



Absorption and desorption of gaseous toluene by an absorbent microcapsules column

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ABSTRACT

Heavy solvents absorption appears to be very attractive in recovering of volatile organic compounds (VOCs) from industrial tail gas. Their high viscosities make good dispersion required but difficult to reduce mass transfer resistance. Microencapsulation techniques provide a candidate solution. In this paper, vapor pressures for toluene + poly(dimethylsiloxane) (PDMS) mixtures were measured at temperature ranging from 273.2 K to 343.2 K. Polyacrylonitrile (PAN) hollow microspheres, prepared by orifice dispersion plus solvent extraction method, was used to immobilize PDMS. The capacity was adjusted from 2.3 g to 9.3 g PDMS/g PAN by addition of cyclohexane in PDMS during solvent impregnation. The breakthrough curves of column packed with PDMS/PAN microcapsules were determined, indicating shapes close to ideality, high absorption efficiencies and considerable absorption capacities before breakthrough. The influence of operational temperature, concentration of feed and gas feed flow rate on the absorption process were investigated as well. A mathematical model, suitable for dilute gas absorption process, was used to simulate the breakthrough curves. This model has proved to be useful to fit curves and analyze the absorption kinetics of PDMS/PAN microcapsules column. After absorption, the column can be regenerated completely by gas stripping. Enrichment of toluene was founded by increasing desorption temperature. Through absorption and desorption by turns, the stability of PDMS/PAN microcapsules column was verified.

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1. Introduction

Volatile organic compounds (VOCs) are organic substances characterized by their high vapor pressure at ambient temperature and their low boiling point [1]. They are among the most common air pollutants emitted from chemical, petro-chemical, and allied industries, leading to serious environmental and health problems. There are many techniques available to abate the emission of VOCs (destruction based and recovery based), such as thermal or catalytic oxidation [2], condensation [3], adsorption [4–7], absorption [8–10], membrane separation [11,12] and biological treatments [13]. They all have many advantages and limitations in different conditions. Evaluation and selection of an appropriate VOC abatement technology depends on the concentration and nature of the compounds, the flow rate of the vent gas and other factors such as safety or economic considerations. Absorption is generally considered to be a fast, safe, and economically feasible method for removing medium/high concentration VOCs from gas stream with a removal efficiency of 95–98% [14]. The most important factor in this process is the choice of a suitable absorbent [15–17]. In addition

to large capacity to absorb VOCs, absorbent should have low vapor pressure and high boiling point in order to reduce the loss and secondary pollution caused by evaporation. With the consideration of environment, the absorbent is preferred to be non-toxic.

Poly(dimethylsiloxane) (PDMS), usually named in the liquid form as silicone oil, has very good chemical and thermal stability, physiological safety, low vapor pressure and high boiling point. It is considered to be a good sorbent of non-polar VOCs [18]. However, PDMS is not suitable to apply in conventional gas–liquid contact equipment directly because it is difficult to be dispersed effectively due to extremely high viscosity. Some researches had been carried out on the basis of introducing PDMS in hollow fiber module to solve such problem [19–21]. But slower gas flow rate or faster PDMS flow rate was required to obtain higher absorption efficiency, which was still limited by the viscosity of PDMS and caused large consumption of absorbent and energy. Microencapsulation could realize the pre-dispersion of absorbent and proved convenient in application of these heavy solvents [22–24]. Due to the elimination of solvent back-mixing, it is possible to achieve high theoretical plate number and absorption efficiency in microcapsules column.

In the present work, toluene, a common and widely used compound was chosen as a model VOC, vapor pressures for toluene + poly(dimethylsiloxane) (PDMS) mixtures were measured. Polyacrylonitrile (PAN) hollow microspheres having good

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endurance to toluene were prepared, and then impregnated with PDMS or PDMS + cyclohexane mixtures to obtain PDMS/PAN microcapsules with controllable loading ratio. The absorption performances of a microcapsules packed column were investigated by experimental approach. A mathematical model previously described for dilute concentration adsorption was applied to the experimental data for checking its suitability for microcapsules column absorption simulation. The gas stripping was executed to regenerate the saturated microcapsules column. The maintenance of absorption capacity in recycling use was also tested.

