



## Sorption of *p*-nitrophenol by anion–cation modified palygorskite

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### ABSTRACT

A series of anion–cation modified palygorskites were prepared by microwave irradiation, and characterized with FT-IR. Compared with natural palygorskite, the *p*-nitrophenol sorption capacity with anion–cation modified palygorskite was significantly improved. The maximum sorption capacity ( $137.74 \text{ mg g}^{-1}$ ) was obtained with the mass ratio 1:4 and 3:10 of hexadecyltrimethylammonium bromide to sodium dodecyl sulfate and the surfactant to palygorskite of at 313 K. The sorption equilibrium was in accordance with the Langmuir isothermal sorption equations. Thermodynamic function calculated showed that the sorption was a spontaneous and exothermic process. Free energy of the sorption increased with the increase of the temperature. The sorption of *p*-nitrophenol followed the pseudo-second-order process (sorption rate constant:  $0.00453 \text{ mg}^{-1} \text{ min}^{-1}$ ).

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### 1. Introduction

*p*-Nitrophenol is a common pollutant found particularly in the effluents from pesticides, pharmaceuticals, petrochemicals and other industries [1]. Its presence in the environment has become of great concern in recent years because of the increased amount of discharge, its toxic nature and adverse effects on the receiving water bodies. Hence, the removal of this organic pollutant from the water stream is critical to ensure the safety of water supplies. A considerable effort has been made concerning the removal of *p*-nitrophenol from wastewater. Several methods have been proposed and developed, and the most extensively used was sorption process. Various types of adsorbent, such as activated-carbon [2], organobentonite [3–5], kaolinite-type clay [6], polymeric adsorbents [7–9], chitosan and modified chitosan [10–11], membrane [12], silica and modified silica [13–15], charred saw-dust [1], and fly ash [16] have been developed.

Compared with other adsorbents, clay minerals are less expensive and more efficient due to their chemical and mechanical stability, high surface area and their structural properties. Palygorskite is a hydrated magnesium silicate mineral with a fibrous morphology, and with a structure consisting of parallel ribbons of 2:1 layers. In contrast to the close relative sepiolite, palygorskite contains cations other than  $\text{Mg}^{2+}$  in the octahedra sheets in insignificant amounts, such as  $\text{Al}^{3+}$  and  $\text{Fe}^{3+}$ . The presence of trivalent cations in octahedral positions creates an excess of positive charge,

which is compensated by vacancies, conferring dioctahedral characteristics to the mineral [17]. In recent years, palygorskite and modified palygorskite have been used as important adsorbents for heavy metal ions [18–21] and organic contaminated wastewaters [22,23].

Studies have found that the mixture of the anionic and cationic surfactants can form mixed micelles, which can produce synergies solubilization to organic compounds [5]. With this in mind, we used the anionic and cationic surfactants to modify palygorskite in an attempt to increase the synergies between the organic matter and the adsorbent to enhance the adsorption capacity. In this study, modified palygorskite was prepared by incorporating cationic and anionic surfactants to the palygorskite and modified by microwave irradiation. The sorption properties of *p*-nitrophenol on the modified palygorskite were investigated systematically.

### 2. Experimental

#### 2.1. Materials and instruments

Palygorskite (PGS) was purchased from Yixing Gaojing Chemical Corp. (Jiangsu, China). *p*-Nitrophenol, the cationic surfactants of tetramethylammonium bromide (TMAB), hexadecyltrimethylammonium bromide (HDTMAB), dihexadecyl dimethyl ammonium bromide (DDMAB) and the anionic surfactant of sodium dodecyl sulfate (SDS), were purchased from Tianjin Chemical Corp. (Tianjin, China) and used without further purification.

