudo-second-order models.

The pseudo-first-order equation is generally represented as follows [40]:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t, \tag{3}$$

where  $q_e$  is the amount of dye adsorbed at equilibrium (mg/g),  $q_t$  the amount of dye adsorbed at time  $t$  (mg/g),  $k_1$  the pseudo-first-order rate constant ( $\text{min}^{-1}$ ) and  $t$  the time (min).

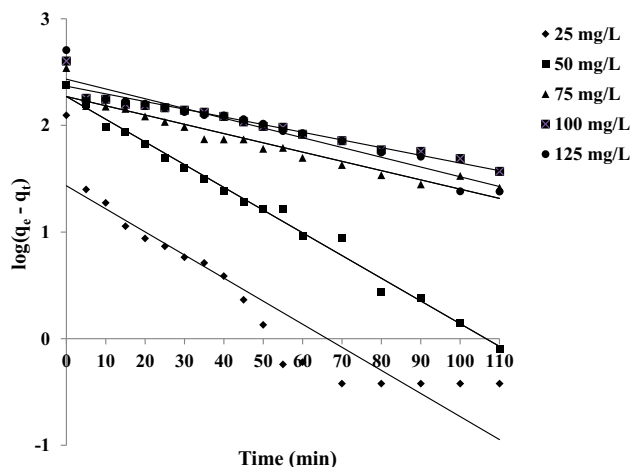
The values of  $k_1$ ,  $q_e$  calculated from the equation and the correlation coefficient ( $R^2$ ) values of fitting the first-order rate model at different concentrations are presented in Table 1. The linearity plots of  $\log(q_e - q_t)$  versus time at different initial dye concentrations (Fig. 8) suggested that the process of dye adsorption did not follow the pseudo-first-order rate kinetics. Also from Table 1, it is indicated that the values of the correlation coefficients are not high for the different dye concentrations. Furthermore, the estimated values of  $q_e$  calculated from the equation  $q_e$  differ substantially from those measured experimentally. This gives confirmation that the adsorption process of BB41 onto C-PAN did not obey the pseudo-first-order model.

The pseudo-second-order equation is generally represented as follows [41]:

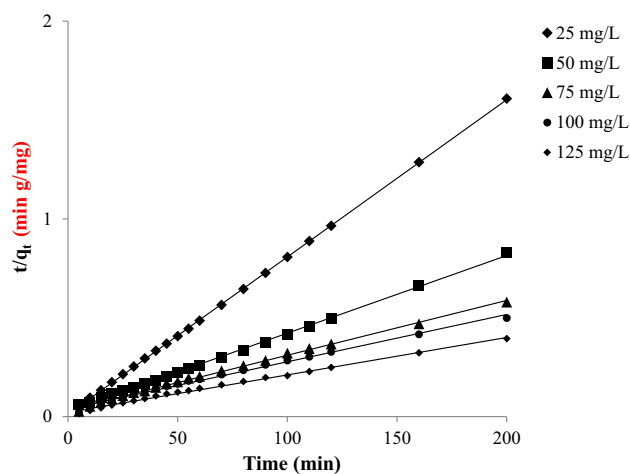
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t, \tag{4}$$

where  $k_2$  is the pseudo-second-order rate constant ( $\text{g}/\text{mg min}$ ).

A plot of  $t/q_t$  and  $t$  should give a linear relationship if the adsorption follows pseudo-second-order model. To understand the applicability of the model, a linear plot of  $t/q_t$  vs.  $t$  under different dye concentrations was plotted as in Fig. 9. The constants  $k_2$ ,  $q_e$  and correlation coefficients ( $R^2$ )



**Fig. 8** Pseudo-first-order plots for different initial dye concentrations removal using C-PAN adsorbent. C-PAN adsorbent 30 mg; temperature 20 °C



**Fig. 9** Pseudo-second-order plots for different initial dye concentration removal using C-PAN adsorbent. C-PAN adsorbent 30 mg; temperature 20 °C

were calculated from the plot and are given in Table 1. The  $q_e$  determined from the model along with correlation coefficients indicated that  $q_e$  was very close to  $q_{exp}$  and the

**Table 1** The pseudo-first-order and pseudo-second-order kinetic parameters for BB41 removal using C-PAN

Concentration of BB41 (mg/L)	Pseudo-first order				Pseudo-second order			
	$q_{e,exp}$ (mg/g)	$k_1$ ( $\text{min}^{-1}$ )	$q_{e,cal}$ (mg/g)	$r^2$	$k_2 \times 10^{-4}$ (g/mg min)	$q_{e,cal}$ (mg/g)	$r^2$	
25	124.41	0.0493	027.03	0.862	59.65	123.45	0.999	
50	241.90	0.0483	186.63	0.989	5.248	256.41	0.999	
75	345.84	0.0184	234.96	0.918	2.548	344.82	0.997	
100	401.45	0.0161	233.34	0.928	1.689	400.00	0.992	
125	508.13	0.0207	271.01	0.906	1.616	502.51	0.995	

correlation coefficient was also greater than 0.99. As a matter of consequence, the system BB41–C-PAN could be well described by the pseudo-second-order model. This adequate model shows that the adsorption of BB41 onto C-PAN is controlled by chemisorption mechanism.