2. Experimental

2.1. Reagents

PDMS (Kinematic viscosity: 300 cSt) was purchased from Beijing Pinghua Shangmao Co. Ltd., and subjected to a low-pressure vacuum to remove the residual volatile impurities. PAN (Mean molecular weight: 60,000) was purchased from Beijing Trihigh Membrane Technology Co. Ltd. *N,N*-Dimethylformamide (DMF, purity > 99.5%), toluene (purity > 99.5%), cyclohexane (purity > 99.5%), ethanol (purity > 99.5%) were of analytical grade and purchased from Beijing Xiandai Dongfang Chemical Industry. All the materials but PDMS were used as received without any further purification.

2.2. Vapor pressure measurements

Headspace method was employed in vapor pressure measurement of toluene + PDMS mixtures. Toluene + PDMS mixtures of known composition were introduced into headspace cells of about 50 mL and sealed. The cells were then placed in a superthermostat (SC-15) with temperature fluctuation within 0.1 K for at least 2 h to establish phase equilibrium. 100 μ L gaseous samples

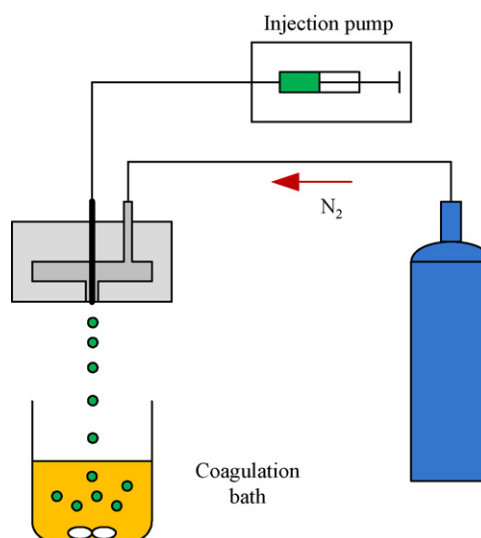


Fig. 1. Set-up of hollow microspheres preparation.

were drawn out from the headspace of the cells using a gas-tight syringe, and directly analyzed by gas chromatography (GC, Agilent 6890) for the content of toluene. The equilibrium liquid phase composition is substituted as the initial. The equilibrium vapor pressures of toluene in gas phase were calculated according to the ideal gas equation, considering the sample displacement due to the pressure difference between the headspace cell and atmosphere.

2.3. Microcapsules preparation

There are various methods to prepare microcapsules, such as solvent evaporation/extraction method [25–28], polymeriza-