The FT-IR spectra were recorded on an AVATAR FT-IR spectrophotometer (Nicolet, USA). The acidity analyses of solution were performed on a PHS-3C meter (Shanghai, China). The concentra-

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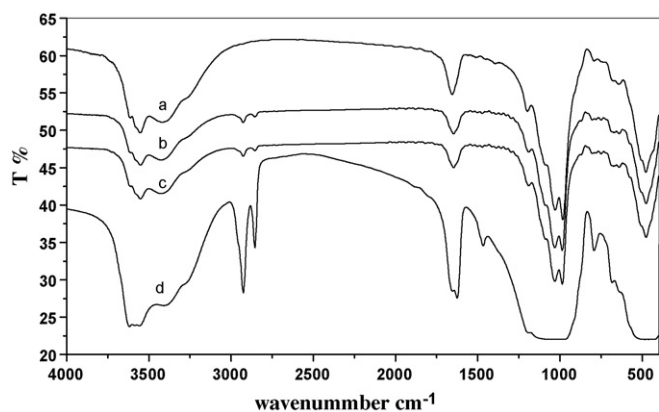


Fig. 1. IR spectra of PGS (a), TMAB-SDS-PGS (b), HDTMAB-SDS-PGS (c) and (d) DDMAB-SDS-PGS.

tion analyses of *p*-nitrophenol were measured on an UV-8453A spectrophotometer (Agilent, USA) at  $\lambda = 318$  nm. The sorption experiments of *p*-nitrophenol were carried out using a SHA-B temperature-controlled mechanical shaker (Jiangsu, China).

## 2.2. Preparation of surfactant-modified palygorskite

Dried palygorskite (5 g) was acidified by HCl (20 mL, 1 mol L<sup>-1</sup>), and mixed with 40 mL of aqueous solution containing 1 g of surfactant. The mixture was stirred in a water bath at 365 K for 3 h. The mixture was put into a 320 W microwave oven and irradiated for 3 min. The mixture was filtrated and washed with distilled water, and 4.8 g modified palygorskite was obtained after dried at 353–363 K. After activated for 1 h at 378 K, the modified palygorskite mechanically was whetted to 100 meshes with a mortar pestle [5].

## 2.3. Sorption of *p*-nitrophenol

Modified palygorskite (20 mg) was added to the solution of *p*-nitrophenol (100 mg/mL, pH: 7.0–8.0). The mixture was shaken at 293 K for 30 min to reach equilibrium. The aqueous phase was separated by centrifugation at 3500 rpm for 30 min. The equilibrium concentrations of *p*-nitrophenol were measured using the aliquots of supernatant withdrawn from the samples.

The sorption capacity was calculated using the following equation:

$$Q_e = \frac{C_0 - C_e}{m} V_L \quad (1)$$

where  $C_0$  and  $C_e$  denote the initial and equilibrium concentration (mg L<sup>-1</sup>), respectively.  $Q_e$  refers to the amount of *p*-nitrophenol adsorbed on the adsorbents (mg g<sup>-1</sup>),  $V_L$  is the volume of the aqueous solution (L), and  $m$  is the mass of dry adsorbents (g).

## 3. Result and discussion

### 3.1. FT-IR spectra

As shown in Fig. 1, the bands (Fig. 1b–d) at 2924–2856 cm<sup>-1</sup> of modified palygorskite were attributed to the stretching vibrations of C–H groups [23], and no band (Fig. 1a) appeared in this region in unmodified palygorskite spectrum.

### 3.2. Sorption properties

Fig. 2 showed that the sorption capacity of *p*-nitrophenol with HDTMAB-SDS and microwave modified palygorskite exhibited the

