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Effect of ionic strength on the adsorption behavior of phenol over modified activated clay

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ABSTRACT

In this paper, the effect of several inorganic salts (NaCl, KCl, $MgCl_2$, $ZnCl_2$, Na_2SO_4, Na_3PO_4) on phenol adsorption at different ionic strength by cetyl trimethyl ammonium bromide and CaO modified activated clay was studied. The adsorption isotherm, adsorption kinetics and thermodynamic in the presence of NaCl were also investigated. The results showed that phenol removal efficiency has been influenced by the co-existed ions. With the increase of ionic strength from 0.5 to 3 $mol\,kg^{-1}$, the removal of phenol slightly increased, except for $ZnCl_2$. The adsorption kinetics showed that the adsorption of phenol can be represented by pseudo-second-order kinetic. The Freundlich model fitted the two processes well. The thermodynamic parameters showed that the two adsorption processes were both exothermic, spontaneous nature, randomness decreasing process.

Keywords: Modified activated clay; Adsorption; Phenol; Ionic strength

1. Introduction

With the development of industry, the discharge of wastewater is more and more serious. Phenol and its derivatives are important pollutants in wastewater, generally existing in chemical industries, especially in processing plants, paper mills, coal conversion plants, chemical manufacturers, pesticide, herbicides industries and plastic etc. [1–3]. Since phenol is poisonous to organisms even at very low concentration, it can destroy the structure of protein, cause headache, nausea, shock, memory loss and other symptoms, it is considered as priority pollutants [4,5]. Meanwhile, phenol also has low solubility and a weak ionization capacity [6]. Consequently, before discharge, it is necessary to remove phenol from wastewater [7].

There are many methods to remove phenol from wastewater, such as adsorption [8], biological degradation [9,10], photo-degradation [11], electro-oxidation [12], and ion exchange, etc. Among these methods, adsorption by clay mineral is the most common method.

With large specific surface area, strong adsorption and cation exchange, low cost and easy to obtain, activated clay (AC) is considered to be an efficient adsorbent to remove phenol from wastewater [13,14].

Phenol adsorption on AC is dependent on a large amount of conditions, such as pH, ionic strength, temperature, adsorbent dose and concentration of substrate

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[15]. In actual wastewater, there exists high concentration salt, so it is worth to investigate the adsorption behavior of phenol in the presence of salts [16,17].

Previous researches indicated the effect of ionic strength on the adsorption of phenol is complex. Al-Asheh et al. [2] reported either NaCl or KCl had no effect on the adsorption of phenol with different types of activated bentonites. Senturk et al. [18] found that the adsorption of phenol onto organo-bentonite decreased with the increase of salt concentration when various concentrations (0.01–0.20 M) of NaCl, Na_2SO_4 and $NaNO_3$ solutions added individually. To the best of our knowledge, the removal of phenol by organo and CaO modified activated clay (MAC) in the presence of various salts has not been systematically investigated.

In this paper, the effect of ionic strength on the adsorption of phenol onto cetyl trimethyl ammonium bromide (CTAB) and CaO MAC was studied. The adsorption kinetics, adsorption isotherms, and adsorption thermodynamics of adsorption of phenol in the presence of 1 mol kg^{-1} NaCl were also investigated.

2. Experimental methods

2.1. Reagent

All reagents used in this work were analytical grade. Stock solution of 2,000 mg L^{-1} phenol was prepared with distilled water. The AC purchased from Anhui province, China, was used as primary material.

2.2. Preparation of MAC

MAC was prepared by semi-dry method. In detail, AC (10 g, treated at 353 K for 4 h before modification) and CaO (3.5 g) mixed by grinding, with 20 ml distilled water added, calcined at 323 K for 12 h, washed with distilled water for several times, the filter cake was heated in an oven at 323 K and grounded (named as CAC). A mixture of CAC and CTAB in a 10:3 mass ratio were blended by grinding, with 20 mL distilled water added, then treated at 323 K for 12 h, washed with distilled water for several times, then heated at 323 K to obtain the final sample, designed as MAC.

