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Journal of Hazardous Materials

Journal of Hazardous Materials 152 (2008) 715-720

www.elsevier.com/locate/jhazmat

Adsorption equilibria and kinetics for phenol and cresol onto polymeric adsorbents: Effects of adsorbents/adsorbates structure and interface

Fu-Qiang Liu^{a,*}, Ming-Fang Xia^{b,c}, San-Li Yao^a, Ai-Min Li^a, Hai-Suo Wu^{b,c}, Jin-Long Chen^{a,c}

^a State Key Laboratory of Pollution Control and Resource Reuse, School of the Environment, Nanjing University, Nanjing 210093, PR China ^b Research Institute of Environmental Sciences of Jiangsu Province, Nanjing 210036, PR China

^c Engineering Technology Research Center of Organic Toxic Substance Control and Resource Reuse of Jiangsu Province, Nanjing 210046, PR China

Received 23 August 2006; received in revised form 16 July 2007; accepted 16 July 2007 Available online 27 July 2007

Abstract

Phenol and cresol (*o-*, *m-*, and *p-*) were selected as the adsorbates with different dipole moment (cresol > phenol, methyl being electron-drawing group) and solubility (phenol > cresol, methyl being hydrophobic group). Macropore polymers (NDA-1800 and XAD-4), hypercrosslinked polymers (NDA-100), and chemically modified adsorbents (NDA-150 and NDA-99), were comparatively used to investigate the adsorption properties including equilibria, thermodynamics and kinetics. First, all of the results about equilibria show that the adsorption data fit well to the Freundlich model. The adsorption capacity of NDA-99 and NDA-150 especially for phenol is larger in a certain extent than other three types of polymers. The hydrophobic interaction from large specific surface was mainly occurred, while the polar groups containing oxygen and amine markedly enhance the adsorption process via hydrogen interaction. Furthermore, the adsorption amount for NDA-99 and XAD-4 decrease linearly with the solubility of solutes tested. Then, the negative values of enthalpy demonstrate the predominantly exothermic and physical solid-extraction processes. Finally, the relatively more rapid adsorption process could be found onto NDA-150 than NDA-99, with the reason of the double larger pore size of the former. In conclusion, solubility of solute, together with surface area, pore size and modified groups, extremely exerts influences to the adsorption performances.

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Keywords: Phenol; Cresol; Polymeric adsorbent; Equilibria; Kinetics

1. Introduction

Wastewater containing phenolic compounds presents a serious disposal problem. This is particularly true of very low or very high concentrations of phenol in water when the biological degradation cleaning works too slowly or does not work at all [1]. Both phenol and cresol are priority pollutants from the view of EPA, thus the stringent regulatory standards have been enacted by regulatory agencies [2,3]. Consequently there has been a growing interest in developing and implementing various methods of removing specific phenols from polluted aqueous media. During the past several years, a number of ref-

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erences can be found in the literatures concerning the adsorption of phenols by polymeric adsorbents, which own the wide range of pore structures and characteristics and have been increasingly viewed as an alternative to activated carbon for the selective removal of these specific organics from contaminated streams [4–8]. At present, polymeric adsorbents are widely employed from the isolation and purification of organic substances, treatment of waste streams, chromatographic analysis, solid phase extraction, adsorption of organic vapors, and so on [9].

Macroporous polymer of Amberlite XAD-4, as widely reported, is considered the best adsorption material for removing phenolic compounds from aqueous media, and then much work has been done to get rid of phenolic compounds applying this kind of polymeric adsorbents. However, because its extreme hydrophobic surface results in poor contact with aqueous solutions, some activation solvents (such as methanol, acetone or

^{*} Corresponding author. Tel.: +86 25 86087695; fax: +86 25 85572627. *E-mail address:* jogia@163.com (F.-Q. Liu).

acetonitrile) have to be used during the pretreatment process [10-12].

Nowadays, much mention has been made on an important contribution to the 'hypercrosslinked' polymeric adsorbent methods since their development by Davankov and Tsyurupa [13]. This technique yielded polystyrene adsorbents of unusual 'hypercrosslinked' structure and exceptionally high adsorption capacity [14]. The sorption capacity of hypercrosslinked copolymers is reported up to three times higher than that of macroporous samples, and the kinetics of adsorption for organic compounds from water is faster [1].

