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Removal of Acid Violet 17 from Aqueous Solutions by Adsorption onto Activated Carbon Prepared from Pistachio Nut Shell

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Adsorbents prepared from pistachio nut shell, an agricultural waste biomass, were successfully used to remove Acid Violet 17 from an aqueous solution. The activated carbons PNS1, PNS2, and PNS3 were characterized by scanning electron microscope (SEM), Fourier Transform – Infra Red spectroscopy (FTIR) and (BET). The effect of pH, adsorbent dosage, and temperature on dye removal was studied. Maximum color removal was observed at pH 2. The adsorption increased with the increase in adsorbent dosage. As the adsorption capacity increased with the increase in temperature, the process was concluded to be endothermic. The experimental data were analyzed by the Langmuir and Freundlich isotherm models of adsorption. Equilibrium data fitted well with the Langmuir model. The rates of adsorption confirmed the pseudo-second order kinetics with good correlation values. The results indicated that the activated carbon prepared from pistachio nut shell can be effectively used for the removal of Acid Violet 17 from aqueous solution.

Keywords Acid Violet 17; adsorption; isotherms; kinetics; pistachio nut shell

INTRODUCTION

Colored compounds comprising of pigment and dyes are widely used in textile, plastic, food, dyeing, paper, printing, pharmaceutical, and cosmetic industries. The dyes impart color to the receiving water and are aesthetically objectionable and they also reduce light penetration into water decreasing the efficiency of photosynthesis in aquatic plants, thereby, having an adverse impact on their growth (1). Wastewater containing dyes create a serious environmental problem because of its high toxicity and possible accumulation in the environment. Many of these dyes, especially acid dyes, are highly water–soluble due to the presence of sulphonic acid groups. Among these dyes, Acid Violet 17 is used by several industries, such as textile, paper, printing, and plastics to color their products. The effluent discharged from these industries is highly colored and disposal of this colored water into the receiving water body not only causes damage to aquatic life, but also to human beings, by producing carcinogenic and mutagenic effects (2). Due to a large degree of organics present in these molecules the conventional physico-chemical and biological treatment methods are not effective for their removal (3), because these processes are costly, need a large land area, and cannot be effectively used to treat the wide range of dye wastewaters (4). Adsorption is one of the important techniques used for the dye removal due to simplicity of design, ease of operation, insensitivity to toxic substances, and ability to treat dyes in more concentrated forms (3). Activated carbon is the most effective and common adsorbent for the adsorption process due to its effectiveness and versatility. However, commercially available activated carbons are very expensive. Therefore, there is a need to produce low-cost and effective carbons for the treatment of dyes in wastewater (5).

In India, agricultural by-products and waste materials that are available in large quantities may have the potential to be used as low-cost adsorbents. The conversion of agricultural waste materials into activated carbon would add considerable economic value, helps to reduce the cost of waste disposal, and most importantly provides a potentially inexpensive alternative to the existing commercial activated carbons. There are many studies in the literature relating to the preparation of activated carbons from agricultural wastes such as sunflower seed hull, peanut hull, almond shell, wheat bran, bagasse, coir pith, banana pith, date pits, cotton stalks, palm tree, orange peel, corncob, barley husk, palm kernel shell, rice husk, pinewood, and soy hull (6–20). Pistachios are native of Afghanistan, Iran, and Turkey, and are now cultivated widely in the

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United States, Syria, China, Greece, etc. In 2005, the world production of pistachio was about 501 thousand metric tonnes i.e., a huge amount of pistachio nut shells is available. However, there are very few research works that report on the preparation of activated carbon using pistachio nut shells for the removal of dyes from waste water (21). Normally pistachio nut shells are used as boiler fuel or in landfills. In the present study it was proposed to utilize this abundant solid waste as starting material for the preparation of activated carbon for the removal of Acid Violet 17 dye (AV 17) from aqueous solution.

EXPERIMENTAL

Materials and Methods

Pistachio nut shells were collected from Taiwan. The adsorbate AV 17 was obtained from Hindustan Ciba-Geigy, Mumbai, India and it was used as such without any further purification. The chemical structure and the characteristics of AV 17 are shown in Fig. 1 and Table 1 respectively. Sulphuric acid used for the activation of pistachio nut shell, hydrochloric acid, and sodium hydroxide used for the pH studies were of analytical reagent grade and were purchased from Qualigens Fine Chemicals, Mumbai, India.