2.3. Batch adsorption experiment

A certain amount of MAC adsorbent was transferred into 250 mL conical flasks containing 100 mL 100 mg L^{-1} of phenol aqueous solution with various type and concentration of salts, with a temperature-controlled shaker at a constant speed (800 rpm) and temperature. The samples were taken at predetermined time intervals and filtrated with filter paper. The phenol concentration was analysed by UVCPôvis spectrometer (Beijing new century, China) at $\lambda_{\max}$ 270 nm for phenol. The removal rate of phenol was calculated by the following equation:

$$\text{Removal rate } (\%) = (A_0 - A_t)/A_0 \times 100\% \tag{1}$$

where A_0 is the initial absorbance of phenol at $\lambda_{\max}$, A_t is the absorbance of phenol at time t (min) at $\lambda_{\max}$.

The ionic strength can be calculated according to Lewis Eq. (2):

$$I = 1/2 \times \sum m_B z_B^2 \tag{2}$$

where I (mol kg^{-1}) is the ionic strength, m_B (mol kg^{-1}) is the molality of B ion, z_B is the valence number of B ion.

$q_{e,\exp}$ was calculated by Eq. (3):

$$q_{e,\exp} = (C_0 - C_e)V/m \tag{3}$$

where c_0 (mg L^{-1}) is initial concentration of phenol, c_e (mg L^{-1}) is the concentration of phenol at equilibrium time, V (mL) is the volume of phenol, m (g) is the mass of adsorbent.

3. Results and discussion

3.1. Effect of various salts on phenol removal

The effect of various salts on phenol removal has been investigated systematically and the results are shown in Fig. 1. It can be found that the type of cations and anions have a significant effect on phenol removal.

The removal rate of phenol decreased with the introduction of various inorganic salts. However, with the increase of ionic strength from 0.5 to 3 mol kg^{-1}, the removal rate of phenol slightly increased, except $ZnCl_2$. The possible reason may be due to that, the presence of salts leads to polymerization of phenol molecules in solution and causes the molecules volume smaller and more hydrophobic, which is beneficial for phenol adsorption [16,19,20]. Meanwhile, the presence of salts can cause a salting-out effect. The dissociative ions in solution form well-organized ionic atmospheres through bind water molecule tightly, thus decreasing the solubility of non-electrolyte (phenol) in solution and improving the removal rate of phenol [21]. On the other hand, in the presence of

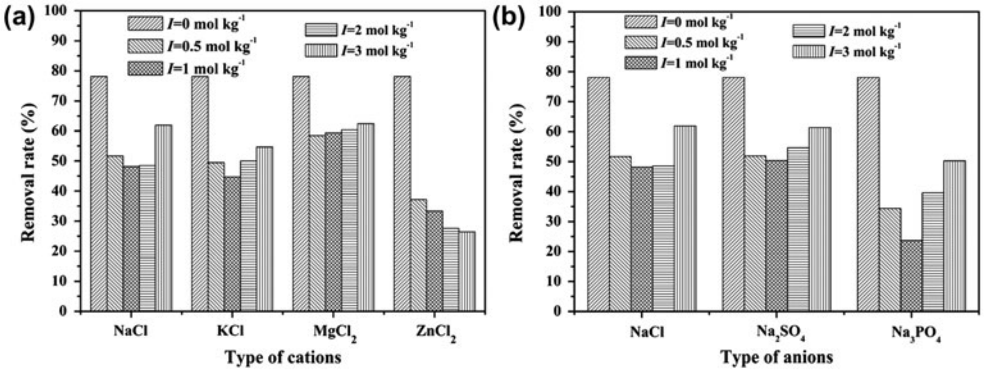

Fig. 1. The effect of different cations (a) and anions (b) on phenol removal (T: 298 K, c_0: 100 mg L^{-1}, V_{sol}: 100 ml, pH 6.8, m_{MAC}: 3.0 g).

salts, the active sites of MAC may be blocked, so phenol molecules are hindered to combine the surface of adsorbent, therefore, lead to a decline in phenol removal rate.

However, as for ZnCl$_2$, with the increase of ionic strength, the removal rate decreased, this mainly because of the decreasing of pH, as Fig. 2 shown. Under acid condition, the removal rate of phenol increased with the increase of pH, however, when the pH value exceeded 8, the adsorption of phenol decreased abruptly. Because as polar compound, phenol can be ionized into phenol anion at alkalinity condition. In the meanwhile, the surface of MAC was negatively charged when pH higher than the pH$_{\text{zpc}}$ of MAC (8.30), at higher pH values, and the electrostatic repulsion between MAC and phenol anion increases [18]. Therefore, phenol removal onto MAC decreased. The pH of solution decreased with the introduction of ZnCl$_2$. When pH lower than pH$_{\text{zpc}}$, the surface of MAC positive charged, which can adsorbed phenol molecular by hydrogen bond force, consequently, with the increase of pH lower than pH$_{\text{zpc}}$, the removal rate of phenol by MAC increased, as Fig. 3 shown.