To prepare serials of new water-compatible hypercrosslinked polymeric adsorbents with higher removal capability to phenolic compounds from aqueous solutions, the surface modification with chemical groups containing oxygen or amine is further carried out. This could overcome the difficulties of common polystyrene adsorbent mentioned above [15,16].

Since phenolic molecules and related compounds are common water contaminants, many attempts have been made to understand how the different ring substitutes affect the adsorption, and the following conclusion could be made, i.e., some adsorption processes trend with the compounds' solubility, and other with the electron density of the aromatic ring or pK_a values [17–22]. For the instance of chlorinated phenols, the *m*-chlorophenol showed higher adsorption selectivity than the *ortho* and *para* compounds because of its solubility in aqueous medium, moreover, solubility and polarity also influence the adsorption phenomenon [23]. According to the adsorption to cresol involving *o*-, *m*-, and *p*-cresol, the position of the substituted group on phenol had no effect on the respective phenol derivative adsorption [24].

To compare the adsorption behaviors, five types of polymeric resin adsorbents were selected herein. To further investigate the effects of the site of methyl on the aromatic ring of phenols, and of the physico-chemical properties of adsorbents, this paper focused on the following aspects: (1) measure and compare the adsorption static isotherms of phenolic compounds including phenol, *p*-cresol, *m*-cresol, and *o*-cresol on macroporous resins of Amberlite XAD-4 and NDA-1800, hypercrosslinked polymeric adsorbent of NDA-100, oxygen-modified resin of NDA-150, and amine-modified resin of NDA-99; (2) calculate and discuss the adsorption thermodynamic parameters involving enthalpies, entropy and Gibbs-energy; (3) investigate and compare the kinetic behavior of phenol and *p*-cresol on such chemically modified adsorbents as NDA-150 and NDA-99.

2. Materials and methods

2.1. Chemicals

Phenol and cresol are of A.R. grade bought from Shanghai Chemical Reagent Plant (Shanghai, PR China), and are used without any further purification. The spherical Amberlite XAD-4 resin was obtained from Rohm & Haas company (Philadelphia, USA). Other adsorbents were all supported by NanGe Environmental Co., Ltd. (Nanjing, PR China). The aqueous solutions were prepared by dissolving the corresponding phenolic com-

Table 1	
Typical properties of phenol and cresol [25]	

Characteristics	o-Cresol	m-Cresol	p-Cresol	Phenol
Solubility (aq. 40/100)	3.1	2.5	2.3	6.7
Dipole moment (D)	1.45	1.61	1.54	1.224
pK _a	10.26	10.00	10.26	9.99
$\log K_{\rm ow}$	2.53	2.44	2.70	2.24

pounds in deionized water. The physical properties of the tested adsorbates are tabulated in Table 1 [25].

2.2. Adsorption assay

Prior to their initial use, all adsorbents were extracted by acetone for 8 h and then dried for 2 h under vacuum at 333 K.

Equilibrium adsorptions of the four phenolic compounds at three different temperatures (283, 303 and 323 K) were performed as follows: 0.100 g of dry resin was introduced into a flask directly, while Amberlite XAD-4 resin was firstly "wetted" with 0.5 mL of methanol and then rinsed three times with deionized water before use. One hundred milliliters of an aqueous solution of phenolic compound with pre-determined concentration was added into each flask. The flasks were placed in a G-25 model incubator shaker (New Brunswick Scientific Co. Inc., Germany) at a pre-settled temperature and shaken under 130 rpm. After adsorption reached equilibrium, the concentrations of phenolic compounds (C_e) were analyzed with HPLC workstation. The initial concentrations (C_0) of the solutions were set 200, 400, 600, 800, and 1000 mg/L, respectively. Thus the solid-phase concentrations of adsorbates, q_e (mmol/g) were calculated according to Eq. (1)

$$q_{\rm e} = \frac{V_1(C_0 - C_{\rm e})}{MW} \tag{1}$$

where V_1 is the volume of solution (L), W the weight of dry resin (g), and M is the molecular weight of corresponding phenol.

For a batch kinetic process, at the initial solute concentrations of 1000 mg/L, the changes of adsorption amounts were measured at definite time intervals until equilibrium was achieved at the moderate temperature of 283 K. The adsorption amount, q (mmol/g), was calculated according to Eq. (2)

$$q = \frac{(C_0 - C)V}{MW} \tag{2}$$

where C (mg/L) is the concentration tested at a certain time.

