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Removal of VOCs by activated carbon microspheres derived from polymer: a comparative study

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Abstract It is of great concern to develop a high-efficient adsorbent for removing halomethanes, because they are harmful to human health. In this work, the activated carbon microspheres (ACMs) derived from spherical copolymer of vinylidene chloride and styrene were used to adsorb these hazardous pollutants from gases. The BET surface area of the ACM was 1104 m^2/g and mostly dedicated by the micropores between 0.4 and 1.5 nm. The micropore dominating the nature of the ACM renders eminent adsorption capacity. The uniform scales on the surface and expedite passages inside the ACM offer excellent mass transfer performance during adsorption process. To assess the adsorption behavior of the ACM, dynamic adsorption tests were conducted at different temperatures, feed concentrations, and gas velocities. For comparison, a typical commercial activated carbon made from coconut shell was also tested under identical conditions. The results show that the ACM has large adsorption capacity, good adaptability and easy recyclability, which renders high potential and cost-effectiveness for commercial utilization.

Keywords Activated carbon microspheres · Dynamic adsorption · Halomethanes · Recyclability · Adsorption thermodynamics · Volatile organic compounds (VOCs)

1 Introduction

Volatile organic compounds (VOCs) are ubiquitous and hazardous to human health, and halomethanes are among the most toxic VOCs. Halomethanes are widely used as reactants, solvents, refrigerant, fire extinguishing agent, fumigant, anesthetic, spraying agent, and foaming agent. But they may cause serious detriments to neural system, respiratory system, liver, kidney, skins, and mucosa of human beings. Halomethanes may also cause severe air pollution, especially the iodomethane containing radioactive iodine (González-garcía et al. 2011; Metts and Batterman 2006; Yamamoto et al. 2010). The atmospheric dispersal of the radioactive iodomethane leaked from nuclear power plant is a disaster to mankind all over the world (Liu et al. 2009), just as we have recently encountered after the explosion of Fukushima nuclear plant in Japan. Consequently, removal of halomethanes from gases is of great importance. Various methods have been utilized to treat this problem (such as adsorption, catalysis, and biotechnology), among which adsorption plays a predominant role.

Activated carbons are widely investigated as adsorbents to remove VOCs from gases and they can recover valuable organic vapors (Vu et al. 2009; Shim and Kim 2010; Lillo-Rodenas et al. 2005; Carrasco-Marin et al. 2009; Ramos et al. 2010). The triethylenediamine-impregnated activated

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carbons are mostly used to get higher adsorption capacity of halomethanes. However, such type of adsorbent exhibits undesirable high water affinity and the adsorption performance can be seriously thwarted by water vapor (Kopečni et al. 1990; Park et al. 1995; Peterson et al. 2010). Moreover, triethylenediamine-impregnated activated carbons are difficult to regenerate and recycle when they are chemically saturated. Therefore preparation of activated carbons with outstanding adsorption performance is crucial for disposing of halomethanes in gases. Compared with natural materials, synthetic polymers are competitive raw materials for preparing activated carbons, because particular pore structure and surface morphology can be obtained by tailoring polymer structure (Qian et al. 2008; Zhu et al. 2008). Despite some scientific researches on the synthetic polymers derived activated carbons (Gao et al. 2014; Sun et al. 2014), there are seldom publications on halomethanes removal from gases with polymer derived activated carbons.

In this paper, we report the preparation of polymer derived activated carbon microspheres (ACMs) and their excellent performance in removing halomethanes from gases in dynamic adsorption tests. For comparison, we tested a typical commercial activated carbon (AC) made from coconut shell at identical adsorption conditions. The AC possessed high adsorption performance (iodine number: 1169 g/kg), and it can represent the activated carbons with high-class adsorption properties (Wu et al. 1999). The adsorption thermodynamics for the high-efficient adsorption of halomethanes on the ACM were also investigated. Moreover, the performance of the regenerated ACM was also tested to evaluate its reusability. This work is also instructive for the design and preparation of other adsorbents for removing hazardous halomethanes.

2 Materials and methods

2.1 Adsorbates

As ozone-depleting agent, the usage of methyl bromide in industrialized countries has been banned since 2005 (Gan et al. 2001). Consequently, we selected chloromethanes (CH_2Cl_2, $CHCl_3$, CCl_4) and iodomethane (CH_3I) as adsorbates. The CH_3I was the crux of this paper, because CH_3I is not only highly toxic but also a major carrier of radioactive iodine (González-garcía et al. 2011; Metts and Batterman 2006; Yamamoto et al. 2010; Liu et al. 2009). The adsorbates used in the experiments were analytical grade and purchased from Sinopharm Chemical Reagent Beijing Co., Ltd. The properties of the adsorbates are listed in Table 1.

