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Adsorption of xylene isomers on ordered hexagonal mesoporous FDU-15 polymer and carbon materials

Zhangxiong Wu · Yunxia Yang · Bo Tu · Paul A. Webley · Dongyuan Zhao

Abstract The oil industry has been facing the challenges of separation of xylene isomers, *o*-xylene, *m*-xylene and *p*-xylene or removing them from the environment. In our present work, we investigated the adsorption of the three isomers on two mesoporous materials, FDU-15-350 polymer and FDU-15-900 carbon materials. The isomer adsorption capacities are well correlated with their physical pore properties. It is found that the micropores are very crucial for the adsorption of these three isomers. The more micropore volume the adsorbent has, the better the adsorption capacity is. Henry's constants were also calculated for the three isomers on the two adsorbents. Both on FDU-15-350 polymer and FDU-15-900, the Henry's constants for the three isomers show the same trend $o > m > p$ xylene which is coincidently in accordance with their polarity trend, indicating more polar adsorbate is preferred for adsorption on the two adsorbents. The isosteric heats of adsorption are correlated with the microporosity and the size of the adsorbate molecule. More microporosity and smaller molecules give higher heats of adsorption. Extracted information on pore properties of adsorbents by using the three isomers has very similar results as that resolved from nitrogen adsorption, indicating the feasibility of using the three isomers as adsorbates to extract pore information.

Keywords Mesoporous materials · FDU-15 · Adsorption · Xylene · Carbon

1 Introduction

Xylene as a very important organic hydrocarbon is normally composed of three isomer components: *o*-xylene, *p*-xylene, and *m*-xylene. The extreme difficulty in separating these isomers has lead to one of the most challenging issues in the oil industry because distillation is only feasible for removing *o*-xylene but fails in the other two isomers because of the similarity of their boiling points (*p*-xylene: 138 °C, *m*-xylene: 138–139 °C) (Meyers 2003). Meanwhile, these isomers are very toxic volatile organic compounds (VOCs) and failure in separating them from the industrial emissions results in the discharge of the mixture into the air threatening the environment. Adsorption is a widely adopted approach to separate xylene isomers. For example, Zeolites X and Y exchanged with cations such as Na^+, K^+, and Ba^{2+} can separate selectively the different xylene isomers and are used as industrial adsorbents (Hulme et al. 1991; Ruthven and Goddard 1986). Metal-organic frameworks (MOFs) are a new class of microporous crystalline materials and have also been investigated for xylene separation. It was found that *p*-xylene is selectively included inside zinc 2,6-naphthalene dicarboxylate coordination polymer during its synthesis (Nandini Devi et al. 2004). Different affinities for *p*-xylene and *o*-xylene are also observed in a zinc terephthalate MOF (Huang et al. 2003). However, all these reports concerning adsorption and separation of xylene isomers are based on microporous materials. It is believed that there exist pore constraints and poor diffusion

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This work is devoted to commemorating the 60th birthday of Professor Mieczyslaw (Mietek) Jaroniec.

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properties in microporous materials because of their small pore sizes.

Ordered mesoporous materials are known to have very high surface areas and large pore volumes. Many attempts have been tried to apply them as industrial adsorbents especially for removing VOCs in water and air (Bathen et al. 1997). Ordered mesoporous silica materials have been used as adsorbents for many hydrocarbons. For example, ordered hexagonal mesoporous silica MCM-41 (Kresge et al. 1992) and SBA-15 (Zhao et al. 1998a, 1998b; Wan and Zhao 2007) were used for adsorption and separation of light hydrocarbons such as C1 $\sim$ C3 (Newalkar et al. 2002, 2003), long-chain hydrocarbons such as C7 (Vinh-Thang et al. 2005) and aromatic hydrocarbons such as toluene and benzene (Hartmann and Vinu 2002; Ravikovitch et al. 2006). But there is very limited report about using ordered mesoporous polymer and carbon materials as adsorbents for hydrocarbons.

Recently, we reported a series of novel ordered mesoporous polymer and carbon materials (Zhang et al. 2005; Wan et al. 2006). Among them, the novel FDU-15 materials (Meng et al. 2005, 2006) are of special interest in this study. FDU-15-350 is a polymer. FDU-15-900 is a corresponding ordered mesoporous carbon. Both of these materials have two-dimensional (2-D) hexagonally ordered mesopores. Most importantly, hierarchical pore structures are present in these materials with mesopores interconnected with each other through the micropore windows on the walls, which we believe will promote diffusion of adsorbate molecules from big pores to smaller pores and enhance the adsorption properties. In our present study, we are aiming to investigate the adsorption properties of ordered mesoporous FUD-15 polymer and its corresponding carbon material towards xylene isomers by measuring the adsorption isotherms, modeling the isotherms, evaluating the adsorption affinity, estimating the isosteric heats of adsorption, and assessing the potential of FDU-15 materials as industrial adsorbents for hydrocarbons. The results can provide necessary information for a better understanding of the hydrocarbon adsorption and separation involving mesoporous FDU-15 molecular sieves.