The concentrations of phenolic compounds were measured at 275 nm using a HPLC assembled by Waters 1525 pump and dual λ absorbance detector. The mobile phase was 30% CH₃OH:70% H₂O (v/v).

3. Results and discussion

3.1. Characterizations of polymeric adsorbents

Some key characteristics of polymeric adsorbents tested, with obvious differences in both chemical surface and pore size dis-

Table 2	
Typical properties of polymeric resin adsorb	ents

Characteristics	XAD-4	XAD-1800	NDA-100	NDA-150	NDA-99
Polarity	Non-polar	Weak-polar	Weak-polar	Weak-polar	Moderate-polar
BET specific surface area (m^2/g)	880.2	785.2	934.0	906.1	819.1
Average pore size (nm)	5.83	8.06	1.2	2.5	1.2
Micropore surface (m^2/g)	3.1	_	561.3	529.2	463.3
Weak basic exchange capacity (mmol/g)	-	_	-	-	1.51

tribution (PSD), are collected in Table 2. And the PSD curves could be found in Fig. 1. It is very obvious that the pore of XAD-4 concentrates mainly in the mesopore range, while that of NDA-1800 shifts to a larger size. Meanwhile, all of the other three hypercrosslinked adsorbents possess abundant micropores. And the infrared spectra (IR) are shown in Fig. 2. The strong band at 2769.17 cm⁻¹ could be found as an indicative of methyl groups from N-CH₃, which suggests that the dimethylamine group is successfully bonded on NDA-99 but NDA-100. The strong bands at 1700 and 3421.79 cm⁻¹ in Fig. 2(b) testify the existence of such oxygen-containing group as hydroxyl and carboxyl on NDA-150.

3.2. Equilibrium adsorption

The equilibrium adsorption isotherms of phenol and cresol were usually described by the Freundlich model what owns the following linear form [26]:

$$\log q_{\rm e} = \log K_{\rm F} + \frac{1}{n} \log C_{\rm e} \tag{3}$$

where $K_{\rm F}$ and *n* are characteristic constants.

From Table 3, the results clearly show that the adsorption data for the studied adsorption systems fit well to both of the Langmuir and the Freundlich models for the correlative factors r^2 larger than 0.98. The adsorption capacity K_F decreases with the increase in temperature according to a certain adsorbent, which shows an exothermic process. The adsorption capacity for cresol is in the following order of NDA-99 > NDA-150 > NDA-100 > NDA-1800 > XAD-4, while for phenol NDA-99 > NDA-100 > NDA-150 > NDA-1800 > XAD-



Fig. 1. PSD curves of polymeric resin adsorbents.

4. NDA-100 and NDA-150 have a larger specific surface area. Additionally, the basic group on NDA-99 strengthens the Lewis interaction. The oxygen groups help to enhance the interaction with more polar molecules such as cresol, since the methyl drags electrons from π -bonding of aromatic ring. Thus, the main difference is caused by the hydrophobic interaction with the hydrogen-interaction enhancement for cresol in comparison with phenol.

At the same residual concentration (C_e) of 4.0 mmol/L, under the temperature of 283 K, the adsorption capacities (calculated values) of the four studied phenolic compounds onto NDA-99 and XAD-4 decrease linearly with solubilities of solutes, as shown in Fig. 3. The similar trends have been reported earlier for the different adsorption systems [21]. Thus, it is considered that the hydrophobic interaction puts the predominant influence onto the overall adsorption process.



Fig. 2. IR spectra of NDA-100 and NDA-99.

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Fittling results with Freundlich equation for various adsorption systems