2.2 Adsorbents

The ACM was prepared by carbonization and steam activation of spherical copolymer of vinylidene chloride and styrene. Briefly, distilled vinylidene chloride and styrene (0.01–0.02 mol%) was polymerized in suspending agent (saturated Na_2SO_4 aqueous solution). The initiator was azobisisobutyronitrile (ABVN) [1 mol% of monomers]. Polyvinyl alcohol (PVA) was used as dispersing agent. Suspension polymerization was kept at a constant temperature of 40 °C for 12 h and then the polymeric beads were obtained. The polymeric beads were washed with hot water and purified in an acetone stream. The carbonization was carried out in a quartz tube, and the temperature was controlled at 180 °C for 24 h, then the temperature was gradually elevated to 1000 °C and kept for 5 h, at this temperature, the chlorine atom inside the precursor could be totally removed, which derived more micropores. All the above processes were conducted in an argon stream of 50 mL/min. The slight steam activation was conducted at 700 °C for 5 min to make the inner pores more accessible. After these steps, the ACM of about 20–60 mesh in size was prepared.

The AC made from coconut shell was purchased from Tangshan Jianxin Activated Carbon Corporation. The AC was in irregular shape and the particle size of AC was 20–60 mesh. The iodine number of AC was 1169 g/kg. The adsorption performance of activated carbons for low-molar-mass species is generally indicated by the iodine number determined according to standard test method (Gergova et al. 1993). The iodine number of commercially available activated carbons is mainly between 600–1000 g/kg (Gan et al. 2001). Therefore the AC selected in this paper can represent the activated carbons with excellent adsorption performance. The adsorbents were fastened in

Table 1 Chemical properties of the adsorbates

Adsorbate	CAS no.	Molecular weight (Da)	Boiling point (°C)	Dipole moment (Debyes)	Liquid density (g/cm^3) (20 °C)	Cross sectional area (nm^2)
CH_2Cl_2	75-09-2	84.93	39.8	1.8	1.327	0.25
$CHCl_3$	67-66-3	119.38	61.6	1.1	1.489	0.28
CCl_4	56-23-5	153.82	76.8	0	1.594	0.32
CH_3I	74-88-4	141.95	42.5	1.59	2.279	0.17

the adsorption column. The packed densities of ACM and AC in the adsorption bed were 0.46 and 0.41 g/cm^3 respectively. The SEM images of ACM were obtained by Hitachi S-4300 Scanning Electron Microscope (SEM). The porosity properties of the adsorbents were characterized by nitrogen adsorption conducted on Quantachrome Autosorb Automated Gas Sorption System. The surface chemistry of the adsorbents was analyzed by X-ray photoelectron spectroscopy (XPS) from VG Scientific using 300 W AlKα radiation. The X-ray diffraction (XRD) analysis was tested on Rigaku D/max2500 with Cu-Kα radiation. The ACM sample was crushed to fine powder before XPS and XRD analysis.

2.3 Adsorption test

Dynamic adsorption test was conducted in a fixed bed adsorber (Fig. 1). The adsorber was a U-type glass tube with 10 mm diameter and placed in an water bath to control the adsorption temperature. The adsorbent loaded in the adsorber was 8 mL (The weights of ACM and AC were 3.68 and 3.28 g respectively) and was fastened and fixed with asbestos cloth at both ends. In order to regulate the gas temperature passing through the adsorbent, 20 mL glass beads (30 mesh) were placed before the inlet end of the adsorbent and also fixed with asbestos cloth. To supply the mixed gas, we let nitrogen gas pass through a vessel containing the specified adsorbate. The adsorbate content in the mixed gas can be regulated and stabilized by controlling the temperature of the vessel. When we conducted the adsorption experiment, the mixed gas as feed passes through the adsorber, and the adsorbate content in the gas before and after the adsorber was determined by gas chromatography with thermal conductivity detector. The stationary phase used in packed column of the chromatography was GDX104. The gas velocity can be regulated with a valve and was determined by a volumetric flow meter. The breakthrough time was set at the ratio of the breakthrough concentration (C) to the initial concentration (C_0) equal to 0.01. Then the breakthrough capacity, denoted as 'q', of halomethane adsorption was calculated using the gas velocity, feed content, and breakthrough time and weight of carbon sample.

