Adsorption behavior of CO_2 on amine-functionalized polyacrylonitrile fiber

Yizhu Kuang[1] · Hui He[1,3] · Shuixia Chen[1,2] · Jingjie Wu[1] · Fenglei Liu[1]

Abstract

Using efficient and stable solid amine adsorbents to capture CO_2 has been considered as an effective technology to reduce the CO_2 concentration in the atmosphere. In this paper, a series of solid amine-containing fibrous adsorbent for CO_2 capture were prepared by direct modification of polyacrylonitrile (PAN) fibers with amination reagents, including diethylenetriamine (DETA), triethylenetetramine (TETA), tetraethylenepentamine (TEPA) and polyethyleneimine (PEI). Abundant amine groups can be introduced on the PAN surface owing to the reactivity between cyano group and amino group. The effects of the type and structure of amination reagents on the swelling properties and CO_2 adsorption capacities of the as-prepared adsorbents were investigated. The results indicated that chemical modification of PAN fibers with amine compounds could greatly increase the CO_2 adsorption capacity of the aminated adsorbents. The adsorption capacities of the adsorbents were correlated well with the content of amino groups. PAN–TETA and PAN–TEPA showed higher CO_2 adsorption capacities and better stability than PAN–DETA and PAN–PEI. Besides, as water molecules could take part in and facilitate the CO_2 adsorption, the CO_2 adsorption capacity of amine-modified fibers would be strongly dependent on the swelling property.

Keywords PAN · Solid amine · Adsorbent · CO_2 adsorption · Swelling

1 Introduction

Atmospheric CO_2 concentration has increased rapidly over the past century owing to the increase of consumption of fossil fuels, and as one of the primary greenhouse gases (GHGs), CO_2 with overhigh concentration could lead to serious environmental issues such as global warming (Li et al. 2011) and natural disasters (i.e. widespread melting of ice and snow, floods, droughts and increasingly average sea levels) (Parry 2007; Kenarsari et al. 2013). Therefore the development of efficient and selective CO_2 capture techniques to reduce atmospheric CO_2 concentration has attracted great research interest. Quantities of processes have been developed for the removal of carbon dioxide from flue gases and other gas streams, including physical/chemical absorption (Rubin et al. 2012; Padurean et al. 2012), adsorption (Gargiulo et al. 2014; He et al. 2016), and membrane separation technology (Geyer et al. 2017). Among them, the most widely applied technology is to use liquid amine sorbents with high CO_2 absorption capacity to absorb CO_2. However, there are some obvious inherent drawbacks, such as high absorbent consumption caused by its high volatility, serious reactor equipment corrosion caused by its high alkalinity and high energy consumption to regenerate the absorbents. (Chaffee et al. 2007; Kang et al. 2016). In order to avoid these disadvantages, many researchers have tried to develop some promising adsorbents to adsorb CO_2. The solids containing amines that prepared through bonding active amine materials to high surface area supports like carbon based materials (Chowdhury et al. 2015; Creamer and Gao 2016), zeolite (Song et al. 2017; Daeho et al. 2017), metal organic frameworks (MOF) (Masala et al. 2017) and polymers resin (Chen et al. 2013; Liu et al. 2017) can be the competitive adsorbents. Compared with liquid amine absorbents, these amine-modified materials could exhibit many merits, including high adsorption capacity, low cost, low pressure, well

✉ Shuixia Chen
 cescsx@mail.sysu.edu.cn

1 PCFM Lab, School of Chemistry, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China

2 GDHPPC Lab, Materials Science Institute, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China

3 College of Light Industry and Food Engineering, Guangxi University, Nanning 530004, People's Republic of China

regeneration performance and low regeneration energy consumption (Samanta et al. 2012).

There are two methods to introduce amine groups to the substrates to prepare amine-modified solid adsorbents. One method is to physically impregnate amine materials into suitable support materials. But generally the recycle performance of the adsorbents prepared with this method are not good enough, because the physically impregnated amine could leak easily in the adsorption and regeneration processes. Another method is to chemically graft amine groups to the substrates. Due to their wide range of sources, light weight, plentiful functional groups, short transfer distance and highly modifiable surface, the polymeric fibers have been considered as good substrates for preparation of amine-modified solid adsorbents for CO_2 capture. In comparison with impregnated-amine adsorbents, the most important advantages of amine-modified fibrous adsorbents are their high stability during adsorption-regeneration process owing to covalent bonding of organoamine through polymerization method, as well as their high CO_2 adsorption capacity for the high density of grafted amino groups on the surface of the fibrous adsorbent.

