



## Rapid removal of bisphenol A on highly ordered mesoporous carbon

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### Abstract

Bisphenol A (BPA) is of global concern due to its disruption of endocrine systems and ubiquity in the aquatic environment. It is important, therefore, that efforts are made to remove it from the aqueous phase. A novel adsorbent, mesoporous carbon CMK-3, prepared from hexagonal SBA-15 mesoporous silica was studied for BPA removal from aqueous phase, and compared with conventional powdered activated carbon (PAC). Characterization of CMK-3 by transmission electron microscopy (TEM), X-ray diffraction, and nitrogen adsorption indicated that prepared CMK-3 had an ordered mesoporous structure with a high specific surface area of 920 m<sup>2</sup>/g and a pore-size of about 4.9 nm. The adsorption of BPA on CMK-3 followed a pseudo second-order kinetic model. The kinetic constant was 0.00049 g/(mg·min), much higher than the adsorption of BPA on PAC. The adsorption isotherm fitted slightly better with the Freundlich model than the Langmuir model, and adsorption capacity decreased as temperature increased from 10 to 40°C. No significant influence of pH on adsorption was observed at pH 3 to 9; however, adsorption capacity decreased dramatically from pH 9 to 13.

**Key words:** mesoporous carbon; CMK-3; powdered activated carbon; bisphenol A; endocrine disrupting chemicals

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### Introduction

Bisphenol A (BPA) is widely used in the plastics industry as a monomer for production of epoxy resins and polycarbonate. It can be released into the aquatic environment through various routes, contaminate surface water and raw water supply of drinking water treatment plants (Jin et al., 2004; Suzuki et al., 2004; Loos et al., 2010), and pose a risk to the reproductive health and fecundity of aquatic animals and humans (MOE, 2005; Nakamura et al., 2010). Bisphenol A has been recognized, therefore, as an endocrine disrupting chemical (EDC), and significant effort must be made to remove it from the aqueous phase.

To date, however, many studies have revealed that conventional treatment processes (clarification, disinfection, and sand filtration) are unable to efficiently remove BPA from water (Nakada, 2007; Stackelberg et al., 2007). Consequently, recent research has focused on developing advanced treatment techniques to remove BPA. Some oxidation processes, including photocatalysis, chlorination, and ozonation have proven to be efficient for BPA abate-

ment (Nakada et al., 2007; Nomiyama et al., 2007; Sharma et al., 2009). Membrane filtration, such as nanofiltration and reverse osmosis, can also remove BPA efficiently from water by membrane adsorption or rejection (Kimura et al., 2004; Zhang et al., 2006). Activated carbon adsorption, which has a low capital cost and does not produce more toxic intermediates than BPA itself (Mohanty et al., 2006), seems a promising treatment process for BPA removal. Yoon et al. (2003) tested six types of powdered activated carbon (PAC), and found that 31% to > 99% of BPA could be removed in model or surface waters at an activated carbon dosage of 5 and 15 mg/L. The effect of factors such as initial pH on adsorption kinetics and isotherms have also been investigated (Bautista-Toledo et al., 2005; Tsai et al., 2006a). Most commercial PAC has a predominance of micropores, which might not be easily penetrated by BPA molecules. Compared to commercial PAC, however, CMK-3 possesses a large specific surface area (over 1000 m<sup>2</sup>/g), appropriate meso-scale pore size and pore volume, and has therefore shown better performance than activated carbon on the adsorption of various organic pollutants (Asouhidou et al., 2009; Shi et al., 2009; Ji et al., 2010). Reactive dyes have been selected by Asouhidou et al.

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(2009) and Shi et al. (2009) as adsorbates, and CMK-3 exhibited higher adsorption rates and larger adsorption capacity than commercial activated carbon. Liu et al. (2006) studied the adsorption behavior of nonylphenol ethoxylates (NPE), the parent compounds of EDCs, over CMK-3, and found NPE was effectively removed from the aqueous phase. To the best of our knowledge, however, the adsorption behavior of BPA over CMK-3 has not yet been reported.