2 Experimental

2.1 Synthesis of FDU-15 materials

FDU-15 materials were prepared according to the method reported elsewhere (Meng et al. 2005). Typically, triblock copolymer poly(ethylene oxide)-*b*-poly(propylene oxide)-*b*-poly(ethylene oxide) (PEO-PPO-PEO, Pluronic F127; 1.00 g) was dissolved in ethanol (20.0 g), then a phenolic resin precursor containing phenol (0.61 g) and formaldehyde (0.39 g) was added and stirred for 10 min to form a homogeneous solution. The solution was transferred into glass dishes and the ethanol was evaporated at room temperature for 5–8 h to produce transparent films. The films were then heated in an oven at 100 °C for 24 h to thermopolymerize the phenolic precursor. The products were calcined at 350 or 900 °C under nitrogen for 5 h with a temperature ramp rate of 1 °C min^{-1} below 600 °C and 5 °C min^{-1} above 600 °C to obtain the porosity and decomposition of the triblock copolymer template. The sample obtained at 350 °C was an ordered mesoporous polymer and was denoted as FDU-15-350, while the carbon sample obtained at 900 °C was denoted as FDU-15-900.

2.2 Characterization of the materials

Small-angle X-ray diffraction (XRD) patterns were recorded on a Brucker D4 powder X-ray diffractometer by using CuKα radiation (40 kV, 40 mA). N_2 adsorption isotherms were measured using a Micromeritics ASAP2020 analyzer at 77 K. Before measurements were taken, all samples were degassed at 473 K for more than 12 h.

2.3 Adsorption equilibrium measurement

The organic xylene isomers used for the adsorption experiments were purchased from Aldrich with high purity. Adsorption equilibrium measurements were carried out using a digital microbalance (IGA Instrument, Model 001) connected to a high-vacuum system. In the first step, about 10–20 mg of powdered sample was loaded on a small pan. Under a high vacuum of 10^{-5} mbar or better, the sample was heated to 180 °C and kept at this temperature for at least 6 h to remove all the possible adsorbed impurities and hygroscopic moisture in particular. Then the sample was cooled to the required experimental temperature. Isotherm measurements were carried out by introducing a dose amount of hydrocarbon vapor directly to the sample chamber, and recording the weight change after reaching a stable equilibrium pressure. Further consecutive measurements were taken by increasing the vapor pressure by steps.

2.4 Calculation methods and procedures

BET specific surface areas of the materials under study were evaluated from N_2 adsorption data at 77 K in the relative pressure range from 0.02 to 0.2. The total pore volume was estimated on the basis of the volume adsorbed at p/p_0 of $\sim$0.99. Pore size distributions (PSD) of the materials were evaluated by density functional theory (DFT) (Ravikovitch et al. 1997) for the micropores ($<$2 nm) based on kernels of adsorption of N_2 on slit-like pores of carbon based materials and BJH model for the mesopores ($>$2 nm) with cylinder-like pore model. Micropore volumes and micropore surface areas were calculated through the t-plot method. All adsorption isotherm data associated with all adsorptive hydrocarbon molecules were well fitted with the Langmuir equation:

$$1/q = 1/q_m + 1/(q_m kp) \quad (1)$$

where q (mmol g^{-1}) is the specific equilibrium amount adsorbed at pressure p and temperature T, and q_m is the limiting molar saturation capacity of the monolayer. The quantity $q_m k$ (k being the Langmuir constant) is equal to K_{H} (the Henry's constant). The parameters of q_m and $q_m k$ can be both evaluated graphically by plotting $1/q$ versus $1/p$. The isosteric heats of adsorption $-\Delta H_{\text{ads}}$ (kJ mol^{-1}) at different adsorbate loadings can be evaluated from the adsorption isotherms data through the Clausius–Clapeyron type equation:

$$[\partial \ln p/\partial T]_q = \Delta H_{\text{ads}}(q)/RT^2 \quad (2)$$

where p and T are the equilibrium pressure and temperature at a given adsorbate loading q. R is the gas constant.

3 Results and discussion

3.1 Structural and textual properties of the FDU-15 materials

Small-angle X-ray diffraction patterns (Fig. 1a) of both the FDU-15 polymer and carbon materials show one strong diffraction peak and one weak peak, which can be indexed to the 100 and 110 diffractions based on 2-D hexagonal structure with *p6mm* symmetry, confirming the periodicity of the pore structures of the two samples. The cell parameter (Table 1) of the polymer FDU-15-350 is about 12.6 nm, while that of the carbon FDU-15-900 is 9.9 nm,

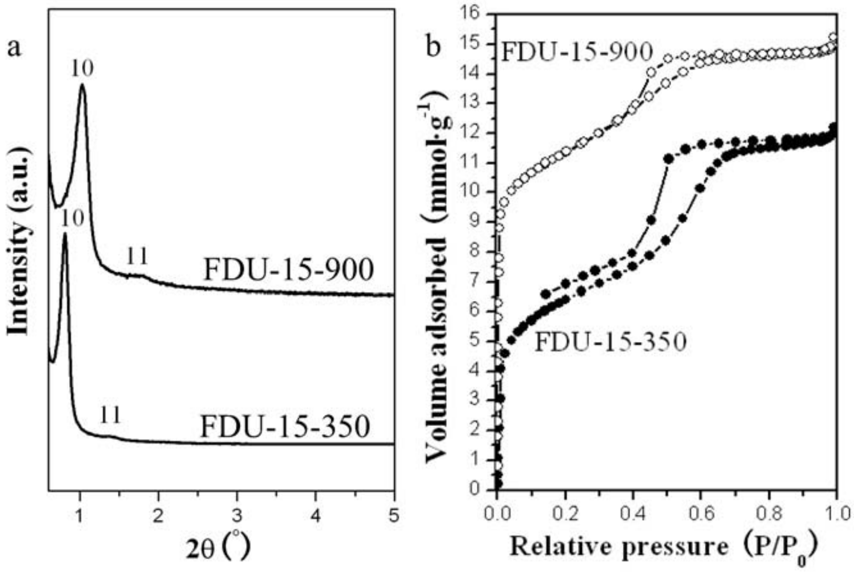

Fig. 1 Powder small-angle XRD patterns (**a**) and nitrogen sorption isotherms (**b**) of ordered mesoporous polymer FDU-15-350 and carbon FDU-15-900

