An Efficient Activated Carbon for the Wastewater Treatment, Prepared from Peanut Shell

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ABSTRACT
Peanut shell was converted into activated carbon and its surface was then modified through impregnation with KOH. The activated carbon was characterized by BET surface area with pore size analyzer, EDX, Tap and apparent density, pH, PZC and titratable surface functional groups. The activated carbon was used for the removal of pyridine from artificially contaminated aqueous solutions. Adsorption studies were carried out in a batch system by considering the effect of various parameters like contact time, initial concentration of adsorbate, pH of solution and temperature. Langmuir and Freundlich models were applied to experimental data. The results show that the Langmuir adsorption isotherm model gives better fit as compared to Freundlich model.

Keywords: Peanut Shell; Activated Carbon; Adsorption; Pyridine

1. Introduction
The increasing contamination of environment by waste materials as a result of human activities poses a continuously growing and serious problem. If these waste materials are used for preparing adsorbents, it will be used in overcoming the contamination of environment and waste materials disposal. Materials having high carbon and low inorganic contents such as wood, peanut shells, rice husk, wheat straw, corn cob, waste cellulosic material, etc., can be used as raw materials for the preparation of activated carbon [1-11]. The raw material can be converted into activated carbon by physical and/or chemical activation. In chemical activation, an activating agent such as KOH, ZnCl₂ and H₃PO₄, etc., is used. The raw material can be treated with activating agent before or after pyrolysis.

Pyridine is a colorless liquid with unpleasant odor and is toxic. It is either derived from coal tar or manufactured by chemical synthesis [12] and is widely used as a solvent and an intermediate in the production of piperidine, agricultural chemicals, drugs, dyestuffs and paints, rubber products, polycarbonate resins and textile water-repellents, and in laboratories [13]. It is a major component in the basic fraction of oil shale retort waters. Pharmaceuticals agents such as isoniazid, ceptylpyridinium bromide, analgesic dermal and cephalaxin are manufactured using pyridine as a catalyst. Therefore, increasing amounts of pyridine containing wastewater are let as effluent by various industries. Many of the pyridine compounds are hazardous and persist for long periods in the environment. The removal of pyridine from wastewater is therefore of great importance. The methods used for the removal of pyridine from water/wastewater include biodegradation [14-17], adsorption [18-20], adsorption and electro-sorption [12], ozonation [21], and ion exchange [18]. Among these adsorption systems, those using activated carbons prepared from agricultural waste material are rapidly gaining importance due to their economic feasibility [22-24].

The present work deals with the preparation of activated carbon from peanut shell with KOH activation, under oxidative atmosphere in air, and non-oxidative atmosphere (under nitrogen), at moderate temperature and compares the efficiency of the prepared samples for the removal of pyridine from aqueous solution. The influence of several operating parameters, such as pH, contact time, temperature and initial concentration of the adsorbent on the adsorption capacity was investigated.

2. Experimental
2.1. Raw Material, Chemicals and Impregnating Agent
Peanut shells were collected from the local market. All
other chemicals used were AR-grade chemicals and were used without further purification. The impregnating agent used for the chemical activation of peanut shells was KOH.

2.2. Procedure

Peanut shells were washed, dried, ground and mixed with 10 weight % KOH solution (6:100). The sample was then transferred to a series of 100 mL conical flasks and kept in ultrasonic cleaner for ten minutes at 30°C. The sample was then shaken on a shaker bath for six hours at 50°C. At the end of impregnation process, the sample was immediately filtered, washed with hot double distilled water for removal of its alkalinity and water soluble components, and dried.

2.3. Carbonization

The impregnated sample was heated at 170°C (±5°C) in a tube furnace in quartz reactor for one hour under nitrogen flow (flow rate 100 mL/minute). The temperature was then increased to 450°C (±5°C) in the same atmosphere for one hour and then lowered down to room temperature in nitrogen flow/breathing grade air. The cooled sample was washed in modified Soxhlet apparatus, dried and sieved by US standard sieve (100 Mesh).