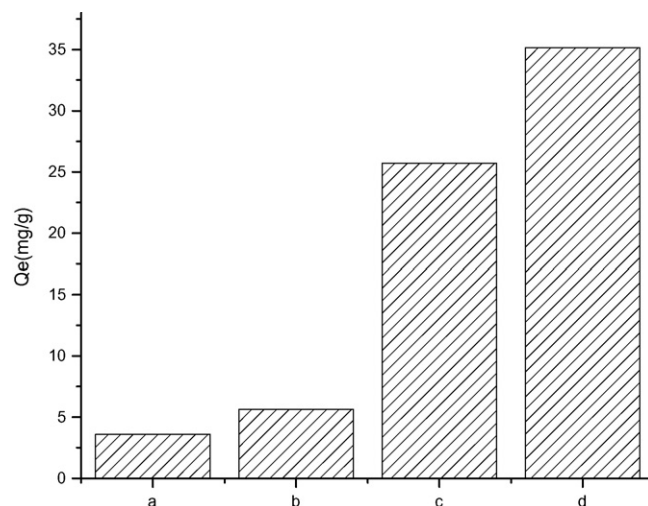


Fig. 2. Sorption capacity of (a) PGS, (b) HDTMAB-PGS, (c) HDTMAB-SDS-PGS and (d) HDTMAB-SDS-PGS ((a)–(c) were prepared by conventional method, but (d) was prepared by microwave irradiation) (the amount of the adsorbent: 20 mg; temperature: 293 K; initial concentration of *p*-nitrophenol: 100 mg/ml; pH: 7.0–8.0).

highest value among the four types of palygorskite. After sorption of the surfactants, the hydrophilic surface of the palygorskite was transformed to hydrophobicity, which has a great affinity for nonionic organic compounds. The result is consistent to the literature [5]. This may due to the mixed surfactants on anion–cation organopalygorskite created a partition medium for *p*-nitrophenol in water. The sorption capacity of *p*-nitrophenol is improved by synergistic solubilization in both anion and cation surfactants of the organopalygorskite. So, the sorption capacity of the anion–cation organopalygorskite is higher than that of the natural palygorskite and cation–palygorskite.

### 3.2.1. Effect of the type of the surfactants on the sorption

A comparative study of the effect of the type of cationic surfactants on the sorption capacities of *p*-nitrophenol was undertaken. The result of sorption capacities of *p*-nitrophenol of modified palygorskite with anionic surfactant SDS and different cationic surfactants were given in Fig. 3. Fig. 3 showed that the highest and lowest values of the sorption capacity were obtained with HDTMAB-SDS modified and DDMAB-SDS modified palygorskite, respectively. The possible reason of the low value of DDMAB-

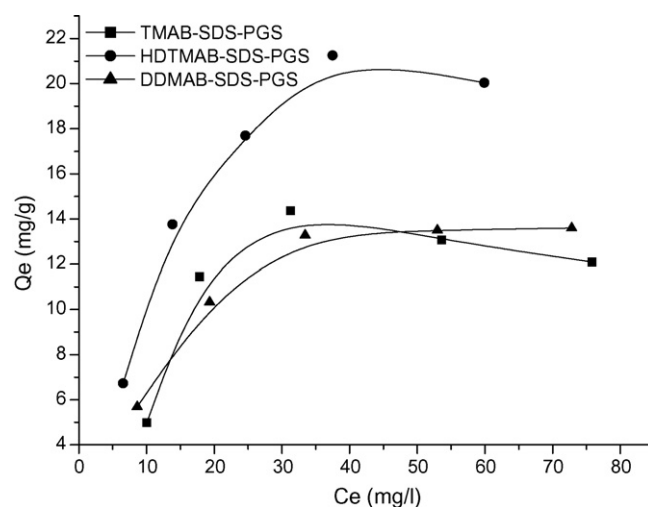
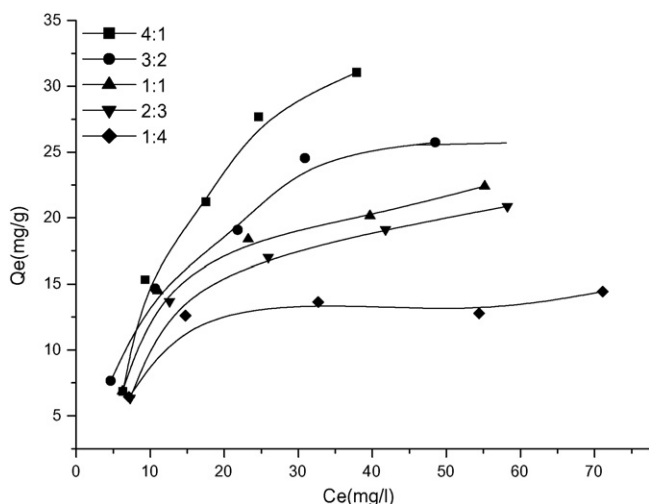


Fig. 3. Effect of the type of the cationic surfactant (temperature: 293 K; adsorbent amount: 20 mg; pH: 7.0–8.0).