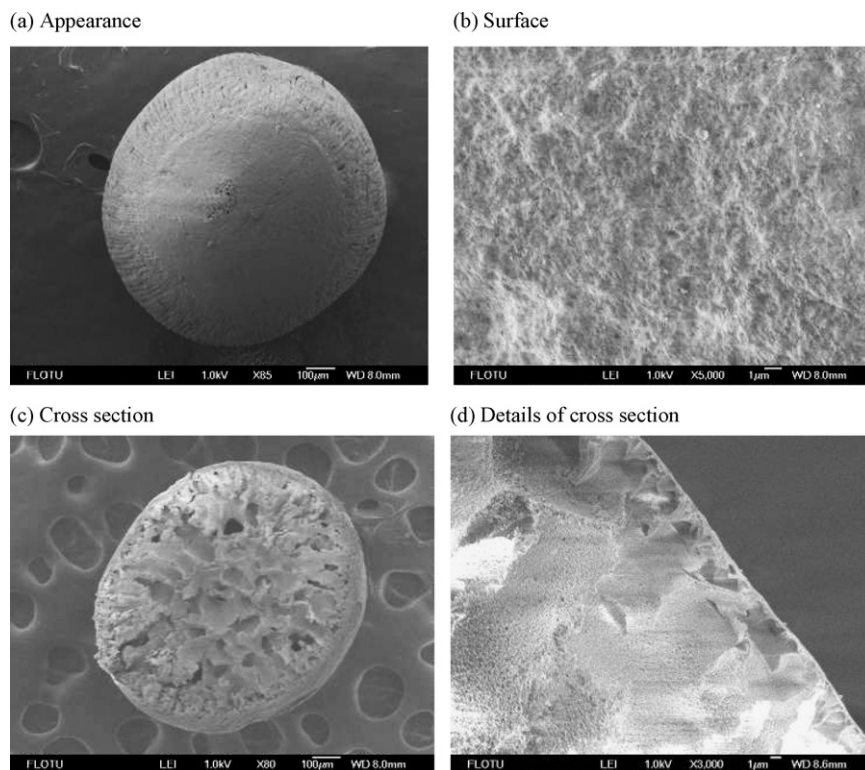


Fig. 2. SEM photographs of PAN microsphere.

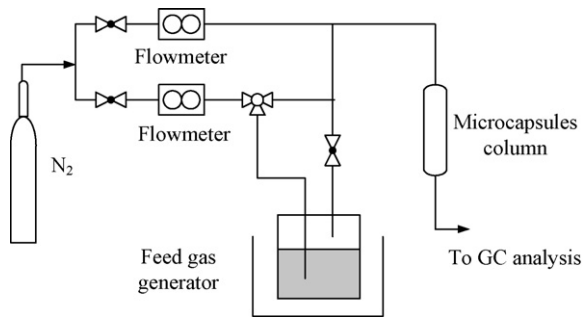


Fig. 3. Schematics of the experimental apparatus used for absorption/desorption experiments.

tion [29]. With the consideration of large absorbent loading ratio and low mass transfer resistance, solvent extraction method was chosen to prepare hollow microsphere, and then absorbent was impregnated with aid of ultrasound. The set-up of hollow microsphere preparation is shown in Fig. 1, including a co-axial micro-dispersion device and a coagulation bath. PAN+DMF mixture of 1:15 (w/w) was extruded from the nozzle by an injection pump, nitrogen gas was introduced at 200 L/h to flow along the outside of nozzle to facilitate the dislodgement of droplet. The coagulation bath is ethanol+water of 3:1 (v/v), which can extract DMF from the droplet, leading to PAN solidification to form hollow microsphere. A gentle stirring was exerted to homogenize the coagulation bath and avoid droplets coalescence before complete solidification. When the solidification process was finished, the PAN hollow microspheres were filtrated out of the coagulation bath, washed three times with deionized water, and dried in vacuum. They were stored in a desiccator before use. Their mean diameter is 1.03 mm. Fig. 2 shows some representative SEM photographs of PAN microsphere. The solvent impregnation of microsphere was carried out under the action of ultrasonic field for 24 h to assure a full impregnation. Besides PDMS, PDMS+cyclohexane mixtures were used as impregnated solvents to control the loading ratio of PDMS in PAN microspheres. After impregnation, cyclohexane was totally removed by vacuum. The loading ratio of microcapsules defined as grams of PDMS per gram of PAN microspheres was determined by weight. Solvent impregnation has little effect on microcapsules' size.

2.4. Absorption (breakthrough curves) and desorption measurements

Absorption/desorption experiments were performed under dynamic conditions at atmospheric pressure with a homemade apparatus (Fig. 3) that permits (i) to prepare model gas mixture and flow rate control, (ii) to perform temperature controllable absorption and desorption tests in a stainless steel column with water jacket, (iii) to collect gas sample for analysis. The size of the column was 10 mm inner diameter and 180.40 mm height. The volume of the column was 15.32 mL. The column was fully packed with PDSM/PAN microcapsules (about 5.5 g). The void volume fraction of the column was about 0.35. The toluene gas flow was generated by bubbling air through a saturator containing toluene placed in a thermostated bath. The saturated toluene stream was diluted with N₂, choosing the flow rate to get toluene stream of required concentration. Absorption was carried out using the prepared model mixture until saturation was reached in order to obtain breakthrough curves. For the desorption process, the model gas mixture was switched to pure N₂ flow. The concentration of toluene at the outlet of microcapsules column was analyzed by gas chromatography.

