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Adsorption of naphthalene onto a high-surface-area carbon from waste ion exchange resin

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Abstract
A high-surface-area carbon (KC-1) was prepared from waste polystyrene-based ion exchange resin by KOH activation and used for naphthalene adsorption. The carbon exhibited a good hydrophobic nature with developed porous structure, favoring the adsorption of organic compounds. The Brunauer-Emmett-Teller surface area and total pore volume of KC-1 were 3442.2 and 1.68 cm³/g, respectively, which can be compared with those of KOH-activated carbons prepared from other precursors. Batch experiments were carried out to investigate the adsorption of naphthalene onto KC-1. The equilibrium data were analyzed by the Langmuir, Freundlich, and Polanyi-Manes isotherms and agreed with the Polanyi-Manes Model. The adsorption of naphthalene depended greatly on the porosity of the carbon, and the dispersive interactions between naphthalene and carbon could be relatively weak. The pH variation in aqueous solution had little effect on the adsorption process. The equilibrium time for 0.04 g/L of carbon dose was around 5 hr. Different models were used to evaluate the kinetic data and the pseudo second-order model was suitable to describe the kinetic process of naphthalene adsorption onto KC-1. Regeneration of spent carbon could be carried out effectively by alcohol treatment. The results indicated that KC-1 was a promising adsorbent for the removal of polycyclic aromatic hydrocarbons from aqueous solutions.

Key words: waste ion exchange resin; activated carbon; high surface area; naphthalene; adsorption

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Introduction
Polycyclic aromatic hydrocarbons (PAHs), which occur in oil, coal and tar deposits, and are also formed during incomplete combustion of carbon-containing fuels, are some of the most widespread organic pollutants. PAHs in the environment are found primarily in soil, sediment and oily substances, and they can also enter the water through atmospheric deposition, urban runoff and sewage discharges (Wassenberg and Di Giulio, 2004). In recent years, PAHs have been found in a variety of waters and wastewaters (Douben, 2003). As a class of environmental pollutants, they are of concern because some have been identified as carcinogenic, mutagenic and teratogenic compounds. Furthermore, most PAHs are hydrophobic with high boiling and melting points and electrochemical stability, which means they can exist and accumulate in the environment for a long time (Chang et al., 2004).

Currently, various methods have been used for the removal of PAHs in water treatment such as biodegradation, adsorption and sonication (Xia et al., 2006; Liu et al., 2011; Sponza and Oztekin, 2010). Owing to its high performance and practicality, adsorption has proved to be one of the most attractive techniques to remove PAHs from aqueous solutions. Activated carbon is the most widely used adsorbent, with extensive surface area and high adsorption ability.

Ion exchange resins are insoluble polymers containing cationic or anionic groups which are able to be exchanged with other ions in solutions. They have been widely used in different industrial processes, such as water softening and purification, metal separation, catalysis, juice purification, sugar manufacturing, pharmacy and petrochemical industry (Apell and Boyer, 2010; Gomes et al., 2001; Liu et al., 2012; Achaerandio et al., 2002). After service, tons of resins are spent, and then discarded as waste, which becomes a potential concern to the environment. Thus, there is an urgent need for effective ways of utilization of such types of waste. Some polymeric resins have been successfully utilized as precursors for production of activated carbons (Przepiorksi et al., 2002; Cai et al., 2004). However, society’s growing concern about environmental pollution and energy shortage makes it necessary...
to prepare activated carbons with high surface areas and pore volumes, which are valuable in pollutant removal, gas storage and manufacture of electric capacitors. A number of low-cost materials, such as cork waste, olive stones, argan seed shells and unburnt coal, have been investigated for the preparation and application of high-surface-area carbons (Carvalho et al., 2003; Ubago-Pérez et al., 2006; Elmouwahidi et al., 2012; Wu et al., 2010). Cabal et al. (2009) studied the adsorption of naphthalene onto bean pod-derived carbons and found that carbon composition including mineral matter might play an important role in naphthalene retention. Yuan et al. (2010) prepared a series of porous carbons and used them for PAHs adsorption, the results of which showed that the equilibrium data fit the non-linear Freundlich equation well and the adsorption of PAHs seemed to be a two-stage process controlled by diffusive transport processes.

The main objectives of this study were to prepare an activated carbon from polystyrene-based ion exchange resin by chemical activation with potassium hydroxide and to evaluate the feasibility of using it for the removal of PAHs from aqueous solutions. Naphthalene, one of the most abundant PAHs in wastewater, was chosen as the target pollutant. In the study, the effects of temperature, pH and contact time on adsorption were analyzed. Some common isotherms (e.g., Langmuir, Freundlich and Polanyi-Manes isotherms) and kinetic models (e.g., pseudo first-order, pseudo second-order and intra-particle diffusion equations) were adopted to investigate the adsorption mechanisms. Furthermore, the regeneration of spent carbon was also studied.