2.4. Characterization of Sample

The pH of the prepared activated carbon was determined by standard reported method [24]. To measure PZC of the carbon samples, 0.1 M NaCl solution at pH 7 was used. 40 ml of 0.1 M NaCl solution were taken in 100 mL of titration flasks. The pH of the solutions was adjusted to a value between 3 and 11 by adding HCl 0.1 M or NaOH 0.1 M solutions. This pH was recorded as initial pH using pH meter model pH 300 with a combination of pH electrode of research grade. Then 0.2 g sample was added to each flask and shaken for 24 hours on a shaker bath at 25°C. Final pH was recorded and the difference was noted, PZC values were calculated from ΔpH vs pH plot. The tap density was calculated by tapping carbon samples in a calibrated pre-weighed 5 ml graduated cylinder until a minimum volume was recorded. The volume of the carbon was noted and the cylinder was weighed again to obtain the weight of the carbon for the determination of the tap density [25]. Carbon samples were taken in a pre-weighed clean and dried specific gravity bottle and weighed to determine the weight of carbon. Triply distilled water, n-octane, toluene and benzene were used as a liquid medium in the specific gravity bottle to determine the volume of the carbon for the determination of the apparent density [25,26]. Boehm’s titration method was used to determine the acidic and basic surface functional groups on the carbon samples [25]. 0.2 gm of carbon was added to 40 ml of 0.05 M NaOH, 0.05 M NaHCO₃, 0.05 M Na₂CO₃, 0.05 M C₂H₅ONa and 0.05 M HCl solutions in 100 ml conical flasks and shaken on a shaker at 25°C for 24 hrs. The solutions were then filtered using Whatmann filter paper No 44, and 10 ml of the filtrate was added to 15 ml of 0.05 M HCl solution, which was then back titrated against 0.05 M NaOH solution. The value of NaOH required to neutralize the solution was converted to titratable surface (acidic) functional groups (m·mol/g). The number of acidic sites of various types were calculated under the assumption that NaOH neutralizes carboxyl, phenolic and lactonic groups, Na₂CO₃ neutralizes carboxyl and lactones, NaHCO₃ neutralizes only carboxyl and C₂H₅ONa neutralizes only quinine groups [8]. The BET surface area and pore structure characteristics of the carbon samples were determined by Nitrogen adsorption at 77 K, using Quanta chrome NOVA 2200e, surface area and pore size analyzer. Prior to adsorption measurements, respective carbon samples were degassed at 150°C for a period of 4 hrs, in nitrogen atmosphere [26]. The S BET values of the samples were calculated by the Brunauer, Emmett, and Teller (BET) method using adsorption isotherms in the range 0.05 ≤ relative pressure (P/Po) ≤ 0.30. The total pore volume was estimated by BJH Method, from the amount of nitrogen adsorbed at P/Po 0.95. The micropores volume and average pore width was determined by Dubinin-Radushkevich (DR) equation from the value of nitrogen (77K) adsorption isotherm. The DA plot method was used for the determination of pore diameter. The volume of Mesopores was calculated by subtracting the volume of micropores from the total pore volume [27] at a relative pressure of 0.95. The elemental analysis of the prepared carbons was performed by EDX. For this purpose the carbon particles was mounted on the sample stubs and coated with gold using gold coating machine (JEOL-JSM-420, Japan).

2.5. Sorption Procedure

Stock solutions of the test reagents were made by dissolving pyridine in double distilled water. The pH of the test solutions was adjusted using either dilute HCl (0.1 M) or NaOH (0.1 M). The adsorption experiments were performed by the batch technique. Concentration of pyridine in the solution was recorded at the λ max = 256 nm, corresponding to pyridine by UV spectrophotometer model UV-1800 SHAMIDZO JAPAN. The effect of pH was observed by studying the adsorption of pyridine over a wide pH range of 2 to 12 on carbons (oxidized and unoxidized). For isotherm studies, a series of 100 mL conical flasks each filled with 40 mL of pyridine solution of varying concentrations (10 - 100 mg/L) was taken and adjusted to the desired pH and temperature. A known amount of adsorbent was added to each flask and agitated.
intermittently for the maximum contact time required to attain equilibrium. The amount of pyridine adsorbed was calculated using the relation (1).

\[ q_e = \frac{(C_0 - C_e)V}{W \times 1000} \]  

(1)

Where \( C_0 \) and \( C_e \) are the initial and equilibrium concentrations (mg/L) of pyridine in solution, \( V \) is the volume and \( W \) is the weight adsorbent in grams. Same procedure was adopted for other temperatures.