2.4 Regeneration of the saturated ACM

To investigate the reusability, the ACM saturated by CH_3I was regenerated in situ. The temperature of the adsorber containing saturated ACM was elevated to 220 °C in a nitrogen gas stream of 300 mL/min. The outlet gas of the adsorber was cooled and passed through a conical flask with *n*-octane to recover the CH_3I vapor, and the outlet gas of the conical flask was passed through another conical flask with triethylenediamine solution to remove the CH_3I residue. During the regeneration process, the outlet gas of the adsorber was analyzed by gas chromatography. After 3 h, the CH_3I in the outlet gas could not be detected, hence the regeneration of saturated ACM was finished. The regenerated ACM was then tested at the same conditions as the new adsorbent to assess its reusability.

3 Results and discussion

3.1 Characterization of the adsorbents

3.1.1 Pore structure and surface morphology

The ACM is in regular round shape (Fig. 2a). The ACM has orderly scale-like surface (Fig. 2b), and the uniform

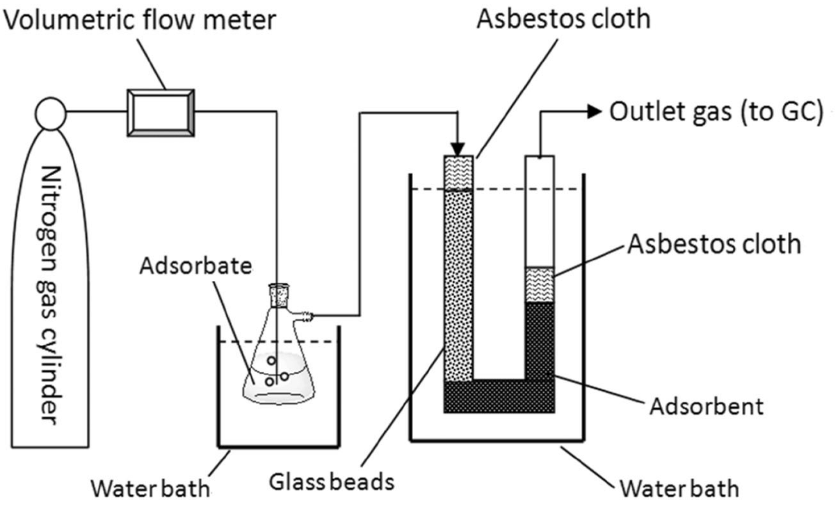

Fig. 1 Schematic diagram of the set-up for the experiments

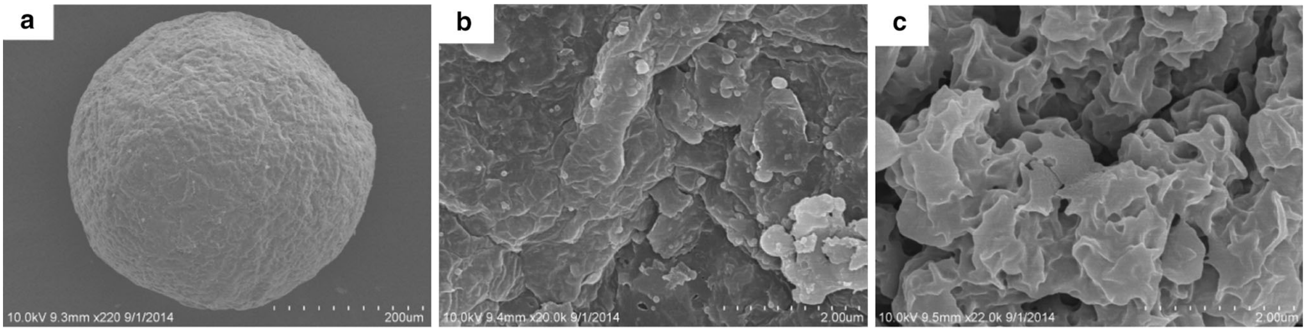

Fig. 2 The SEM images of ACM: **a** global; **b** surface; **c** cross sectional

gaps between scales offer open entry to the bulk. In addition, there are plenty of expedite passages within the bulk of the ACM (Fig. 2c), which join the inner pores well with the environment outside the ACM. It was reported that the diffusion of adsorbate in the micropores on the surface layer of the adsorbents is rather low and open channels to the micropores are critical to mass transfer rate (Yamamoto et al. 2004). Thus the surface morphology and inner structure of the ACM were beneficial to the adsorption performance. The data of nitrogen adsorption analysis of ACM and AC are revealed in Fig. 3, from which we can obtain the porosity properties of the adsorbents. The isotherm of ACM is best described as type I according to IUPAC classification. The porosity properties of the adsorbents are listed in Table 2. The specific surface area of ACM is 1104 m^2/g and mostly dedicated by micropores (94.4 %). Whereas for AC the specific surface area is 1421 m^2/g and only about 30 % is contributed by micropores. The pore size distribution of ACM and AC is revealed in Fig. 4. The micropore dominating nature and peculiar morphology of ACM is mainly formed by the dehydrochlorination of the selected polymer precursor, and the aim of the steam activation procedure is just to make the inner pores easier to access by the adsorbate molecules.

