Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2015.1029007

57 (2016) 9498–9509
April

Taylor & Francis
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Adsorption of polycyclic aromatic hydrocarbons on banana peel activated carbon

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Received 22 August 2014; Accepted 6 March 2015

ABSTRACT

Polycyclic aromatic hydrocarbons (PAHs) are potential pollutants, released in aqueous systems through various sources such as industrial wastewaters, agricultural run-off, and deposition from the polluted air. The present study proposes an efficient and inexpensive approach to remove PAHs from aqueous system using activated carbon synthesized from waste banana peel. The Brunauer–Emmett–Teller N_2 surface area of the developed banana peel-activated carbon (BPAC) is more than 900 m^2/g. Effects of contact time, adsorbent dose, pH, and temperature on adsorption of PAHs have been examined. Results were fitted to Langmuir and Freundlich models, and characteristic parameters for both the adsorption isotherms were calculated. Data is better fitted to Freundlich model. The equilibrium time for the adsorption on BPAC is found to be 80 min. Equilibrium constants obtained from Langmuir adsorption isotherms have been used to calculate the thermodynamic parameters, such as ΔG°, ΔH°, and ΔS°. The result revealed that adsorption of PAHs onto BPAC is spontaneous and endothermic. Quantitative desorption of PAHs from BPAC has been achieved using sodium hydroxide and ethanol mixture, indicating the reuse of prepared activated carbon. The study thus provides cheap and easily acquirable adsorbent which effectively removes PAHs from aqueous system.

Keywords: PAH; Banana peel; Activated carbon; Adsorbent; Desorption

1. Introduction

PAHs are persistent organic pollutants, present ubiquitously in the environment, and composed of two or more fused aromatic rings. PAHs are recognized as potent carcinogens or mutagens and are listed as priority pollutants by the United States Environmental Protection Agency and European commission. Sources of PAHs can be natural or anthropogenic. Natural sources of PAH are forest fires, volcanic eruptions, oil spills, whereas anthropogenic sources can be petrogenic or pyrogenic. Petrogenic sources are petroleum products, fossil fuels, gasoline, asphalt, etc. Pyrogenic PAHs are produced by industrial releases, cigarette smoke, domestic and household burning, and incomplete combustion of fuels [1]. Major sources of PAHs in the environment are atmospheric fallouts, oil spills, petroleum industry, coking plants, paper industry, municipal wastewater treatment plants, etc. The petroleum industry releases large amounts of effluents containing PAHs produced during the refining of the

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crude oils, [2,3]. Chalbot et al. [4] reported the use of excessive chemicals, heat, and pressure during paper production as a source of PAHs. The presence of PAHs in the municipal wastewaters and sewage sludge has been confirmed by various authors [5–7]. Production of coke is another major source of PAHs in surface waters [8,9]. The treated coke industry wastewater containing PAHs is released into the water systems or used for the coke quenching. Coke quenching leads to the PAHs release to the atmospheric air. Therefore, the development of suitable methods for the removal of PAHs from surface waters as well as industrial wastewaters is required. Water solubility of PAHs is quiet low and it decreases with increasing molecular weight of PAHs. According to the legislation, the permissible limit of PAHs in industrial wastewater released to the sewer is 0.2 mg L^{-1} [3], whereas, the permissible limit of PAHs in drinking water according to WHO is 0.002 mg L^{-1} [10]. Higher levels of PAHs in various water and wastewater systems have been monitored by various workers [11–13]. A variety of methods such as biodegradation, catalytic, and photocatalytic degradation [14–16] have been employed for their removal from aqueous and industrial wastewater systems. These methods have several limitations such as low biodegradability of PAHs, the metabolites formed during degradation might be more hazardous and persistent than parent compound. Therefore, the studies are being focused on their adsorptive removal from aqueous systems utilizing activated carbons [17–19].

Adsorption is a simple, versatile, and powerful technique for the removal of pollutants from liquid systems. Activated carbon is the most widely used adsorbent for the removal of pollutants. Several raw materials such as rice husk [20], coconut husk [21], waste tyre [22], plant bark [23], vegetable waste [24], and chicken waste [25] have been used for the synthesis of activated carbon. Memon et al. [26] and Owabor et al. [27] have used banana peel and orange peel as such for the removal of Cr(III) and naphthalene, respectively. In the present study, banana peel has been used to synthesize activated carbon with high adsorption capacity.

The synthesized activated carbon was used for the removal of three PAHs, namely naphthalene (Naph), fluorene (Flu), and phenanthrene (Phen) from aqueous system. Naphthalene, fluorene, and phenanthrene are low molecular weight PAHs, present naturally in coal tar and are used as moth repellent, wood preservative, and are starting material for the synthesis of several compounds such as resins [28]. The three PAHs with less than four rings were chosen for the study due to their high availability in different water and wastewater systems. The adsorption data were subjected to Freundlich and Langmuir isotherms. The effect of various parameters such as contact time, adsorbent dose, pH, and temperature on the adsorption was evaluated. Different thermodynamic parameters were calculated to ascertain the nature and spontaneity of the adsorption. The main objective of the work is to develop a suitable activated carbon from highly available waste which can be used in treatment plants of different industries such as petroleum and coke industry. The study not only provides an efficient method for the removal of naphthalene, fluorene, and phenanthrene, but also serves as a base for the removal of other PAHs.