1 Materials and methods

1.1 Materials

Polystyrene-based ion exchange resin containing sulfonic acid groups (D001) was supplied by Zhejiang Zhengguang Industrial Co., Ltd. (Zhejiang, China).

Naphthalene was purchased from Jiangsu Yonghua Fine Chemical Co., Ltd., China. A stock solution of 25 mg/L was prepared by dissolving an appropriate amount of naphthalene in tert-butyl alcohol (Chengdu Kelong Chemical Co., Ltd., China) and subsequently diluted by deionized water to the final concentration. The volume fraction of tert-butyl alcohol in the stock solution was approximately 0.004.

1.2 Preparation of activated carbon

The ion exchange resin was first extracted with ethanol for 10 hr to remove adsorbed organic components, treated with 5% HCl solution and then washed with deionized water until the eluate had neutral pH. Subsequently, the wet resin was dried under vacuum at 60°C for 12 hr.

Carbonization and activation were carried out in a horizontal cylindrical furnace under N2 flow (150 mL/min). The clean resin was placed into a combustion boat and heated from room temperature to 450°C at a rate of 5°C/min, where it stayed for 30 min. After carbonization, the sample was crushed, mixed with ground KOH at a ratio of 1:3 and activated at 800°C for 1 hr. After cooling, the product was washed, dried, ground and then sieved to 80–140 mesh. The resulting carbon was identified as KC-1.

1.3 Characterization

Chemical compositions of the samples were estimated by elemental analysis using a Vario EL II analyzer (Elementar, Germany).

Nitrogen adsorption-desorption isotherms were measured (ASAP 2010, Micromeritics Instrument Co., USA) at 77 K to evaluate the pore structures of D001 and KC-1. The specific surface area (S BET) was calculated using the BET equation. The total pore volume V t was estimated to be the liquid volume of nitrogen at a relative pressure of about 0.974. The micropore volume (V mic) and micropore area (S mic) were obtained by the t-plot method, and the pore size distribution was investigated using the BJH and HK models.

1.4 Equilibrium experiments

The effects of pH and temperature on adsorption process were investigated in static equilibrium experiments. Thus, an appropriate amount of KC-1 was weighed and added to a flask containing naphthalene solution at a given pH, stoppered and mechanically stirred for 10 hr at a constant temperature. In pH experiments, the initial pH of the solution was adjusted with 0.1 mol/L HCl or 0.1 mol/L NaOH. The effect of temperature was studied at 298, 308 and 318 K. A blank was checked for each experiment to verify that there was no naphthalene volatilization or adsorption on the wall. After equilibration, the residual concentration of the aqueous phase, C e (mg/L), was determined by measuring its absorbance with a UV-1800 spectrophotometer (Shimadzu, Japan) at a wavelength of 276 nm. The amount of naphthalene adsorbed at equilibrium, q e (mg/g), can be calculated according to Eq. (1):

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

where, C0 (mg/L) and Ce (mg/L) are the initial and equilibrium concentrations of naphthalene, respectively. V (L) is the volume of the solution and W (g) is the mass of carbon.

1.5 Kinetics studies

The effect of contact time on adsorption was studied in the kinetic experiments. For this, a certain amount of carbon was accurately weighed and placed in a conical flask containing 975 mL naphthalene solution with an initial concentration of 25 mg/L. Then the suspension was sealed and vigorously stirred at a speed of 110 r/min. At predetermined time intervals, a 1.5 mL sample of the solution was taken out and analyzed as above. A blank
was also used here for verification purposes. The amount of naphthalene adsorbed at time $t$ (min), $q_t$ (mg/g), was also evaluated from Eq. (1), where $C_t$ (mg/L) is the concentration of the solution at time $t$.

1.6 Regeneration of spent carbon

In the experiment, 65 mg of KC-1 was placed into 975 mL naphthalene solution (25 mg/L) and agitated at room temperature. After equilibration, the spent carbon was taken out and immersed in absolute ethanol for 90 min, at a dosage of 1.3 g/L. Then the solvent was totally removed and the carbon was dried under vacuum at 60°C for 12 hr. Subsequently, the adsorption experiment was carried out again and the cycle was repeated three times. The amount of naphthalene adsorbed at equilibrium, $q_e$ (mg/g), was calculated each time.