3.1.2 Surface chemistry

The surface chemistry of the adsorbents was analyzed by XRD and XPS. There were two peaks at nearly the same position of the XRD profile for ACM and AC, revealing that they both had predominantly amorphous structure. The XPS data of the two adsorbents indicated that both adsorbents were mainly composed of C and O elements. The binding energy of C 1s in both adsorbents was 284.8 ± 0.3 eV, which corresponds to ordered carbon, graphite carbon, and carbon bonded with oxygen. The binding energy of O 1s was 532.7 ± 0.3 eV, which coincides with that of carboxyl group and carbonyl group. Furthermore, the contents of C and O in both adsorbents were near. In a word, the adsorbents had little difference in surface chemistry.

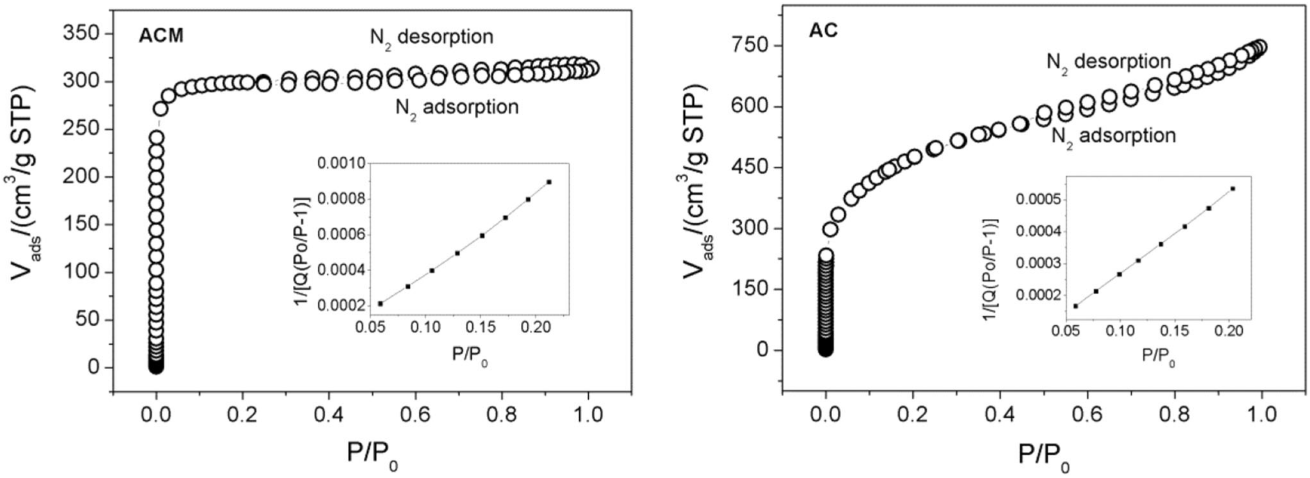

Fig. 3 N_2 adsorption isotherm and BET plot (*inset*) of ACM and AC

Table 2 Porosity properties of the adsorbents

Adsorbent	S^{a}_{BET} (m^2/g)	S^{b}_{micro} (m^2/g)	V^{c}_{t} (cm^3/g)	V^{d}_{micro} (cm^3/g)	D^{e}_{mean} (nm)
ACM	1104	1042	0.448 ($P/P_0 = 0.9929$)	0.439	1.79
AC	1421	415	1.148 ($P/P_0 = 0.9939$)	0.174	2.72