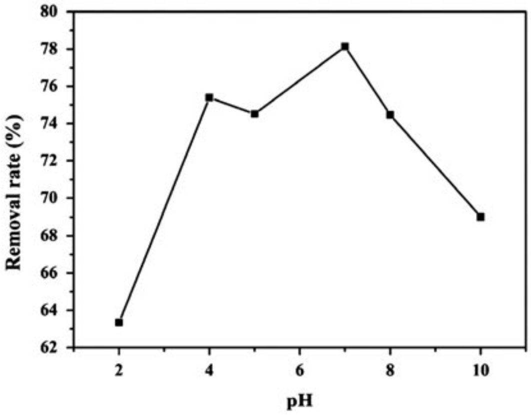

Fig. 2. The effect of pH on phenol removal.

Fig. 1 also shows the effect of various cations and anions on phenol removal. It can be found that as for different valence of cations, the removal rate of phenol in the presence of MgCl$_2$ (2–1) is higher than that of NaCl and KCl (1–1) in the same ionic strength. While for the anions, the higher valence salt (Na$_3$PO$_4$) has much lower removal rate than that of the lower ones (Na$_2$SO$_4$ and NaCl). This maybe suggests that the valence of cations has positive effect, while the valence of anions has the negative effect on phenol removal. More definite investigations need to be further studied.

3.2. Adsorption kinetics [22]

The pseudo-second order kinetic models, are applied to test the experimental data and to assess the adsorption mechanism involved in the absence ($I = 0$ mol kg^{-1}) and presence ($I = 1.0$ mol kg^{-1}) of NaCl for phenol adsorption process. The pseudo-second order models can be expressed as Eq. (4):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{4}$$

where q_t (mg g^{-1}) is the adsorption capacity of phenol at time t, q_e (mg g^{-1}) is the adsorption capacity of phenol at equilibrium time, k_2 (L mg^{-1} min^{-1}) is the rate constant of pseudo-second-order model. The parameters of pseudo-second-order kinetic are shown in Table 1. It can be seen that the pseudo-second-order model fitted the experimental data very well for both $I_{\text{NaCl}} = 0$ mol kg^{-1} and 1 mol kg^{-1}. However, the value of q_e and rate constant decreased with NaCl existed,

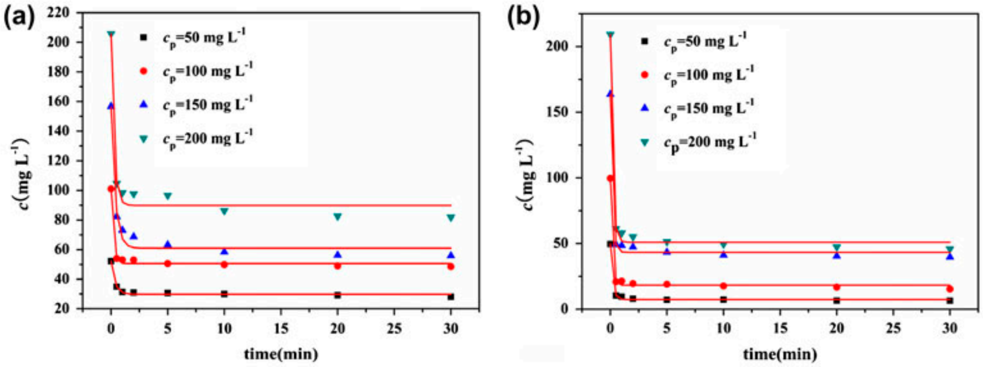

Fig. 3. Changes of phenol concentration with time for $I_{\mathrm{NaCl}} = 1\ \mathrm{mol\ kg^{-1}}$ (a) and $0\ \mathrm{mol\ kg^{-1}}$ (b).