**Fig. 4.** Effect of the ratio of sodium dodecyl sulfate to hexadecyltrimethylammonium bromide on the adsorption (temperature: 293 K; adsorbent amount: 20 mg; pH: 7.0–8.0).

SDS modified palygorskite is that the two big hexadecyl groups of DDMAB could prevent the entrance of *p*-nitrophenol to palygorskite interlayer.

### 3.2.2. Effect of the ratio of anionic surfactant to cationic surfactant on sorption

The relationship of the sorption capacity of modified palygorskites with the ratio of SDS to HDTMAB was shown in Fig. 4. Fig. 4 indicated that the best ratio of the anionic surfactant to the cationic surfactant was 4:1. As well-known, palygorskite contains cations other than  $Mg^{2+}$  (such as  $Al^{3+}$  and  $Fe^{3+}$ ) in the octahedral sheets in insignificant amounts. The presence of trivalent cations in octahedral positions creates an excess of positive charge, so the anionic surfactant can be interacted by electrostatic sorption, while the cationic surfactant can be interacted by ion-exchange reactions and hydrophobic interaction with palygorskite [17]. The value of anionic surfactant sorption capacity would be higher than that of the cationic surfactant [24]. So, the higher ratio of anionic surfactant to cationic surfactant in our experiments should improve the sorption capacity of the organopalygorskite.

### 3.2.3. Effect of the amount of the surfactants

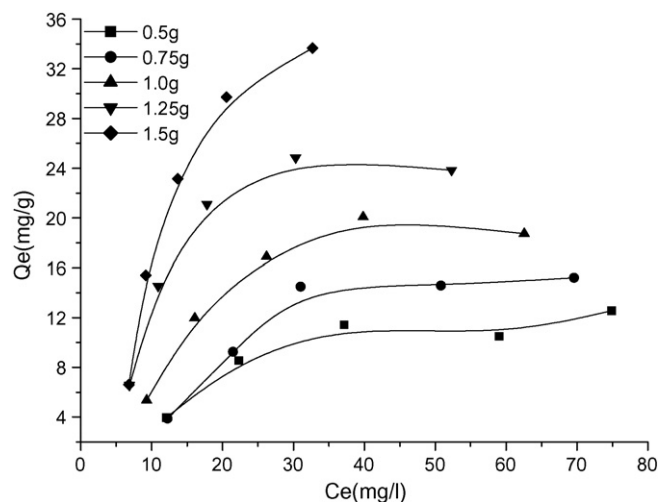
The effect of the amount of the surfactants on the sorption capacity of *p*-nitrophenol on the HDTMAB-SDS (mass ratio was 1:4) modified palygorskite showed in Fig. 5. The sorption capacity increased with the increase of the amount of the surfactant supported to palygorskite in the conditions of our experiments.