For kinetic studies known amounts of sample were taken in a number of flasks (100 mL capacities), each containing 40 mL of solution of pyridine, and were placed on a shaker. A known amount of activated carbon was added to each flask and adjusted to the desired pH and temperature. The solutions were then agitated. At pre-determined intervals of time, the solutions of specified flasks were separated from the activated carbon and analyzed for the uptake of pyridine at the corresponding \( \lambda_{\text{max}} \). The same procedure was adopted for different concentrations. The pyridine concentration retained in the adsorbent phase was calculated by using Equation (1).

3. Results and Discussion

3.1. pH, PZC, Tap Density and Apparent Density

Table 1 shows the pH values of different carbon samples. The pH value of oxidized carbon was greater than the un-oxidized carbon sample. This means that oxidized carbon was more basic than the un-oxidized carbon which may be due to the presence of more basic oxygen surface functional groups on the oxidized carbon.

Table 1 shows the PZC of carbons (oxidized and un-oxidized) respectively, determined by salt addition method. The PZC value of oxidized and un-oxidized carbon was determined to be 8.9 and 8.3, respectively, which shows that PZC value of oxidized carbon was higher than that of the un-oxidized carbon. Table 1 shows the tap and apparent densities of both the oxidized and un-oxidized activated carbons. The tap densities of KOH treated carbons (oxidized and un-oxidized) were smaller than their apparent densities. The volume measured by tapping of both un-oxidized and oxidized carbons includes the void/packing volume while the apparent volume did not include the void/packing volume. The tap density of un-oxidized carbon was greater than that of the oxidized carbon while the apparent densities of un-oxidized carbon were smaller than that of the oxidized carbon. The decrease in the tap density of oxidized carbon might be due to an increase in void packing volume, and an increase in the apparent density might be due to the enlargement of pores and/or sintering of particles.

3.2. Titratable Surface Functional Groups, BET Surface Area and EDS

The surface oxides on a carbon can have acidic as well as basic properties [28-31]. The acidity of a given functional group depends on its chemical environment, i.e. the size and shape of the polyaromatic layers, the presence and position of other constituents, and the charge of neighboring dissociated groups. Table 2 shows the content of functional groups on activated carbon samples as determined by Bohem’s titration. NaOH is a strong base in aqueous medium and has the ability to neutralize acids with strong or weak ionization potential. Therefore NaOH titration is a true acid-base reaction and measures the surface acidity attributed to carboxylic acids, phenols, lactones and other groups with acidic or ionizable hydrogen, present on the surface of activated carbons. It is evident from (Table 2) that the KOH treated un-oxidized carbon has higher NaOH, NaHCO\(_3\), Na\(_2\)CO\(_3\), C\(_2\)H\(_5\)ONa titratable surface groups than that of the oxidized carbon. It may be concluded that the air activation of the un-oxidized carbon decreases the number of acidic groups on the surface of the carbon.

Table 2 shows the HCl titratable surface functional groups for the KOH treated carbons (oxidized and un-oxidized). HCl is a strong acid in aqueous medium.
and has the ability to neutralize basic groups on the surface of activated carbon. It is clear from Table 2 that the KOH treated oxidized carbon has higher HCl titratable surface groups than that of the un-oxidized carbon which may be due to the air activation process. Table 3 summarizes the BET surface area $S_{DR}$, $S_{BJH}$, micro pore volume and average pore width of KOH treated carbons (oxidized and un-oxidized). The BET surface area and pore volume of the oxidized carbons were greater than that of the un-oxidized carbons while the average pore width and pore diameter of the oxidized activated carbons were smaller than that of the un-oxidized carbons. The $S_{BJH}$ and $S_{DR}$ were also larger for oxidized carbon than the un-oxidized carbon. It thus may be concluded that the air activation of un-oxidized carbon increases the BET surface area and pore volume by facilitating the evolution of volatile matter from the precursor.