In the present study, we fabricated CMK-3 using ordered mesoporous silica SBA-15 as a template, and investigated the removal of the typical EDC, BPA, on CMK-3 with ordered mesoporous structure and narrow pore size distribution.

## 1 Materials and methods

### 1.1 Preparation of CMK-3

SBA-15 was fabricated according to Zhao et al. (1998). Briefly, 4.0 g of tri-block copolymer P123 was dissolved in 30 g of deionized water and 120 g of HCl solution (2 mol/L) with stirring at 40°C. Afterwards, 8.50 g of tetraethyl orthosilicate was added and the resultant solution was stirred at 40°C for 20 hr. The final mixture was transferred into a Teflon-lined high pressure autoclave and aged for 2 days at 100°C. The solid was separated by filtration, washed with deionized water, dried at room temperature, and finally calcined in air at 500°C for 6 hr to obtain SBA-15 under a heating rate of 1°C/min.

Mesoporous carbon CMK-3 was obtained using SBA-15 as a template. In a typical synthetic procedure, 1 g of SBA-15 was added to a solution obtained by dissolving 1.00 g of sucrose and 0.02 mL of dense H<sub>2</sub>SO<sub>4</sub> in 5 g of deionized water. The mixture was placed in a drying oven for 6 hr at 100°C, before being increased to 160°C and maintained for 6 hr. Again, after the addition of 0.64 g of sucrose, 0.01 mL of dense H<sub>2</sub>SO<sub>4</sub>, and 5 g of H<sub>2</sub>O, the sample was treated at 100 and 160°C using the same drying process. Carbonization was completed by pyrolysis with heating to 900°C under nitrogen atmosphere at a heating rate of 1°C/min. To remove the silica template, the carbon–silica composite was washed with hydrofluoric acid at room temperature. The template-free carbon product obtained was then filtered, washed with ethanol, and finally dried at 120°C.

### 1.2 Characterization

The X-ray diffraction (XRD) patterns were recorded with a Bruker D8 ADVANCE diffractometer (Germany) operating with Cu K<sub>α</sub> radiation. Transmission electron microscopy (TEM) images were acquired with a FEI Tecnai-20 instrument (USA) operating at 200 kV. Nitrogen adsorption-desorption isotherms were obtained with a Micromeritics ASAP2010 instrument (USA) at –196°C. Zeta potential of CMK-3 was measured by a Delsa nano particle analyzer (Beckman Coulter, USA).

### 1.3 Adsorption experiments

Batch experiments were conducted in an orbital shaker

at 135 r/min. In the adsorption kinetics experiments, 10 mg of CMK-3 or PAC was placed into 200 mL flasks containing 100 mL of BPA solution with an initial BPA concentration of 25 mg/L. The temperature was kept at 25°C. Samples were taken at specific times up to 16 or 18 hr. The isotherm experiments were carried out in 250 mL flasks containing 200 mL of BPA solution. Isotherm studies can be conducted by varying the initial adsorbate concentration (Bautista-Toledo et al., 2005; Liu et al 2006) or the adsorbent dosage (Fukuhara et al., 2006; Yu et al., 2008). In the present study, the CMK-3 dosage varied from 1 to 17.5 mg/L and the initial concentration of BPA was fixed at 25 mg/L. The adsorption temperature was kept at 10, 25, or 40°C. After equilibrium was attained, the samples were filtered through a 0.22 μm PTFE membrane, and immediately analyzed by spectrophotometry (DR5000, Hach, USA) at 275 nm.

The adsorption of BPA was calculated according to Eq. (1):

$$q_e = \frac{(C_0 - C_e)V}{M} \quad (1)$$

where,  $q_e$  (mg/g) is the equilibrium adsorption amount,  $C_0$  (mg/L) and  $C_e$  (mg/L) are the initial and equilibrium concentrations of BPA in the solution, respectively,  $V$  (L) is the solution volume, and  $M$  (g) is the mass of the adsorbent.

## 2 Results and discussion

### 2.1 Characterizations

Parallel to pore direction, the pores were arranged in a hexagonal fashion, while perpendicular to the pore direction, the stripe patterns in the image background were attributed to the lattice fringes of the P6 mm mesostructure of carbon (Fig. 1). The ordered carbon nanorods were interconnected by the carbon in the channel-interconnecting micropores within the SBA-15 wall.