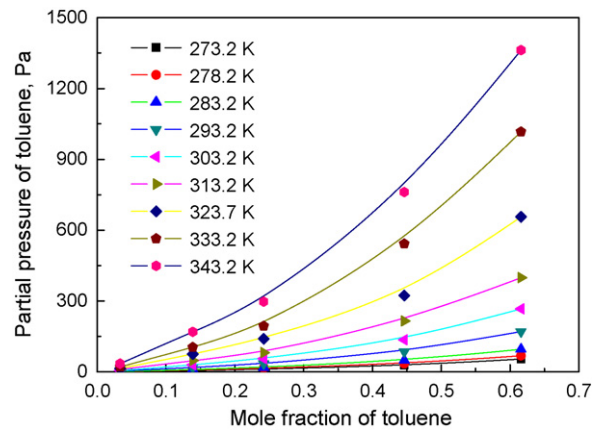


Fig. 4. Gas-liquid equilibrium of toluene + PDMS systems.

3. Results and discussions

3.1. Absorption isotherm

A series of gas-liquid equilibrium experiments were performed at temperature ranging from 273.2 K to 343.2 K. Fig. 4 presents the equilibrium partial pressure of toluene in gas phase versus the mole fraction of toluene in liquid phase. The mole fraction of toluene in liquid phase is calculated from its mass fraction, supposing that the molar mass of PDMS is 14,400 by following correlation.

$$\log v_{298K} = 1.00 + 0.0123M^{0.5} \quad (1)$$

where v_{298K} is the kinematic viscosity of PDMS at 298 K mm²/s; M is the molar mass of PDMS.

When the toluene concentration in liquid phase or the equilibrium temperature increased, the partial pressure of toluene in gas phase increased sharply. Low-temperature absorption was necessary for using PDMS to control toluene vapor. Considering a control on toluene vapor below 60 ppmv at atmospheric pressure by a one-stage contact process, the absorption capacity of 1 g PDMS was only about 1.03 mg at 283.2 K. However, a multi-stage separation process was required for the use of PDMS in toluene abatement.

Assuming that the gas phase obeys the Henry's law as the toluene concentration in liquid phase is infinite low, the Henry's constant was estimated by the experimental results in the system with the lowest toluene content. Fig. 5 clearly shows a linear

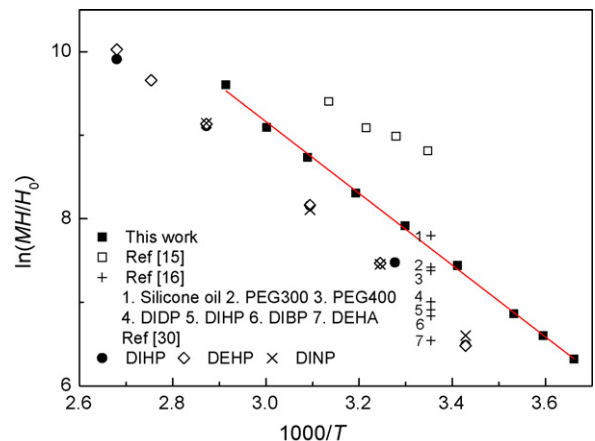


Fig. 5. Temperature dependence of $\ln(MH/H_0)$. $H_0 = 1$ kPa, reference unit for H . DIDP, diisodecyl phthalate; DIHP, diisooheptyl phthalate; DEHA, di(2-ethylhexyl) adipate; DIBP, diisobutyl phthalate; DEHP, di-2-ethylhexyl phthalate; DINP, diisononyl phthalate. M is the mole mass of the solvent.

Table 1
Parameters $\Delta H_{dis\ ij}$ of the temperature dependence law of H .

Solvent	Solute	$\Delta H_{dis\ ij}/(\text{J mol}^{-1})$
PDMS ^a	Toluene	-35708
DIHP ^b		-37618
DEHP ^b		-38630
DINP ^b		-37359

^a This work.

^b Ref [30].