Preparation of Activated Adsorbents

Pistachio nut shells were repeatedly washed with distilled water to remove dirt, dust, and other impurities. The washed shell materials were then dried in sunlight for 48 h. The first sample (PNS1) was prepared by mixing one part of pistachio nut shells and 2 parts of 18 N sulphuric acid (w/v) and kept at room temperature for 24 h. The second sample (PNS2) was prepared by mixing one part of pistachio nut shells and 2 parts of 18 N sulphuric acid (w/v) and kept in a muffle furnace at 333 K for 24 h. The third sample (PNS3) was prepared by mixing one part of pistachio nut shells and 2 parts of 18 N sulphuric acid (w/v) and kept in a muffle furnace at 353 K for 24 h. At the end of these periods, all the carbonized materials were washed several times with distilled water, until the filtrate reached neutral pH. Then the washed materials were dried at 110°C for 4 h in a hot air oven, grounded, and sieved.

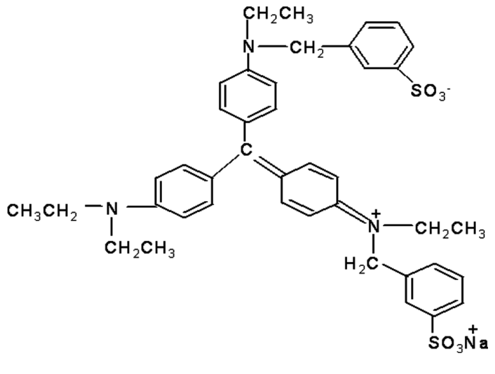

FIG. 1. Chemical structure of Acid Violet 17.

TABLE 1
The physical chemical characteristic of Acid Violet 17

Generic Name	C.I. Acid Violet 17
Color index number	42650
Abbreviation	AV 17
Commercial name	Acid Violet 17
Molecular weight	$C_{41}H_{44}N_3NaO_6S_2$
Purity	90%
Chromophore	Triphenyl methane
Molecular weight	761.93
λ_{max} (nm)	587 nm
Appearance	Dark blue powder
Chemical name (IUPAC)	Sodium 3-[[[4-[(4-diethyl aminophenyl)- [4-[ethyl- [(3-sulfonato phenyl) methyl] azaniumylidene]-1-cyclo hexa-2, 5-dienylidene] methyl] phenyl]-ethylamino] methyl] benzene sulfonate

Characterization of the Adsorbents

The surface area of all the activated carbon samples were measured by nitrogen adsorption at 77 K with an ASAP-2010 porosimeter (Micromeritics Corporation, Norcross, GA). The samples were degassed at 623 K at 10^{-5} mbar overnight prior to the adsorption experiments. The surface area of the materials were then measured. The surface area of activated carbon prepared at 303 K was found to be 25.34 m^2/g. With the increase in temperature from 303 to 333 and 353 K, there might be re-organization of bonds of the activated carbons. In other words, some of the existing bonds might be broken and new bonds may be formed. It should lead to conversion of the micropores into mesopores. As a result, the surface area of PNS2 and PNS3 increased to 32.15 m^2/g and 37.06 m^2/g respectively.

Scanning electron microscope (SEM) images were taken using Jole Jsm-6360 scanning electron microscope. The SEM images were taken to study the surface morphology of the prepared activated carbon samples. The SEM images of activated carbon prepared at 303, 333, and 353 K are shown in Fig. 2. Figure 2(a) appears smoother than others and the presence of H_2O in larger amount in carbon prepared at room temperature might be the cause for the smooth surface. By hydrogen bonding property of polar species, the partly polar grouping such as ester could be brought into some orientation that confers a smooth surface. It is supported by a highly irregular surface for the materials prepared at 333 and 353 K, Figs. 2(b) and (c).

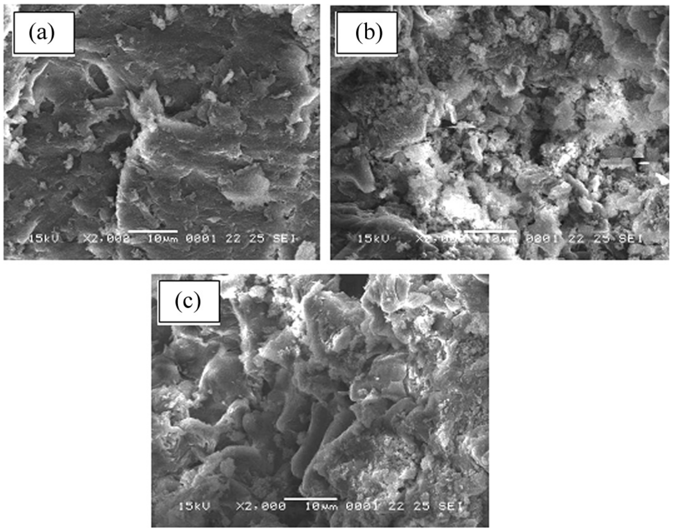

FIG. 2. SEM image of (a) PNS1 (b) PNS2 and (c) PNS3 adsorbents.