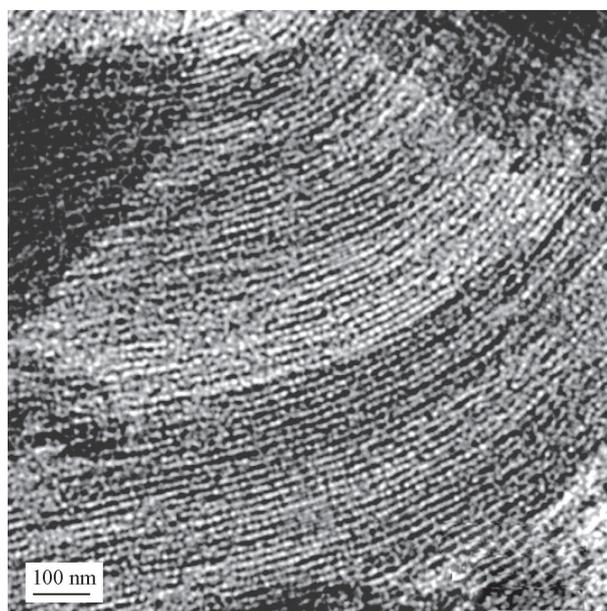


Fig. 1 TEM image of CMK-3 with ordered mesoporous structures.

The XRD characterization was employed to investigate purity of CMK-3 mesoporous materials (Fig. 2). We found diffraction peaks from 0.6 to 4.5 were indexed to 2-D hexagonal P6mm symmetry. The three obvious diffraction peaks observed at  $2\theta$  of 1.05, 1.76 and 2.03, corresponded to reflections of (001), (110), and (200), indicating an ordered hexagonal structure of CMK-3.

The Brunauer-Emmett-Teller (BET) method, which extended the Langmuir theory for monolayer molecular adsorption, to multilayer adsorption (Gregg and Sing, 1982), was used to calculate the surface areas of CMK-3 and PAC. The nitrogen sorption isotherm for the CMK-3 material (Fig. 3a) was type IV with a hysteresis loop of type H1 occurring at  $P/P_0$  0.65–0.80, indicating a uniform structure of the mesopores. Pore-size distributions (Fig. 3b) were calculated by the Barrett-Joyner-Halenda (BJH) method, which is based on the Kelvin equation and linked to pore condensation (Gregg and Sing, 1982). Corresponding pore-size distribution of CMK-3 was very narrow and centered at 4.9 nm. Examining the textural properties of CMK-3 and PAC, including specific surface area, mesoporous pore volume, and pore size (Table 1), showed that CMK-3 possessed a much larger BET surface area (920  $\text{m}^2/\text{g}$ ) and mesoporous pore volume (1.309

**Table 1** Textural property of CMK-3 and PAC adsorbents

Adsorbent	BET surface area ( $\text{m}^2/\text{g}$ )	BJH pore diameter (nm)	Mesoporous pore volume ( $\text{cm}^3/\text{g}$ )*
CMK-3	920	4.87	1.309
PAC	780	3.06	0.058

\* Based on BJH method.

$\text{cm}^3/\text{g}$ ) compared to commercial PAC adsorbent (780  $\text{m}^2/\text{g}$  and 0.058  $\text{cm}^3/\text{g}$ , respectively), which was considered the most important factor related to adsorption performance.

## 2.2 Adsorption kinetics of BPA

Kinetics of BPA adsorption on PAC and CMK-3 was assessed at an initial concentration of 25  $\text{mg}/\text{L}$  and pH of  $6.8 \pm 0.1$ . As shown in Fig. 4, adsorption of BPA on CMK-3 reached equilibrium within 60 min, while adsorption of BPA on PAC was much slower and reached equilibrium after 480 min.