Table 1 shows the EDS elemental analysis of different carbon samples. The major elements present in activated carbons samples were carbon and oxygen, and a small amount of potassium, which may be due to the process of activation with KOH. It is evident from Table 1, that the percentage of carbon in the oxidized carbon was greater than that in the un-oxidized carbon while the percentage of oxygen in the un-oxidized carbon was greater than that in the oxidized carbon. The elemental analysis can be compared with oxygen surface functional groups present on the surface of activated carbons.

### 3.3. Effect of Various Parameters on Adsorption

Pyridine behaves as a base ($P_{Ka} = 5.23$) due to the lone pair of electron on nitrogen atom which is readily available for protonation. Pyridine removal was studied on KOH treated carbons as a function of pH at the initial concentration of 50 mg/L and 80 mg/L, respectively, and the results are shown in Figure 1. It is clear from Figure 1 that the percent adsorption of pyridine on oxidized (KOH treated) sample increases with an increase in pH from 2 to 4. After that there is a decrease in adsorption reaching a minimum at pH 7, and at pH > 7 it becomes nearly constant.

| Table 3. Surface area and pore volume of activated carbon. |
|-------------------------------------------------------------|
| **S.No** | Properties | Oxidized | Un-oxidized |
|----------|-------------|----------|-------------|
| 1        | BET Surface area m$^2$/g | 1126.37 | 1115.24 |
| 2        | DR Surface area m$^2$/g | 1132.62 | 1128.16 |
| 3        | DR Average pore width ($\AA$) | 156.48 | 403.48 |
| 4        | DR adsorption E kJ/mol | 1.66 | 0.64 |
| 5        | DR Micropore V cc/g | 0.04 | 0.00 |
| 6        | DR Pore dia $\AA$ | 19.2 | 9.60 |
| 7        | BJH Surface area m$^2$/g | 1121.61 | 1113.25 |
| 8        | BJH Pore volume cc/g | 0.81 | 0.73 |
| 9        | BJH Pore dia $\AA$ | 131.71 | 135.19 |
| 10       | Mesopore V cc/g | 0.31 | 0.28 |

The pH dependence of pyridine adsorption can largely be related to the type and ionic state of these functional groups and to the adsorbate chemistry in the solution. At lower pH [pH $\leq (pK_a = 5.2)$] the pyridine molecule is mostly converted to Py$H^+$, resulting in the low adsorption of protonated pyridine on the positively charged carbon surface, while at higher pH [pH $\leq (pK_a = 5.2)$] pyridine molecule is un-protonated, resulting in the higher uptake on the charged activated carbons due to dispersion interaction. [23]. These observations suggest that a significant amount of pyridine is adsorbed through van der Waals forces. It is also possible that small amount is adsorbed through the formation of protonated pyridine on the surface at lower pH. Thus it may be concluded that at higher pH electrostatic interactions also take place as pointed out by Radovic et al. [33].

The amount of pyridine adsorbed on KOH treated (oxidized and un-oxidized) carbons was studied as a function of shaking time at different initial concentrations of pyridine at 25°C. Other parameters such as dose of adsorbent and pH of solution were kept constant. The

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![Figure 1](image-url)  
**Figure 1.** Effect of pH$_i$ on % removal of pyridine from aqueous solution at 25°C (KOH treated carbon).
effects of contact time for the four different concentrations of pyridine (30, 40, 50, 60 mg/L) for KOH treated carbon were studied. For KOH treated carbon, the adsorption equilibrium was established with in 24 hours in case of oxidized carbon, and at 30 hrs in case of un-oxidized carbon. The removal of pyridine was found to be dependent on the initial concentration. The amount adsorbed increased with an increase in initial concentration. Furthermore, the adsorption is rapid in the early stages and then gradually decreases and finally becomes almost constant after the equilibrium point. At low concentrations the ratio of the available surface to the initial pyridine concentration is larger, so the removal is independent of initial concentrations. However, in the case of higher concentrations this ratio is low; the percent removal thus depends upon the initial concentration. The curves also indicate that the adsorption leads to saturation, suggesting the possible monolayer coverage of pyridine on the surface of adsorbent [34].