2. Materials and methods

2.1. Adsorbate

Naphthalene, $C_{10}H_8$, fluorene, $C_{13}H_{10}$, and phenanthrene, $C_{14}H_{10}$ (>99%) were supplied by Merck. Ultrapure water was used throughout the analysis. Stock solutions of PAHs were obtained by dissolving a definite amount of PAH in methanol. Working solutions of PAHs (20 mg L^{-1}) were prepared from stock solutions using 20% methanol for dilution. The solutions were stored in airtight glass bottles in deep freeze at 4˚C. The solution pH was adjusted using conc. H_2SO_4 and NaOH, and determined by digital pH meter (Toshniwal Industries, India). The effect of temperature on adsorption was studied using a thermostat (Julabo GmbH, Germany).

2.2. Adsorbent

Banana peel was used as a precursor for the preparation of activated carbon using the method described by Srinivasakannan and Bakar [29]. Banana peels were dried overnight in an oven at 70˚C. The dried banana peels were allowed to soak with 60% phosphoric acid for overnight. The soaked samples were kept in the muffle furnace at 200˚C for semicarbonization for 4 h. The semicarbonized material was dried and cooled to room temperature. The dried material was again heated in furnace at 500˚C for 2 h for activation. The activated material (BPAC) thus obtained was repetitively washed with distilled water to remove all the acid from the material, and washing was continued till the wash liquor was neutral. The obtained carbonaceous material was finally washed with 0.1-M sodium hydroxide solution, followed by distilled water. The product was dried in hot air oven for 5 h at 105˚C and subjected to size reduction, so that the material passes through 75 μm mesh.

Powder X-ray diffraction (XRD) pattern of the adsorbent was obtained by Bruker AXS D8 powder diffractometer employing Cu K_α radiation (40 kV, 45 mA, $\lambda = 1.5418$ Å) and goniometer speed of 2°/min was used for the analysis. The Brunauer–Emmett–Teller surface area was assessed using Micrometics Instrument (Norcros, USA). ZEISS Ultra plus microscope at 15 kV was used for field emission scanning electron microscope (FE-SEM) images. Functional groups present on the BPAC surface were analyzed by Fourier transform infrared spectroscopy (FTIR).

2.3. Adsorption studies

Adsorption studies were carried out in batch mode at 25 ± 2°C. Thirty milliliters of a PAH solution of a known concentration (20 mg L^{-1}) were taken in a conical flask and 2 mg of the adsorbent was added. The mixture was shaken on a mechanical shaker for 80 min at pH 7 (unless otherwise mentioned) and left for 5 min to attain equilibrium. The samples were filtered through Whatmann filter No. 42 and analyzed using UV spectrophotometer at 275, 262, and 250 nm for naphthalene, fluorene, and phenanthrene, respectively. The analysis was carried out in triplicate. The recovery percentage of naphthalene, fluorene, and phenanthrene were 97 ± 2% in all the cases. The amount of PAH adsorbed per gram of adsorbent q_e was evaluated from the mass–balance equation:

$$q_e = V(C_0 - C_e)/W \tag{1}$$

where V is the volume of the solution (L), C_0 is the initial concentration of the adsorbate solution (mg L^{-1}), C_e is the concentration of the adsorbate in liquid phase at equilibrium (mg L^{-1}), and W is the mass of the adsorbent (g).

The adsorption data obtained at different temperatures were used to draw Langmuir and Freundlich isotherms. The Langmuir isotherm predicts the existence of monolayer coverage of the adsorbate at the outer surface of the adsorbent [30]. The linear form of Langmuir adsorption model is given below:

$$1/q_e = (1/Q^\circ b)(1/C_e) + (1/Q^\circ) \tag{2}$$

where Q° is the measure of adsorption capacity of monolayer (mg/g) and b is the Langmuir constant. The plot between $1/Q_e$ and $1/C_e$ is a straight line with a slope corresponding to $1/Q^\circ b$ and intercept to $1/Q^\circ$. Based on the Langmuir constant b and initial concentration C_o, the values of separation factor, R_L, were calculated using the equation:

$$R_L = 1/(1 + bC_o) \tag{3}$$

Freundlich isotherm [30] demonstrates the multilayer adsorption involving heterogeneous surfaces. The isotherm is expressed by the following equation:

$$q_e = K_f C_e^{1/n} \tag{4}$$

where K_f and n are Freundlich constants. The linear form of the equation obtained by taking logarithm on both the sides is given below:

$$\log q_e = \log K_f + (1/n) \log C_e \tag{5}$$

A plot of log q_e vs. log C_e gives a straight line with a slope of $1/n$ and intercept of K_f.

3. Result and discussion

3.1. Characteristics of adsorbent

Fig. 1 shows the XRD spectrum of the adsorbent BPAC. Two broad peaks around 24° and 42° in the spectrum show the formation of amorphous activated carbon [31]. The specific surface area of the adsorbent BPAC is 984.51 m^2/g.

The FE-SEM images of BPAC depict the surface morphology of the activated carbon, before and after adsorption of PAHs (Fig. 2).

