

Adsorption of catechol from aqueous solution by aminated hypercrosslinked polymers

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Abstract: Adsorption of catechol from aqueous solution with the hypercrosslinked polymeric adsorbent NDA-100 and its derivatives AH-1, AH-2 and AH-3 aminated by dimethylamine, the commercial resin Amberlite XAD-4 and weakly basic anion exchanger resin D301 was compared. It was found that the aminated hypercrosslinked resins had the highest adsorption capacities among the tested polymers. The empirical Freundlich equation was successfully employed to describe the adsorption process. Specific surface area and micropore structure of the adsorbent, in company with tertiary amino groups on matrix affected the adsorption performance towards catechol. In addition, thermodynamic study was carried out to interpret the adsorption mechanism. Kinetic study testified that the tertiary amino groups on the polymer matrix could decrease the adsorption rate and increase the adsorption apparent activation energy.

Keywords: hypercrosslinked polymer; catechol; adsorption; thermodynamics; kinetics

Introduction

Catechol, an important derivative of phenol is widely used as intermediates for manufacture of pesticides, medicines and dyestuffs (Zhang, 1992). Catechol is strongly irritant to eyes, skin and respiratory tract, and it is also the inducement of DNA damage, vascular collapse, coma and death (Arinjay, 2003). Wastewater from catechol producing will arouse serious environment problems because of its high strength and low biodegradability. Furthermore, it contains catechol, which is high valuable to reclaim. Consequently, the removal of catechol from such wastewater has become significant environmental and economic concern. During the past decades, resin Adsorption technology as a method of reusing resource has been widely adopted for the treatment of effluents containing phenolic compounds (He, 1992; Lin, 2002; Deosarkar, 2004). In comparison with such classical adsorbents as silica gels, aluminas and activated carbons, polymeric adsorbents are of particular interest for their high chemical stability, easy regeneration and excellent selectivity (He, 1992). Among them, the commercial resin Amberlite XAD-4 had ever been considered the best one for the removal of phenolic compounds in wastewater since it was developed (He, 1992; Xu, 1997). While, the hypercrosslinked polymers developed by Davankov (Davankov, 1990) showed better contact with aqueous phase and larger adsorption capacities towards phenols from aqueous solution (Tsyurupa, 2002; Xu, 1999). In recent years, scientists have made more efforts on chemical modification of polymeric adsorbents to improve their adsorption properties due to the presence of some interaction between adsorbates and adsorbents (Masque, 1997; Li, 2001; Pan, 2002; 2003). In the present study, three aminated hypercrosslinked polymeric adsorbents (AH-1, AH-2 and AH-3) were prepared to remove catechol from aqueous solutions, and further, the adsorption thermodynamics and kinetics were comparatively discussed.

1 Materials and methods

1.1 Chemicals

Catechol was purchased from Shanghai Chemical Reagent Plant (Shanghai, China) and dissolved by deionized water in adsorption tests without pH adjustment. The

chemicals used to synthesize hypercrosslinked polymeric adsorbent (NDA-100) and modified adsorbents (AH-1, AH-2 and AH-3) were all supplied by Langfang Electrical Resin Co. Ltd. (Hebei Province, China).

1.2 Adsorbents

Amberlite XAD-4 resin was purchased from Rohm and Haas (Philadelphia, PA, USA). The weakly anion exchanger resin D301 was supplied by Jiangsu Nange Environmental Technology Co. Ltd. (Jiangsu Province, China). The hypercrosslinked polymeric adsorbent NDA-100 together with the hypercrosslinked polymeric matrixes of AH-1, AH-2 and AH-3 was prepared by controlling the post-crosslinking reaction following the method performed by Li (Li, 2002). AH-1, AH-2 and AH-3 were obtained by amination of the residual chloromethyl groups on the different hypercrosslinked polymeric matrixes with dimethylamine (Pan, 2003). Prior to use, all the polymeric adsorbents tested were extracted by ethanol in a Soxhlet apparatus for 8 h and then dried under vacuum at 333 K for 8 h. The specific surface area and the pore size distribution of resins were measured with a Micromeritics ASAP-2010 surface area instrument (Micromeritics Instrument Corp., Norcross, GA, USA) with nitrogen as the adsorbate with the BET method.