Our group has systematically studied the preparation of fibrous adsorbents and their CO_2 adsorption performance, proving that fibers have great potential as a matrix for CO_2 adsorption (Luo et al. 2016; Xu et al. 2015; Zhuang et al. 2013; Yang et al. 2010). Luo et al. 2016 chose natural sisal fiber (SF) as matrix to prepared amine modified adsorbent through grafting acrylamide and then aminating. The adsorption capacity of SF-AM-TETA could reach 4.20 mmol/g. Xu et al. 2015 has used polypropylene (PP) fibers as substrate to prepare hyperbranched solid amine fibers through co-irradiation grafting copolymerization with glycidyl methacrylate and then amination with ethylenediamine. The hyperbranched solid amine PP fibers adsorbent showed a CO_2 adsorption capacity of 5.35 mmol/g. Yang et al. 2010 have grafted allylamine onto polyacrylonitrile (PAN) fibers through preirradiation grafting copolymerization. The CO_2 adsorption capacity of this amine-modified PAN adsorbent reached up to 6.22 mmol/g. Although all of them showed a good CO_2 adsorption performance, we noticed that they require introducing intermediate active groups or pretreatment in order to graft amine functional groups, which have the relatively complicated synthesis steps and restrict their application in the CO_2 adsorption field.

Zhao and Liu 2014 have prepared an amino-modified PAN (AMPAN) fiber through the reaction of PAN fiber and triethylenetetramine (TETA) in the presence of water. Although the highest adsorption capacity of the AMPAN fibers was only 0.09 g/g even at 15 bar and 50 °C, it showed that the polymeric PAN fiber contains a large number of cyano groups which could be aminated by amination reagent under appropriate reaction conditions. Moreover, in addition

to inherent merits of fibers, PAN fibers even show excellent characteristics such as commercial availability, desirable chemical resistance, high thermal stability, good mechanical properties and non-toxicity in nature (Nirmala et al. 2013; Patel and Hota 2016). Therefore, in this paper, we consider that cyano groups on PAN fibers could directly react with amino groups of amination reagents at an appropriate temperature in order to reduce energy consumption and lower processing cost. Different amination reagents, including diethylenetriamine (DETA), triethylenetetramine (TETA), tetraethylenepentamine (TEPA) and polyethyleneimine (PEI), were applied to react with PAN fibers. The factors that would determine the adsorption property of PAN fibers modified with amine have been investigated.

2 Experimental section

2.1 Materials and reagents

Polyacrylonitrile (PAN) fiber was provided by Lanzhou Refinery, China. CO_2 and N_2 were purchased from Guangzhou Zhuorui Gas Company (Guangdong, China). Diethylenetriamine (DETA), triethylenetetramine (TETA), tetraethylenepentamine (TEPA) and acetophenone were purchased from Guangzhou Reagent Company (Guangzhou, Guangdong, China). Polyethyleneimine (PEI) was purchased from Aladdin Industrial Corporation (Shanghai, China). All chemicals were purchased as analytical grade and used without further purification. Distilled water was used to prepare all solution in the study.

2.2 Synthesis of amine-modified PAN fibrous adsorbents

In a general preparation procedure, 3.0 g PAN fibers were immersed with 120 mL 70 vol% TETA aqueous solution in a round-bottom flask, and heated at 120 °C for 6 h to complete the amination reaction. After that, the amine-modified fibers were rinsed with cold water and hot water (treating them into boiling water about 30 min) repeatedly until the filtrate was neutral. The washed samples were then dried under vacuum at 90 °C. The obtained amine-modified fiber was named as PAN–TETA. DETA, TEPA and PEI were also used to prepare amine-modified fibers, and the resulting fibers were labeled as PAN–DETA, PAN–TEPA and PAN–PEI, respectively. All these amine-modified fibers are generally called as solid amine fibers, or solid amine adsorbents. In order to study the effect of concentration and co-solvent on the amination degree and adsorption performance of PAN–TETA, three reaction systems including pure TETA, TETA aqueous solution and

TETA mixed with acetophenone were used in the preparation of PAN–TETA. The reaction scheme is shown in Fig. 1 (taken TETA as an example).

2.3 Determination of aminating degree of fiber

The aminating degree of fiber was characterized by the weight gain rate (G, %) of the fiber after amine modification, which was calculated with the following Eq. (1) :

$$G\% = \frac{W_2 - W_1}{W_1} \times 100\% \tag{1}$$

where W_1 (g) and W_2 (g) are weights of PAN and PAN-X (X = DETA, TETA, TEPA or PEI), respectively.

2.4 Determination of the content of amino groups

0.50 g Solid amine fibers were put into 75.0 mL 0.050 mmol/L hydrochloric acid standard solution in a conical flask and oscillated at room temperature for 24 h. After that, 10 mL of solution was taken into the conical triangular bottle. The unreacted hydrochloric acid was titrated with 0.050 mmol/L sodium hydroxide standard solution using phenolphthalein as indicator. The content of amino groups of the solid amine fiber was calculated by

by subtracting unreacted hydrochloric acid amount from total hydrochloric acid amount.