Table 1 Detailed textual properties of the adsorbents mesoporous materials FDU-15-350 and FDU-15-900 obtained from liquid nitrogen sorption results at 77 K

Sample name	a_0[a] (nm)	S_{BET}[b] (m^2/g)	S_{mic}[c] (m^2/g)	V_{t}[d] (cm^3/g)	V_{mic}[c] (cm^3/g)	D[e] (nm)
FDU-15-350	12.6	560	220	0.51	0.11	0.80, 1.26, 1.50, 5.30
FDU-15-900	9.9	890	540	0.58	0.24	0.73, 1.10, 1.60, 3.10

[a] Cell parameters calculated from XRD patterns

[b] BET specific surface areas evaluated from N_2 adsorption data at 77 K in p/p_0 from 0.02 to 0.2

[c] Micropore volumes and micropore surface areas calculated through the t-plot method

[d] Total pore volumes estimated on the basis of the volume adsorbed at $p/p_0 \sim 0.99$

[e] Pore size distributions evaluated using DFT model for the micropores (<2 nm) and BJH model for the mesopores (>2 nm)

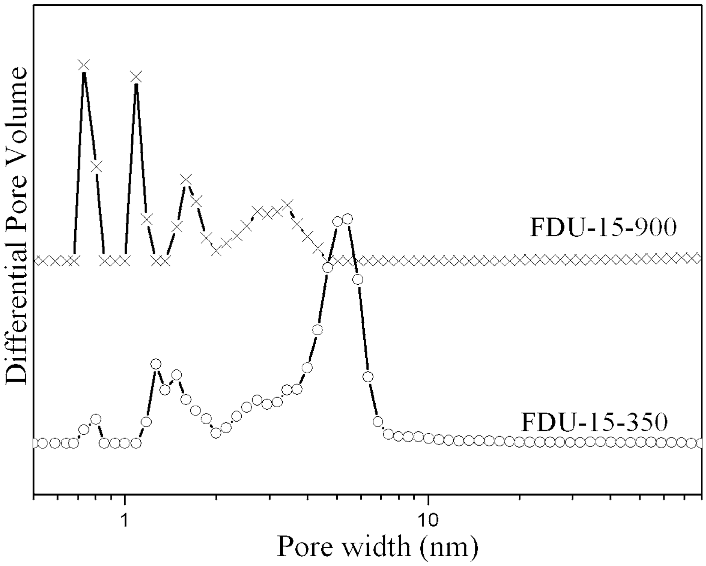

Fig. 2 Pore size distributions of ordered mesoporous polymer FDU-15-350 and carbon FDU-15-900 calculated from nitrogen sorption isotherms using DFT model for the micropores (<2 nm) based on kernels of adsorption of N_2 on slit-like pores of carbon based materials and BJH model for the mesopores (>2 nm) with cylinder-like pore model

much smaller because of the large structural shrinkage during the carbonization process at high temperature. Nitrogen adsorption-desorption isotherms at 77 K on the FDU-15-350 and FDU-15-900 materials (Fig. 1b) are both combination of type-I and type-IV, indicating typical bimodal porous materials. The isotherms displayed two significant adsorption increases at the relative pressure $p/p_0 < 0.1$ and $p/p_0 = 0.4$–0.7, respectively. The former one is due to adsorption in the micropores, while the latter one due to capillary condensation in the mesopores (Gregg and Sing 1982). The textual parameters of the materials, i.e., specific surface area (S_{BET}), micropore surface area (S_{mic}), total pore volume (V_{t}), micropore volume (V_{mic}) and pore diameter (D) are summarized in Table 1. In order to explore the effects of the pore properties on xylene adsorption, Density functional theory (DFT) was employed to calculate the detailed micropore size distributions of the samples from nitrogen sorption isotherms. The micropore distributions in the two materials are both very narrow (Fig. 2), centered at 0.73, 1.10 and 1.60 nm for the ordered mesoporous carbon FDU-15-900, and 0.80, 1.26 and 1.50 nm for the mesoporous polymer FDU-15-350, respectively, which are all larger than the kinetic diameters of xylene isomers. Apparently, the samples, especially the mesoporous carbon, are composed of large amount of micropores with high micropore volume and surface area, which we believe is very important for xylene adsorption processes (see below).