[a] BET surface area

[b] t-plot micropore area

[c] Single point adsorption total pore volume

[d] t-plot micropore volume

[e] Mean pore diameter

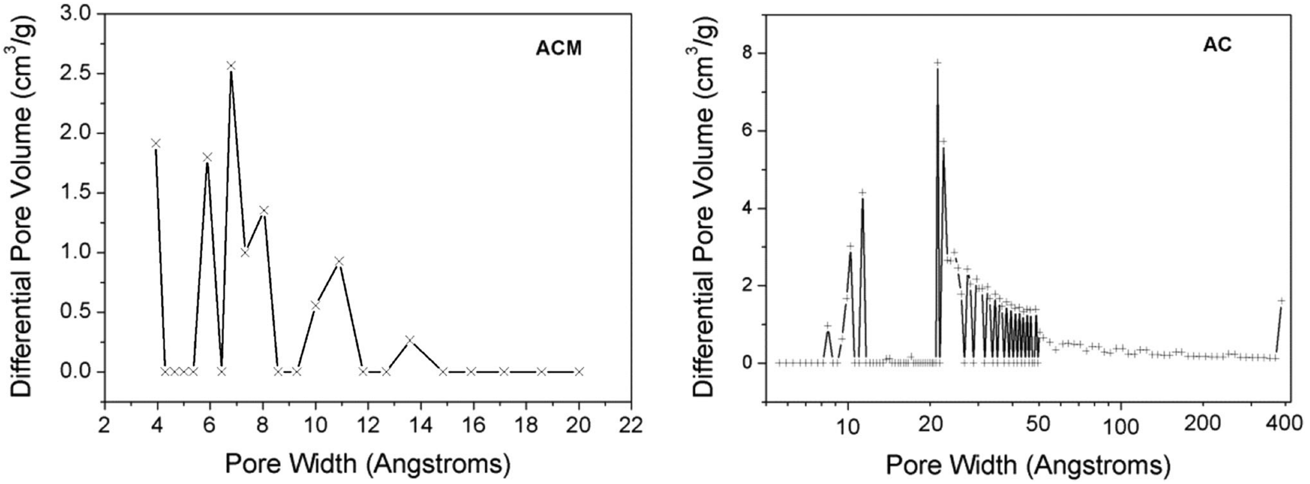

Fig. 4 The pore size distribution of ACM and AC

3.2 Removal of halomethanes under different conditions

3.2.1 Impact of adsorption temperature

Generally speaking, the adsorption of VOCs on activated carbons can be attributed to physical adsorption, in which temperature plays an important role (Dou et al. 2011). To evaluate the impact of adsorption temperature, the dynamic adsorption test of halomethanes was conducted at 20 and 60 °C, respectively. The results of the adsorption test are shown in Fig. 5, in which the breakthrough curves are steep. Hence the adsorption capacity at different conditions can be roughly compared according to the breakthrough time. As a whole, the breakthrough time of the adsorbents decreased evidently with elevating temperature, which is the typical feature of physical adsorption. It is clear that the adsorption capacity of ACM was higher than that of AC at the same temperature, especially for CH_2Cl_2 and CH_3I. However, the adsorption capacities of CH_2Cl_2 and CH_3I on the adsorbents were greatly affected by temperature. When the adsorption temperature increased from 20 to 60 °C, the adsorption capacities of CH_2Cl_2 and CH_3I on ACM decreased 46.2 and 47.4 %, respectively.

The data of adsorption capacity of ACM, indicated as '*q*', are listed in Table 3. The unit of *q* was converted into mL/g according to the data of liquid adsorbate densities at 20 °C from Table 1. It is obvious that the *q* values of all adsorbates at 60 °C are close to the micropore volume of ACM. From Table 3, we also learn that the *q* values of different adsorbates at 60 °C directly correlate with their molecular size. In particular, *q* value of CCl_4 at 60 °C is less than those of other adsorbates, although the boiling point of CCl_4 is the highest among them. This fact further demonstrates the predominance of the adsorbate molecular size on adsorption within micropores. This phenomenon agrees well with the results of other researchers. Carrasco-Marin confirmed that adsorption mainly takes place in micropores commensurate with the size of adsorbate, although adsorbent-adsorbate interactions are nonspecific (Carrasco-Marin et al. 1991). Koresh also proved that maximum interaction occurs if the pore size fits the adsorbate size (Koresh et al. 1985).