To investigate adsorption characteristics of BPA, processes were described by both pseudo first- and second-order kinetics. The Lagergren equation is often used for pseudo first-order kinetics (Lagergren, 1898),

$$\ln \frac{(q_e - q_t)}{q_e} = -k_1 t \quad (2)$$

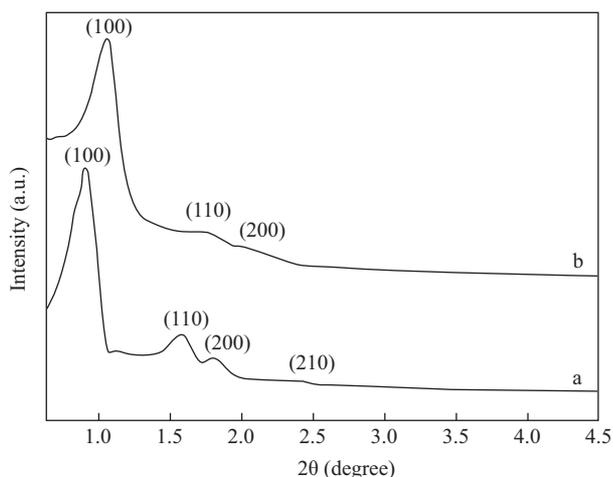
where,  $q_e$  ( $\text{mg}/\text{g}$ ) is the amount of BPA adsorbed at equilibrium,  $q_t$  ( $\text{mg}/\text{g}$ ) is the amount of BPA adsorbed at time  $t$ , and  $k_1$  ( $1/\text{min}$ ) is the pseudo first-order constant.

Pseudo second-order kinetics, the hypothesis of which states that the adsorption rate is controlled by chemical adsorption (Ho and McKay, 1999), can be described by Eq. (3) (Ho and McKay, 1998)

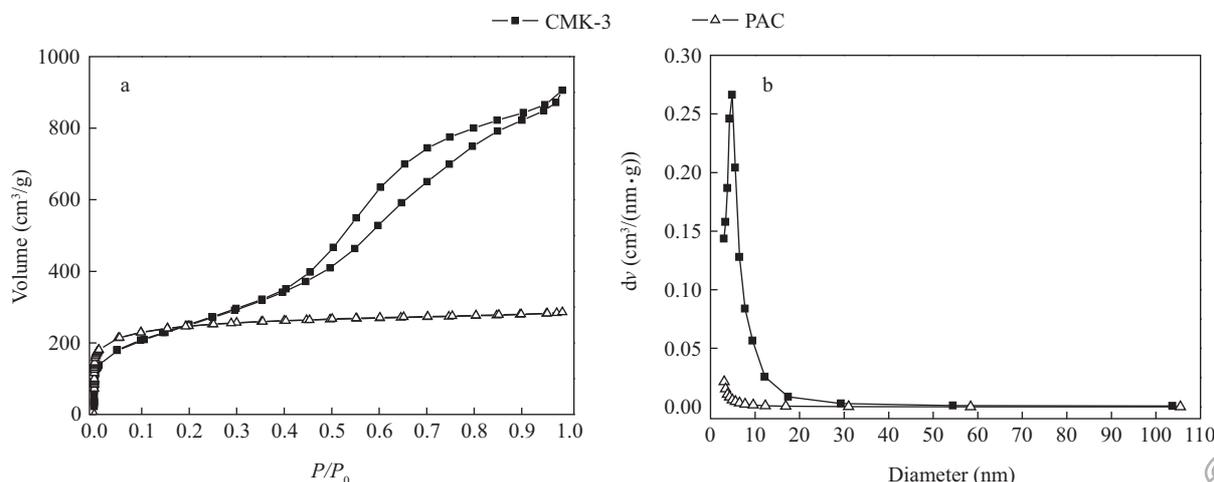
$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (3)$$

where,  $q_e$  ( $\text{mg}/\text{g}$ ) is the amount of BPA adsorbed at equilibrium,  $q_t$  ( $\text{mg}/\text{g}$ ) is the amount of BPA adsorbed at time  $t$ , and  $k_2$  ( $\text{g}/(\text{mg}\cdot\text{min})$ ) is the pseudo second-order constant.

