



Sorption enhancement of 1-naphthol onto a hydrophilic hyper-cross-linked polymer resin

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ABSTRACT

A hydrophilic hyper-cross-linked polymer resin NDA-150 was developed to remove 1-naphthol from the contaminated waters. The sorption performance of 1-naphthol on NDA-150 was explored and compared with that on the commercial hydrophobic resin XAD-4. The sorption rates of 1-naphthol onto both of the two resins obey the pseudo-second-order kinetics, and are limited by the successive steps of film diffusion and intraparticle diffusion. The greater sorption rate on XAD-4 than NDA-150 is probably due to the larger average pore diameter of XAD-4. All the adsorption isotherms can be represented by Langmuir equation. The larger capacity and stronger affinity of NDA-150 than XAD-4 probably result from the abundant microporous structure and polar groups of NDA-150. In aqueous phase hydrophobic XAD-4 adsorbs 1-naphthol driven principally by enthalpy change, while the adsorption onto hydrophilic NDA-150 driven mainly by entropy change. The breakthrough and the total sorption capacity of NDA-150 to 1-naphthol were obtained to be 1.10 and 1.58 mmol mL⁻¹ resin at 293 K, respectively. Nearly 100% regeneration efficiency for the resin was achieved by ethanol at 313 K.

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1. Introduction

Contamination of surface water and groundwater with synthetic aromatic compounds is one of the most urgent environmental problems that threat both human being and wildlife. 1-Naphthol, widely used as an industrial intermediate, is of particular concern due to its acute toxicity and negative environmental impacts once released to the receiving water systems via industrial waste streams. Many techniques were developed to remove or separate the chemical from wastewaters, among which, adsorption by activated carbon is presumed to be one of the most effective options [1–3].

In recent years, synthetic polymeric resins were studied as an alternative to activated carbon in industrial wastewater treatment in China due to its high removal efficiency, feasibility in application, as well as the potential to extract valuable organic compounds from the waste streams [4–7]. For example, the commercial-available Amberlite XAD-4 resin was reported as an ideal adsorbent for a wide variety of aromatic compounds, especially for phenols [8]. However, XAD-4 resin has hydrophobic surface and a low capacity

for most organic chemicals compared to activated carbon, which limits its applications in industrial wastewater treatment [9]. To obtain larger adsorption capacity and better selectivity for a specific organic pollutant, chemical modification of ordinary polymeric resins is often adopted by grafting some special functional groups onto the matrix of the resins [5,10].

The purpose of the study was to synthesize a hydrophilic hyper-cross-linked adsorbent (NDA-150) with high capacity and strong affinity to 1-naphthol. This was achieved by modification of a commercial macroreticular adsorbent CHA-101 through post-cross-linking Friedel–Crafts reaction. The sorption kinetics and equilibrium were thereafter explored in batch experiments and compared with that of XAD-4. Thermodynamic analysis was used to elucidate the adsorption mechanism of 1-naphthol on NDA-150. Ethanol was used to evaluate the regeneration efficiency of NDA-150 in fixed-bed column runs.

2. Experimental

2.1. Materials

CHA-101, a macroreticular polymer, was provided kindly by Langfang Electrical Resin Co. Ltd (Hebei province, China). The spherical macroporous Amberlite XAD-4 resin was purchased from Rohm & Haas Company (Philadelphia, USA).

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Acetone, ethanol, iron chloride, and 1-naphthol are analytical grade and were purchased from Shanghai Reagent Station (Shanghai, China). All the solvents were used without additional purification. The background solution used in the adsorption experiments contained 10^{-3} M KH_2PO_4 , and the pH values of the aqueous solutions were about 5.4–6.6, in which more than 99% of 1-naphthol molecules were nonionic (in supporting materials).

2.2. Synthesis of NDA-150 resin

CHA-101 is a macroreticular polystyrene adsorbent with some residual chloromethyl groups on its polymeric matrix during the synthetic process [5]. Post-cross-linking can take place at these groups via Friedel–Crafts reaction [11].

The hydrophilic NDA-150 was synthesized from CHA-101 according to the following post-cross-linking steps: 50 g CHA-101 was swollen in 300 g nitrobenzene in a 500-mL round-bottomed flask for 12 h. Under mechanical stirring, 6 g iron chloride was slowly added into the flask at 293 K, and the mixture was kept stirring for 12 h at 373 K. Finally, the mixture was poured into the acetone bath containing 1% hydrochloric acid and filtered after 0.5 h.