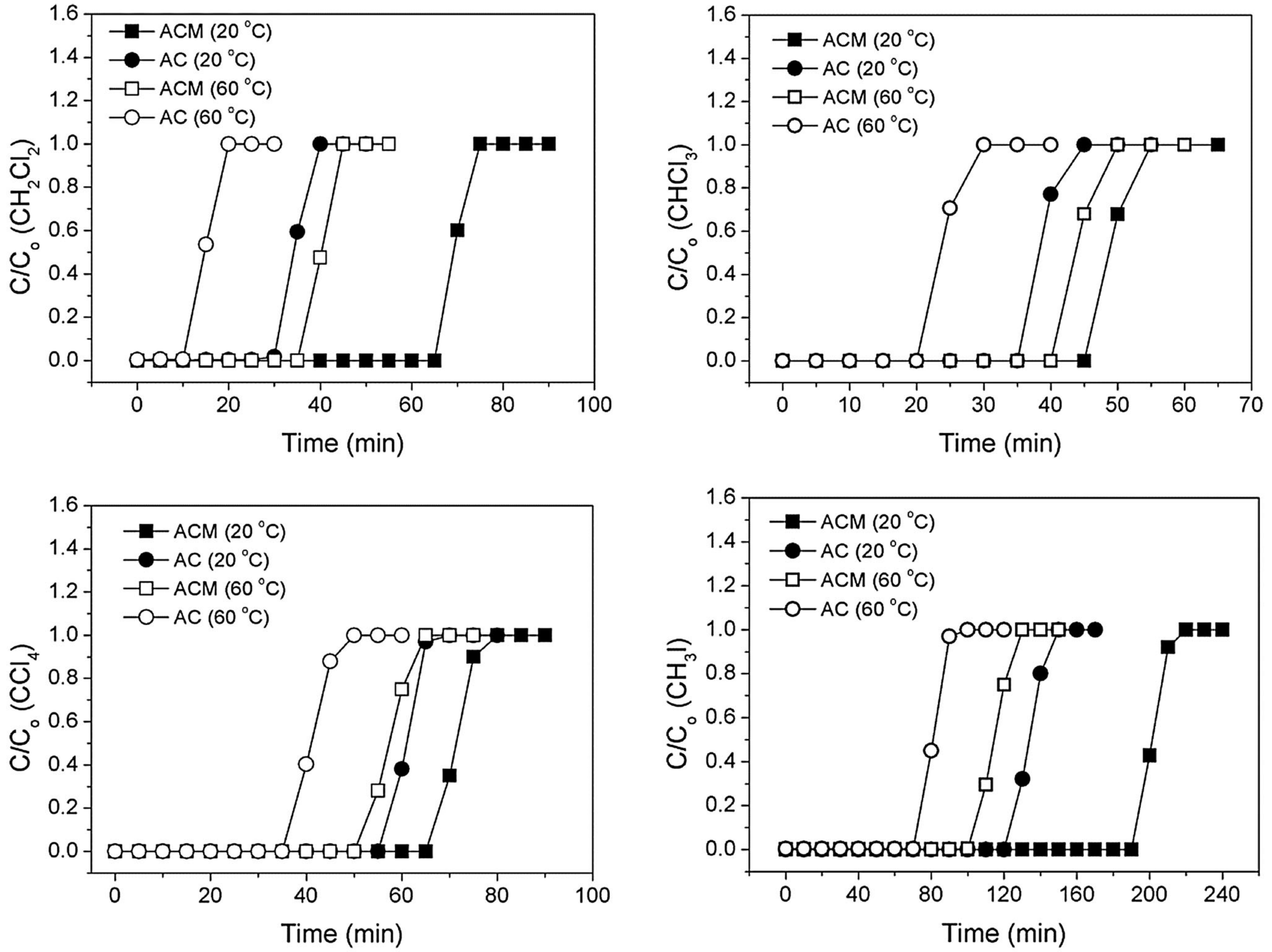

Fig. 5 Impact of temperature on dynamic adsorption of halomethanes (Gas velocity: 200 mL/min; feed contents: CH_2Cl_2 11.0 mol%, $CHCl_3$ 6.0 mol% CCl_4 4.0 mol%, CH_3I 4.0 mol%)

Table 3 Adsorption capacity of the adsorbates on ACM

Adsorbate q (mL/g)	CH_2Cl_2	$CHCl_3$	CCl_4	CH_3I
$q_{20\,°C}$	0.856	0.453	0.469	0.880
$q_{60\,°C}$	0.461	0.404	0.361	0.463

The t-plot micropore volume of ACM is 0.439 mL/g. (The q values were calculated based on the liquid densities of adsorbates at 20 °C)

Interestingly, the q values of CH_2Cl_2 and CH_3I at 20 °C were nearly twice of those at 60 °C, although both adsorbates have lowest boiling points. Whereas the q values of other two adsorbates at 20 and 60 °C had moderate difference. It is probably because of the higher dipole moment of CH_2Cl_2 and CH_3I (see Table 1). The dipole–dipole interaction can lower intermolecular potential energy between adsorbate molecules and render more stable adsorption (Do and Do 2007). To make clear the possible condensation of the CH_2Cl_2 and CH_3I in the adsorption column, we conducted blank tests where the vapors directly passed through the column without adsorbent, but no obvious adsorbate condensation on the column surface can be observed during an hour. Thus at 20 °C the CH_2Cl_2 and CH_3I were mostly adsorbed by the adsorbent inside the column. We can divide the adsorption of halomethanes at 20 °C into two parts: adsorption within micropores and adsorption outside micropores (i.e., meso- and macropores of the ACM) caused by dipole–dipole interaction. At 60 °C, the kinetic energy of CH_2Cl_2 and CH_3I molecules may surmount dipole-dipole interaction between adsorbate and adsorbent outside the micropores, but the adsorbates in micropores are mostly retained.