The parameters  $q_e$  and  $k_2$  were obtained from the intercept and slope of the plot  $t/q_t$  against  $t$ . Half time  $t_{1/2}$



**Fig. 2** Small angle XRD patterns of SBA-15 (line a) and CMK-3 (line b).



**Fig. 3**  $\text{N}_2$  sorption isotherm (a) and BJH pore-size distribution (b) for CMK-3 and PAC.

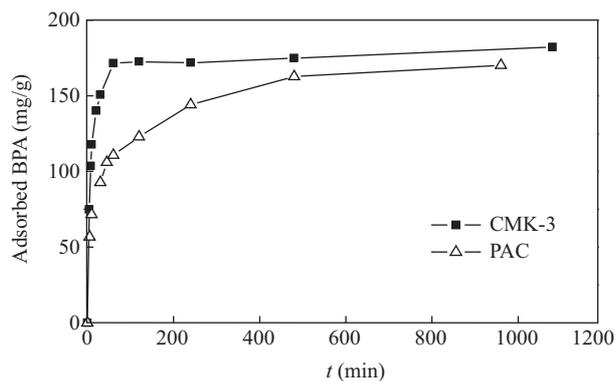


Fig. 4 Time-resolved uptake of BPA onto CMK-3 and PAC.

(min), which is the time required for  $1/2q_e$  of BPA to be adsorbed, is calculated as Eq. (4). The initial adsorption rate,  $h$  (mg/(g·min)), can be obtained at initial adsorption time ( $t = 0$  and  $q_t = 0$ ) as the value of  $q_e$  (Eq. (5), Tsai et al., 2006a):

$$t_{1/2} = 1/(k_2q_e) \quad (4)$$

$$h = k_2q_e^2 \quad (5)$$

As shown in Fig. 5, the pseudo second-order model fitted the kinetics data better than the pseudo first-order model. Parameters describing the adsorption processes on CMK-3 and activated carbon are summarized in Table 2. The higher rate constant, initial adsorption rate, and lower half-life time of CMK-3 demonstrated better adsorbent for BPA removal than PAC, which was likely due to limited contact time in the water treatment processes achieving more efficient elimination by CMK-3.

Table 2 Pseudo second-order parameters for adsorption of BPA onto CMK-3 and activated carbon

Adsorbent	$q_e$ (exp) (mg/g)	$q_e$ (cal) (mg/g)	$k_2$ (g/(mg·min))	$t_{1/2}$ (min)	$H$ (mg/(g·min))	$R^2$
CMK-3	186	180	0.00049	11.3	15.9	0.997
PAC	178	164	0.00015	40.7	4.0	0.937

$q_e$  (exp): the amount of BPA adsorbed at the equilibrium (experimental data);  $q_e$  (cal): the amount of BPA adsorbed at the equilibrium (calculated from pseudo second-order model);  $k_2$ : pseudo second-order constant;  $t_{1/2}$ : half time;  $h$ : initial adsorption rate.

### 2.3 Adsorption isotherm of BPA

Understanding adsorption isotherms are critical for understanding adsorbate-adsorbent interactions. The adsorption isotherms of BPA on CMK-3 at different temperatures are described in Fig. 6. Although temperature had only minor influence on adsorption capacity, the observed trend showed that adsorption capacity decreased when adsorption temperature increased.

To better elucidate the mechanism of BPA on CMK-3, the commonly used Langmuir (Eq. (6)) and Freundlich (Eq. (7)) models were adopted to simulate the experimental data.