Adsorbates- Adsorbents	Temperature (K)	Linear form of Freundlich equation	K _F	n	r^2
	283	$\ln q_{\rm e} = 0.5394 \ln C_{\rm e} - 0.2313$	0.7935	1.8539	0.9944
o-Cresol-XAD-4	303	$\ln q_{\rm e} = 0.7925 \ln C_{\rm e} - 0.7484$	0.4731	1.2618	0.9808
	323	$\ln q_{\rm e} = 0.9074 \ln C_{\rm e} - 1.1406$	0.3196	1.1021	0.9836
	283	$\ln q_{\rm e} = 0.4934 \ln C_{\rm e} + 0.4416$	1.5552	2.0268	0.9867
o-Cresol-NDA-99	303	$\ln q_{\rm e} = 0.5248 \ln C_{\rm e} + 0.3374$	1.4013	1.9055	0.9881
	323	$\ln q_{\rm e} = 0.531 \ln C_{\rm e} + 0.1238$	1.1318	1.8832	0.9948
	283	$\ln q_{\rm e} = 0.4518 \ln C_{\rm e} - 0.205$	0.8146	2.2134	0.9871
m-Cresol-XAD-4	303	$\ln q_{\rm e} = 0.5254 \ln C_{\rm e} - 0.4145$	0.6607	1.9033	0.9881
	323	$\ln q_{\rm e} = 0.5537 \ln C_{\rm e} - 0.7161$	0.4887	1.8060	0.9940
	283	$\ln q_{\rm e} = 0.3803 \ln C_{\rm e} + 0.5581$	1.7473	2.6295	0.9944
m-Cresol-NDA-99	303	$\ln q_{\rm e} = 0.4247 \ln C_{\rm e} + 0.4121$	1.5100	2.3546	0.9986
	323	$\ln q_{\rm e} = 0.5163 \ln C_{\rm e} + 0.1454$	1.1565	1.9369	0.9914
	283	$\ln q_{\rm e} = 0.3662 \ln C_{\rm e} - 0.1843$	0.8317	2.7307	0.9994
p-Cresol-XAD-4	303	$\ln q_{\rm e} = 0.4456 \ln C_{\rm e} - 0.4381$	0.6453	2.2442	0.9991
	323	$\ln q_{\rm e} = 0.5266 \ln C_{\rm e} - 0.8714$	0.4184	1.8990	0.9990
	283	$\ln q_{\rm e} = 0.3719 \ln C_{\rm e} + 0.526$	1.6922	2.6889	0.9952
p-Cresol-NDA-99	303	$\ln q_{\rm e} = 0.4437 \ln C_{\rm e} + 0.3548$	1.4259	2.2538	0.9922
	323	$\ln q_{\rm e} = 0.5629 \ln C_{\rm e} - 0.0098$	0.9902	1.7765	0.9907
	283	$\ln q_{\rm e} = 0.4802 \ln C_{\rm e} - 0.7355$	0.4793	2.0825	0.9995
Phenol-XAD-4	303	$\ln q_{\rm e} = 0.6102 \ln C_{\rm e} - 1.3516$	0.2588	1.6388	0.9997
	323	$\ln q_{\rm e} = 0.7067 \ln C_{\rm e} - 1.9682$	0.1397	1.4150	0.9993
	283	$\ln q_{\rm e} = 0.4976 \ln C_{\rm e} - 0.0377$	0.9630	2.0096	0.9969
Phenol-NDA-99	303	$\ln q_{\rm e} = 0.7301 \ln C_{\rm e} - 0.5112$	0.5998	1.3697	0.9777
	323	$\ln q_{\rm e} = 0.8207 \ln C_{\rm e} - 1.0402$	0.3534	1.2185	0.9900

Table 4

Adsorption thermodynamics parameters

Adsorbates	Adsorbents	$q_{\rm e} \; ({\rm mmol/g})$	ΔH (kJ/mol)	ΔG (kJ/mol)			$\Delta S (J/mol K)$		
				283 K	303 K	323 K	283 K	303 K	323 K
		1.2	-13.2	-4.4	-3.2	-3	-30.9	-32.9	-31.4
	XAD-4	1.4	-10.9	-4.4	-3.2	-3	-23.1	-25.5	-24.6
		1.6	-9	-4.4	-3.2	-3	-16.3	-19.2	-18.6
o-Cresol		1.2	-12	-4.8	-4.8	-5.1	-25.4	-23.7	-21.3
	NDA-99	1.4	-11.6	-4.8	-4.8	-5.1	-23.9	-22.3	-20
		1.6	-11.2	-4.8	-4.8	-5.1	-22.6	-21.1	-18.8
		0.8	-17.6	-5.2	-4.8	-4.8	-43.8	-42.2	-39.6
	XAD-4	1	-15.8	-5.2	-4.8	-4.8	-37.6	-36.5	-34.2
0 1		1.2	-14.4	-5.2	-4.8	-4.8	-32.6	-31.8	-29.8
<i>m</i> -Cresol		1.4	-18	-6.2	-5.9	-5.2	-41.7	-40	-39.7
	NDA-99	1.6	-16.3	-6.2	-5.9	-5.2	-35.6	-34.2	-34.2
		1.8	-14.7	-6.2	-5.9	-5.2	-30.1	-29.1	-29.5
		0.8	-25.3	-6.4	-5.7	-5.1	-66.7	-64.6	-62.5
	XAD-4	1	-21.8	-6.4	-5.7	-5.1	-54.3	-53	-51.6
n Crasal		1.2	-18.9	-6.4	-5.7	-5.1	-44.1	-43.5	-42.6
p-Clesol		1.4	-21.3	-6.3	-5.7	-4.8	-52.8	-51.3	-50.9
	NDA-99	1.6	-18.9	-6.3	-5.7	-4.8	-44.7	-43.7	-43.8
		1.8	-16.9	-6.3	-5.7	-4.8	-37.5	-37	-37.5
		0.4	-35.5	-4.9	-4.1	-3.8	-108	-104	-98.1
	XAD-4	0.6	-30.3	-4.9	-4.1	-3.8	-89.9	-86.6	-82.1
		0.8	-26.7	-4.9	-4.1	-3.8	-76.9	-74.5	-70.8
Phenoi		1	-22.6	-4.7	-3.4	-3.3	-63.3	-63.4	-59.8
	NDA-99	1.2	-19.8	-4.7	-3.4	-3.3	-53.5	-54.3	-51.2
		1.4	-17.5	-4.7	-3.4	-3.3	-45.3	-46.6	-44.0