3.2 Adsorption equilibrium of xylene isomers

Adsorption equilibrium isotherms of *o*, *m*, and *p*-xylene on polymer FDU-15-350 and carbon FDU-15-900 materials measured at different temperatures from 25 to 40 °C are shown in Figs. 3 and 4. All the isotherms are similar to type I isotherm according to IUPAC classification. Because of the large amount of micropores in the materials, the isotherms are characterized by a steep increase at low pressure, corresponding to micropore filling. Especially the mesoporous carbon FDU-15-900 shows much steeper increase at low pressure region and reaches the maximum at high pressure. However, unlike reaching a flat plateau at high pressure in type I isotherm, these isotherms show gradual adsorption increase with increasing pressure in the high pressure region. The normal capillary condensation step in regular mesopores is not found in these isotherms. This may be because the vapor pressures of the xylene isomers are not high enough to condense in the mesopores. Considering the hierarchical pore structure in FDU-15, we believe that the mesopores are just bridging the gap between bulk xylene gases and micropores, providing transport channels for xylene molecules to access the micropores. Once the micropores are saturated, the mesopores can gradually take up gas molecules with the increase of pressure, thus showing the gradual adsorption increase in the isotherms. We further calculated the micropore volume and micropore surface area of the two adsorbents (Table 1) by using the adsorption capacities of xylene isomers near the saturation pressure at different temperatures based on the assumption that the densities of the xylene isomers adsorbed in the pores are the same as those of the corresponding bulk liquid substances. These data are plotted in Fig. 5, together with the micropore volumes and surface areas from the nitrogen adsorption results for comparison. It is noteworthy that the total surface areas and pore volumes calculated from adsorption isotherms of xylene isomers are very close to the micropore volumes and micropore surface areas obtained from nitrogen sorption results (Fig. 5), further confirming that the adsorption of xylene isomers mainly happens in the micropores at a pressure

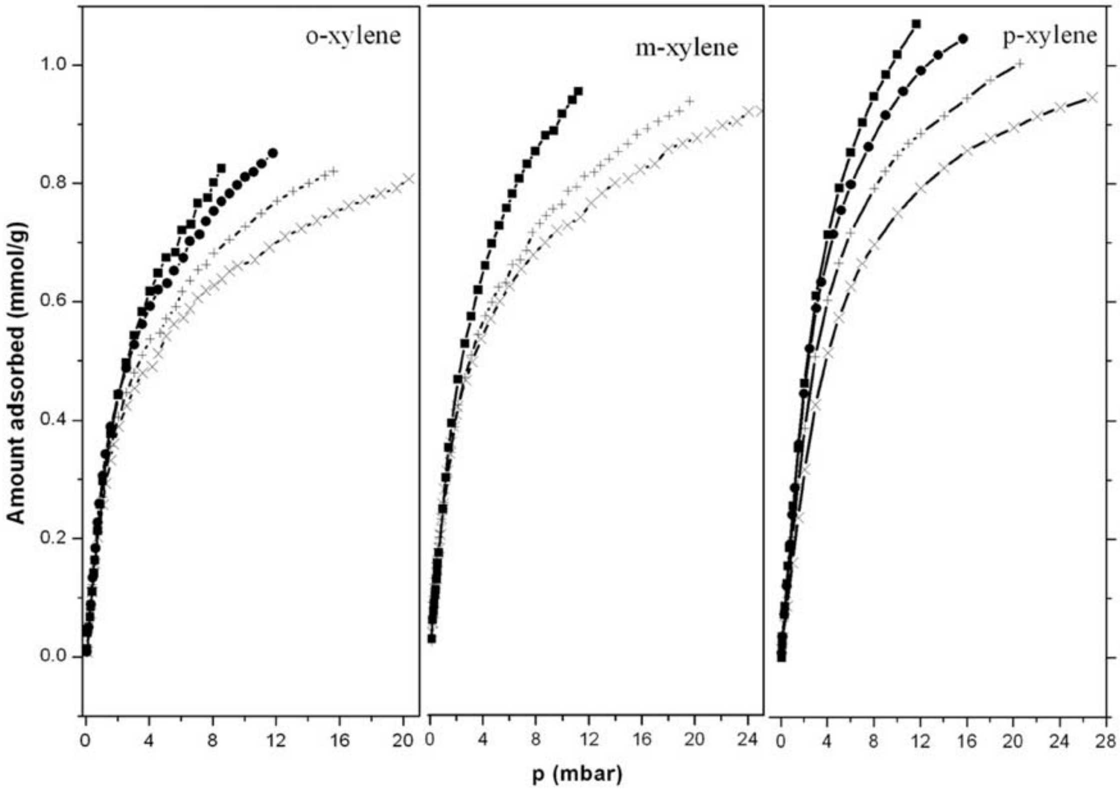

Fig. 3 Adsorption isotherms of xylene isomers (*left*, *o*-xylene; *middle*, *m*-xylene; *right*, *p*-xylene) on ordered mesoporous FDU-15-350 polymer at different temperatures, ■, 25; ●, 30; +, 35; ×, 40 °C