Table 1
The kinetic parameters for adsorption of phenol in 0 and 1 mol kg^{-1} of NaCl

		$I = 0$ mol kg^{-1}				$I = 1$ mol kg^{-1}			
T (K)	c_0 (mg L^{-1})	$q_{e,\mathrm{exp}}$ (mg g^{-1})	q_e (mg g^{-1})	k_2 (L mg^{-1} min^{-1})	$R_2{}^2$	$q_{e,\mathrm{exp}}$ (mg g^{-1})	q_e (mg g^{-1})	k_2 (L mg^{-1} min^{-1})	$R_2{}^2$
298	50	1.44045	1.44361	7.46147	0.99994	0.80215	0.80090	3.00530	0.9990
	100	2.77819	2.77600	2.45582	0.99998	1.75104	1.75568	3.45680	0.9999
	150	4.13304	4.14164	2.15122	0.99994	3.36268	3.39305	1.01484	0.9999
	200	5.44143	5.44900	1.37749	0.99996	4.13304	4.16910	0.72096	0.9997
308	100	2.72683	2.72881	3.73484	0.99990	1.63121	1.64658	1.93982	0.9997
318	100	2.68681	2.57566	6.90452	1	1.50893	1.50996	1.87541	0.9994

showing that the adsorption ability and reaction rate of phenol decreased in the presence of NaCl.

Meanwhile, the apparent kinetic was studied and the apparent kinetic model can be expressed as Eq. (5):

$$r = -\frac{\mathrm{d}c_t}{\mathrm{d}_t} = kc_p^{\beta} \tag{5}$$

Take the logarithm on both sides:

$$\log r = \log k + \beta \log c_p \tag{6}$$

where r (mol^{-1} L^{-1} min^{-1}) is the reaction rate, c_p (mg L^{-1}) is the initial concentration of phenol, β is the reaction order for phenol.

The concentration of phenol changing with time for $I_{\mathrm{NaCl}} = 1$ and $0\ \mathrm{mol\ kg^{-1}}$ are showed in Fig. 3. The reaction orders calculated in the presence of NaCl is 1.31479, while 1.00076 for the absence of NaCl. This result indicated the presence of salt has played a significant role on phenol adsorption process.

3.3. Adsorption isotherm

Several widely used isotherm models: Langmuir, Freundlich and Temkin were applied to study the mechanism of adsorption system. Langmuir model is based on the assumption that adsorbent surface is homogeneous, adsorbate molecules are independent and the adsorption is monolayer [23]. The Langmuir isotherm model can be expressed by the following equation:

$$\frac{c_e}{q_e} = \frac{1}{q_m + K_L} + \frac{c_e}{q_m} \tag{7}$$

where q_m (mg g^{-1}) is the maximal adsorption capacity, q_e (mg g^{-1}) is the equilibrium adsorption capacity, K_L (L mg^{-1}) is a constant related to the free energy of the adsorption, c_e (mg L^{-1}) is the equilibrium concentration of phenol solution. q_m and K_L can be determined by the intercept and the slope of the linear plot of c_e/q_e vs. c_e.

Freundlich model is an empirical equation representing heterogeneous adsorption on the adsorbent surface [24], which are widely applied for physical adsorption, chemical adsorption and solution adsorption. The Freundlich model can be expressed by the following equation:

$$\ln q_e = \ln K_F + \frac{\ln c_e}{n} \tag{8}$$

where q_e (mg g^{-1}) is the equilibrium adsorption capacity, c_e (mg L^{-1}) is the equilibrium concentration of phenol solution, K_F ((mg g^{-1})(mg L^{-1})^n) is the Freundlich adsorption equilibrium constant, $1/n$ is the heterogeneity constant. K_F and 1/n can be calculated by the intercept and the slope of the liner plot of ln q_e vs. ln c_e, respectively.

The Temkin model can be represented by the following equation:

$$q_e = A + B \ln c_e \tag{9}$$

where $B = RT/b$, b (J mol^{-1}) is a constant related to adsorption heat, A (L g^{-1}) is the Temkin constant, R (8.314 J mol^{-1} K^{-1}) is the gas constant, T (K) is the temperature. A and B can be obtained by the intercept and the slope of the liner plot of q_e vs. lnc_e.

The experimental data fitted Langmuir, Freundlich and Temkin isothermal models and the parameters are given in Table 2. It can be found that the Freundlich model fitted the two adsorption processes best. When n value lies in the range of 1–10, indicating a favorable adsorption of phenol [24], the calculating values of n are 0.99899 and 1.56387, respectively, demonstrating that the phenol adsorption in the presence of NaCl is unfavorable, however, as for the phenol adsorption without NaCl, is a favorable process. This result also can be identified by Fig. 1.