2.3. Batch sorption experiments

Prior to use, both resins were extracted with ethanol for 8 h in a Soxhlet apparatus and desiccated in vacuum at 325 K for 24 h. In the following experiments, hydrophilic NDA-150 can be used directly, whereas hydrophobic XAD-4 should be firstly wetted with 0.5 mL methanol and then rinsed three times with deionized water.

Sorption kinetics was determined by analyzing adsorptive uptake of 1-naphthol from its aqueous solution of 1 and 8 mM concentrations at different time intervals until the equilibrium reached at 293 K.

Equilibrium experiments were carried out at 293 K, 303 K and 313 K in 100 mL glass flasks. All flasks contained a fixed amount (0.0250 g) of the adsorbent and a predetermined amount of 1-naphthol (0.6, 1.0, 2.0, 4.0, 6.4, and 8.0 mM, respectively). The flasks were then transferred to a G 25 model incubator shaker with thermostat (New Brunswick Scientific Co. Inc.) and shaken under 150 rpm until the adsorption process reached equilibrium.

For all above systems, control experiments were conducted using flasks prepared similarly but containing no adsorbent, and assessed loss of solutes to the flask components during adsorption tests. Results of triplicate flasks showed that the average solution phase concentrations were within 98–101% of the respective initial concentration of the same solution analyzed similarly. Hence, compound loss was negligible.

2.4. Fixed-bed column tests

Dynamic sorption was conducted in a 10 cm × 4.6 mm-I.D. stainless-steel column packed with the freshly prepared hydrophilic adsorbent NDA-150 at 293 K. 1-Naphthol solution of 8 mM was pumped through the column with the superficial liquid velocity (SLV) and the empty bed contact time (EBCT) equal to 0.30 m h⁻¹ and 6 min, respectively. The effluents from the column were quantitatively analyzed.

2.5. Analysis

The solute concentrations were determined using a reverse-phase HPLC (Waters 600) equipped with dual UV absorbance detector (Waters 2487). 1-Naphthol was quantified at the wavelength of 274 nm. Methanol–water (7:3 in volume ratio) was used as mobile phase, and the flow rate was 1 mL min⁻¹.

Table 1
Main properties of adsorbents [8]

Sorbent	XAD-4	NDA-150
Matrix	Polystyrene	
Polarity	Nonpolar	Moderate polar
BET surface area (m ² g ⁻¹)	914	1225
Micropore area (m ² g ⁻¹)	74	740
Micropore volume (cm ³ g ⁻¹)	0.012	0.336
Average pore diameter (nm)	5.8	3.0
Particle size (mm)	0.4–0.6	0.4–0.6
Residual chloride content (%)	0	2.7
Oxygen content (%)	0	2.9

The specific surface area and the pore distribution of the three adsorbents were, respectively, measured by BET and BJH methods [11] using a Micrometrics ASAP-2010 automatic surface area analysis instrument (Micromeritics Instrument, Norcross, USA). Infrared spectra of the adsorbents in the range of 650–4000 cm⁻¹ were collected by a Nexus 870 FT-IR spectrometer (USA) with a pellet of powdered potassium bromide. The chlorine content was measured according to the method of Volhard [12].

3. Results and discussion

3.1. Characterization of the polymeric adsorbents

Typical properties of XAD-4 and NDA-150 are presented in Table 1. These resins are quite different in the chemical composition and the pore structures. The pore size distribution of the two resins (in supporting materials) indicates that the micropore (<2 nm) dominates in NDA-150, whereas mesopore (2–18 nm) plays the main role in the XAD-4 resin. The strong bands of 1700 cm⁻¹ (carbonyl group) and 3650 cm⁻¹ (hydroxyl group) in the IR spectrum of NDA-150 (in supporting materials) indicate polar groups in its polymeric matrix. Carbonyl groups on NDA-150 might be resulted from oxidation of the chloromethyl group and the pendant double bonds of the St-DVB copolymer structure during post-cross-linking reaction at high temperature [13]. Thus, the moderate polarity of NDA-150 analyzed in this study is different from the nonpolar nature of NDA-100 analyzed in our previous study [8].

