Chemical Engineering Journal 155 (2009) 722–727

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Adsorption of *p*-nitroaniline by phenolic hydroxyl groups modified hyper-cross-linked polymeric adsorbent and XAD-4: A comparative study

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ARTICLE INFO

Article history:
Received 3 March 2009
Received in revised form 3 September 2009
Accepted 7 September 2009

Keywords:
Polymeric adsorbent
Adsorption
p-Nitroaniline
XAD-4

ABSTRACT

Phenolic hydroxyl groups modified hyper-cross-linked polymeric adsorbent HJ-02 was prepared and it was applied to remove *p*-nitroaniline in aqueous solution as compared with Amberlite XAD-4. The results indicated that the adsorption at the solution pH of 3.7–7.8 was efficient for HJ-02 resin while that at the solution pH higher than 4.1 was favorable for XAD-4. The ionic strength affected the adsorption slightly and NaCl at a low concentration (<10%) had a positive effect. The adsorption kinetic curves obeyed the pseudo-second-order rate equation and HJ-02 resin had lower rate constant than XAD-4. The adsorption isotherms could be fitted by Langmuir isotherm and the adsorption capacity onto HJ-02 resin was much larger than that onto XAD-4. The adsorption enthalpy, adsorption free energy, and adsorption entropy onto HJ-02 resin were a little more negative than those onto XAD-4. Analysis of the adsorption mechanism suggested that the phenolic hydroxyl groups uploaded on HJ-02 resin and the predominant mesopores (2–5 nm) were the main reasons for its much larger adsorption capacity.

1. Introduction

Water pollution by aromatic compounds has attracted much attention in recent years, more and more stringent limits for these compounds with acceptable environmental levels are established gradually in many countries. In some developing countries like China, how to deal with large volume of wastewater containing toxic aromatic compounds is a pressing environmental problem. Aniline compounds such as aniline and *p*-nitroaniline are important intermediates to produce dyes, fuel additives, antioxidants, and pesticides [1,2]. Nevertheless, existence of these compounds in water, even at a low concentration, is extremely harmful to creatures. Consequently, their efficient removal or destruction is a big scientific problem.

To remove these aromatic compounds, many disposal processes are developed and adsorption is proven to be an effective method [3–6]. In contrast with the typical inorganic activated carbons, polymeric adsorbents are more attractive alternatives due to their diverse structure and easy regeneration [7–10]. The Amberlite XAD-4 resin is one of the best polymeric adsorbents for removing phenolic compounds from wastewater, especially for phenol [11]. However, its adsorption capacity is much small for hydrophilic aromatic compounds due to its hydrophobic polystyrene skeleton. Recently, chemical modification of polymeric adsorbents is adopted frequently by introducing some special functional groups onto the skeleton of the adsorbent, and then the chemical composition of the adsorbent surface is modified and hence the adsorption is improved for some special compounds [12–14]. There are report a large number of reported references which focus on the adsorption properties of polymeric adsorbents modified with hydrogen bonding acceptors such as amino-groups [15], formaldehyde groups [16], and amide groups [17], and it is seen that their adsorption is enhanced for phenolic compounds. In the same way, if hydrogen bonding donators like phenolic hydroxyl groups can be introduced onto the skeleton of the adsorbent, the adsorption behavior should also be improved for aromatic amine.

This paper aims at preparation of phenolic hydroxyl groups modified hyper-cross-linked polymeric adsorbent HJ-02 by Friedel-Crafts post-cross-linked reaction and esterified reaction from macroporous low cross-linked poly(styrene-co-divinylbenzene) (PS), and its adsorption behavior for *p*-nitroaniline was tested in aqueous solution as compared with XAD-4. Their adsorption kinetics and thermodynamics were analysed and the adsorption mechanism was expounded.