FTIR spectra of BPAC before and after adsorption of PAHs are shown in Fig. 3. In the FTIR spectrum of the adsorbent, major bands appeared at the peak

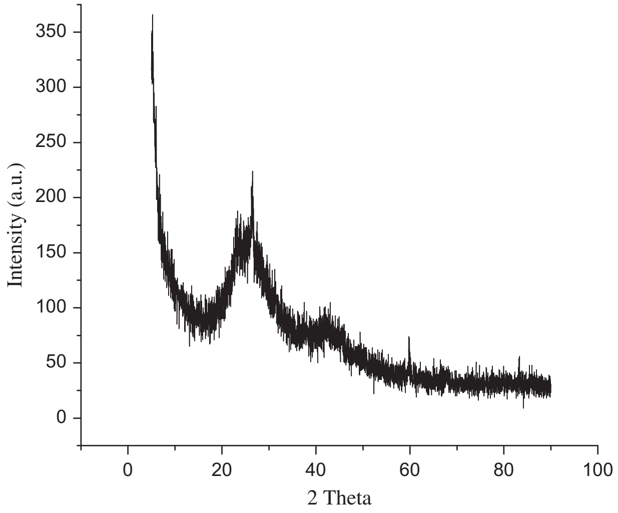
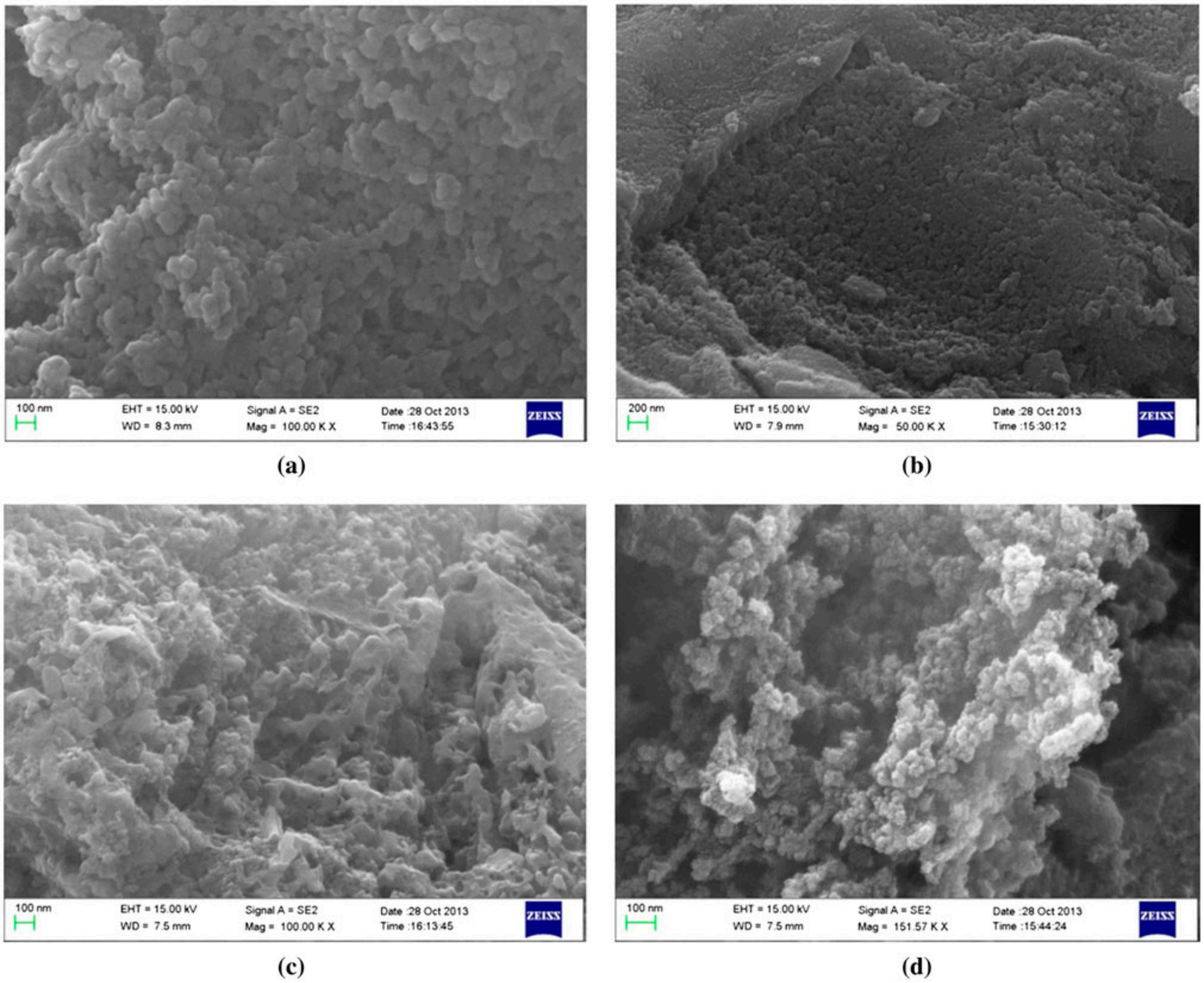


Fig. 1. XRD Spectrum of BPAC.



