Steam regeneration of acetone and toluene in activated carbon and dealuminated Y-zeolite beds

Dong-Geun Lee, Yun-Jin Han, and Chang-Ha Lee[†]

Department of Chemical and Biomolecular Engineering, Yonsei University,
262, Seongsan-ro, Seodaemun-gu, Seoul 120-749, Korea

Abstract—The dynamics of steam regeneration in activated carbon and dealuminated Y-zeolite (DAY-zeolite) beds was studied to recover acetone and toluene from effluent gases. Due to the higher adsorption capacity of acetone on both adsorbents, the desorption breakthrough of toluene was faster than that of acetone, but the breakthrough shape of acetone was steeper than that of toluene. The variation in breakthrough time as a function of steam flow rate was less in the activated carbon bed than in the DAY-zeolite bed. The inside temperature profiles during regeneration showed stepwise shapes. A temperature plateau was observed near 355 K for toluene, which is lower than its boiling temperature and azeotrope. A temperature plateau for acetone occurred near 330 K, near its boiling temperature. The duration of the temperature plateau at the bed end corresponded with the time period of high desorption concentration during acetone desorption; for toluene, plateau duration was shorter than the duration of high desorption concentration. The maximum effluent concentration of toluene reached only 80% of the feed concentration, while that of acetone was almost 100%. Therefore, the water-miscibility of the adsorbate was an important factor in steam regeneration.

Key words: Steam Regeneration, Acetone, Toluene, Activated Carbon, DAY-zeolite

INTRODUCTION

Volatile organic compounds (VOCs) are used in a wide range of products, such as paints and lacquers, cleaning supplies, pesticides, printed materials, craft materials, and photographic solutions. Some VOCs may have short- and long-term adverse health effects, such as eye irritation and respiratory problems [1,2]. Accordingly, VOC emission control constitutes a major portion of the chemical process industry's (CPI's) environmental activities. Recently, in view of the need to reduce greenhouse gas emissions, the recovery of massive amounts of VOCs from effluent gases has become an important effort to reuse resources through energy-saving processes in the petrochemical industry. Especially, acetone is an important commercial solvent and raw material with wide usage in the chemical explosives and lacquer industry. The effluent gas containing acetone is massively emitted from certain petrochemical processes. It is supplied to the furnace as an auxiliary fuel, but it is needed to recover efficiently as recycling resources.

Several chemical engineering processes are commonly used to deal with VOC emissions [3-6]. Among them, adsorption is the most common because of its energy-saving efficiency [7-10]. Chemical industries use periodic adsorption processes such as thermal swing adsorption (TSA) and pressure swing adsorption (PSA) [11-13]. TSA with hot purge gas or steam is often applied to VOC removal because the adsorbate has significant vapor pressure at normal temperatures.

Activated carbon is the most suitable adsorbent for VOC adsorption because it possesses a high surface area, an intricate pore struc-

ture, and a hydrophobic surface [14]. However, activated carbons promote polymerization or oxidation of some solvents [11] and require humidity control. Also, its flammability and difficulty to regenerate the adsorbed solvents with high-boiling points lead to the limitation of hot nitrogen desorption [15]. Interest has grown in the use of highly dealuminated Y zeolite (DAY) for the recovery of organic solvents because of its known hydrophobic surface properties. Its low water uptake reduces the energy required for treating high-humidity gas streams and allows the recovery of VOCs with low water contents. Another attractive feature of DAY is that adsorbent ignition is inherently prevented because zeolites are nonflammable. Therefore, highly dealuminated Y-zeolites (DAY-zeolites) are of growing interest as alternative adsorbents. The adsorption and desorption of VOCs of on activated carbon and DAY-zeolite have been studied using hot nitrogen [16-20].

Steaming is also widely used in industrial TSA processes to regenerate the adsorption packed bed because it provides significantly more heat than inert gas purges, and is therefore safer with regard to hot spot ignition. In addition, when the adsorbate concentration is high, it may be advantageous to leave water on the adsorbent to limit the temperature increase during adsorption. The increased adsorptivity at low temperatures can more than offset the decrease in adsorptivity caused by the presence of the water.

Regeneration is the most important step in the TSA process for VOC removal or recovery because it is energy-intensive. To design a TSA process with minimum greenhouse gas emissions, it is therefore important to understand the behavior of steam and adsorbate in the bed. Generally, adsorbate-adsorbent affinity contributes significantly to regeneration. However, because steam can be temporarily condensed in the steam regenerated bed, adsorbate-water miscibility or the presence of an azeotrope can significantly affect regener-

†To whom correspondence should be addressed.
E-mail: leech@yonsei.ac.kr

ation efficiency. Therefore, we investigated the steam regeneration of acetone (polar) and toluene (nonpolar) in activated carbon and DAY-zeolite beds.

EXPERIMENTAL

1. Materials

A DAY-zeolite and an activated carbon were chosen as adsorbents. The DAY-zeolite (DAY-20F, silicon/aluminum ratio=20/1), which has hydrophobic and nonflammable characteristics, was supplied in pellet form (Degussa AG, Hanau, Germany), [15]. The activated carbon used in this study was bituminous-based (BPL type, 4-10 mesh), and was supplied in granular form (Calgon Carbon Co., USA). Prior to adsorption experiments, the DAY-zeolite and activated carbon were activated for more than 24 h under dry vacuum conditions at 573 K and 423 K, respectively. The measured and supplied properties of the DAY zeolite and activated carbon are listed in Table 1.