$$\frac{1}{q_e} = \frac{1}{(K_L q_m)C_e} + \frac{1}{q_m} \quad (6)$$

$$q_e = K_F C_e^{1/n} \quad (7)$$

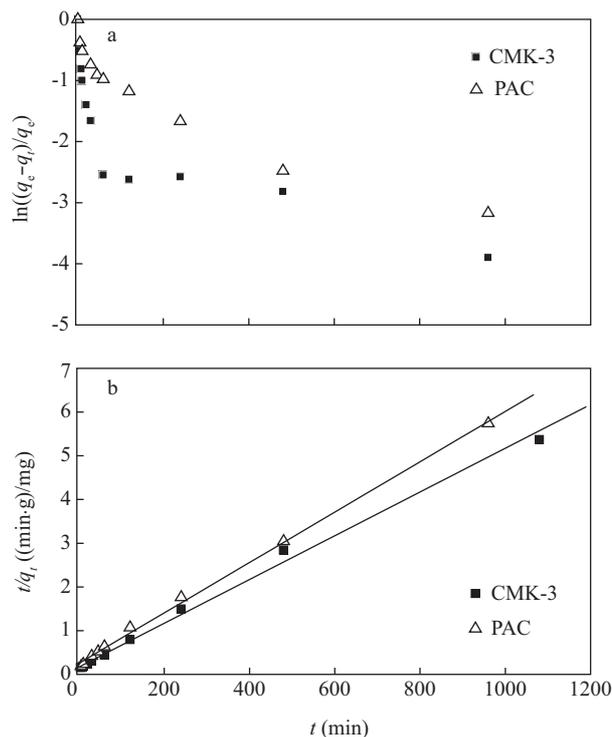


Fig. 5 Simulation of BPA adsorption on CMK-3 and PAC using pseudo first-order kinetics (a) and pseudo second-order kinetics (b).

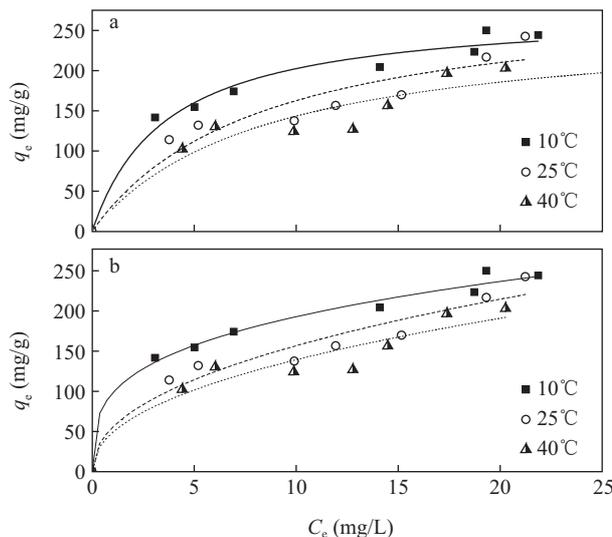


Fig. 6 Adsorption isotherms of BPA over CMK-3 at different temperatures fitted by the Langmuir equation (a) and Freundlich equation (b).

In Eq. (6),  $C_e$  (mg/L) and  $q_e$  (mg/g) are the equilibrium adsorption concentrations in the solution and amount on the adsorbents, respectively.  $K_L$  (L/mg) describes the intensity of the adsorption process, and  $q_m$  (mg/g) reflects maximum adsorption capacity. In Eq. (7),  $K_F$  (mg(L/mg) $^{1/2}$ /g) is a constant related to bonding energy, and represents the general capacity of adsorbate adsorbed onto adsorbents for a unit equilibrium concentration.  $1/n$ , with a range between 0 and 1, reflects adsorption intensity or surface heterogeneity.

The parameters, obtained through the fit of the experimental data to both models, are listed in Table 3. On the basis of  $R^2$  values, adsorption isotherms fitted slightly better with the Freundlich model than the Lang-