(a) (b) (c) (d)

Fig. 2. FE-SEM images of BPAC before adsorption (a) and after adsorption of naphthalene (b), fluorene (c), and phenanthrene (d).

positions 3,413, 1,599, 1,387, 1,212, and 1,072 cm^{-1}. The peak at 3,413 cm^{-1} is due to –OH group stretching and the band at 1,599 cm^{-1} is due to C=C stretching vibration. The peaks at 1,387 and 1,212 cm^{-1} are due to P=O stretch or O–H bending vibration and P–O–C stretch, respectively. The presence of oxy-phosphorus groups on BPAC is due to the use of phosphoric acid in the preparation of BPAC. The peak at 1,072 cm^{-1} is attributed to the phosphate group stretching. The FTIR spectra clearly indicate that the major groups on the surface of BPAC are P=O and –OH groups. The appearance of new peaks and shifting of the former peaks after adsorption are due to the adsorption of PAHs on the surface of the adsorbent. A new peak in the range 958–946 cm^{-1} after adsorption is due to the attachment of aromatic ring with P–O group. The peaks due to P=O, –OH, and PO_4^{3-} groups are shifted on adsorption of all the three PAHs. Additional peaks around 1,500 cm^{-1} are due to aromatic ring stretch. Several new peaks in the reason 900–700 cm^{-1} are due to out-of-plane bending vibration of aromatic C–H which confirms the adsorption of aromatic compounds on BPAC in all the cases. The additional peaks at 2,916, 1,442, and 1,301 cm^{-1} in case of fluorene adsorption are due to stretching and bending C–H vibration of methylene group and stretching of C–C single bond, respectively. The assignments of IR peaks after adsorption of PAHs on BPAC are given in Table 1.

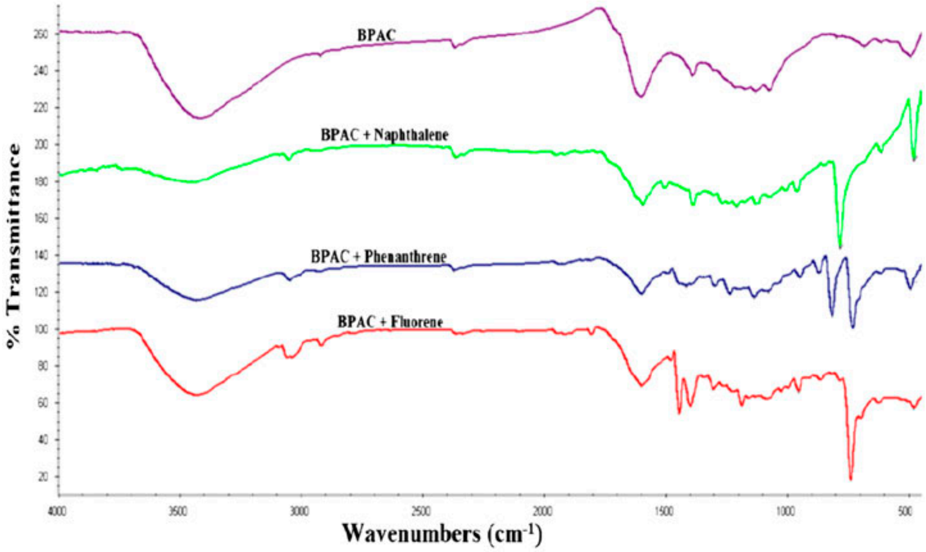


Fig. 3. FTIR spectra of BPAC, before and after adsorption of PAHs.

Table 1
IR frequencies of BPAC and BPAC + PAH (cm^{-1})

BPAC	BPAC + Naph	BPAC + Flu	BPAC + Phen	Assignments
3,413	3,439	3,431	3,432	U (O–H)
	3,055	3,036	3,046	U (Aromatic C–H)
		916		U (C–H)
1,599	1,592	1,597	1,595	U (C=C–C)
	1,500	1,474	1,486	U (C=C–C)
		1,442		δ (C–H)
1,387	1,385	1,396		U (P=O)/δ (O–H)
		1,301		U (C–C)
1,212	1,207	1,184	1,234	U (P–O–C)
1,072	1,065	1,082	1,074	U (PO_4^{3-})
	958	951	946	U (P–O–C)
	850	865	867	γ (C–H)
			813	γ (C–H)
	781	735	726	γ (C–H)

Note: U = stretching; δ = bending; β = in plane bending; γ = out of plane bending.

3.2. *Effect of contact time*

To study the effect of contact time on the adsorption of PAHs on BPAC, a solution of PAH-containing adsorbent was shaken mechanically for varying time (15–120 min). For all the three studied PAHs, adsorption increases up to 60 min and becomes constant thereafter (Fig. 4). For all subsequent studies, a shaking time of 80 min was observed. It was checked that prolonged shaking had no adverse effect on the adsorption.

The results suggest that the adsorption of naphthalene is slightly lower compared to fluorene and phenanthrene. This may be due to the most hydrophilic nature of naphthalene among the studied PAHs (log K_{ow} values: Naph—3.30, Flu—4.18, Phen—4.57). Yakout et al. [32] also found higher adsorption of phenanthrene than naphthalene on activated carbon derived from agricultural wastes. Among phenanthrene and fluorene, fluorene shows slightly higher adsorption than phenanthrene in the present study.

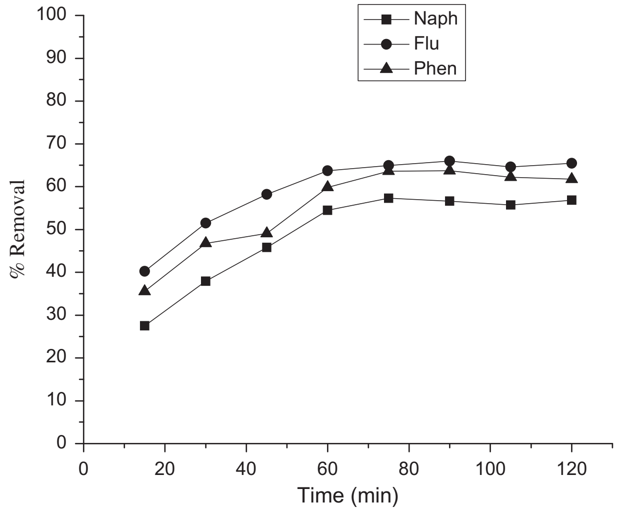


Fig. 4. Effect of contact time on adsorption of PAHs. [PAH] = 20 mg L^{-1}; pH 7; adsorbent = 2 mg; temperature = 25 ± 2˚C.