The FT-IR spectra of activated carbons prepared at (a) 303 K, (b) 333 K, and (c) 353 K are shown in Fig. 3. The spectra showed similar features for all the carbons. The broad band at 3500 cm^{-1} is assigned to (O-H) stretching of H_2O. Its intensity decreased with increase in drying temperature. The peak that appeared just below 3000 cm^{-1} is due to (C-H) vibrations of the alkyl group. The corresponding bending vibrations is seen at about 1357 and 1450 cm^{-1}. The presence of H_2O is once again confirmed by its bending vibrations at about 1630 cm^{-1}. The peak at about 1750 cm^{-1} is assigned to (C=O) stretch of an ester. The broad band at 1250 cm^{-1} confirms the presence of ester functional group. The corresponding alkoxy (C-O) vibration of ester gives a peak at 1000 cm^{-1}.

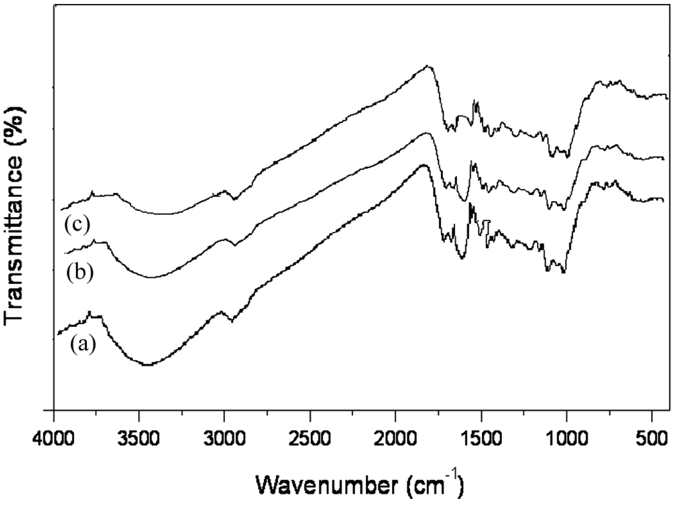

FIG. 3. FT-IR spectra of (a) PNS1 (b) PNS2 and (c) PNS3 adsorbents.

Batch Adsorption Experiments

To evaluate the efficiency of prepared activated carbons as an adsorbent, laboratory batch mode studies were conducted using a thermostatic orbital shaker (Orbitek, Scigenics Biotech, Chennai, India) at a constant speed of 200 rpm at room temperature. The adsorption isotherm experiment was carried out by agitating 50 mL dye solution of various concentrations in a conical flask. After agitating for 4 h (equilibration time), the dye solutions were separated from the adsorbent by centrifugation for 5 min (Research Centrifuge, Remi Scientific Works, Mumbai, India). Centrifugation prior to the analysis was done to avoid potential interference from suspended scattering particles in the UV-Visible analysis. The residual dye concentrations in the supernatant solutions were determined by monitoring the absorbance changes at the wave length of maximum absorbance (587 nm) using a UV-Visible spectrophotometer (Shimadzu model: UV 1601). The effect of pH on the adsorption of dye was studied over a pH range of 2–10. Dilute HCl and NaOH (0.1 M) solutions were used for the pH adjustment. The effect of adsorbent dosage on the adsorption of dye was studied by contacting 50 mL dye solution with different amounts of adsorbent dosage, till equilibrium was reached. Kinetics of adsorption was determined by analyzing the adsorptive uptake of dye color at different time intervals. The percentage of dye removal and amount of dye adsorbed on the activated carbons, q_e (mg/g) were calculated using the following relationships:

$$\text{Percentage removal } (\%) = \frac{C_o - C_e}{C_o} \times 100 \qquad (1)$$

$$\text{Amount adsorbed } (q_e) = \frac{(C_o - C_e)V}{m} \qquad (2)$$

where C_o and C_e are the initial and equilibrium concentrations (mg/L) of dye respectively, m is the mass of adsorbent (g) and V is the volume of dye solution (mL).

RESULTS AND DISCUSSION

Effect of pH

The pH of the system exerts profound influence on the adsorptive uptake of the adsorbate molecule presumably due to its influence on the surface properties of the adsorbent and ionization/dissociation of the adsorbate molecule. The effect of pH on the adsorption of dye solution by various pistachio nut shell activated carbons was studied by varying the pH of the dye solution from 2.0 to 10 for an initial concentration of 150 mg/L, and the results are shown in Fig. 4.