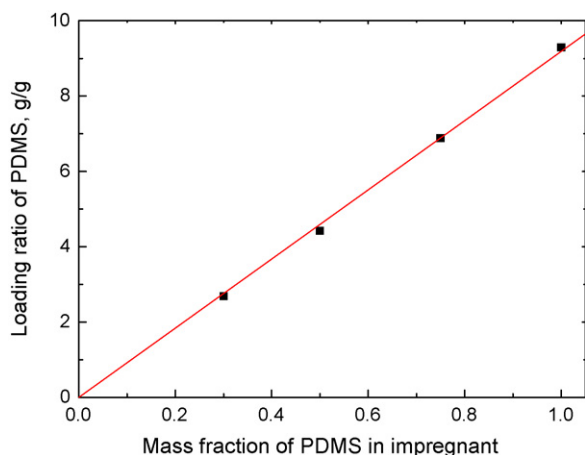


Fig. 6. The effect of impregnant composition on the loading ratio of PDMS in microcapsules.

variation of $\ln H$ versus $1/T$ can be considered in the range of temperatures investigated, similar with those reported earlier [15,16]. Therefore, the temperature dependence of Henry's constants, at a constant pressure, was expressed by

$$\ln H_{ij} = \frac{\Delta H_{dis\ ij}}{RT} + A_{ij} \quad (2)$$

where H_{ij} , $\Delta H_{dis\ ij}$, and A_{ij} are respectively the Henry's constant, the mean enthalpy of dissolution of the solute in the solvent, and a parameter specific for the couple solute (i)–solvent (j); R is the gas constant; and T is the absolute temperature. The values of constant $\Delta H_{dis\ ij}$ and A_{ij} were determined by linear regressions (appearing as the dotted lines in Fig. 4) with very high correlation coefficients close to 1. Table 1 presents parameters $\Delta H_{dis\ ij}$ of PDMS and other three reported heavy solvents, di-2-ethylhexyl phthalate (DEHP), diisooheptyl phthalate (DIHP), and diisononyl phthalate (DINP) [30]. PDMS has the smallest $\Delta H_{dis\ ij}$ in these four solvents, which indicates that the regeneration of PDMS by heating may consume less energy.

3.2. Absorbent capacity of microcapsules

With the aid of ultrasonic, impregnant could occupy the pore volume of microspheres completely. When pure PDMS was used as impregnant, the impregnated PDMS might seep out of microcapsules after dissolving toluene due to the volume expansion. We therefore used mixtures of PDMS+cyclohexane as impregnants. The impregnated cyclohexane can be removed by vacuum, and the non-volatile PDMS preserved. Fig. 6 shows the loading ratio of PDMS in microcapsules with various impregnants. The maximum of the loading ratio for the prepared PAN microcapsules in this work is 9.3 g PDMS/g PAN. The loading ratio of PDMS in microcapsules, directly proportional to the mass fraction of PDMS in impregnant, is easy to control in impregnation process. Considering the stability of loading ratio in absorption/desorption process, microcapsules with the loading ratio of 4.71 g PDMS/g PAN were used hereafter.

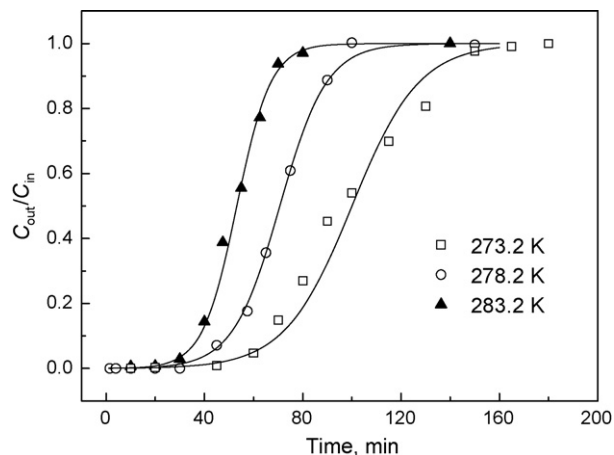


Fig. 7. Breakthrough curves at different temperatures. $C_{in} = 7.5 \text{ mg/L}$, $Q = 200 \text{ mL/min}$.