2. Materials and methods

2.1. Materials

Macroporous low cross-linked chloromethylated PS was provided by Langfang Chemical Co. Ltd. (China), its cross-linking degree

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doi:10.1016/j.cej.2009.09.012

was 6% and chlorine content was 17.3%. The Amberlite XAD-4 was purchased from Rohm & Haas Company (USA). *p*-Nitroaniline, N,N-dimethylformamide (DMF), salicylic acid, sodium bicarbonate, and ethanol were analytical reagents and used without purification.

2.2. *Synthesis of HJ-02 resin*

HJ-02 resin was prepared by Friedel-Crafts alkylation reaction of chloromethylated PS and esterified reaction of the post-cross-linked resin [18]. The Friedel-Crafts alkylation reaction of chloromethylated PS was performed according to the Ref. [16] and thereafter the post-cross-linked resin was obtained. In the esterified procedure, 40 g of the post-cross-linked resin was swollen by 80 ml of DMF at 298 K for 12 h, and 20 g of salicylic acid was added gradually. After that, 13 g of sodium bicarbonate was added and an increase of the temperature from 298 K to 368 K was followed. The reaction mixture was filtered after 10 h at 368 K and the solid particles were rinsed by 1% of hydrochloric acid (w/w) and deionized water, and hence the HJ-02 resin was achieved.

2.3. *Characterization of the adsorbents*

The specific surface area was determined via the N_2 adsorption–desorption isotherms with the temperature at 77 K using a Tristar 3000 surface area measurement instrument. The samples were outgassed at 373 K and equilibrated for about 20 h before measuring the N_2 adsorption–desorption isotherms. The infrared (IR) spectra were collected by KBr disks on a Nicolet 510P Fourier transformed infrared instrument.

2.4. *Bath adsorption*

The equilibrium adsorption was performed at three temperatures: 288, 298, and 308 K, respectively. 0.050 g of dry adsorbent and 50 ml of *p*-nitroaniline solution with known concentration C0 (mg/l) were added into a cone-shaped flask. Hydrochloric acid and sodium hydroxide were applied to adjust the solution pH, and NaCl solids were added directly to adjust the salinity. The flasks were then shaken in a thermostatic oscillator at a preset temperature for about 24 h so that the equilibrium was reached, and the equilibrium adsorption capacity q_e (mg/g) was calculated as:

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

where C_e (mg/l) was the equilibrium concentration of *p*-nitroaniline, *V* was the volume of aqueous solution (l), and *W* was the weight of the dry adsorbent (g). The adsorption kinetic curves were carried by analyzing the *p*-nitroaniline uptakes until the adsorption equilibrium was reached.

3. Results and discussion

3.1. *Characterization of HJ-02 resin*

The specific surface area and pore volume of HJ-02 resin are measured to be 540.0 m^2/g and 0.364 cm^3/g, respectively, much lower than the corresponding ones of XAD-4 (873.1 m^2/g and 1.21 cm^3/g). The N_2 adsorption–desorption isotherms of HJ-02 resin and XAD-4 are described in Fig. 1, it seems the adsorption isotherm of HJ-02 resin is close to type-II isotherm of IUPAC classification while that of XAD-4 is rather of type-IV. For the two resins, at a relative pressure below 0.05, the N_2 uptakes increase rapidly with the increment of the relative pressure, implying that micropores even super-micropores are existent. In addition, the visible hysteresis loops of the two resins indicate that there exist mesopores. These analyses agree with the results of the pore distribution shown in Fig. 2. In particular, the pore distribution of the two resins is distinguishing although the mesopores dominate their pore structure. The mesopores in the range of 2–5 nm are predominant for HJ-02 resin, while the mesopores of 2–18 nm play an important role for XAD-4. Moreover, the pore distribution using density functional theory (DFT) method indicates that HJ-02 resin contains some macropores, and the error of the cylindrical pore model is relatively high (about 30%), which suggests that the pores of HJ-02 resin have complex shape including narrow microporous throats, super-micropores, and mesopores.