This observation may be attributed to the smaller diameter of fluorene than phenanthrene (Flu—1.14 nm, Phen—1.17 nm [33]). The absence of any particular trend in the adsorption of the studied PAHs is probably due to fact that various factors such as hydrophobic character, solubility, molecular size, chemical properties of adsorbate, and nature of adsorbent influence the adsorption process [34].

3.3. Effect of adsorbent dose

Fig. 5 shows the results of adsorption study carried out with varying amounts of BPAC (0.5–10 mg). The results reveal that adsorption of PAHs increases with the increase in adsorbent dose initially and attains a quantitative value (>98%) at 8 mg for fluorene and phenanthrene and 10 mg for naphthalene.

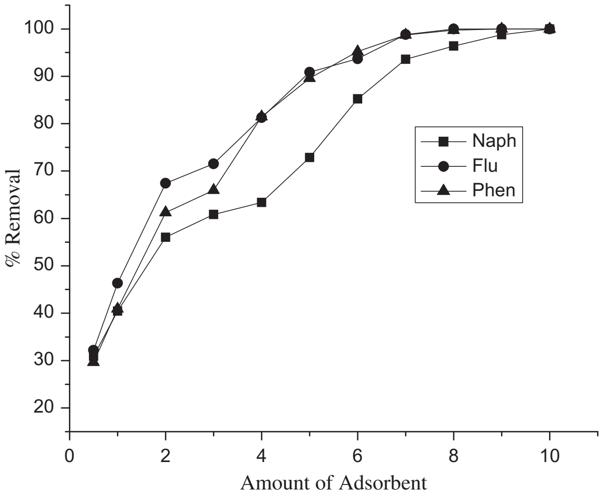


Fig. 5. Effect of adsorbent dose on adsorption of PAHs. [PAH] = 20 mg L^{-1}; pH 7; shaking time = 80 min; temperature = 25 ± 2˚C.

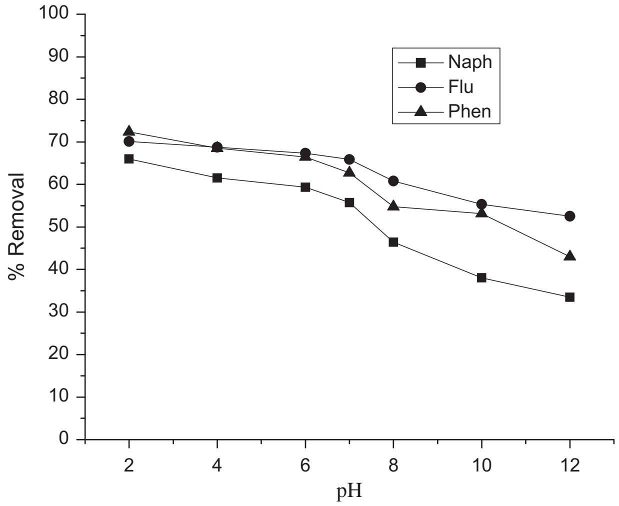


Fig. 6. Effect of pH on the adsorption of PAHs onto BPAC. [PAH] = 20 mg L^{-1}; adsorbent = 2 mg; shaking time = 80 min; temperature = 25 ± 2˚C.

3.4. Effect of pH

The influence of pH on the adsorption of PAHs on BPAC was investigated in the pH range 2–12. The results are shown in Fig. 6 which indicate that the adsorption of all the three PAHs decreases with the increase in pH. The results further suggest that the adsorption of PAHs is higher at low pH due to the presence of positive charge on the surface of the adsorbent. Thus, at low pH, electrostatic attractions are higher between the positively charged adsorbent surface and PAH, accounting for higher adsorption. As the pH increases, the adsorption of PAHs decreases due to decrease in positive charge on the surface, followed by decrease in electrostatic attraction between the adsorbate and adsorbent. At higher pH, the competition between OH^- ions and PAH for adsorption sites may also lead to lower adsorption. Similar behavior was observed by Owabor et al. [27] during removal of naphthalene using unripe orange peel.

3.5. Effect of temperature

To discern the effect of temperature on the adsorption of PAHs, studies were carried out at 20, 30, 40, and

50˚C. The results indicate that adsorption of all the three PAHs increases with rise in temperature (Fig. 7). The trend suggests that adsorption of PAHs on BPAC is endothermic in nature. Similar results were observed by Mittal et al. [35] and Baseri et al. [36] for the adsorption of Tartrazine and basic dyes, respectively.

3.6. Modeling of adsorption isotherm

The plots between $1/q_e$ and $1/ C_e$ are straight line as shown in Fig. 8. Langmuir adsorption constants Q° and b were calculated using the slope and intercept values. Similarly, Freundlich adsorption isotherm plots were drawn between log q_e and log C_e. The straight line obtained was used to calculate Freundlich constants n and K_f (Fig. 9). The Langmuir and Freundlich constants along with r^2 values for adsorption of PAHs at different temperatures are presented in Table 2. The r^2 values suggest that the data are better fitted to the Freundlich isotherm.