### 3.2.4. Effect of the microwave irradiation time on sorption capacity

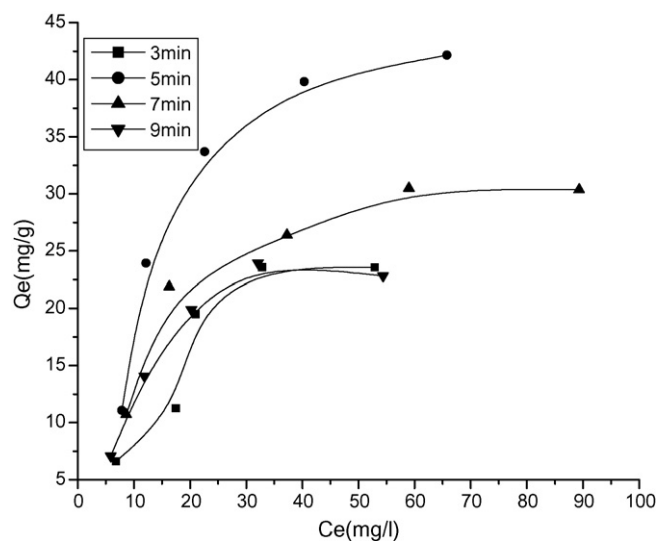
The time and energy consumption of the preparation of the anion-cationic organopalygorskite could be reduced with the microwave irradiation (Fig. 6). The sorption capacity increased with the increase of the microwave irradiation time within 5 min, but after 5 min, the sorption capacity decreased. In addition, the preparation of organopalygorskite would exceed the optimum temperature after 5 min microwave irradiation. The optimum microwave irradiation time was 5 min in the conditions of our experiments.

### 3.2.5. Effect of the adsorbent amount

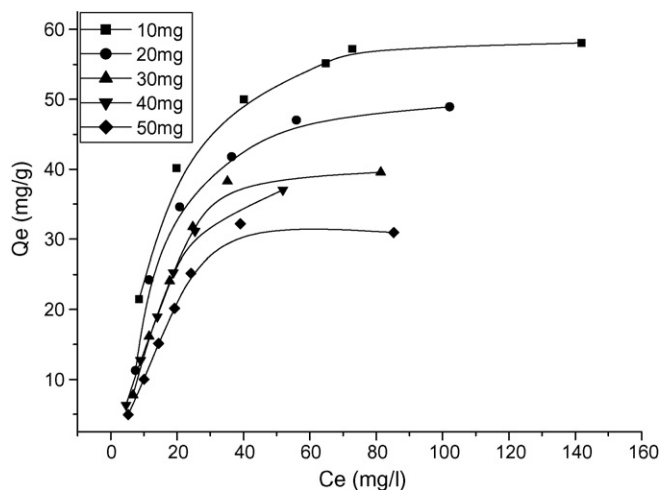
The effect of adsorbent amount on the adsorption capacity of *p*-nitrophenol was investigated (Fig. 7). Obviously, the sorp-



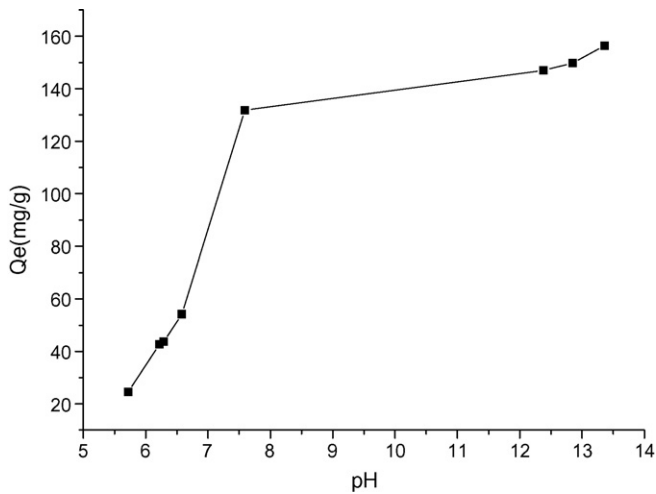
**Fig. 5.** Effect of the amount of the surfactant supported on 5 g of palygorskite (temperature: 293 K; adsorbent amount: 20 mg; pH: 7.0–8.0).



**Fig. 6.** Effect of the microwave irradiation time on adsorption of *p*-nitrophenol by modified palygorskites (temperature: 293 K; adsorbent amount: 20 mg; pH: 7.0–8.0).



**Fig. 7.** Effect of the amount of sorbent on adsorption (temperature: 293 K; anionic surfactant/cationic surfactant = 4/1; irradiation time, 5 min; pH: 7.0–8.0).