1.3 Static adsorption

Static adsorption of catechol on the adsorbents at three temperatures (283, 298 and 313 K) was conducted as follows: 0.100 g of dry resin was introduced into a flask and 100 ml of aqueous solution of catechol was added. The initial concentrations (C_0) of the solutions were 100, 200, 400, 600, 800 and 1000 mg/L respectively. The flasks were completely sealed and placed in a constant temperature shaker (Taicang Guangming Experimental Instrument Co. Ltd., Jiangsu Province, China) at a preset temperature and shaken at 200 r/min for 24 h to ensure the adsorption process reaching equilibrium. The equilibrium concentration (C_e) of catechol was determined using a Helios Beta UV-Vis spectrometer (Unicam, Cambridge, UK). The corresponding equilibrium adsorption capacity Q_e (mmol/g) was calculated via the following equation:

$$Q_e = V(C_0 - C_e)/WM, \quad (1)$$

where V is the volume of solution (L), W is the mass of dry

resin(g) and M is the molecular weight of catechol.

1.4 Kinetic adsorption

Kinetic adsorption of catechol onto the hypercrosslinked resins NDA-100, AH-1, AH-2 and AH-3 was carried out in the way similar to the static adsorption tests, except for that the initial concentration of catechol was settled at 1000 mg/L in all cases at 298 K and 313 K. The samples were taken at different intervals of time and the instantaneous catechol uptakes on the resins were calculated by measuring the concentration of catechol in solution at different contact time.

2 Results and discussion

2.1 Characteristics of the polymeric adsorbents

The characteristics of the six adsorbents are presented in

Table 1 Characteristics of the six adsorbents

Property	XAD-4	NDA-100	AH-1	AH-2	AH-3	D301
Structure	Polystyrene	St-DVB*	St-DVB	St-DVB	St-DVB	St-DVB
Polarity	Nonpolar	Weak polar	Moderate polar	Moderate polar	Moderate polar	Polar
BET surface area, m ² /g	780	934	819	726	483	38
Micropore area ($r \leq 1$ nm), m ² /g	3	561	463	394	261	8
Micropore volume ($r \leq 1$ nm), cm ³ /g	0	0.25	0.21	0.18	0.12	0
Tertiary amino group, mmol/g	0	0	1.51	2.10	2.75	4.90
Average pore diameter, nm	11.6	2.4	2.4	2.6	3.0	9.0
Average particle size, mm	0.4—0.6	0.4—0.6	0.4—0.6	0.4—0.6	0.4—0.6	0.4—0.6
Colour	White	Yellow	Yellow	Brown	Brown	White

Note: * Abbreviated form of styrene-divinylbenzene

2.2 Static equilibrium adsorption

Fig. 1 shows the equilibrium adsorption isotherms of catechol on the adsorbents at the temperature of 283, 298 and 313 K, respectively. The adsorption capacity appeared to remarkably increase with increasing equilibrium concentration. The data were analyzed using the empirical Freundlich equation (Slejko, 1985):

$$\log Q_e = \log K_F + 1/n \log C_e, \quad (2)$$

where Q_e is the equilibrium adsorption capacity of the

adsorbent (mmol/g); C_e is the equilibrium concentration of adsorbate (mmol/L), K_F and n are the characteristic constants. All the isotherm parameters could be determined from the experimental data by plotting $\log Q_e$ against $\log C_e$ on the basis of Equation (2). The Freundlich isotherm parameters K_F , n , and the correlative coefficients R^2 were listed in Table 2.

All equations were reliable because all the correlative

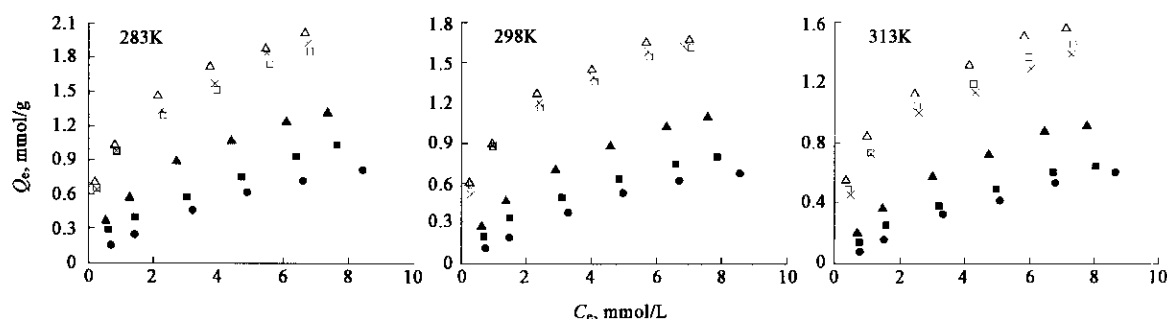


Fig.1 Adsorption isotherms of catechol onto (●) XAD-4, (■) D301, (▲) NDA-100, (□) AH-1, (△) AH-2 and (×) AH-3 at 283 K, 298K and 313K