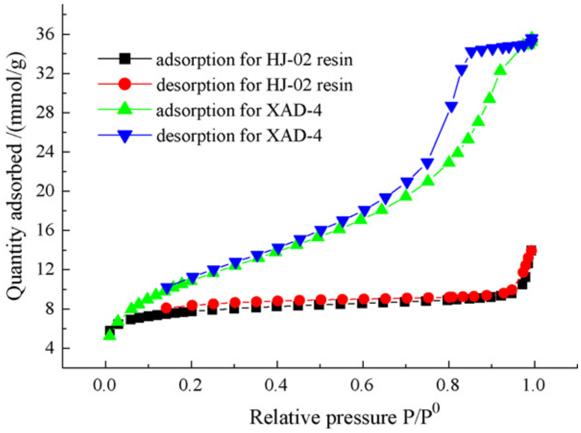

Fig. 1. The N_2 adsorption–desorption isotherms onto HJ-02 and XAD-4 resin.

As displayed in Fig. 3, the IR spectrum of XAD-4 characterizes its polystyrene-type structure based on the representative vibrations at 1600.7 cm^{-1}, 1510.1 cm^{-1}, 1446.4 cm^{-1} (C=C stretching of the benzene ring) and 796.5 cm^{-1} (=C–H bending of 1,4-disubstituted benzene ring) [19]. As for HJ-02 resin, after the Friedel-Crafts post-cross-linked reaction and esterified reaction, two strong characteristic bands related to the CH_2Cl groups at 1265.1 and 669.2 cm^{-1} are weakened greatly, while two other strong vibrations with frequencies at 3378.8 cm^{-1} and 1110.9 cm^{-1} are presented, the band at 3378.8 cm^{-1} is concerned with the O–H stretching of the OH groups and that at 1110.9 cm^{-1} is the C–O stretching between the benzene ring and the OH groups. In addition, the C–O–C stretching of the ester carbonyl groups at 1311.4 cm^{-1} and the C–H out of bending of 1, 2-disubstituted benzene at 754.0 cm^{-1} also confirm the esterified reaction [19].

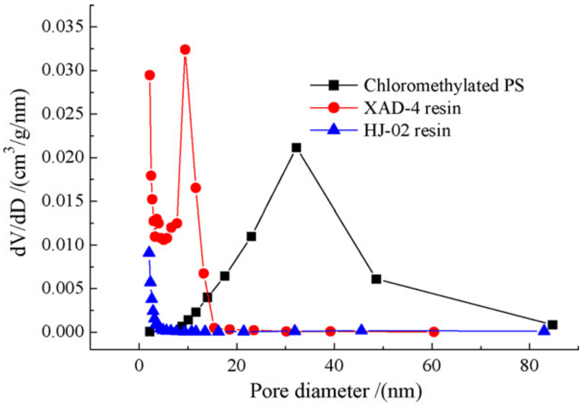

Fig. 2. The pore distribution of the chloromethylated PS, HJ-02 and XAD-4 resin.

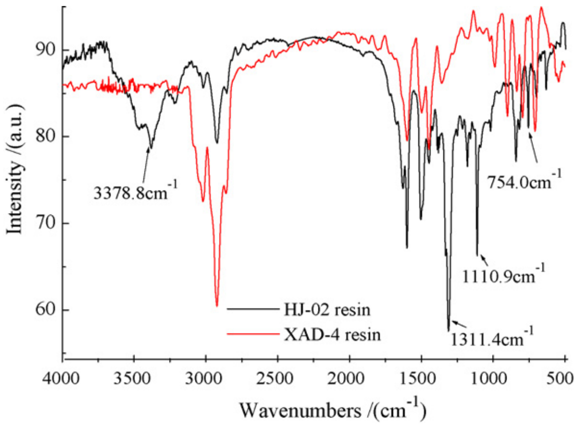

Fig. 3. The IR spectra of HJ-02 and XAD-4 resin.