3.3. Column absorption and desorption

As already noted, a series of toluene absorption experiments in PDMS/PAN microcapsules column were performed to investigate the kinetics characters and the influences of three main factors, temperature, gas feed concentration and flow rate. The analysis of the absorption kinetics was performed using a simple empirical model proposed by Yoon and Nelson [31]. They showed that a breakthrough curve can be expressed by the equation

$$t = t_{0.5} + \frac{1}{k} \ln \left(\frac{C_{out}}{C_{in} - C_{out}} \right) \quad (3)$$

Being

$$W_e = \frac{C_{in} Q}{t_{0.5}} \quad (4)$$

where Q is the gas flow rate, L/min; C_{in} is the toluene inlet concentration, mg/L; W_e is the equilibrium mass absorption capacity of the column, mg; $t_{0.5}$ is the stoichiometric time, s; k is the global kinetic constant, s^{-1} . Plotting $\ln((C_{in} - C)/C)$ versus time, the kinetic constant (k) and the stoichiometric time ($t_{0.5}$) can be calculated from the slope and the intercept, respectively. With these two parameters, the theoretical mass absorption capacity (W_e) can be calculated.

Figs. 6–8 show the experimental results and breakthrough curves obtained by calibration. Except for the experiment performed at 273.2 K (as shown in Fig. 6), the calculated results

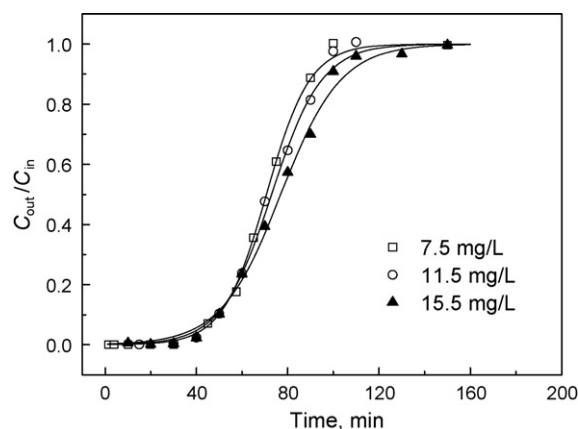


Fig. 8. Breakthrough curves of gas feeds with different toluene concentrations at 278.2 K. $Q = 200 \text{ mL/min}$.

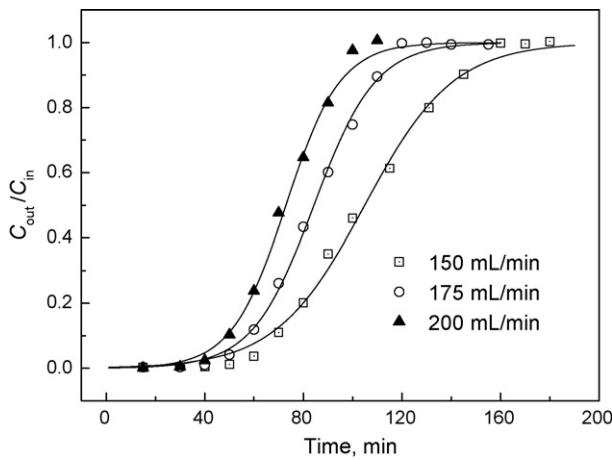


Fig. 9. Breakthrough curves at different gas feed flow rates at 278.2 K. $C_{in} = 11.5$ mg/L.

agree with the experimental results well. It corroborates that the empirical model proposed by Yoon and Nelson, commonly used for adsorption process, is a potential model for PDMS/PAN microcapsules column. The more compact influence of internal diffusion due to the higher viscosity at lower temperature may lead to deviations of experimental and calculated results at 273.2 K. Calculated from breakthrough curves in Fig. 7, the theoretical mass absorption capacity at 273.2 K, 278.2 K and 283.2 K was 170.4 mg, 104.6 mg and 91.8 mg, respectively. The breakthrough time, defined as the time when $C_{out}/C_{in} = 0.05$, was 59.9 min, 42.6 min and 32.8 min, respectively. The absorption capacity at breakthrough point was 89.9 mg, 63.9 mg and 49.2 mg, respectively. The low-temperature absorption is preferable and feasible for PAN/PDMS microcapsules column.