2.5 Physical and chemical characterization

The elemental analysis were obtained using an Analysen systeme GmbH Elementar Vario EL (Germany) to analyze the elemental composition of the fibers. The Fourier transform Infrared (FTIR) spectra of fibers were detected by using FTIR spectrometer (Tensor-27, Germany) equipped with an attenuated total reflection (ATR) objective and FTIR-Raman Spectrometer (Nicolet NXR 9650, America). X-ray powder diffraction (Empyrean, Netherlands) were conducted to determine the crystal structure of fibers. The ^{13}C nuclear magnetic resonance (NMR) spectra of fibers were recorded using a solid NMR (Bruker, Germany) at 400 MHz. The Ultra-depth three-dimensional microscope (VHX-1000C) were employed to observe the morphology and measure the diameter of fibers.

2.6 CO₂ adsorption experiment

The adsorption experiment was conducted in a fixed bed packed with adsorbents in the presence of water. The CO_2 adsorption performances of all samples were characterized by using breakthrough curves. A desired amount of adsorbent was packed into a glass column (1.3 cm inner diameter), from which the air and excess water in the column was removed by introducing dry N_2 with a flow rate of 30 mL/
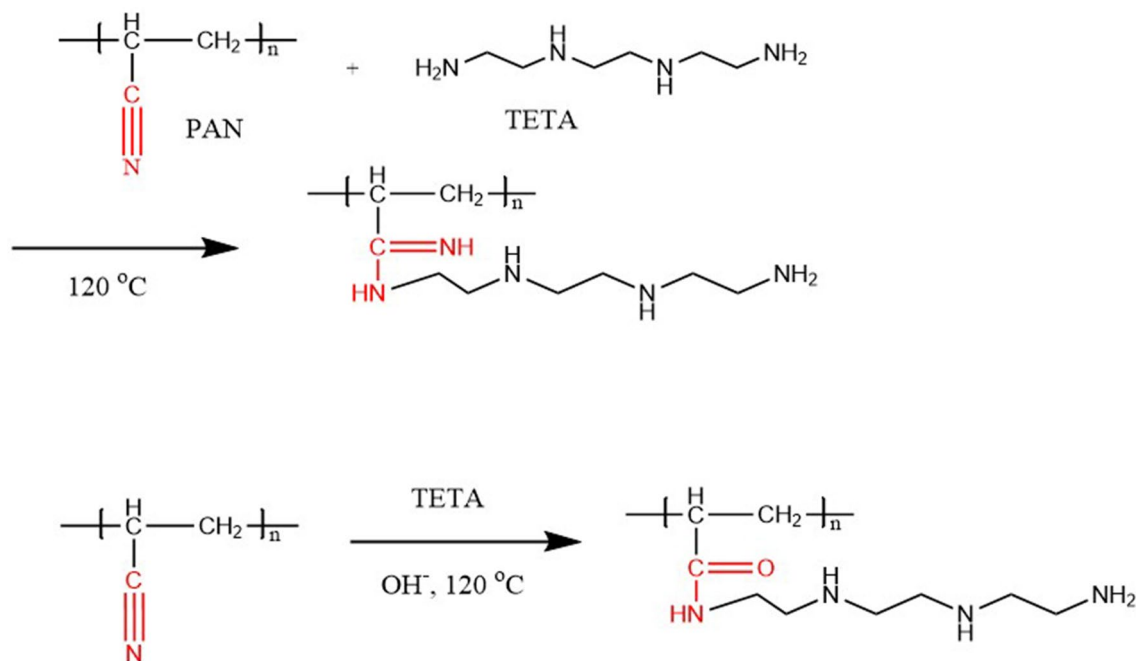

Fig. 1 Reaction scheme of PAN–TETA preparation

min for 0.5 h before starting detection. Then, the CO_2/N_2 mixture gas (10% CO_2/90% N_2) with a flow rate of 30 mL/min was introduced into the adsorption column. The influent/effluent concentration of CO_2 were tested every 2 min, using a gas chromatograph (GC, D7900, Techcomp, China) with a thermal-conductivity detector (TCD). After adsorption, pure N_2 gas at a flow rate of 30 mL/min was introduced through the column at 90 °C to regenerate the spent adsorbent sample.

The adsorption capacity of CO_2 was calculated as follows:

$$Q = \int_0^t \left(C_{in} - C_{eff} \right) V dt / 22.4W \tag{2}$$

where Q represents the adsorption capacity of the adsorbent (mmol CO_2/g), t is the adsorption time (min), and C_{in} and C_{eff} were the influent and effluent concentrations of CO_2 (mL/min), respectively. V is the total flow rate, 30 ml/min; W and 22.4 are the weight of the fiber sample (g) and molar volume of gas (mL/mmol), respectively.