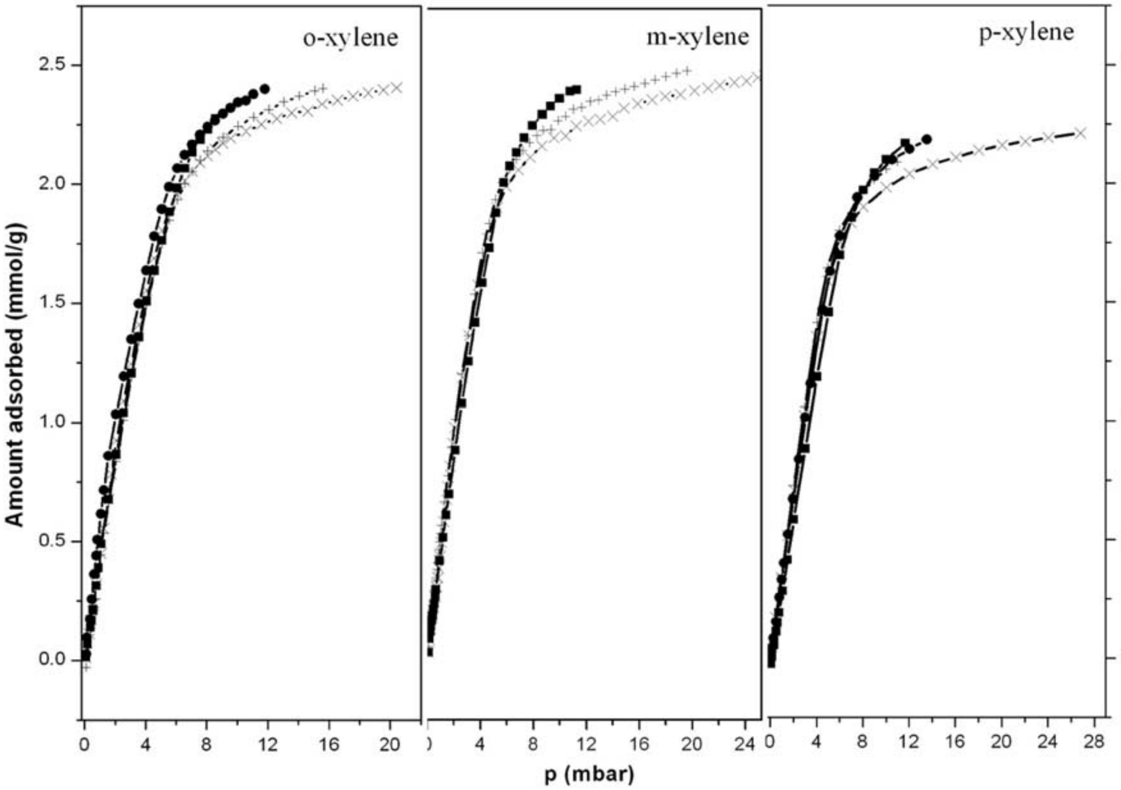

Fig. 4 Adsorption isotherms of xylene isomers (*left*, *o*-xylene; *middle*, *m*-xylene; *right*, *p*-xylene) on ordered mesoporous FDU-15-900 carbon at different temperatures, ■, 25; ●, 30; +, 35; ×, 40 °C