Fig. 8 shows the effect of toluene inlet concentration in gas feed on toluene absorption at 278.2 K. The theoretical mass absorption capacity when $C_{in} = 7.5$, 11.5 mg/L and 15.5 mg/L was 104.6 mg, 167.8 mg and 238.6 mg, respectively. The breakthrough time was 42.6 min, 40.6 min and 38.2 min, respectively. The absorption capacity at breakthrough point was 63.9 mg, 93.4 mg and 118.4 mg, respectively. Evidently, in the experiments, the breakthrough time was almost independent on the toluene inlet concentration, and the absorption capacity increased with the toluene inlet concentration increasing. Aiming at a specific discharge limitation, the PDMS/PAN microcapsules column absorption has good adaptability to variance of gas feed concentration.

Fig. 9 shows the effect of gas feed flow rate on toluene absorption at 278.2 K. The theoretical mass absorption capacity when $Q = 150$ mL/min, 175 mL/min and 200 mL/min was 176.9 mg, 169.7 mg and 167.8 mg, respectively. The breakthrough time was 51.9 min, 47.5 min and 40.6 min, respectively. The absorption capacity at breakthrough point was 89.5 mg, 95.6 mg and 93.4 mg, respectively. In the experiments, the effect of gas feed flow rate on toluene absorption is not sensible.

Desorption, as a prerequisite step for the use of absorbent microcapsules column, was performed by gas stripping in this work. On the demand of waste gas abatement or further treatment, desorption process is expected to have some concentration effect. According to the gas–liquid equilibrium data of toluene + PDMS mixtures, desorption process adopted operation temperature higher than what absorption process adopted. Before desorption the column firstly absorbed toluene under identical conditions for comparisons: absorption temperature was 278.3 K, the toluene concentration of gas feed was 6 mg/L, the flow rate of gas feed was 150 mL/min, and the absorption time was 30 min. Fig. 10 shows the desorption curves at various temperatures. The maximum

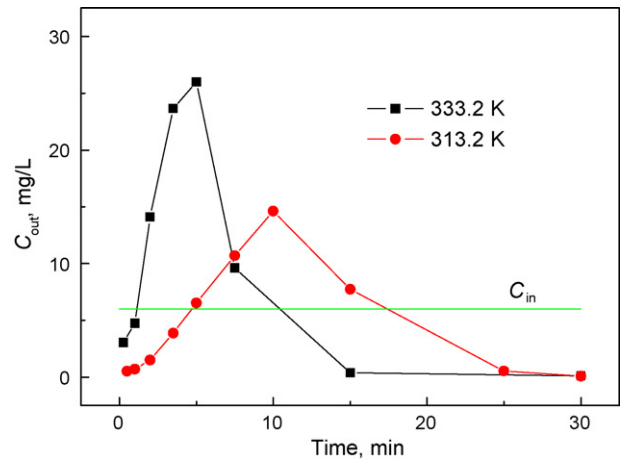


Fig. 10. Desorption curves at different temperatures. $Q = 200$ mL/min.

of toluene outlet concentration increased with the increasing of desorption temperature. Toluene could be concentrated up to 4.5 times at 333.2 K. The higher temperature was beneficial to concentrate toluene and decrease the time for desorption. Increasing the flow rate of stripping gas could also accelerate the desorption process, as shown in Fig. 11. The effect of the flow rate of stripping gas on the maximum of toluene outlet concentration is weak. A batched cycling gas stripping can be introduced to fulfill a concentration task.