The pH of the dye solution itself was found to be 6.1 and the zero point charge (pH_{zpc}) of the adsorbents was determined to be 4.3, 4.0, and 3.8 for PNS1, PNS2, and PNS3 respectively. From the figure, it is evident that

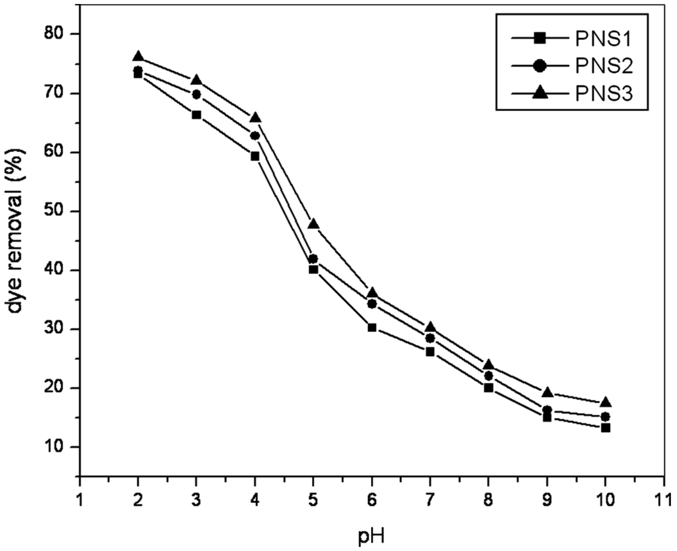

FIG. 4. Effect of pH on the adsorption of Acid Violet 17 onto PNS1, PNS2 and PNS3. Conditions: Co 150 mg/L, temperature 30°C.

maximum adsorption occurs at pH 2 i.e., $pH < pH_{zpc}$. Since the positive charge density on the PNS activated carbon surface increases, the adsorption of anionic dyes also increases. As the $pH > pH_{zpc}$, the negative charge density on the PNS activated carbon surface increases, and this does not favor the adsorption of anionic dyes due to the electrostatic repulsion. Furthermore, lower adsorption of the anionic dyes in alkaline medium is also due to the competition from excess OH^- ions with the anionic dye molecule for the adsorption sites.

Effect of Adsorbent Dosage

The effect of adsorbent dosage on the amount of dye adsorbed was studied by varying the dosage from 10 to 200 mg for the 50 mL of 100 mgL^{-1} dye solution. The percentage removal of AV 17 increased with increase in the adsorbent dosage. The maximum percentage removal of AV 17 dye solution was at 120 mg, after that saturation took place as shown in Fig. 5. Increase in adsorption with dose can be attributed to the increased surface area and availability of more adsorption sites (22).

Effect of Temperature

The degree of adsorption depends on the temperature of the solid-liquid interface. The effect of temperature was studied in the range 30–60°C. The optimized adsorbent dosage was 120 mg for the 50 mL dye solution of 100 mg/L. As shown in Fig. 6, the adsorption capacity of the PNS activated carbons increased with increase in temperature. It was found that higher temperature is to the advantage of adsorption. Thus, the process was concluded to be endothermic and spontaneous.

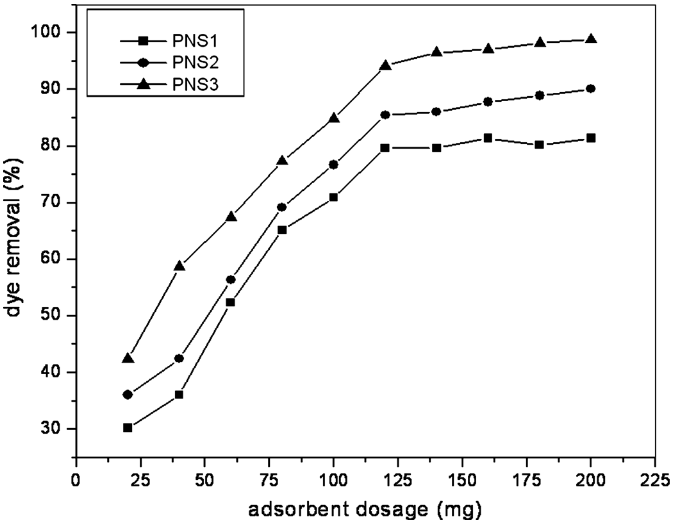

FIG. 5. Effect of adsorbent dosage on the adsorption of Acid Violet 17 onto PNS1, PNS2 and PNS3. Conditions: Co 100 mg/L, temperature 30°C.