Fig. 3. Relation curves for the adsorption capacities according to the same residual concentration (C_e) of 4.0 mmol/L, under the temperature of 283 K with solubilities of adsorbates.

3.3. Thermodynamic properties

The isosteric enthalpies were calculated with a derivative Van't Hoff equation [27]:

$$\log(C_{\rm e}) = -\log(K_0) + \left(\frac{\Delta H}{2.303RT}\right) \tag{4}$$

where ΔH is the isosteric enthalpy, *R* the gas constant, *C*_e the equilibrium concentration of solute in moles per liter at the temperature of *T* and *K*₀ is the constant.

The calculated isosteric adsorption enthalpies of phenolic compounds were always negative and tabulated in Table 4, the indicative of an exothermic process. And their magnitudes (<43 kJ/mol) show a physical adsorption process. The negative value of ΔG , then, indicated the spontaneous nature of all the adsorption processes. Finally, the negative values of the adsorption entropy were consistent with restricted mobilities of the adsorbed molecules of phenolic compounds as compared with those molecules in bulk [28].

3.4. Kinetic performance

Both of the modified materials including NDA-99 and NDA-150 are selected to investigate the kinetic properties towards phenol and *p*-cresol from the views of their relatively larger adsorption capacities, since the kinetics of adsorption is important from the view that it controls the process efficiency. The kinetic curves could be found in Fig. 4.

In the initial time-range, NDA-150 exhibits a faster adsorption process for its relatively larger pore size, which would facilitate adsorbate–molecule approach through pores to active sites of adsorbents [29].

In the time range of 0–20 min, for *p*-cresol, both adsorbents show a clear fluctuation in the adsorption amount on the solid phase, the possible reason lies in the solid phase extraction via the dry and hydrophobic solid and liquid interface and the competition caused by polar water molecule exerts simultaneously the hydrogen interaction onto adsorbates and adsorbents tested.



Fig. 4. Kinetic adsorption performance for phenol and p-cresol with the initial concentration of 1000 mg/L at the temperature of 283 K.

4. Conclusions

The adsorptions for phenol and cresols onto the polymeric adsorbents are exothermic prior processes. The hydrophobic interaction plays a most important role. The polar groups on the matrix of polymers, such as oxygen and amine, strengthen the adsorption affinity, with an extremely larger adsorption of NDA-99 and a little larger adsorption of NDA-150 to cresol. Thus, hydrogen interaction enhances the adsorption processes involving phenol and cresol. Larger pore size is of significance to the kinetic performance, a good example from NDA-150. In conclusion, both modified polymeric adsorbents such as NDA-150 and NDA-99 can be expected to successfully remove both phenol and cresol from aqueous media.

Acknowledgements

The work was supported by Research Institute of Environmental Sciences of Jiangsu Province, and Science & Technology Bureau of Nanjing University. The authors wish to express their appreciation to the Analytical Center at Jiangsu Industrial University, Yancheng Teachers College for the measurement of PSD and IR. Special thanks go to the kind reviewers for their good suggestions.

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