3 Results and discussion

3.1 Structure and morphology of PAN and amine-modified PAN

The ATR-FTIR spectra of PAN, PAN–DETA, PAN–TETA, PAN–TEPA and PAN–PEI and the FTIR-Raman of PAN and PAN–TETA are shown in Fig. 2. For the pristine PAN fibers, the prominent peaks in Fig. 2a at 2243, 2925 and 1452 cm^{-1} can be ascribed to the stretching vibrations of cyano groups (–C≡N), stretching and bending vibrations

of methylene groups (–CH$_2$–), respectively. Besides, the absorption peak at 1738 cm^{-1} is the stretching vibration of bonding carbonyl groups (C=O), which is the second monomer ester in PAN fibers. After reaction with different amine agents, it can be found that the –C≡N absorption peak of PAN-X (X = DETA, TETA, TEPA or PEI) at 2243 cm^{-1} was weaken in varying degrees for the different chemical reactivity of their cyano groups with amino groups. It is noteworthy that the peaks of –C≡N and –C=O at 2243 cm^{-1} and 1738 cm^{-1} almost completely disappeared in the IR spectra of PAN–TETA and PAN–TEPA, which indicated that almost all cyano groups on PAN fibers have been converted into imino or amino groups by TETA and TEPA. The absorption peaks at 1555 and 1263 cm^{-1}, corresponding to amide –N–H bending vibration and –C–N stretching vibration, were strengthened. A strong peak at 1647 cm^{-1}, corresponding to –C=O or –C=N adsorption, appeared in the IR spectra, which indicated that cyano groups in PAN fibers have been converted into –CONH– through amidation reaction and/or into –C=N through addition reaction between –CN group and –NH group. In order to confirm the reactions, FTIR-Raman spectra of PAN–TETA and PAN were conducted, results in Fig. 2b showed that there were two new absorption peaks appearing at 3288 cm^{-1} and 1646 cm^{-1} in the FTIR-Raman spectrum of PAN–TETA, corresponding to the N–H and C=N, respectively. The result confirmed the addition reaction between –CN group and –NH group. On the other hand, elemental analysis results (Table 1) showed that percentage of oxygen atoms in PAN–TETA fibers obviously increased after amination with TETA (5.326 wt% for PAN, 17.452 wt% for PAN–TETA), indication –CN groups in PAN fiber were hydrolyzed into carboxyl groups, which then reacted with amino groups of TETA to form –CONH.
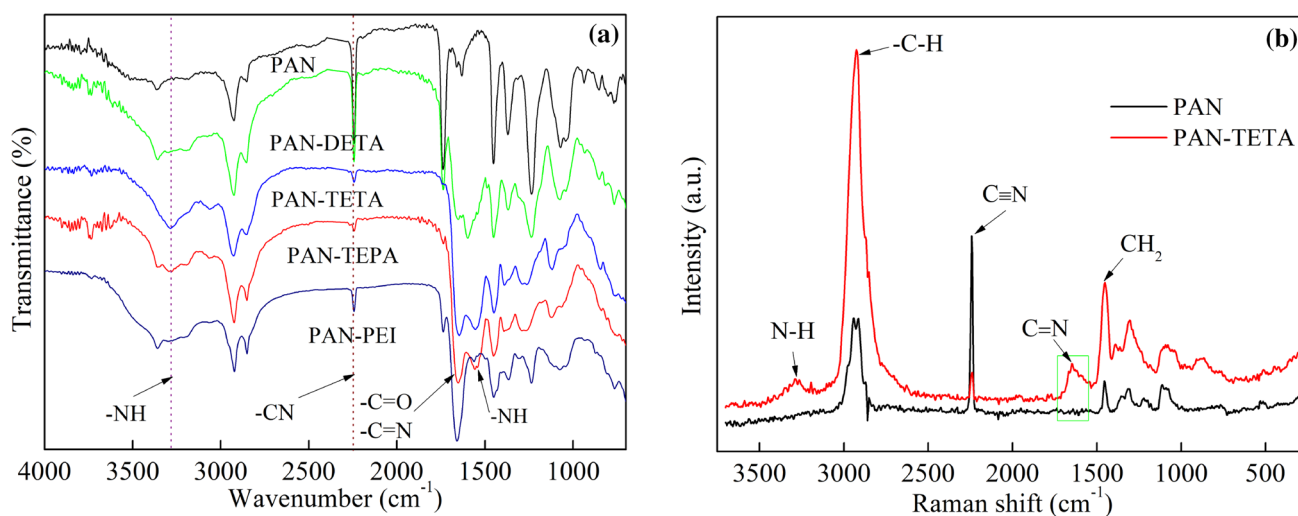

Fig. 2 ATR-FTIR spectra of PAN, PAN–DETA, PAN–TETA, PAN–TEPA and PAN–PEI (**a**) and FTIR-Raman spectra of PAN and PAN–TETA (**b**)