3.2. Sorption kinetics

The percentage sorption (q_t/q_e) at a certain time interval was determined until equilibrium was reached. The removal of 1-naphthol by XAD-4 and NDA-150 at varying contacting time is presented in Fig. 1. All of the curves in Fig. 1 have a similar shape, only differing in the percentage adsorbed. XAD-4 and NDA-150 have different capacities and different uptake rate. From the figure, the loading half-time ($t^{1/2}$) needed to reach 50% sorption of the total capacity was calculated for the lower initial concentration of 1 mM, which has been found to be within 35 min for NDA-150 and within 8 min for XAD-4, indicating that the kinetics of 1-naphthol–adsorbent interaction is sufficiently rapid.

From the kinetic data the dynamics of the sorption process can be understood. Both pseudo-first-order and pseudo-second-order models [14,15] were applied to fit all the data:

$$\text{Pseudo-first-order model: } \log(q_e - q_t) = \log(q_e) - \frac{k_1 t}{2.303} \quad (1)$$

$$\text{Pseudo-second-order model: } \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where q_e is the sorption capacity (mmol g⁻¹), q_t is the uptake at the time t (mmol g⁻¹), and k_1 is the pseudo-first-order rate constant (min⁻¹), and k_2 is the pseudo-second-order rate con-

Table 2
Kinetic parameters of 1-naphthol sorption on XAD-4 and NDA-150 resins at different initial concentrations at 293 K

Sorbent	C_0 (mM)	Pseudo-first-order model		Pseudo-second-order model	
		k_1 (min^{-1})	R^2	k_2 ($\text{g mmol}^{-1} \text{min}^{-1}$)	R^2
XAD-4	1	0.041	0.967	0.046	0.989
	8	0.090	0.901	5.341	0.963
NDA-150	1	0.026	0.955	0.032	0.979
	8	0.014	0.967	0.964	0.989

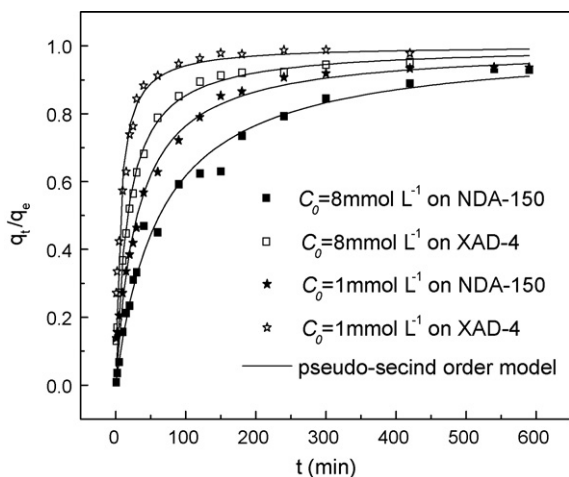


Fig. 1. Effect of contact time on 1-naphthol removal by XAD-4 and NDA-150 resins at 293 K.

stant ($\text{g mmol}^{-1} \text{min}^{-1}$). The fitting data in Table 2 indicate that the pseudo-second-order model describes the sorption kinetics better than the pseudo-first-order model. The uptake rate of 1-naphthol onto XAD-4 is greater than that onto NDA-150, and the value increases with increasing of initial concentration. The different uptake rates between XAD-4 and NDA-150 show that both the average pore diameter and the particle size of the two resins (in Table 1) may play a dominant role.

In general, sorption process can be interpreted by a series of steps: mass transfer from liquid to the particle surface across the boundary layer, diffusion within the pores and deposition on the surface of the particles [16]. To determine which one (film diffusion or intraparticle diffusion) is rate limiting step as well as the corresponding rate constants, the kinetic data were further processed. The two diffusion models can be written as [17]:

$$\text{Film diffusion model: } \ln(1 - F) = -K_f t \quad (3)$$

$$\text{Intraparticle diffusion model: } q_t = K_p t^{1/2} \quad (4)$$

where K_f is the diffusion rate parameter in film diffusion model, K_p is the diffusion rate parameter in intraparticle diffusion model, and F is the fractional attainment of the equilibrium. The fitting results of Eqs. (3) and (4) listed in Figs. 2 and 3 show that both diffusion models can give a perfect fitting to all the sorption kinetic data ($R^2 > 0.99$). And the obtained values of K_f (0.028 min^{-1}) and K_p ($0.102 \text{ mmol g}^{-1} \text{ min}^{1/2}$) on XAD-4 are notably larger than those (0.016 min^{-1} and $0.074 \text{ mmol g}^{-1} \text{ min}^{1/2}$) on NDA-150, respectively. From this figure, it can also be observed that none of the straight lines passes through the origin, which indicates that intraparticle diffusion and film diffusion are both the rate limiting steps for 1-naphthol diffusion onto XAD-4 and NDA-150 [15]. Based on the values of K_f and K_p , 1-naphthol molecules diffuse more rapidly on XAD-4 surface than those on NDA-150, which is in agreement with their sorption rates.