3.2.2 *Impact of feed content*

The adsorption of halogenated VOCs is also affected by the adsorbate content in the gases (Gan et al. 2001). To evaluate the impact of feed content, we selected CH_3I as model adsorbate and conducted dynamic adsorption test at 20 and

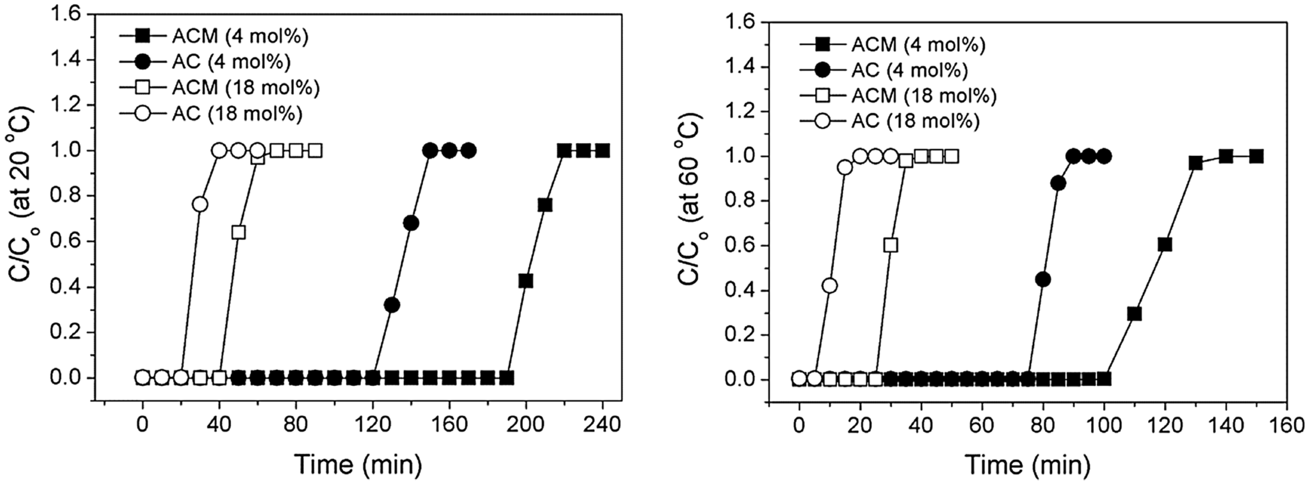

Fig. 6 Impact of feed content on dynamic adsorption of iodomethane at 20 and 60 °C (gas velocity: 200 mL/min)

60 °C. The feed contents were 4.0 and 18.0 mol% respectively, and other conditions were identical. The results are revealed in Fig. 6. The adsorption capacity of ACM has little change with changing feed content. In contrast, the breakthrough time of AC undergoes severe reduction with elevated feed content, especially at higher temperature.

3.2.3 *Impact of gas velocity*

For dynamic adsorption test, the performance of the adsorbent can also be affected by gas velocity (Delage et al. 2000; Rehrmann and Jonas 1978). To assess this factor, we also conducted dynamic adsorption test of CH_3I at different gas velocities (200 and 400 mL/min). The results are shown in Fig. 7. The breakthrough time reduced nearly in proportion to the elevating gas velocity, indicating that the adsorption capacity of ACM had little change. This can be attributed to the peculiar structure of the ACM. There is a strong possibility that the lower tortuosity of the micropores in ACM shortened the diffusion pathway of adsorbates into the adsorption sites. As for AC the mass transfer performance was unsatisfactory, because its adsorption capacity reduced 33 % due to the gas velocity elevation. Many adsorbate molecules may escape unadsorbed as the flow rate increases. In addition, at higher flow rates the already adsorbed molecules tend to desorb and be carried away by the feed flow. These results are also in agreement with those referred to in the literature (Das et al. 2004; Mohan et al. 2009).