Table 1 Elemental analysis of the PAN fibers before and after amino modification

Samples	G (%)	C (wt%)	H (wt%)	N (wt%)	O (wt%)
PAN	–	65.40	5.584	23.69	5.326
PAN-DETA	17.00	63.36	6.567	23.77	6.303
PAN-TETA	124.67	52.13	9.878	20.54	17.452
PAN-TEPA	102.50	53.19	7.108	21.27	18.432
PAN-PEI	10.50	64.03	6.345	22.79	6.835

For DETA and PEI, they can hardly react with the cyano groups on PAN fibers, so that absorption peaks of cyano groups can still be detected evidently by ATR-FTIR. This is because that for DETA, its strong alkalinity could cause fiber degradation during the reaction process. Besides, owing to the limited chain flexibility and tendency to tangling of its macromolecules, PEI can hardly react with PAN fiber even under the same reaction conditions as small molecule amines like TETA and TEPA. Given the above, the desirable solid amine fibers will be prepared using TETA and TEPA for their suitable chain length and basicity.

The ultra-depth 3D microscope images (Fig. 3) showed that the diameter of PAN raw fiber was approximately 13 μm, while diameters of PAN–TETA and PAN–TEPA fibers both increased to nearly 30 μm, confirming the occurrence of a chemical reaction between polyacrylonitrile fibers and amine reagents. The weight gain degree of PAN–PEI was really low, thus the diameter of PAN–PEI was similar to raw fibers. Obviously, amine compounds in amination process had a significant effect on fiber diameter.

From Fig. 4 it could be found that untreated-PAN fibers showed two strong diffraction peaks at 18.6° and 29.8°, indicating the high crystallinity of raw fibers. But in amine-modified fibers, the above two peaks disappeared, instead the wide and weak peaks showed up at 10.0° or 23.0°, suggesting that the modification of TEPA and TETA could significantly damage crystalline structure of fibers. Under the reaction condition, PAN fibers began to relieve at order area segment, and the movement of their medial and branched macromolecule chains could facilitate the diffusion of amine solution into the inner part of the fibers, so as to enhance the interaction between amino groups and cyano groups.
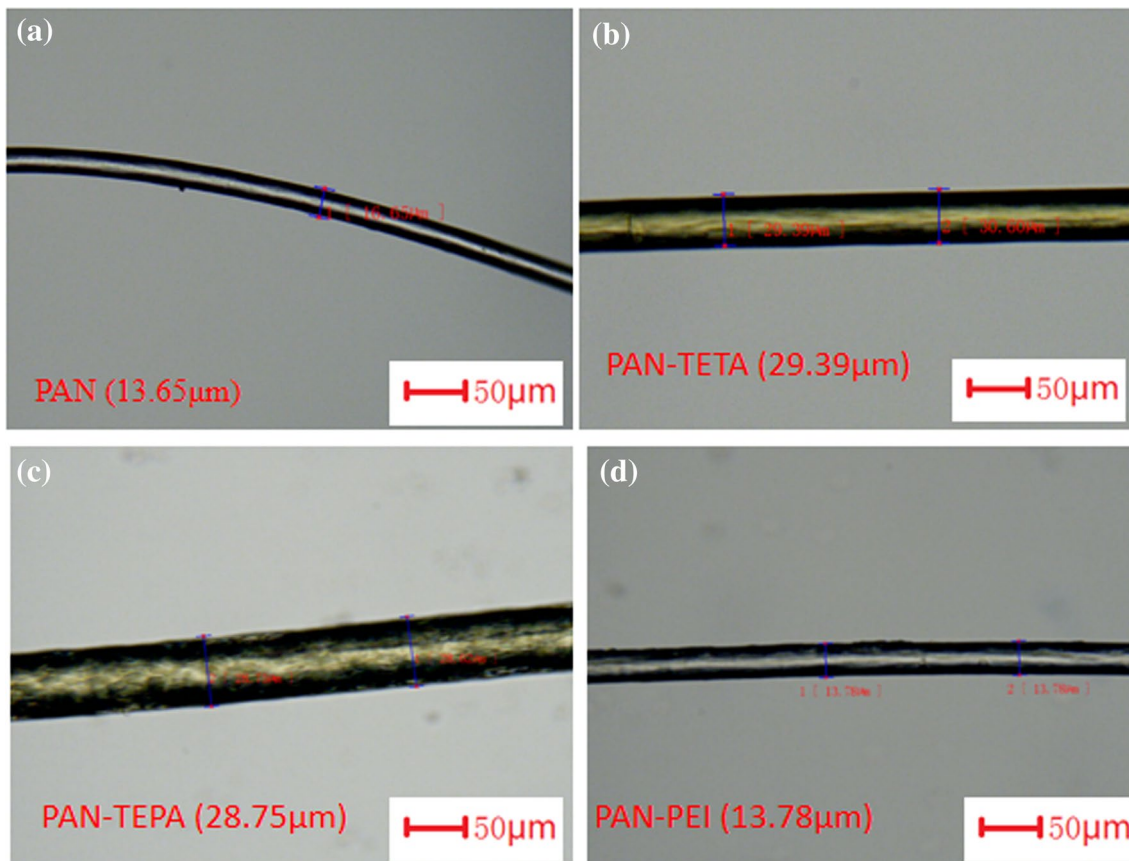