The calculated R_L values were in the range 0.0625–0.7974 for all the three PAHs which indicates the adsorption process to be favorable ($R_L > 1$ Unfavorable, $R_L = 1$ Linear, $0 < R_L < 1$ Favorable, $R_L = 0$ Irreversible) [35].

The synthesized adsorbent BPAC was compared with some other reported activated carbons for PAHs with respect to adsorption capacity, equilibration time, and temperature range (Table 3). The perusal of data suggests that the developed adsorbent has high adsorption capacity than several reported adsorbents (1, 2, 3(b), 5(a), 6, 7(a) & 7(b) in Table 3). In the cases where adsorption capacity of BPAC is comparable (3 (a), 5(b)) or lower (4) than the reported ones, the equilibration time required for the BPAC is much less than the time required for the reported ones.

3.7. Thermodynamic parameters

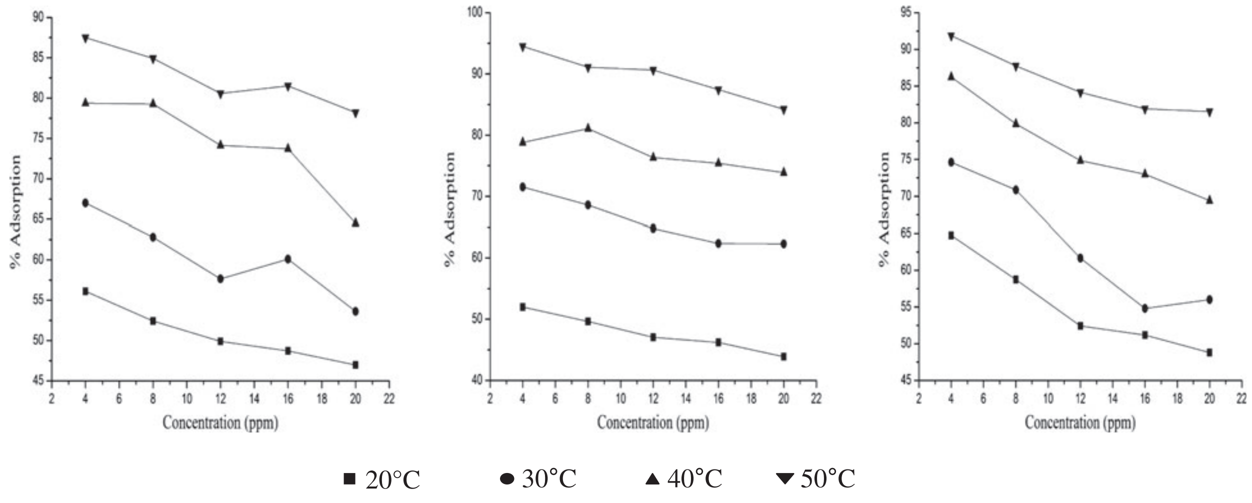
The spontaneity and the nature of an adsorption process are depicted by various thermodynamic parameters. The thermodynamic parameters, Gibb's free energy change, ΔG°, enthalpy change, ΔH°, and entropy change, ΔS°, were evaluated from Langmuir adsorption isotherms using the following equations:

$$\Delta G^\circ = -RT \ln K_a$$

and

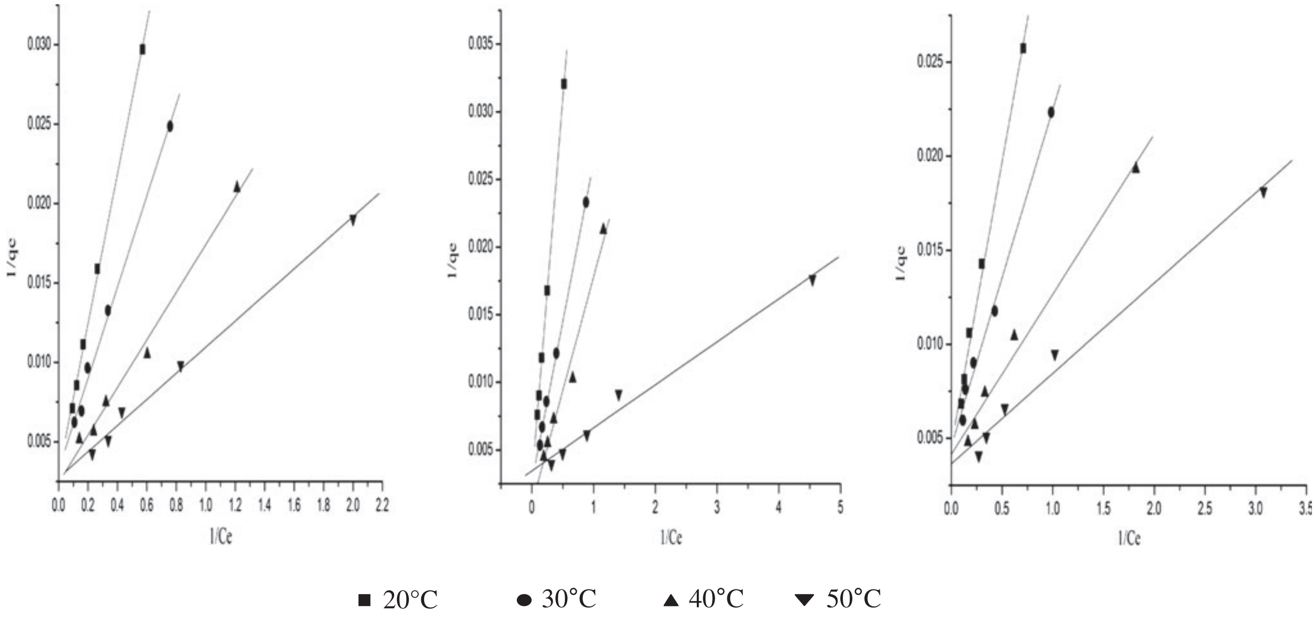
$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ$$