coefficients (R^2) were larger than 0.98. The values of K_F , a relative indicator of adsorption capacity from Freundlich theory (Slejko, 1985), indicated that the adsorption capacities of the hypercrosslinked polymers including NDA-100, AH-1, AH-2 and AH-3 were higher than those of XAD-4 and D301 towards catechol. The exponent n was larger than 1 in all cases, an indication of favorable processes. The adsorption capacities (Q_e) on the adsorbents calculated with Freundlich equations corresponding to the

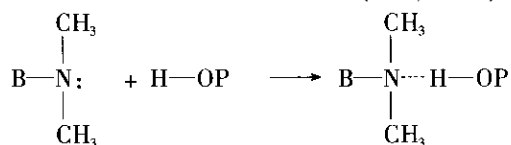
same residual concentration (C_e) of 3.0 mmol/L are also listed in Table 2. At 283K, when $C_e = 3.0$ mmol/L, the adsorption capacities of catechol on NDA-100, AH-1, AH-2 and AH-3 were 2.07—3.74 and 1.43—2.59 times of those on XAD-4 and D301 respectively. Further, the adsorption capacities of AH-1, AH-2 and AH-3 were 62.1%, 80.8% and 68.1% higher than that of NDA-100 respectively. Generally speaking, surface area, micropore structure, polarity as well as chemical surface heterogeneity of an

Table 2 Freundlich isotherms parameters for adsorption of catechol on the adsorbents

Adsorbent	Temperature, K	K_f	n	R^2	Q_e , mmol/g ^a
NDA-100	283	0.5109	2.028	0.9955	0.8782
	298	0.3774	1.834	0.9960	0.6870
	313	0.2682	1.585	0.9914	0.5366
AH-1	283	1.015	3.245	0.9987	1.424
	298	0.8742	3.113	0.9987	1.244
	313	0.7044	2.676	0.9966	1.062
AH-2	283	1.145	3.357	0.9970	1.588
	298	0.9410	3.202	0.9955	1.326
	313	0.8155	2.920	0.9966	1.188
AH-3	283	1.024	3.001	0.9988	1.476
	298	0.8474	2.777	0.9912	1.259
	313	0.6476	2.520	0.9841	1.001
XAD-4	283	0.2017	1.473	0.9969	0.4251
	298	0.1483	1.348	0.9931	0.3351
	313	0.1053	1.180	0.9908	0.2672
D301	283	0.3516	1.969	0.9873	0.6142
	298	0.2608	1.791	0.9961	0.4816
	313	0.1778	1.567	0.9951	0.3585

^a Calculated by isotherm equations when $C_e = 3.0$ mmol/L

adsorbent put much influence on its adsorption capacity (Li, 2001; 2002; Pan, 2003). The specific surface area of hypercrosslinked polymer NDA-100 as well as AH-1, AH-2 and AH-3 is comparable to or even lower than that of Amberlite XAD-4, but the capacity of the latter is much lower, thus the significant difference in adsorption capacity is not only related to the specific surface area. It is reasonable that the higher adsorption capacities of the hypercrosslinked polymers are attributed to the micropore structure in their networks (Lide, 1995; Mattson, 1969). As to AH-1, AH-2 and AH-3 with the moderate polarity, it may be possible that the hydrogen-bond interaction occurred between the tertiary amino group on adsorbents and the hydroxyl group of catechol comparing with NDA-100. The schematic representation of such interaction can be listed as follows (Pan, 2003):



where B represents the polymeric backbone and H-OP stands for catechol molecular. This is probably the primary reason for the increased adsorption capacities of the aminated hypercrosslinked polymers over that of NDA-100. In