Fig. 3 Ultra-depth three-dimensional microscope images of PAN (**a**), PAN–TETA (**b**), PAN–TEPA (**c**) and PAN–PEI (**d**)
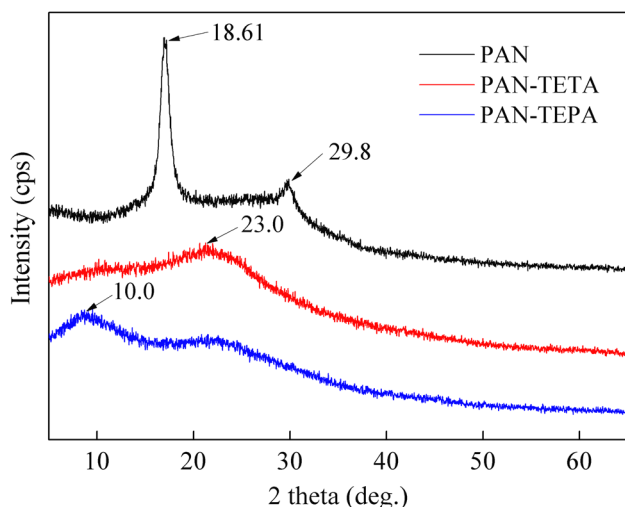
Fig. 4 XRD spectra of PAN, PAN–TETA and PAN–TEPA
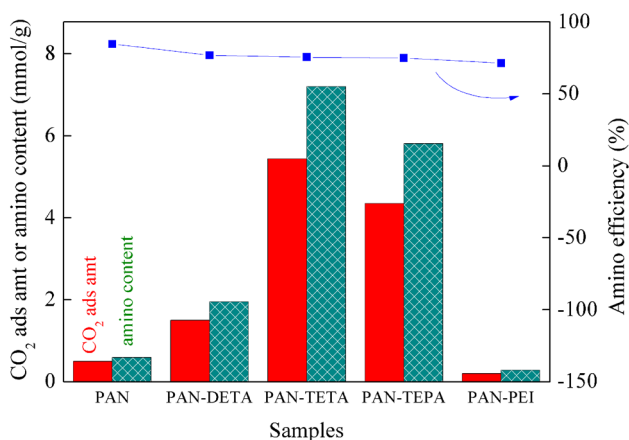

Fig. 5 The CO_2 adsorption capacity and the actual content of amino of amine-modified PAN fibers (CO_2 conc.: 10 vol%, temperature: 25 °C)

3.2 Adsorption behavior of amino-modified adsorbent for CO_2

The chemical compositions of the solid amine fibers were analyzed by elemental analysis, and the results are shown in Table 1. It could be seen that the percentage of oxygen atoms on the solid amine fibers obviously increased due to the hydrolyzation of cyano groups under alkaline conditions.

The actual content of amino groups in amine-modified PAN fibers were determined by acid-base titration, and the results are shown in Fig. 5. Meanwhile, their corresponding adsorption amount and amino efficiency are compared in Fig. 5. It can be clearly seen that PAN–DETA and PAN–PEI contain much lower alkyl amino groups than PAN–TETA and PAN–TEPA. As discussed in the above section, the low

alkyl amino group contents of PAN–DETA and PAN–PEI can be attributed to the strong alkalinity of DETA and the low reactivity of PEI with PAN. Due to low amino contents, PAN-DETA (1.95 mmol amino/g) and PAN–PEI (0.28 mmol amino/g) showed very low CO_2 adsorption capacities. In contrast, the CO_2 adsorption amount of PAN–TETA and PAN–TEPA could reach 5.44 mmol/g and 4.35 mmol/g as their amino content could be as high as 7.20 mmol amino/g and 5.81 mmol amino/g, respectively. Their utilization efficiencies of amino, which is defined as the percentage ratio of CO_2 adsorption amount to amino content of the adsorbent, were all approximately 75%.

We focused on the study of the CO_2 adsorption behavior of PAN–TETA and PAN–TEPA. A series of PAN–TETA and PAN–TEPA samples with different weight gain rates (from 10 to 140 wt%) were prepared and then applied to CO_2 adsorption. Their breakthrough curves of CO_2 adsorption are shown in Fig. 6. It can be seen that PAN–TETA and PAN–TEPA samples showed similar adsorption behavior. All of them were able to thoroughly adsorb CO_2 at the initial phase to keep their effluent CO_2 concentration at zero, and then a breakthrough occurred. It was found that the higher weight gain rate could lead to a longer time to breakthrough, and thus increase the CO_2 adsorption capacities of the amine-modified PAN fibers. The increased content of amino group would be beneficial to the diffusion of CO_2 gas and interaction between CO_2 and amino groups. For example, the PAN–TETA with the highest amino content (124 wt%) showed the highest adsorption capacity among these different PAN–TETA samples, reaching up to 5.44 mmol/g.