3.4. Kinetics of Adsorption

Lagergren equation was applied to the experimental data (Figure 2) and first order kinetics was proposed for pyridine adsorption process [35-37].

\[
\log (q_e - q_t) = -kt/2.303 + \log q_e \tag{2}
\]

Where \( q_e \) and \( q_t \) are the amounts of pyridine adsorbed (mg/g) at equilibrium and at time \( t \), respectively, and \( k \) is the rate constant of first order adsorption (h\(^{-1}\)) given in Table 4.

### Effect of Temperature

The adsorption of Pyridine on KOH treated carbons (oxidized and un-oxidized) was studied at different temperatures, and the results are shown by Figures 3 and 4 respectively. It is clear from these figures that the amount of pyridine adsorbed increases with a rise in temperature, which means that pyridine adsorption from aqueous solutions on both oxidized and un-oxidized carbons is an endothermic process. The enhancement of adsorption capacity may be due to a chemical interaction between adsorbate and adsorbent, creation of some new adsorption sites, or an increased rate of intra particle diffusion into the pores of the adsorbate, at higher temperature [38,39].

To examine the relationship between sorbed \( (q_e) \) and aqueous concentration \( (C_e) \) at equilibrium, sorption isotherm was studied (Figure 3). The results are presented in Table 4.

| Table 4. Reaction rate constant (kh\(^{-1}\)) values for pyridine adsorption on KOH treated carbons (oxidized and un-oxidized). |
|----------------|------------------|------------------|------------------|------------------|
|                | Oxidized         |                | Un-oxidized      |                |
| \( C_e \), mg/L| \( k \) hrs\(^{-1}\) | \( R^2 \)      | \( k \) hrs\(^{-1}\) | \( R^2 \)      |
| 20              | 0.131            | 0.989         | 0.137            | 0.984          |
| 30              | 0.140            | 0.992         | 0.144            | 0.985          |
| 50              | 0.148            | 0.978         | 0.153            | 0.971          |
| 60              | 0.155            | 0.933         | 0.153            | 0.957          |

Figure 2. Lagergren plot for pyridine adsorption on un-oxidized/oxidized carbon (KOH treated) at 25°C and different concentration of pyridine.

Figure 3. Isotherm study of pyridine adsorption on oxidized carbon at different temperatures.
Isotherm models are widely employed for fitting the data, of which Langmuir and Freundlich equations are most widely used. The Langmuir model assumes that uptake of adsorbate molecules occurs on a homogenous surface by monolayer adsorption without any interaction between the adsorbed molecules. Freundlich model assumes heterogeneous adsorption due to the diversity of sorption sites or the diverse nature of adsorbate molecules adsorbed [34]. To get the equilibrium data, initial pyridine concentrations were varied while the adsorbent mass for each sample was kept constant. To ensure equilibrium conditions, the linear form of the Langmuir equation was used.

\[
\frac{C_e}{q_e} = \frac{1}{Q_0b} + \frac{C_e}{Q_0}
\]

(3)

Where \(q_e\) is the solid-phase adsorbate concentration at equilibrium (mg/g), \(C_e\) is the concentration of pyridine solution (mg/dm³) at equilibrium. The constant \(Q_0\) gives the theoretical monolayer adsorption capacity (mg/g). And \(b\) is related to the energy of adsorption (dm³/mg). Straight lines were obtained by plotting \(C_e/q_e\) against \(C_e\) as shown in Figures 5 and 6 for KOH treated carbons. The linear plot of \(C_e/q_e\) against \(C_e\) indicates the applicability of Langmuir adsorption isotherm. Consequently suggesting the formation of monolayer coverage of the adsorbate on the surface of the adsorbent, Langmuir constants \(Q_0\) and \(b\) were calculated from the slopes and intercepts of plots of \(C_e/q_e\) against \(C_e\) respectively and are given in Table 5.