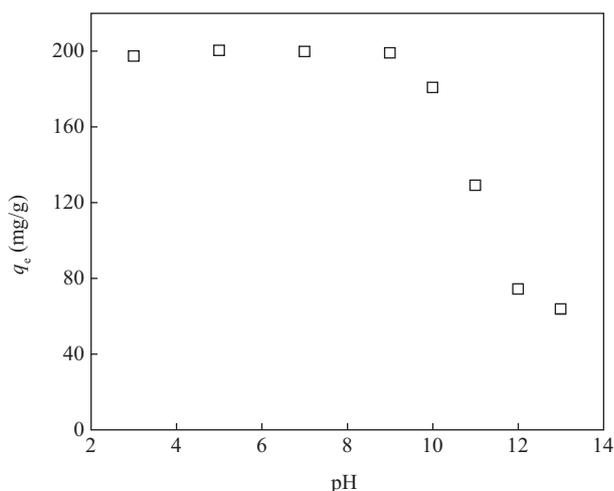
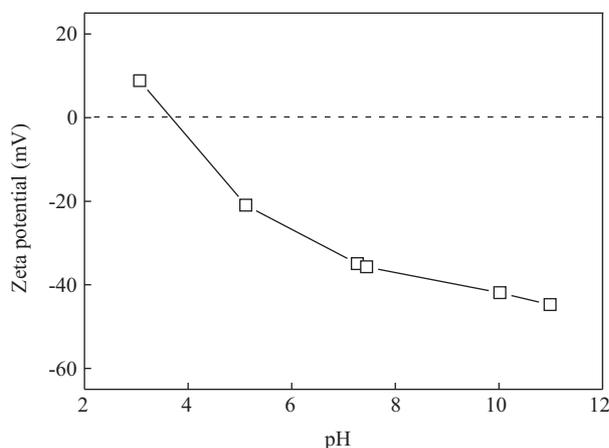
**Table 3** Parameters of adsorption isotherms modeled by the Langmuir equation and Freundlich equation

Temp. (°C)	Langmuir			Freundlich		
	$q_m$ (mg/g)	$K_L$ (L/mg)	$R^2$	$K_F$ (mg (L/mg) <sup>1/2</sup> /g)	$1/n$	$R^2$
10	276	0.276	0.979	97.7	0.30	0.990
25	296	0.121	0.918	55.1	0.45	0.951
40	263	0.120	0.906	48.8	0.46	0.931

muir model. As the Langmuir equation is derived under the assumption of monolayer coverage, the fitting results imply that multilayer adsorption rather than monolayer adsorption occurred. The Freundlich model is an empirical isotherm model usually used in heterogeneous surface energy systems. The increased temperature led to decreased  $K_F$  values, indicating that the adsorption process involved an endothermic reaction. The  $1/n$  values were significantly less than 1, suggesting a favorable adsorption system and heterogeneity of the CMK-3 adsorption sites.

## 2.4 Effect of initial pH

The effect of initial pH on adsorption capacity of BPA onto CMK-3 was evaluated at a CMK-3 dosage of 100 mg/L, initial concentration of BPA of 25 mg/L, contact time of 4 hr and temperature of 25°C. As shown in Fig. 7, adsorption amounts were relatively constant (around 200

**Fig. 7** Effect of initial pH on the adsorption of BPA onto CMK-3.**Fig. 8** Zeta-potential of CMK-3 as a function of pH.

mg/g) at pH 3 to 9. However, adsorption capacity decreased from 200 to 64 mg/g when pH increased from 9 to 13. The  $pK_a$  of BPA ranged from 9.6 to 10.2 (Yoon et al., 2003), therefore for initial pH values > 10, molecules were deprotonated and formed bisphenolate anions. Consequently, electrostatic repulsion between the bisphenolate anions and the adsorbent surface negatively charged (Fig. 8) therefore reduced adsorption capacity. These results were consistent with previous research on adsorption of BPA onto activated carbon (Bautista-Toledo et al., 2005; Tsai et al., 2006a) and Y-type zeolite (Tsai et al., 2006b).

## 3 Conclusions

Mesoporous carbon, CMK-3 was prepared from hexagonal SBA-15 mesoporous silica. Characterization by TEM, XRD, and nitrogen adsorption demonstrated that prepared CMK-3 had an ordered mesoporous structure with a high specific surface area of 920 m<sup>2</sup>/g. The adsorption experiments indicated that BPA was effectively removed from the aqueous phase by prepared CMK-3. Kinetics data obeyed the pseudo second-order model and indicated that adsorption on CMK-3 was much faster than on commercial PAC. The adsorption capacity on CMK-3 decreased with increased temperature, and isotherm data fitted slightly better with the Freundlich model than the Langmuir model. Adsorption amounts were little affected in an initial pH range of 3 to 9, but decreased dramatically from pH 9 to 13, probably due to the deprotonation of BPA.

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