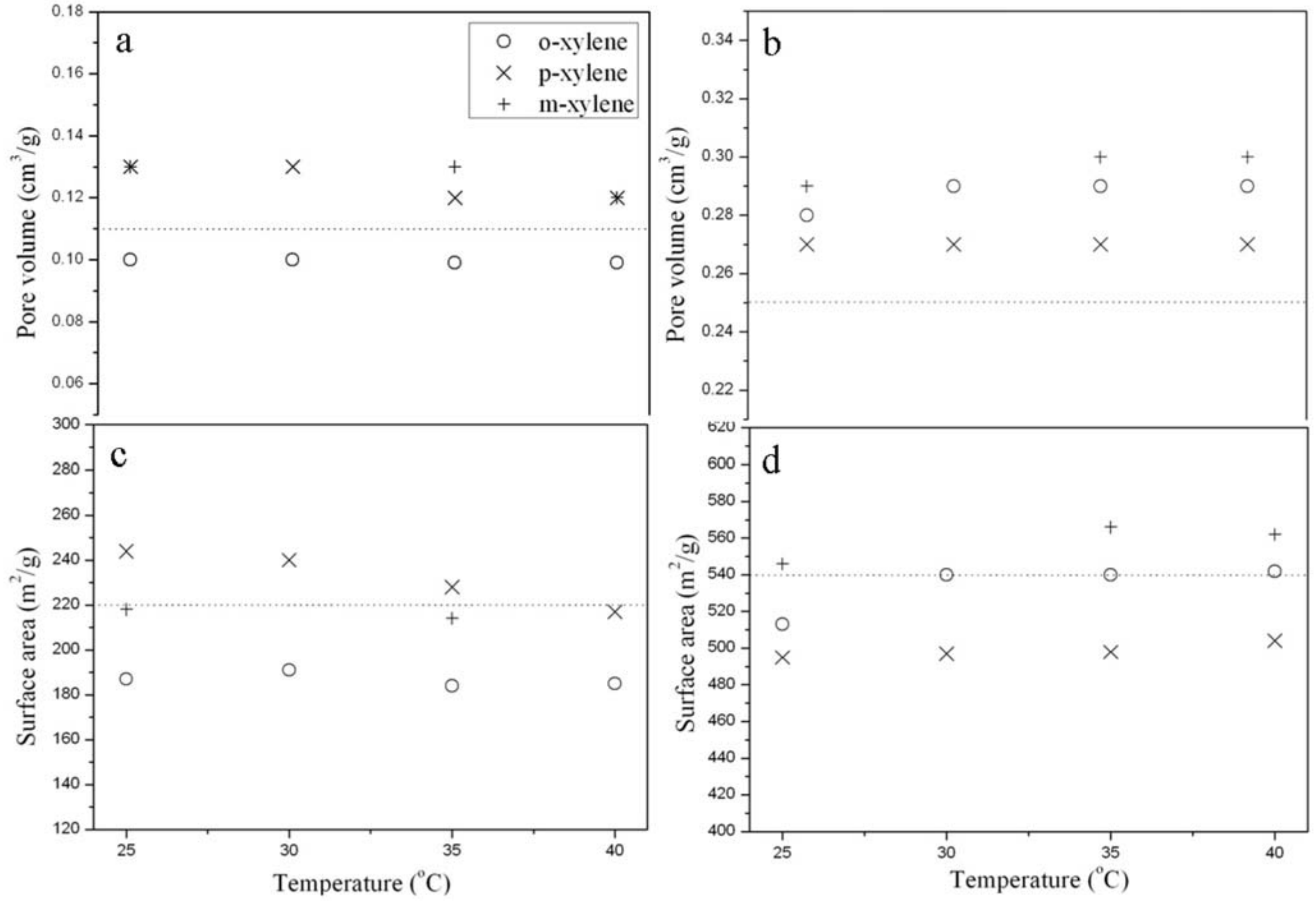

Fig. 5 Pore volumes (**a**, **b**) and surface areas (**c**, **d**) of the mesoporous polymer FDU-15-350 (**a**, **c**) and carbon FDU-15-900 (**b**, **d**) calculated from the adsorption capacities of *o*-xylene (○), *m*-xylene (+), and *p*-xylene (×) at different temperature from 25–40 °C. The *dashing lines* are micropore volumes (**a**, **b**) and micropore surface areas (**c**, **d**) of the corresponding FDU-15 materials obtained from nitrogen sorption at 77 K

less than its saturation pressure. This is the reason why the normal capillary condensation step in regular mesoporous materials was not observed in these isotherms. A similar phenomenon was observed for the adsorption of toluene and *o*-xylene on mesoporous UL-ZSM-5 materials reported by Huang et al. (2006). We summarize the adsorption capacities of xylene isomers on the two adsorbents in Table 2. The saturation adsorption capacities are very close in the temperature range from 25 to 40 °C for a specific adsorbate on both of the adsorbents. However, an adsorption capacity trend is exhibited on FDU-15-350 polymer material as *p*-xylene > *m*-xylene > *o*-xylene, which is just the opposite trend of the kinetic diameters of the adsorbates. We think this is because *o*-xylene has a larger kinetic diameter and less structure flexibility, and can not be as effectively adsorbed as the other two counterparts with smaller kinetic diameters and more structure flexibilities. However, a completely different phenomena is observed on FDU-15-900 (*p*-xylene < *m*-xylene ∼ *o*-xylene), because the carbon adsorbent has a non polar surface and the polarity of the adsorbate may play an important role for high adsorption capacity.

3.3 Henry's constant

Henry's constants for the two mesoporous materials towards xylene isomers are calculated from the adsorption isotherms at low pressures, i.e., $p < 0.52$ mbar (Table 2). The Henry's constants reflect adsorption affinity in the linear region of the isotherms. Generally, all the three xylene isomers are strongly adsorbed. The constants are calculated to be $2\text{–}4 \times 10^{-3}$, which are much higher than the reported values of *o*-xylene adsorbed on mesoporous silica (Huang et al. 2006). This is because the FDU-15 materials are mainly hydrophobic, leading to a high adsorption affinity toward the hydrophobic xylene isomers. Interestingly, Henry's constants on the two mesoporous materials show the same trend, i.e., *o*-xylene > *m*-xylene > *p*-xylene, suggesting the materials show greater affinity to more polar molecules than that to less polar or non-polar molecules. This is because

Table 2 Henry's constants (K_H) and equilibrium adsorption capacities (q) of xylene isomers on ordered mesoporous FDU-15 materials at different temperature (25–40 °C)

Adsorbent	Adsorbate	t (°C)	q (mmol g^{-1})	K_H ($\times 10^{-3}$)
FDU-15-350	*o*-xylene	25	0.83	3.2
		30	0.85	3.3
		35	0.82	3.3
		40	0.81	3.1
	m-xylene	25	0.96	2.6
		35	0.94	2.3
		40	0.93	2.4
	p-xylene	25	1.07	2.6
		30	1.05	2.4
		35	1.00	2.1
		40	0.95	1.6
FDU-15-900	*o*-xylene	25	2.38	4.5
		30	2.40	4.6
		35	2.40	4.4
		40	2.41	4.5
	m-xylene	25	2.40	4.5
		35	2.49	4.4
		40	2.47	4.6
	p-xylene	25	2.17	3.4
		30	2.18	3.5
		35	2.09	3.6
		40	2.21	3.4

FDU-15-350 polymer and even the FDU-15-900 carbon are composed of a high content of oxygen atoms, which endows the pore surface with polarity. Thus the materials show higher affinity to more polar xylene isomer (the polarity is *o*-xylene > *m*-xylene > *p*-xylene). This phenomenon is similar to the adsorption affinity sequence of different hydrocarbons with different polarity adsorbed on mesoporous silica (Zhao et al. 2001). Another interesting phenomenon is that the Henry's constants for adsorption of xylene isomers on FDU-15-900 carbon material are systematically larger than those on FDU-15-350 polymer. It is expected that the Henry's constant for an adsorbate increases with increasing micropore volume of the adsorbent. Because the FDU-15-900 carbon material has much higher micropore surface area and micropore volume, it shows larger adsorption affinity towards the xylene isomers than polymer FDU-15-350 does. All the Henry's constants at 25–40 °C are comparable (Table 2). However, the ratios of Henry's constants or selectivities at the same temperatures for the xylene isomers are low (<2). It suggests that both of the materials have low potential for separation of these xylene isomers. Nevertheless, because the FDU-15 materials show good adsorption capacities and large adsorption affinities towards xylene isomers, they are still promising adsorbents for removing these toxic organic compounds in waste water or air resources.