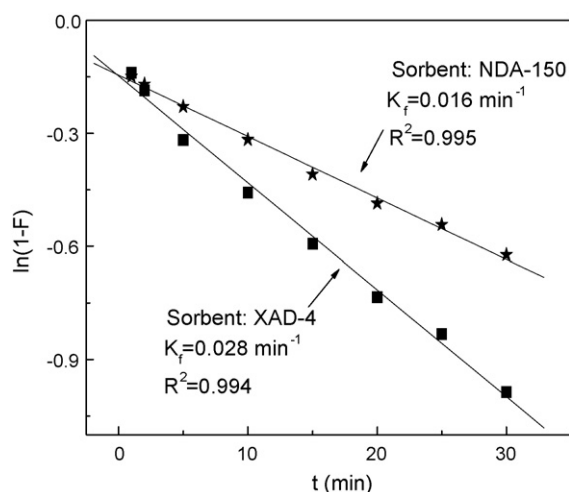


Fig. 2. The straight lines of $\ln(1 - F)$ vs. the contact time t from 1-naphthol adsorbed onto XAD-4 and NDA-150 resins at 293 K.

3.3. Adsorption isotherms and thermodynamics

Equilibrium data concerning the sorption of 1-naphthol onto XAD-4 and NDA-150 are presented in Fig. 4. Langmuir and Freundlich [18,19] parameters obtained by nonlinear regression of the experimental data with Levenberg–Marquardt algorithm are listed in Table 3. Langmuir model yields a better correlation for all the equilibrium sorption curves. Fig. 4 and Table 3 show the adsorbing capacity (q_m) and affinity (K_L) for 1-naphthol to NDA-150 are significantly higher than those to XAD-4. It is probably attributed to the two factors: (1) the micropore ($< 2 \text{ nm}$) in NDA-150 is much higher than that of XAD-4 which deduces the pore-filling driving force;

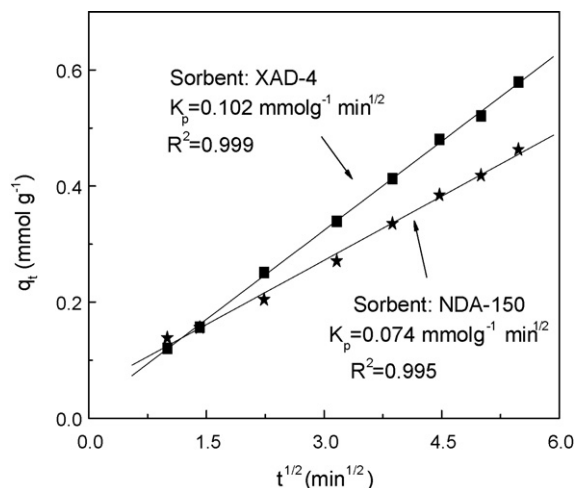


Fig. 3. The straight lines of q_t vs. $t^{1/2}$ from 1-naphthol adsorbed onto XAD-4 and NDA-150 resins at 293 K.

Table 3
Isotherm parameters of 1-naphthol sorption on XAD-4 and NDA-150 resins at different temperatures

Sorbent	T (K)	Freundlich			Langmuir [8]		
		K_f	n	R^2	K_L (L mmol ⁻¹)	q_m (mmol g ⁻¹)	R^2
XAD-4	293	1.661	3.040	0.924	2.057	2.941	0.998
	303	1.402	2.589	0.970	1.285	2.887	0.998
	313	1.315	2.537	0.976	1.152	2.804	0.995
NDA-150	293	2.586	2.789	0.952	2.549	4.330	0.999
	303	2.494	2.894	0.935	2.611	4.153	0.999
	313	2.351	2.916	0.946	2.612	3.926	0.999