Adsorption Isotherm Studies

Adsorption equilibrium data which express the relationship between mass of adsorbate adsorbed per unit weight of adsorbent and liquid-phase equilibrium concentration of adsorbate are represented by adsorption isotherms and provide important design data of adsorption system. The equilibrium data for the removal of AV 17 in the present investigation were analyzed using the Langmuir and Freundlich adsorption isotherm models (23,24). The Langmuir adsorption isotherm is based on monolayer, uniform, and finite adsorption site assumptions, therefore

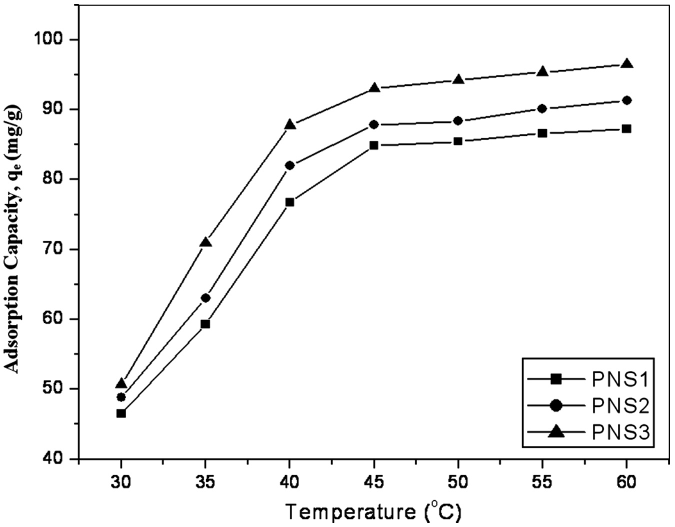

FIG. 6. Effect of temperature on the adsorption of Acid Violet 17 onto PNS1, PNS2 and PNS3. Conditions: Co 100 mg/L, adsorbent dosage 120 mg, volume 50 ml.

a saturation value is reached beyond which no further adsorption takes place (25). It also assumes that there is no interaction between molecules adsorbed on neighboring sites. The Langmuir equation which is valid for monolayer adsorption onto a surface with a finite number of identical sites is given by:

$$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \tag{3}$$

The linear form of the above equation is represented as

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \tag{4}$$

where C_e is the concentration of the dye solution at equilibrium (mg/L), q_e is the amount of dye adsorbed per unit mass of adsorbent (mg/g), and K_L is the constant related to the free energy of adsorption (L/mg). Q_m is the maximum adsorption capacity. The values of Q_m and K_L were calculated from the slope and intercept of the linear plot of C_e/q_e Vs C_e (Fig. 7).

The essential characteristic of the Langmuir isotherm can be expressed in terms of a dimensionless constant called equilibrium parameter (26):

$$R_L = \frac{1}{1 + K_L C_0} \tag{5}$$

where K_L is the Langmuir constant and C_0 is the highest initial dye concentration (mg/L). The value of R_L indicates the type of isotherm to be either favorable ($0 < R_L < 1$), linear ($R_L = 1$), unfavorable ($R_L > 1$), or irreversible $R_L = 0$.

The Freundlich isotherm is an empirical equation for multilayer, heterogeneous adsorption sites. The Freundlich equation is commonly given by:

$$q_e = K_F C_e^{\frac{1}{n}} \tag{6}$$

where q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g), C_e is the equilibrium concentration of solute in the bulk solution (mg/L), K_F is a Freundlich constant indicative of the relative adsorption capacity of the adsorbent (mg/g), and 1/n is the heterogeneity factor. A linear form of the Freundlich expression can be obtained by taking logarithms of nonlinear form:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{7}$$

A plot of log q_e verses log C_e (Fig. 8) enables the constant K_F and exponent 1/n to be determined from the intercept and slope of the line respectively.

The adsorption isotherm data of AV 17 onto various activated pistachio nut shell were fitted with both Langmuir and Freundlich isotherms (Fig. 7 and 8). The calculated Langmuir and Freundlich isotherm constants are presented in Table 2. The Langmuir model is more suitable than the Freundlich model to describe the adsorption isotherm as reflected with higher correlation coefficient R^2. Moreover, the dimensionless constant, R_L lies within the favorable limit indicating favorable adsorption for AV 17 on pistachio nut shell activated carbons.