2 Results and discussion

2.1 Characterization of D001 and KC-1

Elemental analysis was mainly used to determine the total percent values of carbon, hydrogen, nitrogen and sulfur, the results of which are depicted in Table 1. It is obvious that KC-1 had a higher carbon level and lower oxygen and sulfur levels when compared with the precursor resin, which might be caused by the liberation of volatile compounds (CO, CO$_2$, H$_2$O, SO$_2$, etc.) during the pyrolysis process and activation. The low oxygen content indicates the good hydrophobic nature of the carbon, which can reduce solvent competition and improve the affinity between naphthalene molecules and the carbon surface.

Figure 1 shows the adsorption-desorption isotherms of N$_2$ at 77 K on KC-1. It can be observed that the adsorption isotherm belongs to type I according to IUPAC, associated with microporous solids. The initial part of the N$_2$ isotherm represents micropore filling and a low slope of the plateau is due to multilayer adsorption on the mesopore and macropore surface (Long et al., 2008). The isotherm also shows a minor H4-type hysteresis loop, characteristic of slit-shaped pores (Ania et al., 2007). Therefore, KC-1 was considered to be a microporous material with relatively small external area. Table 2 summarizes the analysis of the data obtained from N$_2$ adsorption-desorption isotherms. It can be seen that KC-1 had a very high surface area, 3442.2 m$^2$/g, which was primarily attributed to the presence of extensive micropores. The total pore volume was as high as 1.68 cm$^3$/g, which could also be compared with those of KOH-activated carbons prepared from other precursors, such as corncob, Spanish anthracite and fir wood (Tseng and Tseng, 2005; Lozano-Castelló et al., 2001; Wu et al., 2005). The microporous nature of carbon could provide extensive active sites and favor the adsorption of organic compounds.

2.2 Isotherm studies

The adsorption isotherms of naphthalene onto KC-1 are presented in Fig. 2. All of the isotherms show the same upward trend with increasing equilibrium concentration. The adsorption capacities at a concentration of 5 mg/L were all larger than 500 mg/g, showing good performance of KC-1.

Further analysis of the isotherm data was carried out by fitting them to different models, i.e., Langmuir, Freundlich and Polanyi-Manes isotherms. The Langmuir model assumes monolayer adsorption on the solid surface and the Freundlich model is empirical in nature (Benhamou et al., 2009). The Polanyi-Manes model is based on a pore-filling mechanism, which has been used to describe the

![Fig. 1 N$_2$ adsorption-desorption isotherms of KC-1 at 77 K.](image1)

![Fig. 2 Adsorption isotherms of naphthalene onto KC-1 at different temperatures.](image2)
adsorption process of PAHs by carbon nanomaterials. The equations of the models can be represented as follows (Yang et al., 2006; Anbia and Ghaffari, 2009):

Langmuir model
\[ q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \]  
(2)

Freundlich model
\[ q_e = K_f C_e^{1/n} \]  
(3)

Polanyi-Manes model
\[ \log q_e = \log Q_0 + a(\varepsilon_{sw}/V_s)^b \]  
(4)

\[ \varepsilon_{sw} = RT \ln(C_s/C_e) \]  
(5)

where, \( q_e \) (mg/g) is the amount of naphthalene adsorbed at equilibrium; \( C_e \) (mg/L) is the concentration of naphthalene solution at equilibrium; \( Q_0 \) (mg/g) is the maximum adsorption capacity; \( K_L (L/mg) \) and \( K_f ((mg/(mg/L))^{1/n}) \) are the Langmuir and Freundlich constants respectively; \( 1/n \) can be a measure of adsorption intensity; \( \varepsilon_{sw} \) (kJ/mol) is the effective adsorption potential; \( a \) and \( b \) are fitting parameters; \( R \) is the universal gas constant (8.314 J/mol K) and \( T \) (K) is the absolute temperature; \( V_s \) (mL/mol) and \( C_s \) (mg/L) are the molar volume and solubility of naphthalene, respectively (May et al., 1978).

The model fitting results are shown in Table 3. The mean weighted square error (MWSE), equal to 1/\( v \) (\( \Sigma (q_{measured} - q_{model})^2/q_{measured}^2 \)) and correlation coefficients (\( r^2 \)) were calculated to evaluate the goodness of fit; where \( v \) is the degree of freedom (\( v = N - 2 \) for Freundlich model and Langmuir model; \( v = N - 3 \) for Polanyi-Manes model), \( N \) is the number of experimental data points, \( q_{measured} \) is the measured equilibrium concentration, and \( q_{model} \) is the estimated value by the respective models. It is clear that the Polanyi-Manes model represents a good fit for all the isotherms with the highest \( r^2 \) and lowest MWSE values. This, combined with the fact that extensive micropores exist in KC-1, indicates that volume filling plays a very important role in the adsorption of naphthalene from aqueous solution.