The Langmuir model does not describe isotherm data properly, which considers that all the adsorption sites are identical and each site retains one molecule of the given compound [25]. Besides, the solid surface has a limited adsorption capacity $q_{\max}$, and the isotherm reaches a plateau. In this paper the curves (Fig. 4) which describe phenol adsorption for both conditions of $I_{\text{NaCl}} = 1$ mol kg^{-1} and 0 mol kg^{-1} on MAC are nearly straight line, without a plateaus, indicating that phenol adsorption on MAC are not limited to monolayer adsorption [26].

3.4. Adsorption thermodynamics

The activation energy (E_a) can be calculated by Arrhenius Eq. (10):

$$\ln k = \ln A - \frac{E_a}{RT} \tag{10}$$

where k (L mg^{-1} min^{-1}) is the rate constant of pseudo-second-order model, A is the pre-exponential factor, T (K) is the temperature, R (8.314 J mol^{-1} K^{-1}) is the gas constant, E_a (kJ mol^{-1}) is the activation energy of phenol adsorption. E_a can be determined by the slope of the linear plot of ln(k) vs. $1/T$, as shown in Fig. 5.

The adsorption thermodynamics were investigated to better understand adsorption behaviour.

The standard enthalpy change ΔH° of phenol adsorption can be calculated by Eq. (11) [27]:

$$\ln(K_p) = -\frac{\Delta H^\circ}{R}\frac{1}{T} + \text{Con} \tag{11}$$

where T (K) is the temperature, R is the gas constant (8.314 J mol^{-1} K^{-1}), K_p is the adsorption interaction equilibrium constant.

The plot of ln(K_p) against $1/T$ should be linear, and the slope equal to $-\Delta H^\circ/R$. The standard free energy change (ΔG°) and the standard entropy change (ΔS°) can be calculated by Eq. (12) and Eq. (13).

Table 2
The adsorption isothermal parameters of phenol adsorption onto MAC

Langmuir			Freundlich			Temkin		
$q_{\max}$ (mg g^{-1})	K_L (L mg^{-1})	R^2	K_F	n	R^2	A	B	R^2
$I_{\text{NaCl}} = 1$ mol kg^{-1}								
31.726	0.00131	0.054	0.039	0.998	0.920	−7.557	2.555	0.818
$I_{\text{NaCl}} = 0$ mol kg^{-1}								
13.153	0.0159	0.852	0.448	1.564	0.985	−3.639	2.424	0.903

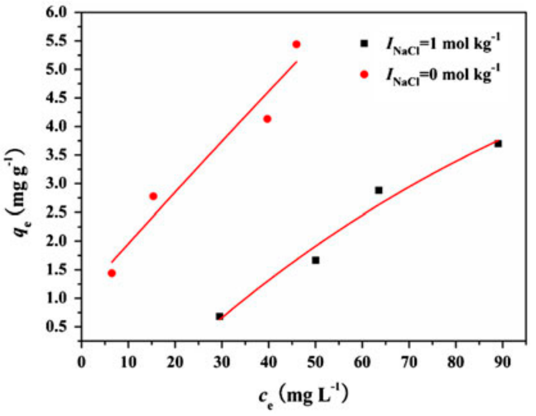

Fig. 4. Isotherms of phenol adsorption onto MAC.

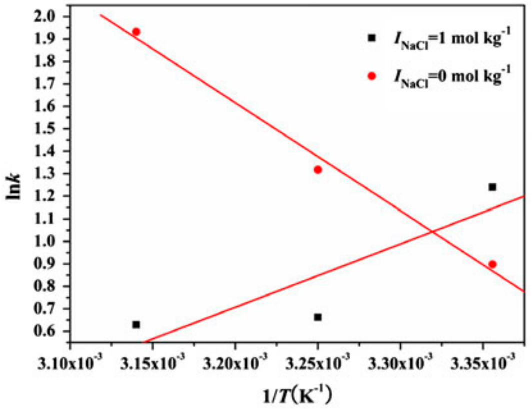

Fig. 5. The relationship of ln$k$ vs. $1/T$.

$$\Delta G^\circ = -RT\ln(K_p) \tag{12}$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{13}$$

Alternatively, ΔS° can be derived directly from the plot $\ln(K_p)$ against $1/T$. Indeed, assuming that both ΔH° and ΔS° change only slightly with the temperature, it can be obtained Eq. (14):

$$\ln(K_p) = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{14}$$

where K_p can be calculated from Eq. (15):

$$K_p = \frac{C_s}{C_l} \tag{15}$$

where C_s is the amount of phenol (mg) adsorbed on the adsorbent per L of the solution at equilibrium. C_l is the equilibrium concentration (mg L^{-1}) of phenol in the solution.