### Adsorption isotherms

Adsorption isotherms are basic requirements for the design of adsorption systems. It can express the relationship between the amounts of adsorbate by unit mass of adsorbent at a constant temperature. Herein, we analyzed our experimental data by Langmuir and Freundlich isotherms models. The best-fitting model was evaluated using the correlation coefficient.

The Langmuir model [42] permits the evaluation of maximum dye adsorption capacity when all sites have equal affinities and active sites of adsorbent are independent of each other. The linear form of the Langmuir isotherm is expressed as follows:

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{1}{q_m} C_e, \quad (5)$$

where  $C_e$  (mg/L) is the equilibrium concentration of the BB41 dye and  $q_e$  (mg/g) is the amount of BB41 adsorbed per unit mass of adsorbent.  $q_m$  (mg/g) and  $K_L$  (L/mg) are the constants related to the maximum adsorption capacity and is the Langmuir constant, respectively.

A straight line with a slope of  $1/q_m$  and intercept of  $1/q_m K_L$  is obtained when  $C_e/q_e$  is plotted against  $C_e$ . Table 2 shows the values of these parameters.

The essential characteristics of the Langmuir equation can be expressed in terms of the dimensionless separation factor,  $R_L$ , defined as:

$$R_L = \frac{1}{1 + K_L C_0}, \quad (6)$$

**Table 2** Adsorption isotherm constants for removal of BB41 onto the C-PAN adsorbent

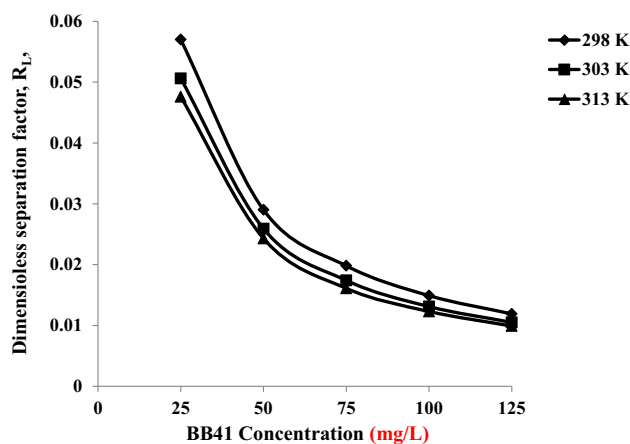
	Temperature		
	298 K	303 K	313 K
Langmuir isotherm			
$q_m$ (mg/g)	500.00	555.55	625.00
$K_L$ (L/mg)	0.66	0.75	0.80
$r^2$	0.997	0.995	0.991
Freundlich isotherm			
$K_F$ (mg/g) (L/g)	213.80	269.15	359.74
$n$	4.03	3.97	4.10
$r^2$	0.982	0.984	0.979

where  $C_0$  is the initial concentration of the BB41 dye; the  $R_L$  value implies whether the adsorption is unfavorable:  $R_L > 1$ , linear:  $R_L = 1$ , favorable:  $0 < R_L < 1$ , or irreversible:  $R_L = 0$ .

Figure 10 depicts the plot of the calculated  $R_L$  values versus the initial dye concentration at 298, 303 and 313 K. It was observed that all the  $R_L$  values obtained were between 0 and 1, showing that the adsorption of BB41 onto C-PAN was favorable. The  $R_L$  values decrease upon increasing the initial dye concentration, which indicates that the adsorption is more favorable at higher BB41 concentrations. The adsorption capacity increases on increasing the temperature, attaining a higher value of adsorption capacity of 625 mg/g at 313 K. The values of  $K_L$  increased on an increase in temperature. Therefore, by increasing the temperature, higher adsorption capacities were achieved.