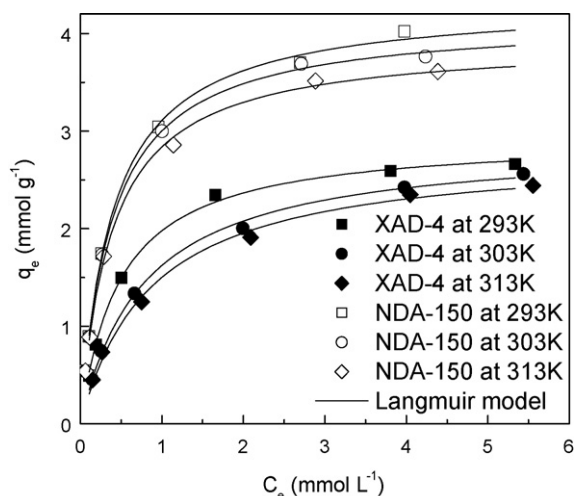


Fig. 4. Sorption isotherms of 1-naphthol on XAD-4 and NDA-150 resins at different temperatures.

(2) the polar groups on NDA-150 polymer matrix are also helpful to increase the affinity to 1-naphthol by the proposed hydrogen bonding interaction.

It can be seen in Fig. 4 that lower temperature is favorable for 1-naphthol adsorption onto XAD-4 and NDA-150, indicating an exothermic nature of the adsorption. The free energy change (ΔG), enthalpy change (ΔH), and entropy change (ΔS) for adsorption process were calculated in the following equations [11,20]:

$$\Delta G = -RT \ln K_L \quad (5)$$

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

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (7)$$

where K_L is the characteristic constant in Langmuir equation, R is the universal gas constant (8.314 kJ mol⁻¹ K⁻¹), T is the temperature (K), and K_0 is a constant. From Eq. (6), ΔH was calculated according to the slope of line plotted by $\ln K_L$ vs. $1/T$ (not shown). Thermodynamic parameters of 1-naphthol sorption onto XAD-4 and NDA-150 are listed in Table 4.

The negative value of ΔG indicates that all the test sorption processes are favorable and spontaneously occurred. Compared to XAD-4, the larger absolute value of ΔG for NDA-150 suggests the greater affinity of 1-naphthol to NDA-150. It is probably attributed to the formation of donor–acceptor adducts between the hydroxyl group on 1-naphthol molecule and the polar group (carbonyl group and hydroxyl group) on NDA-150 matrix, and the pore-filling mechanism due to its abundant microporous structure. This is consistent with their sorption capacities.

The negative value of ΔH indicates an exothermic process of 1-naphthol adsorption onto XAD-4, whereas the slightly positive

value of ΔH shows the endothermic process onto NDA-150. The sorption of 1-naphthol onto XAD-4 was driven mainly by enthalpy change due to the negative values of ΔH and ΔS , whereas the sorption of 1-naphthol onto NDA-150 was driven mainly by entropy change due to the positive values of ΔH and ΔS . Because in aqueous solution water molecules surround hydrophilic NDA-150 resin surface, 1-naphthol must get some heat to kick off water molecules before it could be adsorbed on the surface. The action is called “solvent replacement” [21]. For the bigger molecular size 1-naphthol molecule than water molecule, the number of water molecule replaced was larger than that of 1-naphthol molecule adsorbed (shown in Fig. 5). Therefore, the solvent replacement process results in the increase of entropy and enthalpy.

3.4. Dynamic sorption and desorption

The satisfactory sorption capacity for 1-naphthol on NDA-150 makes it possible to develop NDA-150 as a polymeric adsorbent for the removal and recovery of organics from industrial wastewater. For this reason, the dynamic sorption and desorption was tested. The results of mini-column dynamic sorption and desorption of 1-naphthol on NDA-150 are shown in Fig. 6. Breakthrough point was determined when the concentration of the effluent from the column reached 10% of the initial value, and the corresponding sorption capacity was calculated as 1.10 mmol mL⁻¹ resin base on the total amount of 1-naphthol removed at that time. Similarly, the total sorption capacity was calculated as 1.58 mmol mL⁻¹ resin when the concentration of the effluent from the column reached 100% of the initial value.