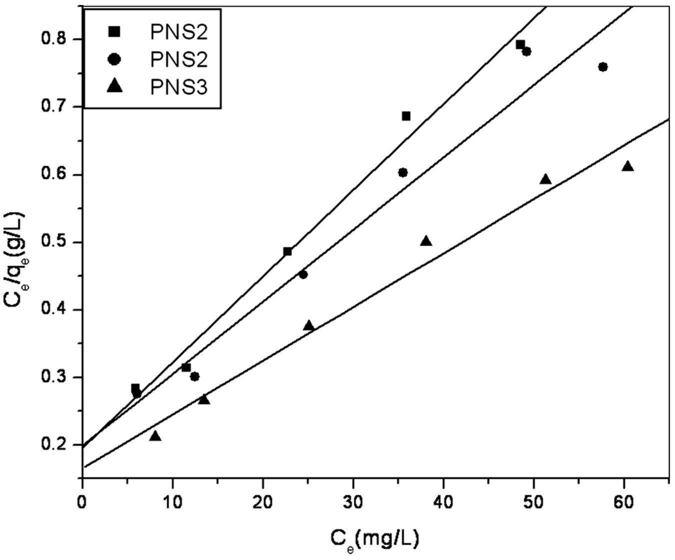

FIG. 7. Langmuir isotherm plots for adsorption of Acid Violet-17 onto PNS1, PNS2 and PNS3. Conditions: adsorbent dosage 120 mg, temperature 30°C.

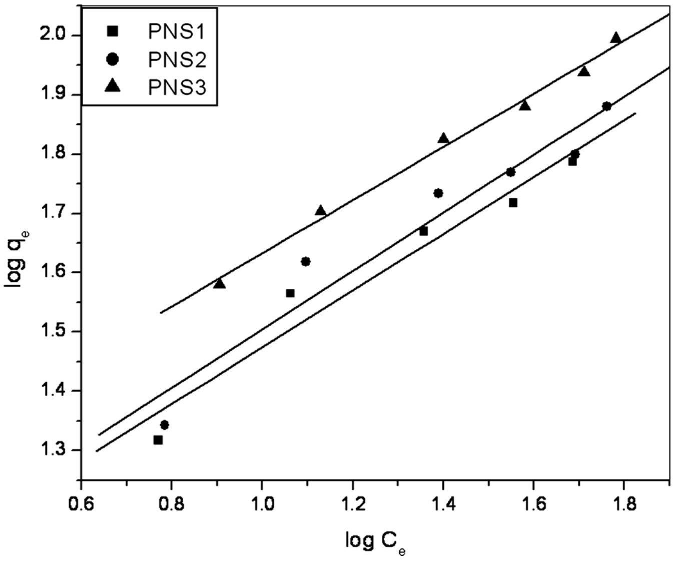

FIG. 8. Freundlich isotherm plots for adsorption of Acid Violet 17 onto PNS1, PNS2 and PNS3. Conditions: adsorbent dosage 120 mg, temperature 30°C.

TABLE 2
The Langmuir and Freundlich constants for the adsorption of Acid Violet 17 onto PNS1, PNS2, and PNS3

	Langmuir Constants				Freundlich Constants		
Adsorbent	Q_m (mg/g)	K_L (L/mg)	R_L	R^2	K_F(mg/g)	n	R^2
PNS1	78.74	0.0650	0.13	0.993	9.90	2.08	0.951
PNS2	93.45	0.0539	0.15	0.992	10.30	2.03	0.899
PNS3	125.00	0.0483	0.17	0.990	15.30	2.23	0.901

As it is evident from the IR studies, the activated carbon surface carries the ester functional group and thus the surface of the carbon will have a dipole-dipole interaction with the anionic dye. Therefore, all the three adsorbents have affinity towards the AV 17 dye. But, the adsorption capacity of AV 17 on PNS activated at 353 K was higher than that of the activated carbon prepared at 303 and 333 K. Because a larger surface area is generally a requirement for an optimum adsorbent, the SEM image of PNS3 (Fig. 2c) shows a carbon structure with high blurred appearance compared to others as discussed earlier. So, there is a good possibility for dyes to be trapped and adsorbed into these pores.

The low adsorption capacity of PNS1 and PNS2 may be because the pore size generated on activation at 303 and 333 K was smaller than the molecular size of AV 17; thus the resulting microporous structure making it inaccessible to the dye molecules on the surface (27) so that the adsorption capacity is reduced as evidenced by the low surface area 25.34 and 32.15 m^2/g.

The maximum adsorption capacity of AV 17, Q_m for the adsorbent used in this study along with that of other adsorbents are presented in Table 3. The adsorption capacity of pistachio nut shell activated carbon was much higher than that of other potential adsorbents such as coir pith, orange peel, bagasse charcoal, and cow dung charcoal. According to the results obtained, pistachio nut shell activated carbon could be employed as low-cost adsorbents and could be considered as an alternative to commercial activated carbons for the removal of color.