3.3 Effect of swelling property of PAN–TETA on its adsorption performance

It is found that the swelling property of amine-modified PAN fibers has been remarkably improved after amination, and the swelling capacity of amine-modified fibers could significantly affect their CO_2 adsorption property. In order to further study the influence of preparation process of fibers on its swelling and adsorption performance, PAN fibers have been aminated in three reaction media, including pure TETA, TETA aqueous solution, and the mixture of TETA and acetophenone. The swelling and adsorption properties of the resulting samples of PAN–TETA were compared.

The spectra of TETA-modified PAN fibers prepared in different media are shown in Fig. 7. It should be noted that the deformation and stretching vibrations of amino groups (~ 3379 cm^{-1}) were identical, indicating that the reaction medium had little effect on the species of amino groups of PAN–TETA fibers. However, it could determine the species and contents of other groups. As shown in Fig. 7, the positions and relative absorption intensities of –C=N and –C=O groups of the samples have changed markedly. In
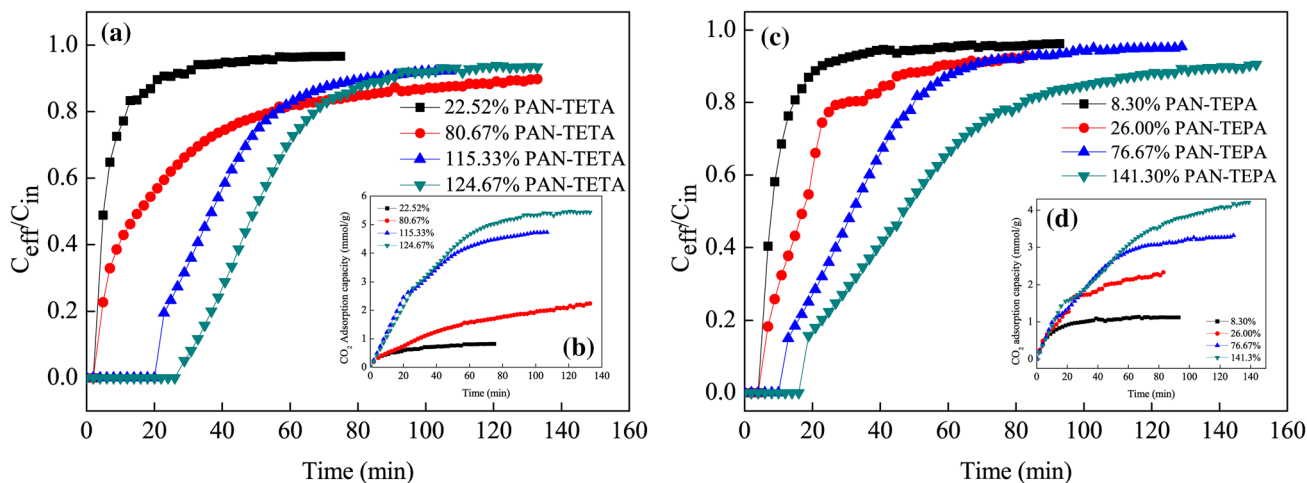
Fig. 6 The breakthrough curves and accumulated adsorption capacity of CO_2 on PAN–TETA (**a, b**) and PAN–TEPA (**c, d**) with different weight gain rate (CO_2 conc.: 10 vol%, temperature: 25 °C)
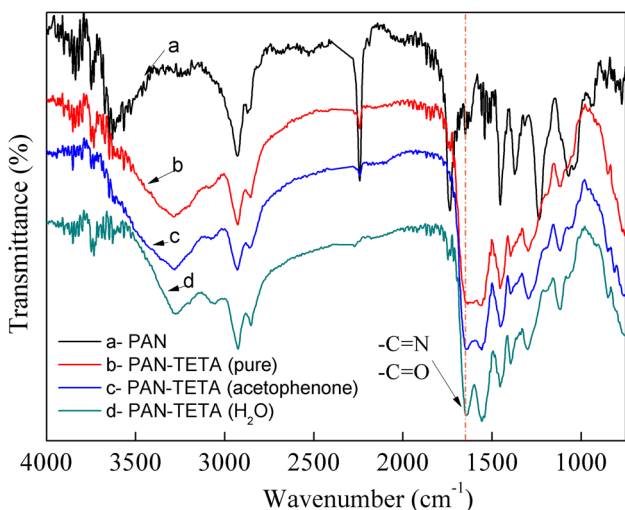

Fig. 7 ATR-FTIR spectra of PAN–TETA fibers prepared in different reaction medium

TETA aqueous solution medium, the PAN could be hydrolyzed in the presence of water and the cyano groups could be converted into carbonyl groups. Therefore, the intensity of absorption peak at 1653 cm^{-1} that corresponding to the

–C=N and –C=O groups significantly increased, compared with that of PAN–TETA prepared in pure TETA medium. For TETA-acetophenone medium, acetophenone with high enough boiling point can decrease the viscosity of TETA, and efficiently promote the amination reaction, so that a fair high amination degree can be obtained.