3.2. Effect of the solution pH on the adsorption

The effect of the solution pH on the adsorption of *p*-nitroaniline onto HJ-02 resin and XAD-4 is illustrated in Fig. 4. Meanwhile, the dissociation curves of *p*-nitroaniline and phenol on dependence of the solution pH are also employed (the dissociation curve of phenol is to simulate HJ-02 resin. The functional groups uploaded on HJ-02 resin are phenolic hydroxyl groups, and they can be represented by phenol. The pK_a values used for simulation of *p*-nitroaniline and phenol are 13.0 and 9.89, respectively) [20,21]. It is seen from Fig. 4 that the pH dependency trend of HJ-02 resin is different from XAD-4. The pH dependency trend of XAD-4 is consistent with the dissociation curve of *p*-nitroaniline, revealing that the molecular form of *p*-nitroaniline is suitable for the adsorption while its protonated form R-NH_3^+ (R stands for *p*-nitrophenyl group) is not favorable.

As for HJ-02 resin, Fig. 4 indicates that the adsorption capacity of *p*-nitroaniline retains approximately equivalent with the solution pH in the range of 3.7–7.8, while the adsorption capacity decreases sharply as the solution pH is lower than 3.7 or higher than 7.8. As can be seen from the dissociation curves of *p*-nitroaniline and phenol in Fig. 4, *p*-nitroaniline is protonated as R-NH_3^+ in strong acidic solution, and phenol will be ionized to be R′-O^- in alkaline solution (R′ represents phenyl group). That is to say, the molecular form of *p*-nitroaniline and the functional groups of HJ-02 resin are suitable for the adsorption, and a maximum adsorption occurs at the molecular state for both *p*-nitroaniline and phenol with the solution pH 3.7–7.8. By introducing phenolic hydroxyl groups on HJ-02 resin, formation of acid–base complex is possible (in the system we considered, the acid–base complex is specifically the hydrogen bonding between the phenolic hydroxyl groups and the basic amino-groups as well as nitro-groups of *p*-nitroaniline). Anyway, different pH-dependent adsorption of HJ-02 resin in contrast with XAD-4 indicates that the phenolic hydroxyl groups uploaded on the matrix of HJ-02 resin have a positive effect on the adsorption of *p*-nitroaniline.

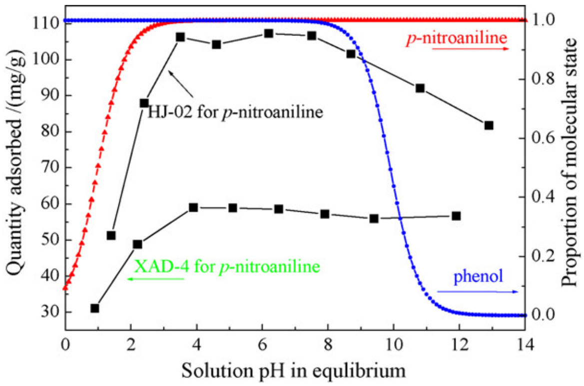

Fig. 4. The effect of the solution pH on the adsorption of *p*-nitroaniline onto HJ-02 and XAD-4 resin, along with the dissociation curves of *p*-nitroaniline and phenol on dependency of the solution pH.

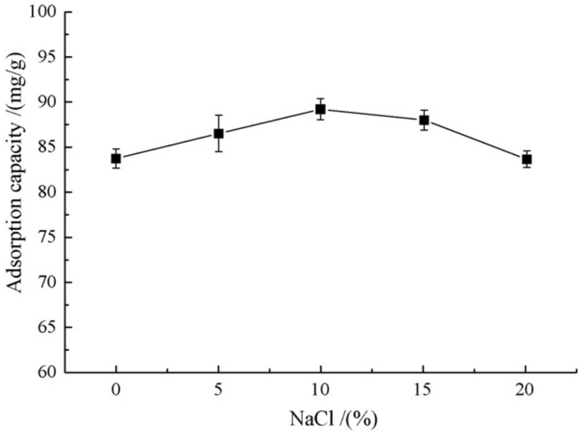

Fig. 5. The effect of NaCl on the adsorption capacity of *p*-nitroaniline onto HJ-02 resin with the temperature at 288 K.