3.4 Evaluation of the equilibrium isotherm model

The adsorption equilibrium data were fitted by the Langmuir isotherm model presented in equation (1). The linear fitting curves at 25 and 40 ° C are shown in Fig. 6. Apparently, the experimental data are well represented by this model. FDU-15-900 carbon shows higher $q_m k$ (equal to K_H values) (Table 2) than FDU-15-350 polymer. This result further reflects the fact that the interaction of xylene isomers with the adsorbents' surface is strongly related to the framework microporosity. The adsorbent with higher framework microporosity has a stronger interaction with aromatic hydrocarbon molecules than its counterpart with lower framework microporosity. Similar observations were reported for alkenes adsorbed on SBA-15 materials by Newalkar et al. (2002, 2003).

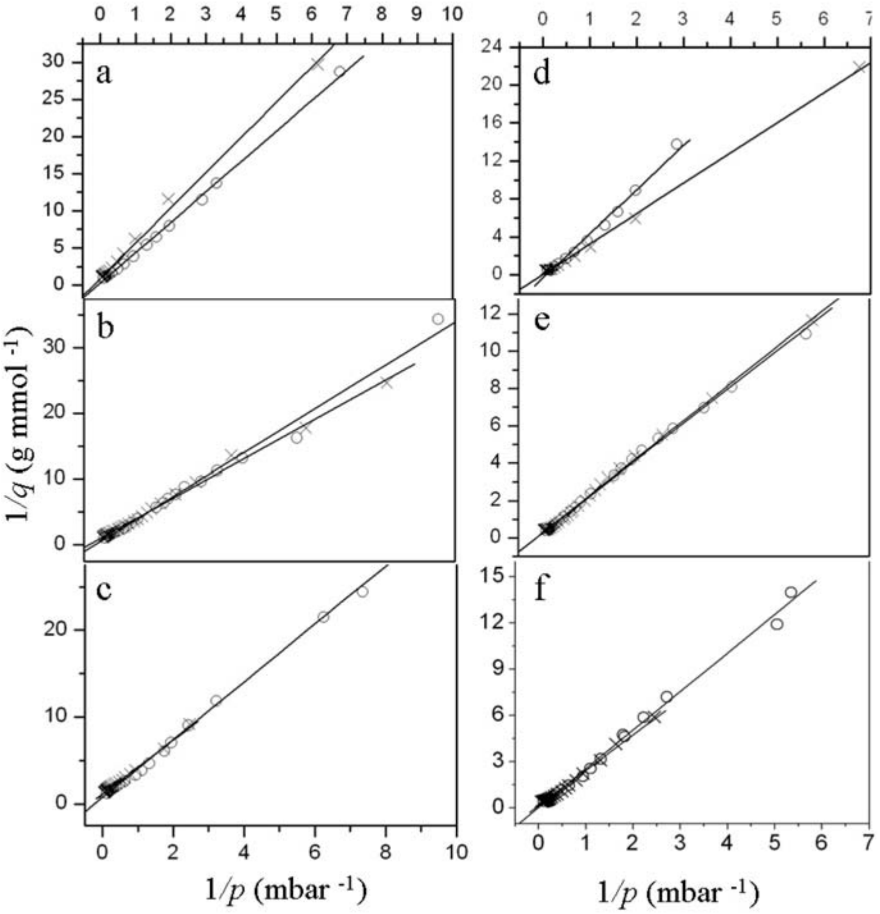

Fig. 6 Langmuir plots for adsorption isotherms of p-xylene (**a**), m-xylene (**b**) and o-xylene (**c**) isomers on mesoporous polymer FDU-15-350 and p-xylene (**d**), m-xylene (**e**) and o-xylene (**f**) isomers on carbon FDU-15-900 materials at 25 (○) and 40 °C (×)

3.5 Isosteric heats of adsorption

Isosteric heats of adsorption (ΔH_{ads}) were calculated from the measured adsorption equilibrium data at different temperatures using the Clausius–Clapeyron equation presented in equation (2). The dependence of isosteric heats of adsorption is related to the combined effect of adsorbate–adsorbent and adsorbate–adsorbate interactions (Cao and Sircar 2001). The relevant information obtained from that analysis can provide useful information to evaluate the energetic heterogeneity of interactions on the surface of the adsorbent. For FDU-15-350, ΔH_{ads} gradually increases when the loading of the xylene isomers is increased (Fig. 7). This fact is probably attributed to the particular adsorbate–adsorbent and adsorbate–adsorbate interactions due to high energetic interactions. It has been reported that high energetic interaction between n-alkane hydrogen atoms and oxygen atoms of silanol groups leads to the phenomenon that the isosteric heats of alkane adsorption on silica materials increase with increasing adsorbate loadings (Hernández and Velasco 2003). Herein, FDU-15-350 has a large number of oxygen atoms and aromatic benzene rings, thus it is reasonably to deduce that there are high energetic interactions between the polymer material and the aromatic xylene isomers. Moreover, ΔH_{ads} increases with loading and the shapes of ΔH_{ads} versus loading plots are indicative of an adsorption phenomenon that roughly occurs on an energetically uniform (with respect to the distribution of adsorption sites) surface of FDU-15-350. However, for FDU-15-900, unlike FDU-15-300, the overall trend of ΔH_{ads} for all the three isomers shows a maximum value at a low loading and then a gradual decrease with increasing adsorbate loading. That is because the interaction between adsorbate and adsorbent surface is higher than the bulk adsorbate interaction. The most energetic sites on the adsorbent surface for isomer adsorption are in the fine micropores because of overlapping of the potential fields generated by the micropore walls. The most energetic sites are taken up first by adsorbate and then less energetic ones are filled, leading to a gradual decrease of heat of adsorption evolved with loading. It is worth mentioning that the kinetic molecular sizes of the three isomers are following a trend $o \sim m > p$ (in one particular orientation). In our case, the highest heats of adsorption on FDU-15-900 are observed for p-xylene isomer which is the smallest adsorbate if only one orientation is considered. Although the adsorbent surface is not polar, the adsorbate sizes are also influential on heats of adsorption.