A plot of adsorbed volume (\( Q_v \)) against adsorption potential (\( \varepsilon_{sw} \)), referred to as the “characteristic curve”, is usually employed to examine whether the Polanyi theory mechanismally captures the adsorption progress. Figure 3 depicts the characteristic curves of naphthalene adsorption onto KC-1 at different temperatures. All of them almost fall onto a single curve, indicating that it is mechanistically useful to apply the Polanyi theory to naphthalene adsorption.

### Table 2 Pore characteristics of KC-1

<table>
<thead>
<tr>
<th>( S_{BET} ) (m²/g)</th>
<th>( S_{mic} ) (m²/g)</th>
<th>( S_{mic} ) (%)</th>
<th>( V_1 ) (cm³/g)</th>
<th>( V_{mic} ) (cm³/g)</th>
<th>( V_{mic} ) (%)</th>
<th>( D_p ) (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3442.2</td>
<td>3289.8</td>
<td>95.6</td>
<td>1.68</td>
<td>1.50</td>
<td>89.3</td>
<td>1.95</td>
</tr>
</tbody>
</table>

\( D_p \): average pore diameter.

### Table 3 Isotherm constants and correlation coefficients for naphthalene adsorption

<table>
<thead>
<tr>
<th>Temperature (K)</th>
<th>( Q_0 )</th>
<th>( K_L )</th>
<th>( r^2 )</th>
<th>MWSE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Langmuir model</td>
<td>298</td>
<td>709.46</td>
<td>0.8677</td>
<td>0.9262</td>
</tr>
<tr>
<td></td>
<td>308</td>
<td>741.23</td>
<td>0.5558</td>
<td>0.9092</td>
</tr>
<tr>
<td></td>
<td>318</td>
<td>871.10</td>
<td>0.3035</td>
<td>0.9379</td>
</tr>
<tr>
<td>Freundlich model</td>
<td>298</td>
<td>307.25</td>
<td>0.4152</td>
<td>0.9904</td>
</tr>
<tr>
<td></td>
<td>308</td>
<td>263.51</td>
<td>0.4486</td>
<td>0.9891</td>
</tr>
<tr>
<td></td>
<td>318</td>
<td>217.71</td>
<td>0.5305</td>
<td>0.9857</td>
</tr>
<tr>
<td>Polanyi-Manes model</td>
<td>298</td>
<td>3.2568</td>
<td>-6.1860</td>
<td>0.7676</td>
</tr>
<tr>
<td></td>
<td>308</td>
<td>3.9150</td>
<td>-4.764</td>
<td>0.4255</td>
</tr>
<tr>
<td></td>
<td>318</td>
<td>5.0656</td>
<td>-5.4781</td>
<td>0.2806</td>
</tr>
</tbody>
</table>

The pH value of the solution is an important controlling factor in the adsorption progress, which can influence the solubility of the adsorbate and the electrostatic and dispersive interactions between the adsorbent and adsorbate. Naphthalene molecules are non-polar, and can interact with the carbon surface due to their polyaromatic structure, involving dispersive interactions with the \( \Pi \)-electron-rich regions located in the graphene layers of carbon (Ania et al., 2007). Therefore, pH variation may affect the adsorption of naphthalene by changing the dispersive forces. To

### 2.3 Interactions between naphthalene and the carbon surface

The pH value of the solution is an important controlling factor in the adsorption progress, which can influence the solubility of the adsorbate and the electrostatic and dispersive interactions between the adsorbent and adsorbate. Naphthalene molecules are non-polar, and can interact with the carbon surface due to their polyaromatic structure, involving dispersive interactions with the \( \Pi \)-electron-rich regions located in the graphene layers of carbon (Ania et al., 2007). Therefore, pH variation may affect the adsorption of naphthalene by changing the dispersive forces. To

![Figure 3 Characteristic curves of naphthalene adsorption on KC-1.](https://example.com/figure3.png)

\[ Q_v (\times 10^{-3} \text{cm}^3/\text{g}) \]

\[ \varepsilon_{sw} \text{(kJ/mol)} \]
verify this, batch experiments were carried out by varying the initial pH of the solution at an initial concentration of 25 mg/L with 0.010 g/125 mL carbon dose. The results showed that the adsorption efficiency of naphthalene was larger than 95%, and was not affected by pH except for minor variations over the range of 2.5–11.5. Therefore, the dispersive interactions between naphthalene and KC-1 could be considered relatively weak and the adsorption of naphthalene depends greatly on the porosity of the carbon.