3.3. Effect of ionic strength on the adsorption

Inorganic salts such as NaCl, Na_2SO_4, and NH_4Cl are often coexistent in industrial wastewater with a comparatively high concentration, and hence the effect of NaCl as the model inorganic salt on the ability of HJ-02 resin to remove *p*-nitroaniline is investigated and the result is depicted in Fig. 5. It is obvious that the ionic strength does not affect the adsorption evidently and NaCl at low concentration (<10%) exhibits a positive effect due to the "salting-out" effect [22].

3.4. Adsorption kinetics

Fig. 6 shows the adsorption kinetic curves of *p*-nitroaniline onto HJ-02 and XAD-4 resin. It is seen that the required time from the beginning to the equilibrium for XAD-4 is about 100 min, much shorter than that for HJ-02 resin. This may be due to the different pore structure of the two resins. Furthermore, the higher temperature or lower initial concentration of *p*-nitroaniline leads to the shorter required time.

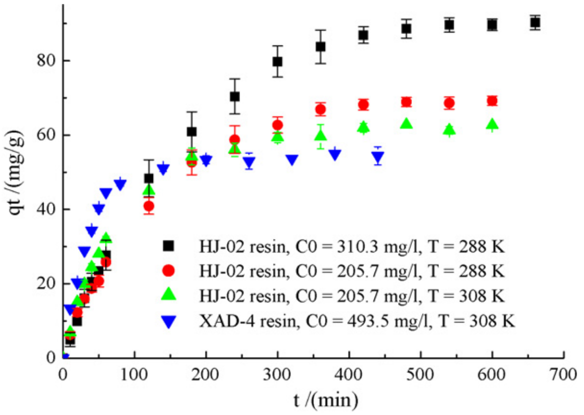

Fig. 6. The adsorption kinetic curves of *p*-nitroaniline adsorbed onto HJ-02 and XAD-4 resin.

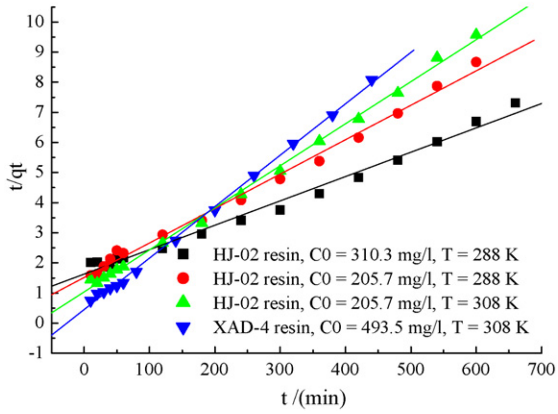

Fig. 7. The fitted adsorption kinetic curves of *p*-nitroaniline adsorbed onto HJ-02 and XAD-4 resin by pseudo-second-order rate equation.

The pseudo-first-order and pseudo-second-order rate equations are employed to fit the adsorption kinetic data [23]:

$$\ln\left(\frac{q_e}{(q_e - q_t)}\right) = k_1 t \tag{2}$$

$$\frac{t}{q_t} = \frac{1}{(k_2 \times q_e^2)} + \frac{t}{q_e} \tag{3}$$

here q_t is the adsorption capacity at the contact time t (mg/g), and k_1 and k_2 are the pseudo-first-order (min^{-1}) and the pseudo-second-order [g/(mg min)] rate constants, respectively.