It is observed from the Table 5 that the \(Q_0\) and \(b\) values are larger for higher temperatures. The essential characteristics of the Langmuir isotherm can be expressed by a dimensionless constant called equilibrium parameter \(RL\) [37]

\[
RL = \frac{1}{1 + bC_i}
\]

(4)

Where \(b\) is the Langmuir constant and \(C_i\) is the initial concentration (mg/g) and \(RL\) values indicate the type of isotherm. The values of \(RL\) at different temperatures and concentrations were found to be less than 1.0, and greater than 0 indicate the favorable adsorption of pyridine on KOH treated carbons (oxidized and unoxidized). The linear form of Freundlich isotherm as expressed by Equation (5) was also applied to the adsorption data of pyridine.

\[
\ln q_e = \ln K_1 + \frac{1}{n}\ln C_e
\]

(5)

Where \(K_1\) (mg/g) and \(1/n\) (g/L) are Freundlich constants, indicating the adsorption capacity and adsorption intensity, respectively. Straight lines were obtained by plotting \(\ln q_e\) against \(\ln C_e\) for KOH treated carbons which shows that adsorption of pyridine fitted into Freundlich
Table 5. Various Langmuir and thermodynamic parameters from $C_e/q$ vs $C_e$ plot of pyridine adsorption on KOH treated carbon (un-oxidized/oxidized).

| Activated Carbon | $T$ (K) | $Q_0$ mg/g | $b$ L/mg | $R^2$ | $\Delta H^\circ \times 10^3$ (kJ·mol$^{-1}$) | $\Delta S^\circ \times 10^{-3}$ (kJ·mol$^{-1}$K$^{-1}$) | $\Delta G^\circ$ (kJ·mol$^{-1}$) |
|------------------|--------|-----------|--------|------|-----------------------------|-----------------|------|
| Un-oxidized      | 293    | 10.86     | 0.021  | 0.95 | -0.87                       |                  |      |
|                  | 303    | 11.76     | 0.024  | 0.98 | -0.90                       |                  |      |
|                  | 313    | 13.17     | 0.027  | 0.96 | -0.93                       |                  |      |
|                  | 323    | 14.73     | 0.031  | 0.98 | -0.96                       |                  |      |
|                  | 293    | 13.97     | 0.025  | 0.93 | -1.04                       |                  |      |
|                  | 303    | 16.34     | 0.025  | 0.95 | -1.08                       |                  |      |
|                  | 313    | 16.92     | 0.029  | 0.97 | -1.12                       |                  |      |
| Oxidized         | 323    | 18.76     | 0.036  | 0.91 | -1.15                       |                  |      |

isotherm reasonably. The thermodynamic parameters such as $\Delta H^\circ$ (enthalpy change) and $\Delta S^\circ$ (entropy change) were calculated from the slopes and intercepts of the plots of $\ln b$ versus $1/T$ as shown in (Figure 7) for KOH treated carbons (oxidized and un-oxidized) respectively by using the following relation.

$$\ln b = \frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \quad (6)$$

Where $\ln b$ was obtained from the Langmuir plots of Figures 5 and 6. The $\Delta G^\circ$ (free energy change) was calculated from the following relation.

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

The values of $\Delta H^\circ$, $\Delta S^\circ$, and $\Delta G^\circ$ are given in Table 5. The value of $\Delta H^\circ$ is positive in case of both oxidized and un-oxidized carbons, indicating endothermic process.

The value of $\Delta G^\circ$ is negative for pyridine adsorption on both oxidized and un-oxidized carbons, indicating spontaneous process of adsorption. The values of $\Delta S^\circ$ are positive in case of both oxidized and un-oxidized carbons which confirms the possibility of favorable pyridine adsorption [40,41].

4. Conclusion

New alternative adsorbents for pyridine removal have been explored by converting peanut shell into activated carbon with surface modification through KOH. The adsorption of pyridine was found to be highly dependent on the pH value of the system, with the best results being obtained at pH 5 for oxidized (KOH treated) sample, at pH 6 for un-oxidized carbon. At lower concentrations, removal is independent of initial concentrations. However, at higher concentrations, removal depends upon the initial concentration. The kinetic data were well fitted by a first order kinetic model. The Langmuir adsorption isotherm model gives better fit as compared to Freundlich model. Thermodynamic parameters suggest that pyridine adsorption on both oxidized and an un-oxidized carbon is endothermic, spontaneous and favorable.

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