where R is universal gas constant (8.314 $\text{JK}^{-1}\,\text{mol}^{-1}$), T is absolute temperature in Kelvin, and K_a is equilibrium constant obtained from Langmuir adsorption isotherms. Gibb's free energy, ΔG°, was evaluated using equilibrium constants at varying temperatures. A plot between ΔG° and temperature, T, gave a straight line with a slope value corresponding to ΔS° and intercept to ΔH°. The values of thermodynamic parameters, ΔG°, ΔH°, and ΔS° are presented in Table 4.



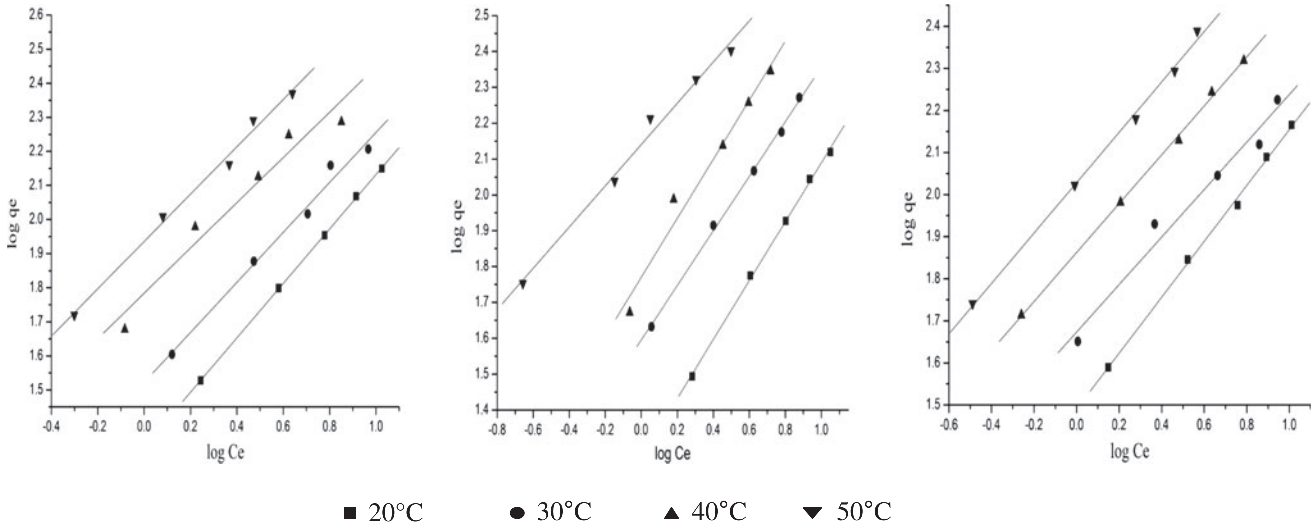
(a) Naphthalene **(b) Fluorene** **(c) Phenanthrene**

Fig. 7. Effect of temperature on adsorption of PAHs on BPAC. [PAH] = 20 mg L^{-1}; pH 7; adsorbent = 2 mg; shaking time = 80 min.



(a) Naphthalene **(b) Fluorene** **(c) Phenanthrene**

Fig. 8. Langmuir adsorption isotherm for PAH-BPAC system at different temperatures.



(a) Naphthalene **(b) Fluorene** **(c) Phenanthrene**

Fig. 9. Freundlich adsorption isotherm for PAH-BPAC system at different temperatures.

The negative values of ΔG° indicate the spontaneity of the studied adsorption processes. Reduction in ΔG° values with the increase in temperature shows the adsorption of PAHs is favorable at higher temperatures. The endothermic nature of the adsorption process is depicted by the positive value of enthalpy change, ΔH°. The positive value of the entropy change, ΔS°, indicates increase in randomness at the interface (solid/liquid) during adsorption of PAHs and suggests good affinity of the PAHs towards the BPAC.

Table 2
Langmuir and Freundlich constants and r^2 values for adsorption of PAHs on BPAC at different temperature

	Naphthalene				Fluorene				Phenanthrene			
	20℃	30℃	40℃	50℃	20℃	30℃	40℃	50℃	20℃	30℃	40℃	50℃
Langmuir parameters												
Q° (mg/g)	333.33	344.82	370	416	285.71	370.37	384.61	476.19	217.39	222.22	243.90	277.77
b (L/mg)	0.0635	0.1006	0.18	0.2926	0.0762	0.1144	0.1538	0.2560	0.1523	0.2513	0.4823	0.75
r^2	0.9658	0.8918	0.9530	0.9228	0.9841	0.9371	0.8893	0.9804	0.9425	0.9359	0.9556	0.9218
Freundlich parameters												
n	1.2521	1.3636	1.44	1.5055	1.217	1.2325	1.3131	1.7382	1.5053	1.5767	1.6329	1.7705
K_f	21.54	33.28	60.77	86.21	18.54	39.34	58.92	138.22	30.92	47.11	72.84	106.73
r^2	0.9998	0.9852	0.9905	0.9922	0.9986	0.9978	0.9743	0.9892	0.9978	0.9743	0.9987	0.9969