The fitted results in Table 1 (The fitted adsorption kinetic curves by the pseudo-second-order rate equation are shown in Fig. 7) indicate that the pseudo-first-order rate equation is not suitable for the adsorption (the fitted results by the pseudo-first-order rate equation are not displayed in Table 1) while the pseudo-second-order rate equation can characterize the adsorption well due to the correlation coefficients $R^2 > 0.99$. In addition, the predicted rate constant for XAD-4 is greater than that for HJ-02 resin, consistent with the shorter required time for XAD-4. The average pore diameter and the pore distribution of the adsorbent determine the diffusion speed of the adsorbates from the solution phase to the adsorption site of the adsorbent. The average pore diameter of XAD-4 resin is determined to be 5.55 nm (larger than that of HJ-02 resin, the average pore diameter of HJ-02 resin is 2.54 nm) and the mesopores in the range of 2–18 nm play a predominant role for XAD-4 (while the mesopores in the range of 2–5 nm dominate the pore for HJ-02 resin), which is favorable for the diffusion of *p*-nitroaniline. In particular, the rate constant of HJ-02 resin with higher temperature or lower initial concentration of *p*-nitroaniline is a little greater, accordant with the above observation that the higher temperature or lower initial concentration leads to the shorter required time.

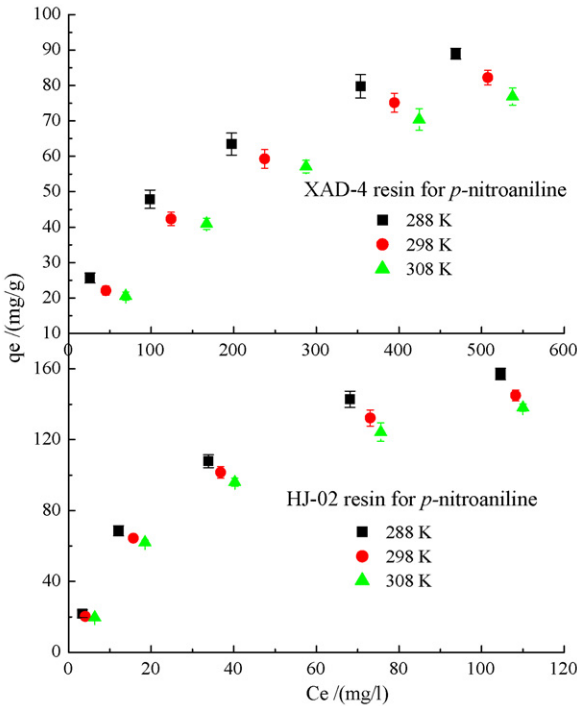

Fig. 8. The adsorption isotherms of *p*-nitroaniline onto HJ-02 and XAD-4 resin in aqueous solution with temperature at 288, 298, and 308 K, respectively.

3.5. Adsorption isotherms and thermodynamics

Fig. 8 shows the adsorption isotherms of *p*-nitroaniline onto HJ-02 and XAD-4 resin in aqueous solution. It is clear that low temperature is more favorable for the adsorption, and the adsorption capacity onto HJ-02 resin is much larger than that onto XAD-4 at the same temperature and equilibrium concentration.

Langmuir and Freundlich isotherm equations are adopted to describe the adsorption equilibrium data [24]:

Langmuir isotherm equation:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{(q_m K_L)} \tag{4}$$

Freundlich isotherm equation:

$$\log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \tag{5}$$

here q_m is the maximum adsorption capacity (mg/g), K_L (l/g) is a binding constant in Langmuir equation [25], K_F and n are the Freundlich constants for the heterogeneous adsorbent [24,25]. The corresponding parameters summarized in Table 2 indicate that Langmuir isotherm is more suitable for the adsorption than the Freundlich one.

Table 1
The correlation parameters for the adsorption kinetic data of *p*-nitroaniline adsorbed onto HJ-02 and XAD-4 resin according to the pseudo-second-order rate equation.

	k_2/[g/(mg min)]	q_e/(mg/g)		R^2
		Exp.	Cal.	
XAD-4 resin				
$T = 308$ K, $C_0 = 493.5$ mg/l	6.719×10^{-4}	54.45	58.51	0.9985
HJ-02 resin				
$T = 288$ K, $C_0 = 310.3$ mg/l	3.362×10^{-5}	90.25	132.28	0.9900
$T = 288$ K, $C_0 = 205.7$ mg/l	8.623×10^{-5}	69.20	87.41	0.9942
$T = 308$ K, $C_0 = 205.7$ mg/l	1.878×10^{-4}	62.66	71.63	0.9976

Table 2
The correlated parameters for the adsorption isotherm data of *p*-nitroaniline adsorbed onto HJ-02 and XAD-4 resin according to Langmuir and Freundlich isotherm equation.