TABLE 3
Comparison of the adsorption capacities of various adsorbents for Acid Violet 17 dye

Adsorbents	Q_m (mg/g)	Reference
Red mud	1.37	(28)
Penicillium	4.32	(29)
Coir pith	8.06	(11)
Banana pith	13.10	(12)
Orange peel	19.88	(15)
Bagasse charcoal	38.32	(30)
Cow dung charcoal	50.30	(30)
Ground nut shell charcoal	100.57	(30)
Sunflower seed hull	116.27	(6)
Pistachio nut shell	125.00	present study

Kinetic Studies

A study of adsorption kinetics is desirable, as it provides information about the mechanism of adsorption, which is important for optimizing the efficiency of the adsorption process. In the present study, kinetic data were treated with the pseudo-first order and pseudo-second order kinetic models. The pseudo-first order equation of Lagergren is given by (31):

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \tag{8}$$

where q_e and q_t refer to the amount of dye adsorbed (mg/g) at equilibrium and at any time, t (min), respectively and K_1 is the equilibrium rate constant of pseudo-first order kinetics (min^{-1}). The values of the adsorption rate

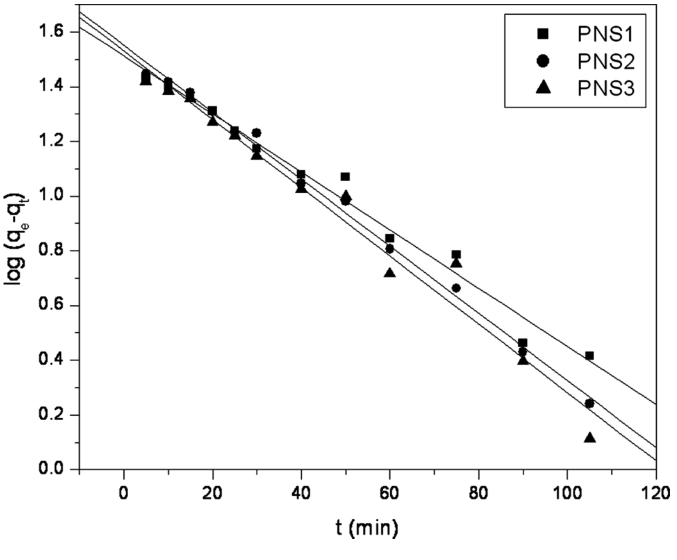

FIG. 9. Acid Violet 17 uptake by PNS1, PNS2 and PNS3 according to Pseudo-first order kinetic model. Conditions: Volume 500 mL, adsorbent dosage 120 mg.

TABLE 4
Kinetic parameters for the adsorption of Acid Violet 17 onto PNS1, PNS2, and PNS3

	Pseudo-first order			Pseudo-second order		
Adsorbents	q_e (mg/g)	K_1 (min^{-1})	R^2	q_e (mg/g)	K_2 ($\times 10^{-3}$ g/(mg·min))	R^2
PNS1	39.23	0.024	0.981	42.54	1.52	0.999
PNS2	51.97	0.053	0.972	56.89	1.99	0.990
PNS3	68.14	0.061	0.985	73.67	2.38	0.994

constant (K_1), the equilibrium adsorption capacity, q_e and the correlation coefficients were determined from the plots of log ($q_e - q_t$) against t (Fig. 9). These values are given in Table 4.

A linear form of the pseudo-second order model (32):

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \tag{9}$$

where K_2 is the equilibrium rate constant of pseudo-second order adsorption (g/mg·min). The second-order rate constant K_2 and the equilibrium adsorption capacity q_e, were calculated from the plot of t/q_t vs t (Fig. 10). The values of q_e, K_2 along with the correlation coefficients for the pseudo-first order and pseudo-second order models are presented in Table 4. For the pseudo-first order kinetics, the experimental data deviated greatly from linearity. This was evident by low q_e and low correlation values. The correlation coefficients were closer to unity for pseudo-second order kinetics than that for the pseudo-first order kinetics. This indicates that the adsorption of AV 17 on to PNS obeyed the pseudo-second order kinetic model.

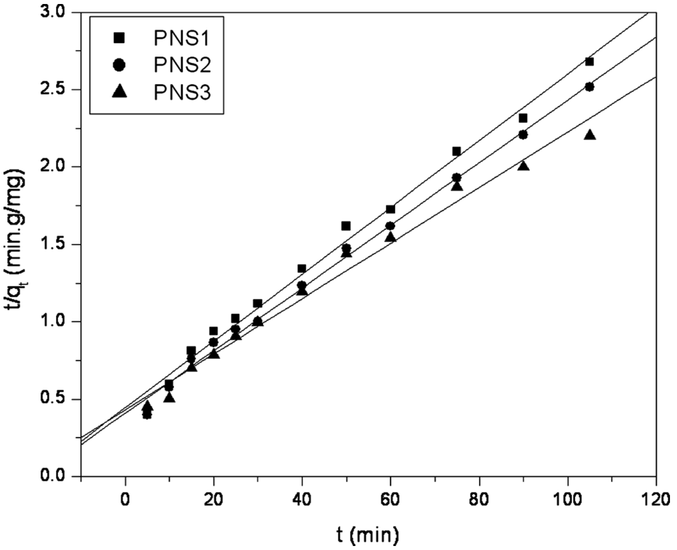

FIG. 10. Acid Violet 17 uptake by PNS1, PNS2 and PNS3 according to Pseudo-second order kinetic model. Conditions: Volume 500 mL, adsorbent dosage 120 mg.