3.4. Recycling use of microcapsules column

The toluene absorption performance of PDMS/PAN microcapsules column in recycling use was tested. The absorption operational parameters included: temperature, 278.2 K; feed gas toluene concentration, 7.5 mg/L; feed gas flow rate, 200 mL/min; duration, 160 min. The desorption operational parameters included: temperature, 333.2 K; stripping gas flow rate, 200 mL/min; duration, 30 min. The absorption kinetics at the first time and the fourth time were shown in Fig. 12. The difference between them was quite little, showing good stability of microcapsules column. The high boiling point/low vapor pressure of PDMS avoided the loss of solvent during the absorption and desorption process. Besides, effectiveness was also proved to regenerate PDMS/PAN microcapsules column by heating and gas stripping. Under proper conditions, toluene can be concentrated in stripping gas, and collected by condensation economically.

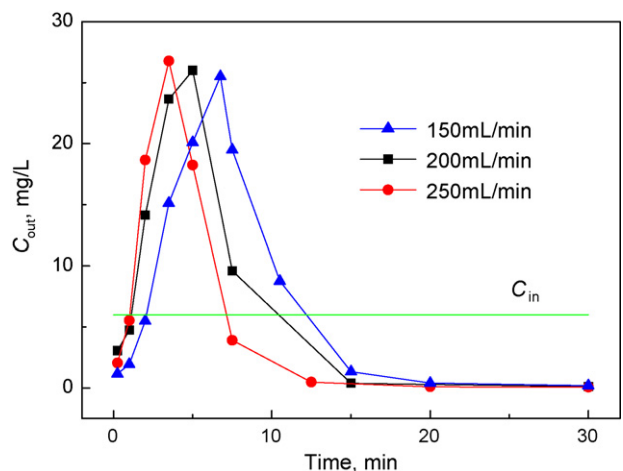


Fig. 11. Desorption curves with different stripping gas flow rates at 333.3 K.

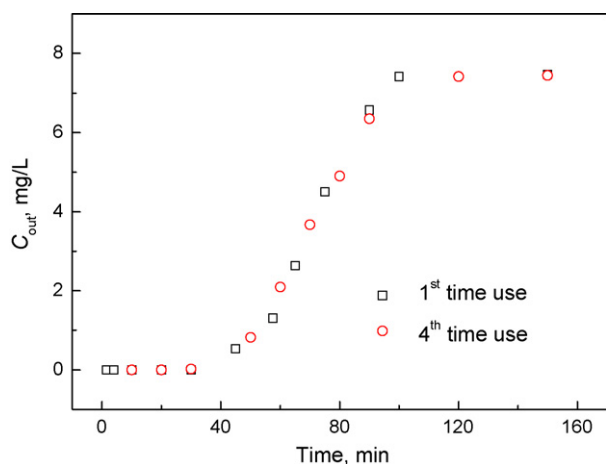


Fig. 12. The toluene absorption performance of microcapsules column in recycling use.

4. Conclusion

From the case of toluene absorption with PDMS/PAN microcapsules column, this work shows the interest of absorbent microcapsules column in environmental technology for abatement of VOCs. Not only could microcapsulation effectively disperse heavy solvent like PDMS as a green VOCs absorbent before application, but also it is possible to achieve high theoretical plate number and absorption efficiency in microcapsules column due to the elimination of absorbent back-mixing.

Vapor pressures for toluene + PDMS mixtures were measured at atmospheric pressure and temperature ranging from 273.2 K to 343.2 K. Polyacrylonitrile (PAN) hollow microspheres, prepared by orifice dispersion plus solvent extraction method, was used to immobilize PDMS. The capacity was adjusted from 2.3 g to 9.3 g PDMS/g PAN by addition of cyclohexane in PDMS during solvent impregnation. The breakthrough curves of PDMS/PAN microcapsules column were determined, indicating high absorption efficiencies and considerable absorption capacities before breakthrough. The influence of operational temperature, concentration of feed and gas feed flow rate on the absorption process were investigated as well. A mathematical model, suitable for dilute gas adsorption process, was used to simulate the breakthrough curves. This model has proved to be useful to fit curves and analyze the absorption kinetics of PDMS/PAN microcapsules column. After absorption, the column can be regenerated completely by gas stripping. Enrichment of toluene was found by increasing desorption temperature. Through absorption and desorption by turns, the stability of PDMS/PAN microcapsules column was verified.

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