The element analysis of the three PAN–TETA samples prepared in different media were listed in Table 2. It is found that the three samples had different nitrogen contents and amino group densities. PAN–TETA (H_2O) showed the highest amino group content among the three sample, reaching up to 7.20 mmol/g. The hydroscopicity of the origin PAN fibers and the three amine-modified PAN–TETA fibers prepared in different reaction media are compared in Table 2. A comparison of the CO_2 adsorption capacity on these samples are also illustrated in Fig. 8.

Although the reaction medium had almost no effect on functional species as demonstrated above, the swelling property of these amine modified PAN fibers had changed (Table 2). Owing to the hydrogen bonding between amino groups and water, the hydroscopicity of all amine-modified PAN fibers were obviously increased compared with origin PAN fibers. The water absorption capacity of origin PAN fiber was just 37.62%, but that of PAN–TETA (H_2O) fibers

Table 2 Element analysis and amino-utilization efficiency of different solvent reaction of PAN–TETA

Samples	G (%)	N (wt%)	N (mmol/g)	Q_a (mmol/g)	Q_q (mmol/g)	E_u (%)	Water absorption
PAN		23.69	16.92	0	0	0	37.62
PAN–TETA (pure)	50.67	21.60	15.42	6.04	4.30	71.19	56.50
PAN–TETA (acetophenone)	86.67	18.04	12.88	6.53	4.80	73.51	84.74
PAN–TETA (H_2O)	124.67	20.54	14.67	7.20	5.44	75.51	97.90

Q_a: the actual of amino; Q_q: CO_2 adsorption capacity; E_u: utilization efficiency of amino ($E_u = Q_q/Q_a$)
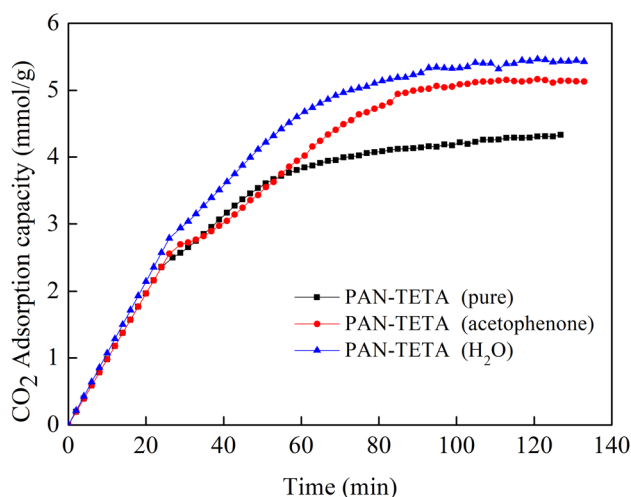
Fig. 8 CO_2 accumulated adsorption capacity curves of different solvent reaction of PAN-TETA (CO_2 conc.: 10 vol%, temperature: 25 °C)

prepared in TETA aqueous system significantly increased to 97.90%, which was also higher than that of PAN–TETA (acetophenone) (84.74%) and PAN–TETA (pure) (56.50%). Based on the essence of the amino-CO_2 interactions, the CO_2 adsorption capacity is actually enhanced by the presence of moisture. Under humidity conditions, one mole of amino groups could theoretically react with one mole of CO_2 to form ammonium bicarbonate. On the other hand, the swollen fiber made CO_2 molecules easy to diffuse into the inner part of the fiber to react with amino groups, which would also increase the utilization efficiency of amino groups in CO_2 adsorption, and further increase the CO_2 adsorption amount.

4 Conclusions

A series of fibrous amine adsorbents were prepared by amination of PAN fiber with different amine agents, including DETA, TETA, TEPA and PEI. The alkalinity of amine reagents would significantly determine the aminating degree of PAN fiber. Amine reagents could react with cyano groups to introduce amino groups onto the fiber, but overhigh alkalinity would break PAN chains and result in the degradation of PAN fibers. TETA and TEPA were appropriate reagents for the amination of PAN fiber because of their moderate alkalinity and high reactivity, and the as-prepared PAN–TETA and PAN–TEPA fibers showed remarkable CO_2 adsorption capacity (5.44 mmol/g, 4.35 mmol/g, respectively). It is also found that high swelling capacity of fibers could significantly improve the CO_2 adsorption capacity as it can promote the CO_2 diffusion and mass transfer during the CO_2 adsorption process. These results demonstrated that the solid

amine-modified PAN fibers would have great potential in the practical application of CO_2 adsorption.

Compliance with ethical standards

Conflict of interest There are no conflicts to declare.

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