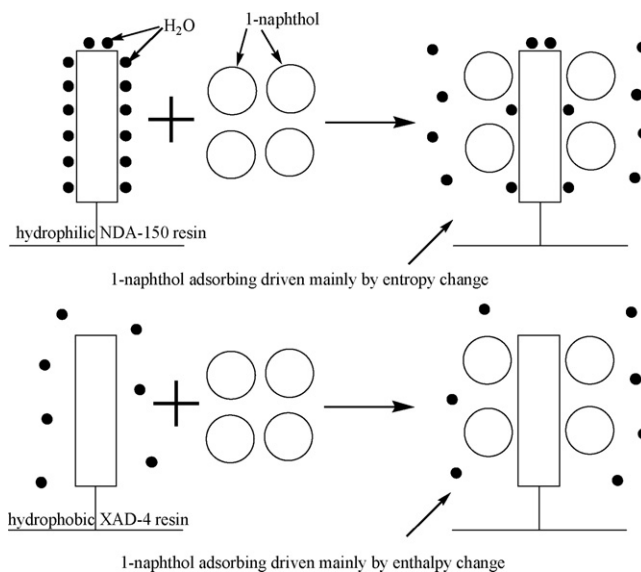


Fig. 5. Schemes for the proposed interaction between solute and adsorbent in aqueous solution.

Table 4
Thermodynamic parameters of 1-naphthol sorption on XAD-4 and NDA-150 resins

Sorbent	ΔH (kJ mol ⁻¹)	ΔG (kJ mol ⁻¹)			ΔS (J mol ⁻¹ K ⁻¹)		
		293 K	303 K	313 K	293 K	303 K	313 K
XAD-4	-22.250	-1.757	-0.633	-0.368	-69.941	-71.344	-69.910
NDA-150	0.939	-2.280	-2.418	-2.499	10.984	11.077	10.982

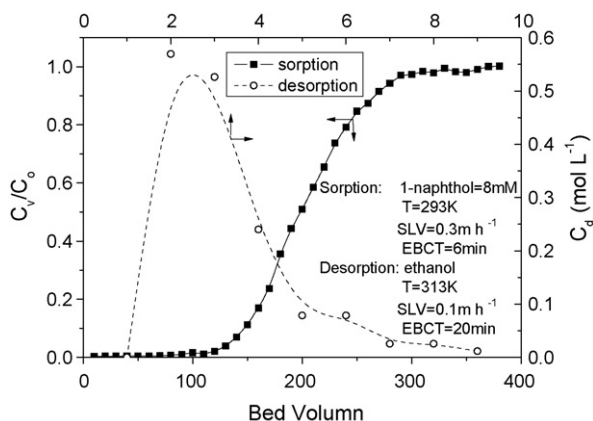


Fig. 6. Sorption and desorption dynamic curves of 1-naphthol on NDA-150 resin. (C_0 is the concentration of initial solution (mM), C_v is the concentration at different bed volume of the effluent (mM), C_d is the concentration at different bed volume of regenerative reagent (mM), SLV is the superficial liquid velocity (m h⁻¹), and EBCT is the empty bed contact time (min).)

Ethanol was used to elute 1-naphthol from NDA-150 column. At the flow rate of 3 BV h⁻¹, nearly 100% regeneration efficiency was achieved at 313 K (shown in Fig. 6). By the additional distilling, both 1-naphthol and ethanol can be easily recovered.

4. Conclusions

A hydrophilic hyper-cross-linked polymer resin NDA-150 for adsorbing 1-naphthol from aqueous solution was synthesized. The batch kinetic tests show that the sorption kinetics of 1-naphthol onto XAD-4 and NDA-150 can be fitted well by the pseudo-second-order model, and the film diffusion and intraparticle diffusion are both the rate limiting steps. The sorption rate of 1-naphthol onto XAD-4 is greater than that onto NDA-150.

The equilibrium sorption capacity and affinity for 1-naphthol on NDA-150 is significantly higher than that on XAD-4, which is attributed to its higher microporous structure and the polar functional groups. All the isotherms can be fitted with the Langmuir equation satisfactorily. The spontaneous sorption of 1-naphthol onto XAD-4 was driven mainly by enthalpy change while onto NDA-150 by entropy change.

The experimental results of dynamic sorption of 1-naphthol by NDA-150 show that the breakthrough capacity and the total capacity are 1.10 and 1.58 mmol mL⁻¹ resin, respectively. 100% regeneration efficiency was achieved using ethanol.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jhazmat.2008.06.054.

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