2.4 Kinetic studies

Figure 4 shows the effect of contact time on the adsorption of naphthalene by KC-1 at two carbon doses (0.04 and 0.08 g/L). It can be observed that the removal of naphthalene was rapid initially, then slowed and eventually reached equilibrium. The equilibrium time for 0.04 g/L of carbon dose was around 5 hr.

Then three models, i.e., pseudo first-order, pseudo second-order and intra-particle diffusion models, were used to investigate the kinetic mechanism of naphthalene adsorption onto carbon.

The pseudo first-order and pseudo second-order equations can be given as follows (Zhang et al., 2008):

**Pseudo first-order model**

\[
\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t
\]

**Pseudo second-order model**

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}
\]

where, \(k_1\) (min\(^{-1}\)) and \(k_2\) (g/(mg·min)) are the rate constants of pseudo first-order and pseudo second-order adsorptions, respectively. The parameters of the two models are listed in Table 4. The correlation coefficients \(r^2\) from the pseudo second-order model were both larger than 0.998 and the theoretical \(q_{cal}\) values were closer to the experimental data, \(q_{exp}\), indicating that the pseudo second-order model is suitable to describe the kinetic process of naphthalene adsorption onto KC-1.

Based on the pseudo second-order model, the initial adsorption rate \((h_0, \text{mg/(g·min)})\) and the half adsorption time \((1/2, \text{min})\) were calculated according to the equations:

\[
h_0 = k_2 q_e^2
\]

\[
\tau_{1/2} = 1/k_2 q_e
\]

As shown in Table 4, the initial adsorption rate at 0.08 g/L was 31.95 mg/(g·min), which is 10 times larger than that of the commercial granular activated carbon; coupled with such a short half adsorption time (9.48 min), this indicates a high affinity between the solute molecules and the substrate in this study (Cabal et al., 2009).

The intra particle diffusion is often the rate limiting step in many adsorption processes, the possibility of which can be explored by using the following equation (Kanna and Sundaram, 2001):

\[
q_t = k_p t^{1/2} + C
\]

where, \(k_p\) (mg/(g·min\(^{1/2}\))) is the rate constant of the intra-particle diffusion process and \(C\) is the intercept. Figure 5 shows the plots of \(q_t\) vs. \(t^{1/2}\) at two carbon dosages, the multilinearity of which indicates that the adsorption progress consisted of several steps. The value of the correlation coefficient \(r^2\) at the dosage of 0.08 g/L revealed the presence of intraparticle diffusion (Table 4). The intercept, \(C\), gives an idea about the boundary layer thickness, the high values of which in the study show the great effect of the boundary layer on the adsorption kinetics.

2.5 Regeneration

The results of regeneration studies are shown in Fig. 6. It can be seen that KC-1 still had a high adsorption capacity after three cycles, indicating the effectiveness of the alcohol treatment for regeneration of the spent carbon. Furthermore, naphthalene in the extract solution can be separated and purified by distillation, which has been widely used in the petrochemical industry. Therefore,
Table 4  Kinetic parameters for the adsorption of naphthalene onto KC-1

<table>
<thead>
<tr>
<th>Carbon dosage (g/L)</th>
<th>q_{e,exp}</th>
<th>Pseudo first-order</th>
<th>Pseudo second-order</th>
<th>Intraparticle diffusion</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>q_{e,cal}</td>
<td>k_1</td>
<td>r_1^2</td>
<td>q_{e,cal}</td>
</tr>
<tr>
<td>0.04</td>
<td>576.17</td>
<td>262.00</td>
<td>0.01359</td>
<td>0.9279</td>
</tr>
<tr>
<td>0.08</td>
<td>297.80</td>
<td>166.84</td>
<td>0.02280</td>
<td>0.9514</td>
</tr>
</tbody>
</table>

Fig. 6  Adsorption capacities of naphthalene onto KC-1 for three cycles.

alcohol treatment is a good choice for the regeneration of naphthalene-exhausted KC-1.

3 Conclusions

Waste ion exchange resin can be used as a precursor for the preparation of high-surface-area carbon. Carbonization and activation reduced oxygen and sulfur levels and developed high microporosity in the carbon. Batch experiments were carried out to investigate the adsorption of naphthalene onto the carbon. The pH variation in aqueous solution had little effect on the adsorption efficiency. Equilibrium data agreed with the Polanyi-Manes Model and the adsorption of naphthalene depended greatly on the porosity of the carbon. The adsorption kinetics was found to follow the pseudo second-order model. Naphthalene-exhausted carbon could be effectively regenerated through alcohol treatment.

Acknowledgments

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References


Aims and scope

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