	Langmuir isotherm equation			Freundlich isotherm equation		
	K_L/(l/g)	q_m/(mg/g)	R^2	K_F/([(mg/g)(l/mg)$^{1/n}$])	n	R^2
XAD-4 resin						
288 K	9.195	106.3	0.9864	6.449	2.327	0.9978
298 K	4.921	113.9	0.9962	2.880	1.832	0.9920
308 K	2.741	129.7	0.9994	1.385	1.542	0.9886
HJ-02 resin						
288 K	39.17	196.1	0.9978	1.763	13.12	0.9512
298 K	31.36	188.7	0.9990	1.666	10.20	0.9602
308 K	20.08	204.1	0.9756	1.485	6.877	0.9434

If the liquid-phase adsorption follows Langmuir isotherm equation, the thermodynamic parameters such as adsorption enthalpy ΔH (kJ/mo1), adsorption free energy ΔG (kJ/mo1), and adsorption entropy ΔS (J/(mo1 K)) can be calculated according to the following equations [26–28]:

$$\ln K_L = -\frac{\Delta H}{(RT)} + \ln K_0 \tag{6}$$

$$\Delta G = -RT \ln K_L \tag{7}$$

$$\Delta S = \frac{(\Delta H - \Delta G)}{T} \tag{8}$$

where R is the universal gas constant, 8.314 J/mol K, T is the absolute temperature, and K_0 is a constant. By plotting $\ln K_L$ versus $1/T$, a straight line can be gained (plot of $\ln K_L$ versus $1/T$ is shown in Fig. 9), and the ΔH can be obtained from the slope of the straight line. As presented in Fig. 9, the negative ΔH and ΔS imply the adsorption is mainly driven by the enthalpy change, and the adsorption is an exothermic and more ordered process. In particular, the greater ΔH onto HJ-02 resin than XAD-4 displays the stronger interaction between HJ-02 resin and *p*-nitroaniline. The more negative ΔS onto HJ-02 resin than XAD-4 suggests a more ordered arrangement of *p*-nitroaniline onto HJ-02 resin after the adsorption. In addition, the negative ΔG indicates a favorable and spontaneous process, and the greater ΔG onto HJ-02 resin than XAD-4 suggests the more favorable process.

3.6. Adsorption mechanisms

It is shown in Figs. 4 and 8 that the adsorption capacity of *p*-nitroaniline onto HJ-02 resin is much larger than XAD-4, and this different adsorption capability of the two resins may be explained by the specific surface area, the polarity matching between the adsorbent and the adsorbate, and the size matching between the pore diameter of the adsorbent and the molecular size of the adsorbate.

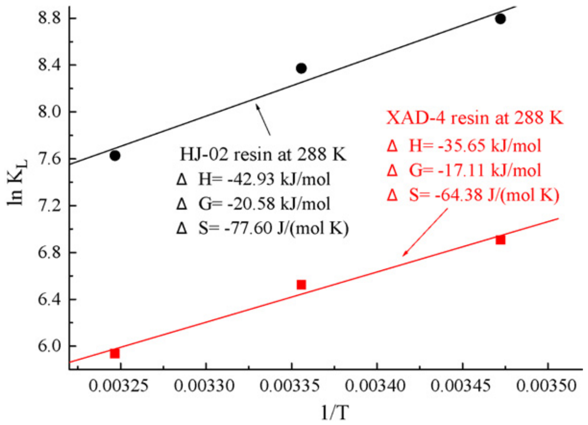

Fig. 9. Plots of $\ln K_L$ with $1/T$ for the adsorption of *p*-nitroaniline onto HJ-02 and XAD-4 resin in aqueous solution.