**Fig. 8.** Effect of pH on adsorption of *p*-nitrophenol by modified palygorskites (temperature: 293 K; anionic surfactant/cationic surfactant = 4/1; irradiation time, 5 min).

tion capacity of *p*-nitrophenol on anion–cation organopalygorskite decreased rapidly with an increase in the amount of anion–cation organopalygorskite.

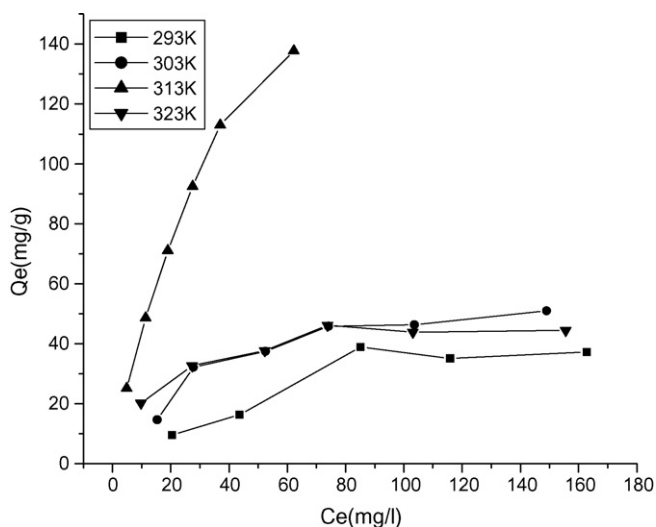
### 3.2.6. Effect of pH on sorption

The sorption behavior of *p*-nitrophenol on the modified palygorskite was studied from pH 5.7 to pH 13.6 (Fig. 8). The uptake of *p*-nitrophenol was observed when the pH is higher than 7.5. In order to avoid further contamination of high concentration of basic solution, pH of 7.0–8.0 was selected in this study.

### 3.3. Sorption isotherms

The sorption behaviors of *p*-nitrophenol at different temperatures were studied (Fig. 9). The sorption capacity increased with the increase of the temperature from 293 K to 313 K, and the sorption capacity decreased above 313 K.

Sorption isotherms show how solutes interact with adsorbents and so are critical in optimizing the use of adsorbents. Thus, the correlation of equilibrium data by either theoretical or empiri-



**Fig. 9.** The adsorption isotherms of *p*-nitrophenol onto modified palygorskites at different temperature (adsorbent amount: 10 mg; anionic surfactant/cationic surfactant = 4/1; irradiation time, 5 min; pH: 7.0–8.0).

**Table 1**

The Langmuir isotherm equations and corresponding parameters of *p*-nitrophenol.

T (K)	Regression equation	R	b	Q <sub>m</sub> (mg/g)
293	$1/Q_e = 0.0124 + 1.903/C_e$	0.992	0.00652	80.65
303	$1/Q_e = 0.0115 + 0.798/C_e$	0.965	0.0144	86.96
313	$1/Q_e = 0.0047 + 0.171/C_e$	0.999	0.0275	212.77
323	$1/Q_e = 0.0199 + 0.292/C_e$	0.994	0.0681	50.25

**Table 2**

The Freundlich isotherm equations and corresponding parameters of *p*-nitrophenol.

T (K)	Regression equation	R	K	n
293	$\log Q_e = 0.0696 + 0.711 \log C_e$	0.970	1.174	1.406
303	$\log Q_e = 0.677 + 0.502 \log C_e$	0.923	4.753	1.992
313	$\log Q_e = 0.959 + 0.683 \log C_e$	0.994	9.099	1.464
323	$\log Q_e = 1.053 + 0.296 \log C_e$	0.947	11.298	3.378

cal equations is essential to the practical design and operation of sorption systems. Langmuir isotherm equation (2) and Freundlich isotherm equation (3) were used to model the sorption equilibrium data:

$$\frac{1}{Q_e} = \frac{1}{Q_m} + \frac{1}{bQ_m C_e} \quad (2)$$

$$\log Q_e = \log K + \frac{1}{n} \log C_e \quad (3)$$

In the above equations,  $Q_m$  is defined as the maximum amount of *p*-nitrophenol per unit weight of modified palygorskites ( $\text{mg g}^{-1}$ ),  $b$  as the Langmuir constant related to the affinity of the binding sites.  $K$  and  $1/n$  are designated as Freundlich constants related to the sorption capacity and the sorption efficiency, respectively.