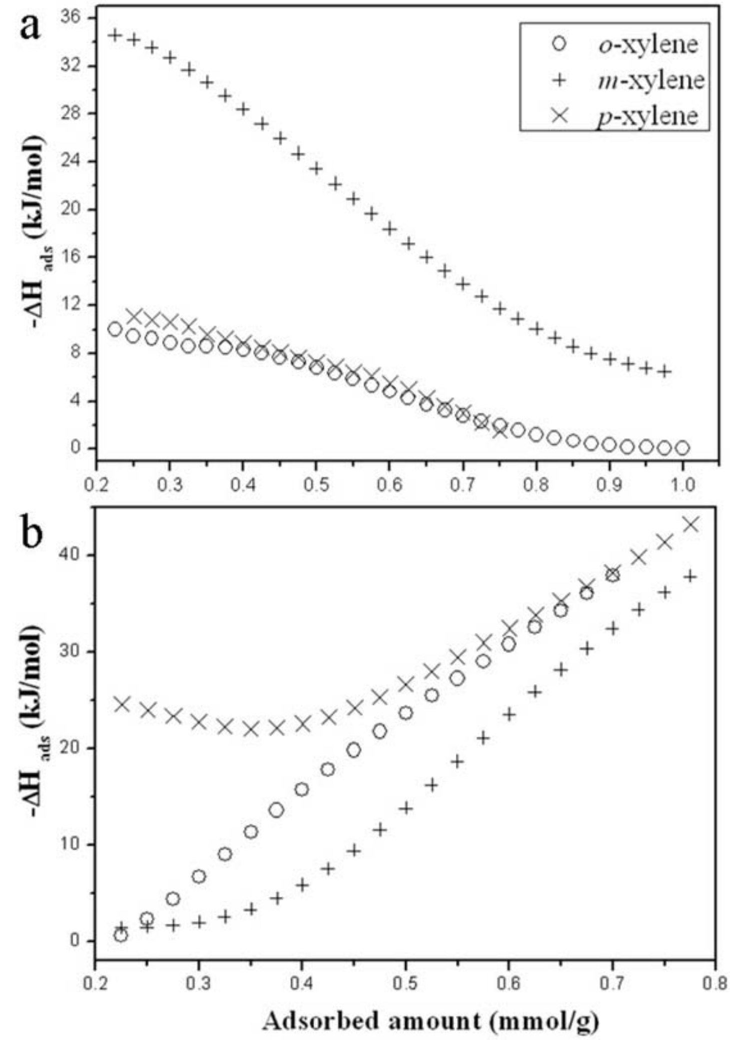

Fig. 7 Dependence of isosteric heats of adsorption ($-\Delta H_{\mathrm{ads}}$) on loadings for *o*-xylene (○), *m*-xylene (+), and *p*-xylene (×) for mesoporous polymer FDU-15-350 (**a**) and carbon FDU-15-900 (**b**) materials

4 Conclusions

By using ordered mesoporous polymer and carbon materials FDU-15 as adsorbents for isomers *o*-xylene, *m*-xylene and *p*-xylene adsorption, the adsorption properties of the three isomers were investigated. Micropores in the adsorbent materials are fully accessible for these three isomers with smaller isomer showing the greatest capacity. Henry's constants reveal that in both of the adsorbents, the most polar isomer is the most preferred. Heats of adsorption of the isomers on polymer FDU-15-350 show an increasing trend with the increase of adsorbate loading, suggesting large energetic interaction between adsorbates and adsorbent. However, an opposite case occurs in carbon FDU-15-900, suggesting that much more heterogeneous interactions between adsorbate and adsorbent surface is developed. An effort is also attempted to extract the pore volume and surface area of the adsorbents by calculating the adsorbed adsorbate volume, assuming that adsorbates have the same density as their liquid phases. They are very close to the micropore volume and micropore surface area obtained from nitrogen adsorption, implying that these small isomers can be used as adsorbates to obtain the pore structure and property information. Nonselectivity between the adsorbates reflected by the close Henry's constants is observed, revealing that the separation of the isomers by these two adsorbents is not feasible. However, promising adsorption capacities and high adsorption affinity of the isomers on these adsorbents still suggest the materials great potential in removing toxic hydrocarbons from the environment.

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