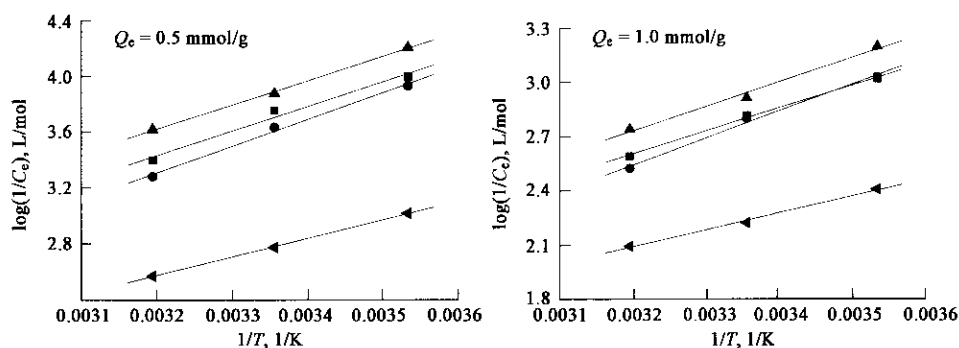


Fig. 2 Determination of isosteric enthalpy changes of adsorption towards catechol onto (▲) NDA-100, (■) AH-1, (▲) AH-2 and (●) AH-3 at adsorption capacities $Q_e = 0.5$ mmol/g and $Q_e = 1.0$ mmol/g

addition, the adsorption capacity towards catechol on AH-2, which has moderate content of tertiary amino groups, is highest among the three aminated hypercrosslinked polymers. It is because that both specific surface area and micropore region of the polymers decrease with increased content of tertiary amino groups. In a word, specific surface area, micropore structure, together with content of tertiary amino groups dominate the adsorption property of catechol on the aminated hypercrosslinked polymers, which is further supported by the fact that the adsorption capacity of catechol on D301 is much lower than all the aminated hypercrosslinked resins although it has the highest content of tertiary amino groups.

2.3 Thermodynamics of the adsorption of catechol onto NDA-100, AH-1, AH-2 and AH-3

As discussed above, the hypercrosslinked polymers demonstrated excellent adsorption capability towards catechol. The thermodynamic study of NDA-100, AH-1, AH-2 and AH-3 was thus carried out to explore the adsorption mechanism. The isosteric enthalpy change of adsorption herein was calculated by Van't Hoff equation (Garcla-Delgado, 1992):

$$\log(1/C_e) = \log K_e + (-\Delta H/2.303RT), \quad (3)$$

where C_e is the equilibrium concentration of solute in mol/L at the absolute temperature T ; ΔH is the adsorption isosteric enthalpy change and R is the gas constant. C_e was obtained from the well-suited isotherms corresponding to a definite Q_e value at different temperatures (283, 298 and 313 K). ΔH was then calculated from the slope of the line plotted by $\log(1/C_e)$ versus $1/T$, and some of the plotted lines are shown in Fig. 2.

The adsorption free energy change was calculated from the Freundlich isotherm by employing the Gibbs equation (Garcla-Delgado, 1992; Juang, 1999):

$$\Delta G = -nRT, \quad (4)$$

where n is the parameter of the Freundlich equation and ΔG is the free energy change in an adsorption process.

The adsorption entropy change then could be calculated via the Gibbs-Helmholtz relationship (Garcla-Delgado, 1992):

$$\Delta S = (\Delta H - \Delta G) / T. \quad (5)$$

Values of the isosteric enthalpy change (ΔH , kJ/mol), free energy change (ΔG , kJ/mol) and entropy change (ΔS , J/(mol·K)) calculated from the data obtained in the present study were thoroughly presented in Table 3.

Table 3 Calculated thermodynamic parameters for adsorption of catechol at different adsorption capacities within the temperature range of 283–313 K

Adsorbent	Q_e , mmol/g	ΔH , kJ/mol	ΔG , kJ/mol			ΔS , J/(mol·K)		
			283K	298K	313K	283K	298K	313K
NDA-100	0.5	-25.31	-4.77	-4.54	-4.12	-72.58	-69.70	-67.70
	1.0	-17.80				-46.04	-44.50	-43.71
AH-1	0.5	-33.76	-7.64	-7.71	-6.96	-92.30	-87.42	-85.62
	1.0	-24.18				-58.45	-55.27	-55.02
AH-2	0.5	-33.26	-7.90	-7.93	-7.60	-89.61	-85.00	-81.98
	1.0	-25.86				-63.46	-60.17	-58.34
AH-3	0.5	-36.72	-7.06	-6.88	-6.56	-104.81	-100.13	-96.36
	1.0	-28.55				-75.94	-72.72	-70.27