The calculated values of the Langmuir and Freundlich sorption isotherm constants and correlation coefficients,  $R$  are listed in Tables 1 and 2. Based on the correlation coefficient obtained, the Langmuir equation gives a better fit ( $R > 0.99$ ) to the experimental data than the Freundlich equation. The exponent  $n$  indicates the favorability of the sorption. If the sorption intensity,  $n$ , is more than 1, it shows a favorable sorption condition. Based on Table 2, the  $n$  values obtained for the sorption of *p*-nitrophenol onto modified palygorskites, shows a favorable sorption.

### 3.4. Sorption thermodynamics

The Gibbs free energy ( $\Delta G$ ), the enthalpy ( $\Delta H$ ) and the entropy ( $\Delta S$ ) for the sorption process were obtained using the formulas as follow:

$$\Delta G = -RT \ln K \quad (4)$$

$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (5)$$

where  $R$  is defined as the ideal gas constant ( $\text{kJ mol}^{-1} \text{K}^{-1}$ ),  $T$  as temperature in K, and  $K$  as the sorption constant. The enthalpy ( $\Delta H$ ) and the entropy ( $\Delta S$ ) can be calculated from a plot of  $\ln K$  vs.  $1/T$ . Sorption thermodynamics parameters are listed in Tables 3 and 4. They show that the free energy of the process at all temperatures is negative and decreases with the increase of temperature, thus the sorption process is spontaneous and the overall process seems to be endothermic. The positive entropy change ( $\Delta S$ ) indicates that the

**Table 3**

Sorption thermodynamic equations of *p*-nitrophenol.

	$\ln K = -\Delta H/RT + \Delta S/R$	R
<i>p</i> -nitrophenol	$\ln K = 24.715 - 7113.567/T$	0.951

**Table 4**  
Thermodynamic functions of *p*-nitrophenol.

T (K)	$\Delta G$ (kJ/mol)	$\Delta H$ (kJ/mol)	$\Delta S$ (J/mol K)
293	-0.39	59.16	205.48
303	-3.93	59.16	205.48
313	-5.75	59.16	205.48
323	-6.51	59.16	205.48

reorientation or restructuring of water around the solute or surface is unfavorable, since it disturbs the existing water structure and imposes a new and more ordered structure on the surrounding water molecules. As a result of sorption of *p*-nitrophenol onto modified palygorskite surface, the liberation of the solvent molecules from the solvated shells is more predominant in the studied system, thus, the degree of freedom of the water molecule increases. Therefore, the positive value of entropy suggests the increased randomness at the solid-solution interface during the sorption of *p*-nitrophenol.