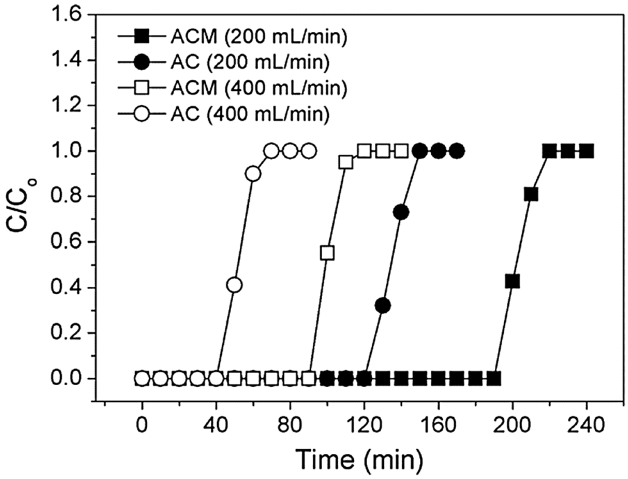

Fig. 7 Impact of gas velocity on dynamic adsorption of iodomethane (temperature: 20 °C, feed content: 4.0 mol%)

3.3 Adsorption thermodynamics of halomethanes on ACM

The adsorption capacity of these four halomethanes decreased with the increased adsorption temperature, as indicated in Table 3. The adsorption properties of halomethanes on ACM can also be estimated by enthalpy change (ΔH). Applying the Vant Hoff equation, the relationship between enthalpy and adsorption coefficients can be quantified as

$$\frac{dlnK}{dT} = \frac{\Delta H}{RT^2} \tag{1}$$

where R is the ideal gas constant, T is the absolute temperature (K), and K is the equilibrium ACM-gas distribution coefficient. In this study, ΔH is assumed constant over the adsorption temperature range. The low negative values of adsorption enthalpy for these four VOCs can be calculated, ranging from −18 to −40 kJ/mol (see Table 4). According to the results, the adsorption of these VOCs must be physical and exothermic process. The greater adsorption enthalpy in this study means stronger interactions

Table 4 Adsorption enthalpies of the adsorbates on ACM

Adsorbate	CH_2Cl_2	$CHCl_3$	CCl_4	CH_3I
ΔH (kJ/mol)	−40	−20	−18	−38

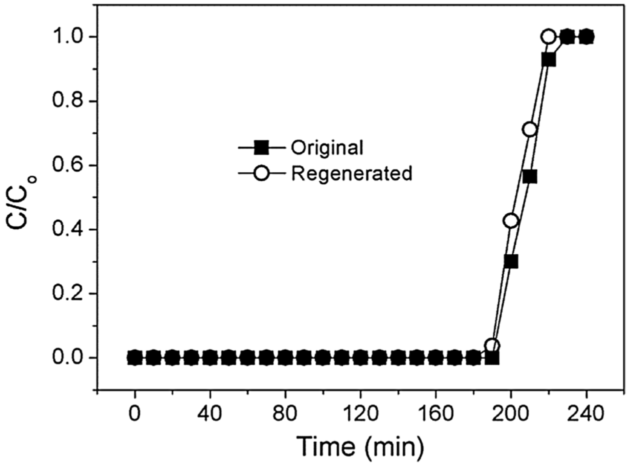

Fig. 8 Comparison tests on dynamic adsorption of iodomethane by new and regenerated ACM (gas velocity: 200 mL/min, feed content: 4.0 mol%, adsorption temperature: 20 °C)

of the adsorbates with ACM. The enthalpy data are in agreement with the results of *q* values from Table 3.

3.4 Reusability of the saturated ACM

To evaluate the reusability of the saturated ACM, we conducted the dynamic adsorption tests of CH_3I on the regenerated ACM at identical conditions to that of the new adsorbent, and the results are shown in Fig. 8. The adsorption capacity of the regenerated ACM was very close to that of the new one. Thus the ACM is suitable to be reused after saturation. The easy recyclability of the ACM offers good cost effectiveness for commercial utilization.

4 Conclusions

The ACM is highly efficient in removing halomethanes from gases. Firstly, the ACM has large adsorption capacity, which is the prerequisite for high-level requirements. Secondly, the ACM can sustain its performance at different feed content and gas velocity, which ensures its adaptability in practical applications. The eminent adsorption properties of ACM originate from its peculiar pore structure and surface morphology. The micropore dominating nature of ACM renders stable adsorption sites, which can resist temperature change and sustain its adsorption capacity. The ACM is in regular round shape, and has orderly scale-like surface and inside expedite passages, which offer outstanding mass transfer properties. The saturated ACM can be easily regenerated and maintain the same adsorption performance as the new adsorbent. In a word, the ACM has large adsorption capacity, good adaptability and easy recyclability, which renders high potential and cost-effectiveness for commercial utilization.

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