From Table 3, exothermic adsorption processes are testified by the negative values of all the enthalpy changes. The larger absolute values of adsorption isosteric enthalpy changes of AH-1, AH-2 and AH-3 mean the interactions between catechol and the aminated hypercrosslinked resins are stronger than that between catechol and NDA-100 due to the hydrogen-bonding interaction. The fact that the absolute values of the enthalpy changes decreased with increased loading indicates that the active sites on the adsorbent surface are not all equivalent energetically and that those with the greater binding energy are occupied firstly. The adsorption free energy changes are always negative proving that the adsorption processes of catechol on adsorbent surface are all spontaneous. In addition, the absolute values of adsorption free energy changes of the aminated hypercrosslinked resins are larger than that of NDA-100, suggesting that the adsorption of catechol is easier onto the former resins. The

entropy changes during the adsorption of catechol on all the employed adsorbents are negative, indicating that the activity of catechol molecules on the adsorbents is weaker than that in the aqueous solution. In comparison with NDA-100, the absolute values of the adsorption entropy changes of AH-1, AH-2 and AH-3 are large, which indicates that the catechol molecules are more tight and ordered on the aminated hypercrosslinked resins.

2.4 Kinetics of catechol adsorbed onto NDA-100, AH-1, AH-2 and AH-3

Kinetic sorption of catechol onto NDA-100, AH-1, AH-2 and AH-3 was carried out to explore the practicability of this type of resins in removal of catechol from water stream. The influence of contact time on the uptake of catechol onto NDA-100, AH-1, AH-2 and AH-3 was shown in Fig. 3.

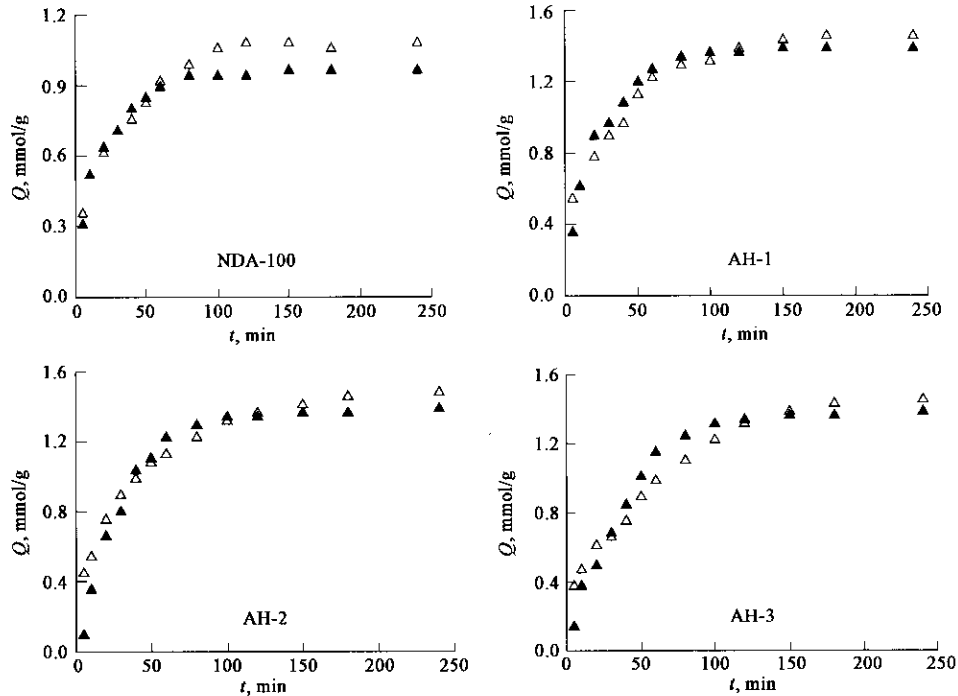


Fig. 3 Effect of contact time on the uptake of catechol onto NDA-100, AH-1, AH-2 and AH-3 at (Δ) 298 K and (\blacktriangle) 313 K ($C_0 = 1000$ mg/L)