The specific surface area of an adsorbent plays an important role in the adsorption. Generally, the higher the specific surface area of the adsorbent is, the larger the adsorption capacity is [29]. XAD-4 has a higher surface area than HJ-02 resin while it has a smaller adsorption capacity than HJ-02 resin, revealing that the specific surface area is not the reason resulting in the enhanced adsorption of *p*-nitroaniline onto HJ-02 resin.

It is known that the adsorbent with non-polar skeleton can adsorb non-polar compounds selectively, and that the adsorbent functionalized with polar groups on its skeleton has much larger adsorption capacity for polar adsorbates [30]. XAD-4 has a non-polar polystyrene skeleton, while *p*-nitroaniline has polar amino- and nitro-groups on its benzene ring. As a result, XAD-4 has a small adsorption capacity for *p*-nitroaniline. Contrary to XAD-4, HJ-02 resin contains a few polar phenolic hydroxyl groups on its skeleton. The polarity matching between HJ-02 resin and *p*-nitroaniline will lead to the larger adsorption capacity. In addition, Fig. 4 suggests that the molecular form of *p*-nitroaniline is favorable for the adsorption, and formation of intermolecular hydrogen bonding is possible between the phenolic hydroxyl groups on HJ-02 resin and the amino-groups as well as nitro-groups of *p*-nitroaniline, which enhances the surface adsorption.

Beside the polarity matching, the size matching between the pore diameter of the adsorbent and the molecular size of the adsorbate should also be taken into account. It was found that the optimum ratio of the pore diameter of the adsorbent to the molecular size of the adsorbate was 2–6 for polymeric adsorbents [31]. If the pore diameter of the adsorbent is too small, intraparticle diffusion of the adsorbate from the adsorbent surface to the adsorption site across the pore will be hindered. On the other hand, the adsorbate cannot be adsorbed on the adsorbent steadily if the pore diameter of the adsorbent is too large. The better the matching between the pore diameter of the adsorbent and the molecular size of the adsorbate is, the larger the adsorption capacity is. The mesopores (2–5 nm) play a predominant role in the pore structure for HJ-02 resin and the average pore diameter is 2.54 nm. The molecular size of *p*-nitroaniline is optimized to be 0.67 nm × 0.43 nm (obtained from the optimized molecular structure performed by Gaussian 03 software package [32]), which is suitable for the adsorption. While the average pore diameter of XAD-4 is measured to be 5.55 nm, inducing the less adsorption capacity. Additionally, Fig. 4 shows that the adsorption capacity onto HJ-02 resin is still considerable even at the strong alkaline solution. The polar phenolic hydroxyl group on HJ-02 resin are ionized as negative oxygenic ions (O^-) at the strong alkaline solution, the one and only reason that HJ-02 resin still has considerable adsorption capacity for *p*-nitroaniline is its mesoporous structure in the range of 2–5 nm.

4. Conclusions

HJ-02 resin is prepared successfully in this study and it is a more excellent adsorbent for removal of *p*-nitroaniline than XAD-4. The solution pH 3.7–7.8 is optimum for the adsorption of *p*-nitroaniline onto HJ-02 resin while that higher than 4.1 is favorable for XAD-4. NaCl does not influences the adsorption remarkably and it poses a positive effect at a low concentration (<10%). The pseudo-second-order rate equation is suitable for the adsorption kinetic curves and Langmuir isotherm characterizes the adsorption isotherms better than Freundlich isotherm. The adsorption thermodynamic parameters are set to be negative and the calculated adsorption enthalpy, adsorption free energy, and adsorption entropy onto HJ-02 resin are greater than the corresponding ones onto XAD-4. The added polarity by introducing phenolic hydroxyl groups on the skeleton and the predominant mesopores of HJ-02 resin induce the much larger adsorption capacity than XAD-4 resin.

Acknowledgments

The National Natural Science Foundation of China (No. 20804058) and the undergraduate innovative experiment project (No. LZ09247) are gratefully acknowledged.

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