Table 3
Adsorption capacities of various adsorbents reported in the literature

Adsorbent	Adsorbate	Adsorption capacity (mg/g)	Temperature	Equilibrium time	References
1. Activated carbon (Coconut Shell)	Phenanthrene	20.22	25℃	1 h	[37]
2. Activated carbon (Commercial)	Naphthalene	271.72	20℃	NA	[38]
3. Activated carbon (Bean Pods)	Naphthalene				[18]
(a) Chemical activation		300	NA	72 h	
(b) Physical activation		85	NA	72 h	
4. Zeolite	Naphthalene	769.23	NA	>200 min	[28]
5. Activated carbon	Naphthalene				[19]
(a) Milk bush kernel shell		21	28 ± 2℃	2 h	
(b) Flamboyant pod		294.118	28 ± 2℃	2 h	
6. Boehmite nanopowder	Naphthalene	200	Room Temp.	1 h	[39]
7. (a) Activated carbon (Commercial)	Anthracene	8.35	25℃	20 min	[40]
(b) Posidonia oceanica		0.14	25℃	20 min	
8. (a) BPAC	Naphthalene	333.33	20℃	80 min	Present study
(b) BPAC	Fluorene	285.71	20℃	80 min	Present study
(c) BPAC	Phenanthrene	217.39	20℃	80 min	Present study

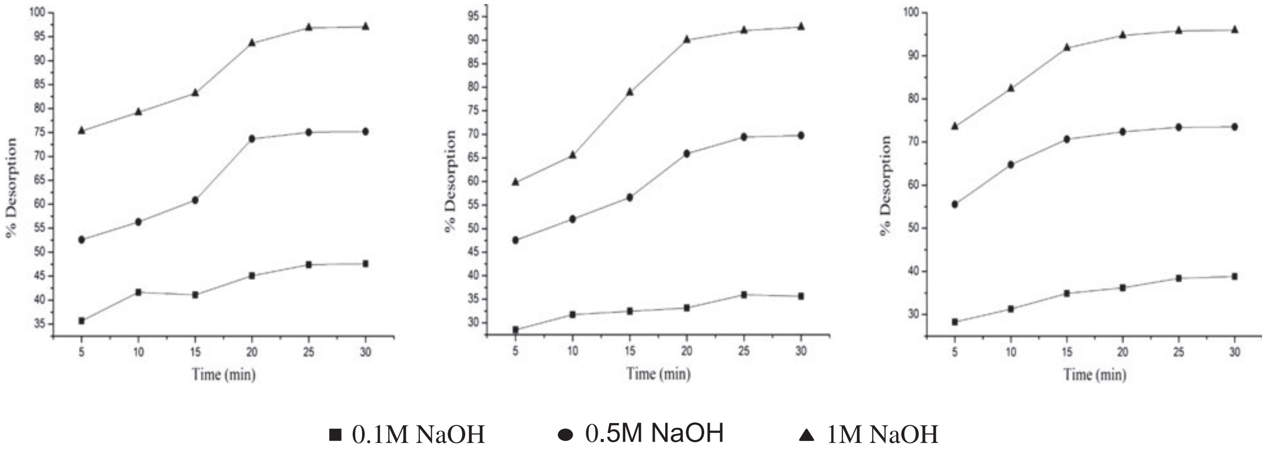
Note: NA—Not available.

4. Desorption studies

Desorption studies were carried out in batch mode. In the first step, adsorption was carried out under optimum conditions to load the BPAC with the respective PAH. In the second step, the loaded BPAC was agitated with NaOH and ethanol mixture [41] of varying compositions for different time (5–30 min). The results are depicted in Fig. 10 which indicate that

Table 4
Thermodynamic parameters for the adsorption of PAHs on BPAC

	$-\Delta G^\circ$ (kJ mol^{-1})					
	20℃	30℃	40℃	50℃	ΔH°	ΔS°
Naphthalene	5.107	6.440	8.167	9.733	40.702	0.1561
Fluorene	6.184	7.419	8.434	10.072	31.024	0.1268
Phenanthrene	8.041	9.577	11.590	13.146	42.782	0.1733



(a) Naphthalene **(b) Fluorene** **(c) Phenanthrene**

Fig. 10. Desorption of PAHs from BPAC with different NaOH concentrations.

1.0 M NaOH in 50% ethanol is most effective for quantitative desorption of naphthalene (97%), fluorene (95%), and phenanthrene (95%).

5. Conclusion

The present study confirms that the activated carbon synthesized from waste banana peel is a potential adsorbent for the removal of PAHs from aqueous system. The adsorption data were feasible with both Langmuir and Freundlich models, but was better fitted to Freundlich model. The synthesized adsorbent has good adsorption capacity and large BET surface area. The negative values of Gibb's free energy change, ΔG°, and the positive value of enthalpy change, ΔH°, reveal the spontaneity and endothermic nature of the adsorption process, respectively. The desorption studies suggest that the adsorbent can be readily regenerated using NaOH solution in 50% ethanol which is cheap and easily available. The study provides cheap, effective, and recyclable adsorbent which is useful for the removal of PAHs and can be easily utilized in the industrial treatment plants for the removal of PAHs. The developed adsorbent is eco-friendly and utilizes readily available banana peels.

Acknowledgments

One of the authors, Himanshu Gupta, would like to acknowledge Ministry of Human Resource Development (MHRD), India for funding the scheme.

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