The plot of $\ln K_p$ vs. $1/T$ was linear and the values of slope and intercept of this line provide ΔH° and ΔS°, respectively, as shown in Fig. 6. These values were used to calculate ΔG° according to Eq. (13).

The thermodynamic parameters are listed in Table 3. It can be found that the E_a value is negative (-23.43 kJ mol^{-1}) for $I_{\text{NaCl}} = 1$ mol kg^{-1}, while positive for the absence of NaCl, maybe due to that the electrostatic interaction facilitate the weak electrolyte phenol migration rate and improve the adsorption process [20].

The positive value of ΔH° indicates that the adsorption is endothermic process, while negative value implies an exothermic adsorption process [28]. In this paper, the values of ΔH° are -5.46 and -10.83 kJ mol^{-1} for $I_{\text{NaCl}} = 1$ and 0 mol kg^{-1}, respectively, indicating that the phenol adsorption in the two processes are both exothermic processes. However, the difference in ΔH° value indicates that in the presence of NaCl, the degree of reaction heat release decreased. The negatively values of ΔG° indicate the adsorption is a feasibility and spontaneous nature process. Furthermore, the values of ΔS° are both negative (-17.01 and -22.79 J mol^{-1} K^{-1}) for the two adsorption processes, suggesting the randomness decreased during adsorption. Meanwhile, ΔS° value also states the degree of randomness decrease is different for two processes. Consequently, in the presence of NaCl, the degree of reaction heat release and randomness are both decreased, indicates the ionic strength can restrain the adsorption and the adsorption process is complex.

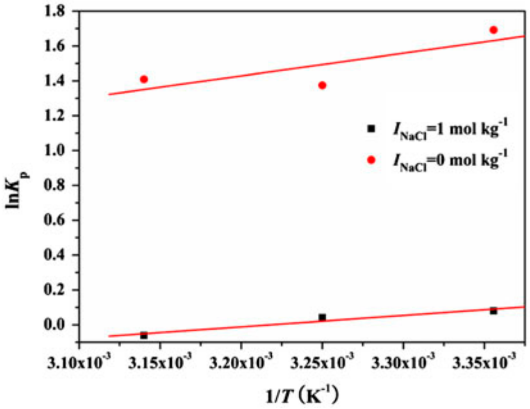

Fig. 6. The relationship of ln K_c vs. $1/T$.

Table 3
The thermodynamic parameters of phenol adsorption onto MAC

	$I_{\mathrm{NaCl}} = 1\ \mathrm{mol\ kg^{-1}}$					$I_{\mathrm{NaCl}} = 0\ \mathrm{mol\ kg^{-1}}$				
T (K)	K_p	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (Jmol^{-1} K^{-1})	E_a	K_p	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (Jmol^{-1} K^{-1})	E_a
298	1.08	−0.39	−5.46	−17.01	−23.43	5.43	−4.04	−10.83	−22.79	39.89
308	1.04	−0.22				3.95	−3.81			
318	0.94	−0.051				4.09	−3.58			

4. Conclusion

In this study, MAC was directly prepared with CTAB and CaO MAC with semi-dry method. The prepared MAC was tested for its adsorption capacity to remove phenol from aqueous solutions in the presence of various salts and ionic strength, and the experiments indicated that MAC possesses excellent adsorption capacity for phenol removal from aqueous solution. The results also indicated that the existence of salts had a negative effect on phenol abatement. However, the removal rate of phenol increased with the increase of ionic strength. The kinetics and isotherm data obtained from the experimental study can be well fitted with the pseudo-second-order kinetic model and the Freundlich isotherm. However, the different values of n indicated that the existence of NaCl has an unfavorable adsorption for phenol comparing with the absence of NaCl. Meanwhile, the differences of thermodynamic parameters indicate that the adsorption process is complex and various. This study demonstrated that CTAB and CaO modified by AC is a promising adsorbent for environmental pollutants, even if in the presence of inorganic salts.

Acknowledgments

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