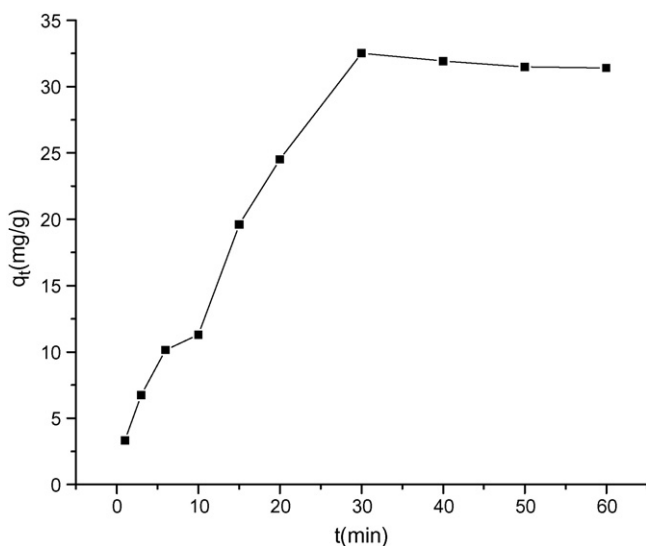
### 3.5. Sorption kinetics

Fig. 10 showed the effect of contact time on the sorption capacity of *p*-nitrophenol on modified palygorskite. As shown in Fig. 10,  $q_e$  increases within 30 min and then it continues to increase at a relatively low speed with contact time until equilibrium is reached at 60 min and remains constantly. In order to investigate the sorption kinetics of *p*-nitrophenol on modified palygorskite, the experimental data were further analyzed using the pseudo-second-order kinetic model and pseudo-first-order kinetic model, expressed respectively as

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2 t} + \frac{1}{q_e} \quad (6)$$

$$\log(q_e - q_t) = \log(q_e) - k_1 t \quad (7)$$

where  $k_2$  ( $\text{g mg}^{-1} \text{min}^{-1}$ ) and  $k_1$  ( $\text{min}^{-1}$ ) are defined as the rate constant of the pseudo-second-order and pseudo-first-order kinetics equations, respectively;  $q_t$  and  $q_e$  are the *p*-nitrophenol uptake per unit weight of anion-cation modified palygorskite ( $\text{mg g}^{-1}$ ) at time  $t$  and equilibrium, respectively.



**Fig. 10.** Effect of contact time on the removal of adsorption at 293 K (initial concentration of *p*-nitrophenol: 200 mg/l; adsorbent amount: 10 mg; anionic surfactant/cationic surfactant = 4/1; irradiation time, 5 min; pH: 7.0–8.0).

**Table 5**  
Sorption kinetics functions of *p*-nitrophenol fit by pseudo-second-order at 293 K.

T (K)	293
$q_{e \text{ exp}}$ (mg/g)	32.52
$q_{e \text{ cal}}$ (mg/g)	28.28
$\Delta q_e$ (%)	15.0
$k_2$ ( $\text{g mg}^{-1} \text{min}^{-1}$ )	0.00453
R	0.984

exp: experimental result; cal: calculated results.

**Table 6**  
Sorption kinetics functions of *p*-nitrophenol fit by pseudo-first-order at 293 K.

T (K)	293
$q_{e \text{ exp}}$ (mg/g)	32.52
$q_{e \text{ cal}}$ (mg/g)	32.69
$\Delta q_e$ (%)	0.52
$k_1$ ( $\text{min}^{-1}$ )	0.0299
R	0.940

exp: experimental result; cal: calculated results.

Tables 5 and 6 exhibited that the pseudo-second-order kinetics model is much better ( $R^2 = 0.968$ ) than that of pseudo-first-order kinetics model ( $R^2 = 0.884$ ). These results are consistent with previous reports [25,23,26].

## 4. Conclusion

A series of modified palygorskites were prepared by microwave irradiation, and well characterized by FT-IR.

The maximum sorption capacity ( $137.74 \text{ mg g}^{-1}$ ) was obtained with the mass ratio 1:4 of hexadecyltrimethylammonium bromide to sodium dodecyl sulfate modified palygorskite at 313 K for 5 min microwave irradiation.

The sorption isotherms were in good agreement with Langmuir and Freundlich equations. The thermodynamic function calculation showed that the sorption was a spontaneous and endothermic process, and the sorption enthalpy and entropy changes of *p*-nitrophenol were  $59.16 \text{ kJ mol}^{-1}$  and  $205.48 \text{ J mol}^{-1} \text{ K}^{-1}$ , respectively. Free energy of the sorption decreased with the increase of temperature.

The sorption of *p*-nitrophenol on anion-cation surfactant modified palygorskite equilibrated within 60 min, and followed the pseudo-second-order rate expression (sorption rate constant:  $0.00453 \text{ mg}^{-1} \text{ min}^{-1}$ ).

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