Freundlich isotherm [43] is observed if the sites with stronger binding affinities are occupied by the dye molecules first and the binding strength decreases with increase in the degree of site occupation. The linear form of the Freundlich isotherm is expressed as follows:

$$\log(q_e) = \log(K_f) + \frac{1}{n} \log(C_e), \quad (7)$$



**Fig. 10** Dimensionless separation factor,  $R_L$ , versus concentration dye

**Table 3** Thermodynamic data for the adsorption of BB41 onto C-PAN

$T$ (K)	$K_C$	$\Delta G^0$ (kJ/mol)	$\Delta H^0$ (kJ/mol)	$\Delta S^0$ (J/mol K)
298	106.51	-11.64	144.6	524.3
303	287.90	-14.26		
313	624.08	-16.88		
323	1354.26	-19.50		

**Table 4** Comparison of the maximum monolayer adsorption capacities of C-PAN with those of various AC adsorbents

AC adsorbent	Dye	Isotherm	$q_m$ (mg/g)	Reference
Pomegranate peel	Remazol brilliant	Freundlich	370.86	[44]
Homemade cocoa shell	Reactive violet 5	Liu	603.3	[45]
Rambutan peel	Acid yellow 17	Langmuir	215.05	[46]
Rice husks	Methylene blue	Langmuir	578	[47]
Pomelo skin	Methylene blue	Langmuir	501.1	[48]
Stricta algae based	Safranin O	Langmuir	526	[49]
<i>Persea americana</i>	Basic Blue 41	Langmuir	625	This study

where  $K_f$  (mg/g) (L/g) and  $n$  are Freundlich constants related to the adsorption capacity and adsorption intensity, respectively.

The Freundlich constants  $n$  and  $K_f$  were obtained from the plot of  $\log(q_e)$  versus  $\log(C_e)$  that should give a straight line with a slope of  $(1/n)$  and intercept of  $\log(K_f)$  (as shown in Table 2). In this study, the values found for  $n$  were superior to 1, which indicates that the adsorption of BB41 onto C-PAN is favorable.

### Thermodynamic study

The thermodynamic data reflect the feasibility and favorability of the adsorption. The parameters such as free energy change ( $\Delta G^0$ ), enthalpy change ( $\Delta H^0$ ) and entropy change ( $\Delta S^0$ ) can be estimated by the change of equilibrium constants with temperature. The free energy change of the sorption reaction is given by:

$$\Delta G^0 = -RT \ln K_C, \quad (8)$$

where  $\Delta G^0$  is the free energy change (kJ/mol),  $R$  the universal gas constant (8.314 J/mol K),  $T$  the absolute temperature (K) and  $K_C$  the equilibrium constants ( $q_e/C_e$ ). The values of  $\Delta H^0$  and  $\Delta S^0$  can be calculated from the Van't Hoff equation:

$$\ln K_C = -\frac{\Delta H^0}{RT} + \frac{\Delta S^0}{R}, \quad (9)$$

where  $\ln K_C$  is plotted against  $1/T$ , and a straight line with the slope ( $-\Delta H^0/R$ ) and intercept ( $\Delta S^0/R$ ) are found. The calculated thermodynamic parameters are depicted in Table 3.

In the study,  $\Delta G^0$  values were determined at different temperatures and decrease from  $-11.64$  to  $-19.50$  kJ/mol when the temperature increases from 298 to 323 K. The negative values of  $\Delta G^0$  suggest that the adsorption of BB41 onto C-PAN is a highly favorable process. The values of  $\Delta H^0$  and  $\Delta S^0$  were obtained as 144.6 and 524.3 J/mol K, respectively. The positive value of  $\Delta H^0$  shows that the adsorption is an endothermic process, while a positive value of  $\Delta S^0$  reflects the increase of randomness state at the solid/solution interface during the adsorption.

### Comparison of adsorption capacities of C-PAN with those of various AC adsorbents

Table 4 depicts the values of maximum monolayer adsorption capacities of many AC adsorbents. It appeared that C-PAN can be classed among the best AC adsorbents with an adsorption capacity of 625 mg/g.

### Conclusion

Based on the results obtained in this study, it appears that the activated carbon prepared from *Persea Americana* nut constitutes a good adsorbent for removing a dye from aqueous solutions:

- The percent (%) removal of BB41 was observed to increase with increasing initial dye concentration and increasing adsorbent dose.
- The Langmuir isotherm best described the equilibrium data with acceptable  $R^2$ , which signifies that a homogeneous adsorption takes place between the BB41 dye and C-PAN.
- The pseudo-second-order equation best describes the kinetics of the C-PAN adsorption system due to its high  $R^2$ . In addition, the theoretical  $q_e$  generated by the pseudo-second-order equation is in good agreement with the experimental  $q_e$  value. This implies that the rate-limiting step is a chemisorption process.
- Thermodynamic studies indicated that the adsorption process is endothermic and spontaneous.

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