Thermodynamic Studies

The thermodynamic parameters reflect the feasibility and spontaneous nature of the process. The thermodynamic parameters such as change in standard free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) of adsorption were determined using the following equations:

$$K = q_e/C_e \tag{10}$$

$$\Delta G^\circ = -RT \ln K \tag{11}$$

$$\log K = \frac{\Delta S^\circ}{2.303R} - \frac{\Delta H^\circ}{2.303RT} \tag{12}$$

where R is the gas constant, K is the equilibrium constant, and T is the solution temperature in Kelvin. ΔH° and ΔS° are calculated from the slope and intercept of the Van't Hoff plots of log K Vs 1/T (Fig. 11) and the results are given in Table 5. The positive values of ΔH° show the endothermic nature of adsorption and also indicate the possibility of physical adsorption (33).

The negative values of ΔG° (Table 5) showed that the adsorption is highly favorable for AV 17; it indicates that

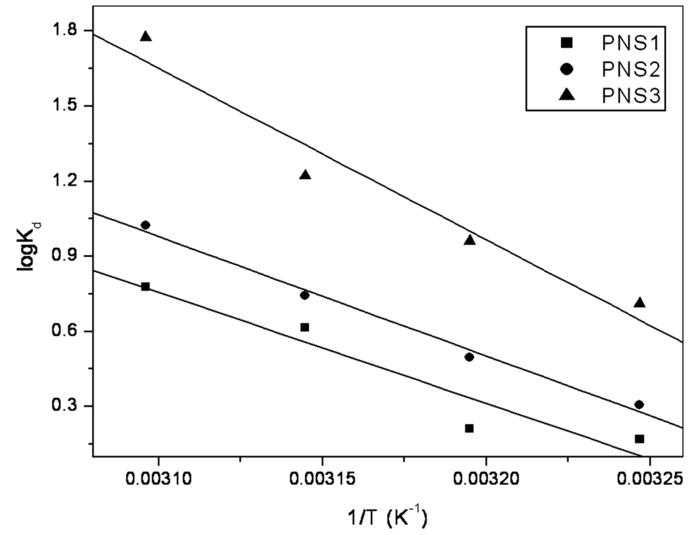

FIG. 11. The plot of log K_d Vs 1/T for PNS1, PNS2 and PNS3.

TABLE 5
Thermodynamic parameters calculated for the adsorption of Acid Violet 17 onto PNS1, PNS2, and PNS3

Adsorbent	ΔH° (kJ/mol)	ΔS° (kJ/mol)	ΔG° (kJ/mol)
PNS1	84.89	0.277	−1.26
PNS2	91.28	0.301	−2.96
PNS3	131.07	0.437	−5.76

the dye adsorption is spontaneous. The positive values of ΔS° (Table 5) show the increased disorder and randomness at the solid-solution interface during the adsorption of dye on the adsorbent (PNS). The adsorbed water molecules, which are displaced by the adsorbate species, gain more translational entropy than is lost by the adsorbate molecules, thus allowing the prevalence of randomness in the system. Enhancement of adsorption capacity of PNS activated carbons at higher temperature may be attributed to the enlargement of pore size and/or activation of the adsorbent surface (34).

CONCLUSION

In this work, three different activated carbons were prepared from pistachio nut shell and used successfully as an adsorbing agent for the removal of AV 17 dye from aqueous solutions. Adsorption was influenced by initial pH and adsorbent dosage. The maximum adsorption of AV 17 dye by PNS3 occurred at an initial pH of 2. The Langmuir and Freundlich adsorption isotherm models were used for the description of the adsorption equilibrium. The data were in good agreement with the Langmuir isotherm, thus confirming the monolayer adsorption capacity. It was shown that the equilibrium adsorption data of AV 17 onto pistachio nut shell was best fitted by the pseudo-second order kinetic model. According to the results obtained, pistachio nut shell (PNS) activated carbon is expected to be economically viable and has the potential to compete with the commercial one. Thus, pistachio nut shell could be employed as a low-cost adsorbent alternative to commercial activated carbons for the removal of various anionic dyes as well as for the removal of industrial effluents containing the AV 17 dye.

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