The adsorption rate constants for the removal of catechol by the resins were determined using the first-order kinetic expression (Yu, 2001):

$$\ln(1 - Q_t/Q_e) = -k t, \quad (6)$$

where Q_t is the adsorption uptake (mmol/g) of catechol at time t ; Q_e is the equilibrium uptake (mmol/g); k and t are the rate constant and time respectively. The kinetic data obtained within 80 min were plotted relating $\ln(1 - Q_t/Q_e)$ with the contact time t at 298 and 313 K respectively, and k

was therefore obtained from the slope of the linear relationship. The apparent activation energy was calculated via the Arrhenius equation (Fu, 1990):

$$k = k_0 \exp(-E_a / RT), \quad (7)$$

where k is the rate constant (1/min); k_0 is a parameter related to temperature (1/min); E_a is the apparent activation energy (J/mol); R is the gas constant (8.314 J/(mol·K)); and T is the absolute temperature (K). The values of the rate constants and the correlation coefficients along with the

apparent activation energy are listed in Table 4.

Table 4 Kinetic parameters for adsorption of catechol onto NDA-100, AH-1, AH-2 and AH-3

Adsorbent	Temperature, K	$\ln(1 - Q_t/Q_e) = -k t$		E_a , kJ/mol
		k , min^{-1}	R^2	
NDA-100	298	0.0258	0.9806	24.82
	313	0.0417	0.9851	
AH-1	298	0.0238	0.9839	26.19
	313	0.0395	0.9881	
AH-2	298	0.0187	0.9899	32.70
	313	0.0352	0.9939	
AH-3	298	0.0148	0.9874	35.13
	313	0.0292	0.9833	

Fig. 3 shows the sorption equilibrium completed within about 4 h of contact time with the initial catechol concentration C_0 of 1000 mg/L and the higher temperature is favorable to attaining equilibrium but to low sorption uptake. From Table 4, it will be found that the adsorption of catechol onto the four resins conformed to be the first-order reaction because all the correlation coefficients (R^2) are higher than 0.98. Under the same temperature, the values of rate constants k decrease in the order: AH-3 < AH-2 < AH-1 < NDA-100. Some researchers (Marcus, 2000) had studied the effect of chemical surface heterogeneity on the adsorption of dissolved aromatic compounds on activated carbon and found that water adsorption happened on the hydrophilic, polar oxygen groups and then water clusters were built up on these groups by hydrogen-bonding interaction. The water clusters can effectively reduce the width of the pores and then the diffusion rate will slow down. Similarly it may occur on AH-1, AH-2 and AH-3 for the tertiary amino group on the resin matrix and then water adsorption can be formed by hydrogen-bonding interaction between water molecule and tertiary amino group to slow down the adsorption rate (Cornelia, 2001). The order of apparent activation energy required for the adsorption of catechol onto the four resins is: NDA-100 < AH-1 < AH-2 < AH-3. This may be attributed to the hydrogen-bonding interaction.

3 Conclusions

Surface modification of hypercrosslinked polymeric matrix with dimethylamine was studied and aminated derivatives AH-1, AH-2 and AH-3 were obtained. The adsorption capacities of catechol on the aminated hypercrosslinked resins AH-1, AH-2 and AH-3 are higher than that on the hypercrosslinked resin NDA-100. Specific surface area, micropore structure and content of tertiary amino groups play a combined role during the adsorption of catechol onto the aminated hypercrosslinked polymers.

Negative values of the isosteric enthalpy changes indicate that the adsorption towards catechol on the hypercrosslinked resins is exothermic. The adsorption free energy changes are always negative, demonstrating that the adsorption is a spontaneous process. The entropy changes are negative, indicating the weaker activity of catechol molecules on the adsorbents than that in the aqueous solution. The absolute values of the three thermodynamic parameters of

AH-1, AH-2 and AH-3 are all larger than those of NDA-100 indicating that the aminated hypercrosslinked resins exhibited better adsorption property towards catechol.

Kinetic sorption study of catechol on NDA-100, AH-1, AH-2 and AH-3 indicates a first-order kinetic mechanism for the adsorption system. The amination reaction on the hypercrosslinked polymeric backbone leads to a lower adsorption rate maybe for the water clusters built up. The values of apparent activation energy required for the adsorption increase with increased content of tertiary amino groups on the resin matrix for the occurrence of hydrogen-bonding interaction.

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