The adsorbates investigated in this study were toluene, acetone and water. The toluene (mass fraction 99.5% purity) and acetone

Table 1. Physical properties of DAY zeolite (DAY-20F) and activated carbon (BPL)

Property	DAY	BPL
Bulk density [kg/m^3]	500	480
Density [kg/m^3]	1550*	2100**
BET surface area [m^2/g]	704	993
Micropore area [m^2/g]	676	956
Micropore volume [cm^3/g]	0.268	0.414
BJH desorption average pore diameter [nm]	1.40	1.40
Average pore diameter by BET [nm]	2.17	2.00

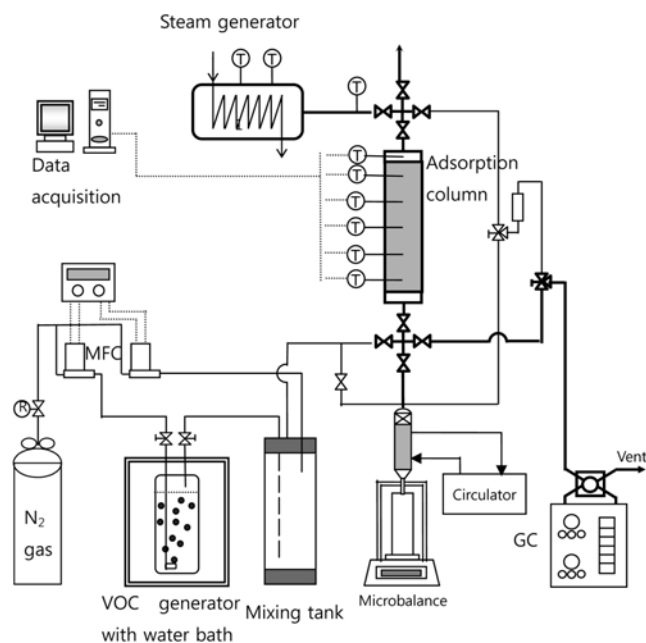*: Solid density, **: Real density by He displacement

Fig. 1. Schematic diagram of the apparatus of the regeneration experiment.

(mass fraction 99.5% purity) were used as received. Purified, distilled, ion-exchanged water was used.

2. Steam Regeneration

The schematic of the experimental apparatus is shown in Fig. 1. Prior to any regeneration experiments, the bed was packed with DAY-zeolite or activated carbon and saturated by adsorbate, acetone or toluene. After the steam regeneration experiment, the bed was packed by a new adsorbent for another saturation and regeneration experiment. Details of the adsorption methods used to saturate the bed are described in our previous studies [16,17].

The adsorption bed was made of a stainless steel pipe with a length of 30 cm, an ID of 2.2 cm, and a wall thickness of 0.15 cm. The bed was insulated with ~5.0 cm thick fiberglass. Both ends of the bed were packed by glass beads. Six calibrated K-type thermocouples were installed at positions 0, 3, 9, 15, 21, and 27 cm from the top of the bed. Since the first thermocouple was inserted into glass beads, its temperature was considered as the temperature of the inlet steam.

For the bed saturation prior to regeneration, the nitrogen gas line was divided into two branches: one was for pure nitrogen gas as a carrier, and the other was connected to a vessel filled with toluene or acetone liquid. In the adsorption experiment, the desired concentration was produced by adjusting the two nitrogen flow rates. The VOC vapors concentration produced by nitrogen streams was identified several times by gas chromatography before each experimental run. The acetone or toluene vapors were carried by nitrogen gas during adsorption step. The adsorption bed saturated through the breakthrough experiment was applied for a steam regeneration experiment. While the feed flowed upward in the adsorption bed in the adsorption breakthrough, hot steam flowed downward along the bed in the regeneration breakthrough.

The water produced from an ultra pure system (Barnstead, D4641) was fed to the bed with nitrogen as a carrier gas. To generate a desired steam, the water was injected into the nitrogen flow through a solenoid valve that was controlled by an in situ hygrometer (VAISARA, HMD40U). In addition, the vapor-generating tube was wound with a heating tape to protect the generated steam from condensation, and the flow rate was controlled by a mass flow controller (Bronkhorst High-Tech, F-201C) pre-calibrated for nitrogen. Then, the steam was supplied to the bed through the connected line with a heating tape and a temperature controller. The steam flow rate from the electric steam generator was set with a metering valve and measured with a balance by condensing the steam through the condenser. Even though superheated steam (typically 393 K) was produced from the steam generator, the input steam temperature in the bed reached 373 K because the bed was exposed to room temperature in the initial stage. As a result, saturated steam was applied because the purpose of this study was to elucidate the condensation phenomena of steam regeneration.

The desorbed adsorbates were condensed and collected in a vial filled with carbon disulfide. At that time, the weight of the collected liquid sample was measured as a function of time with a microbalance. Since the sample was collected for 10 sec at a constant interval (2 min) in a vial, the experimental data represented the average value during the 10 sec regeneration. Then, the amount of desorbed adsorbates was measured periodically by GC (Hewlett Packard 6890 series II). Since too diluted sample in the carbon disulride solution gener-

ated analysis errors, the steam regeneration was applied to the bed within 1 hr. After removing moisture from the column in a drying oven, new adsorbents were packed to the bed for each regeneration experiment.

RESULTS AND DISCUSSION

1. Toluene/Activated Carbon System

Fig. 2 shows temperature profiles and concentrations of regenerated toluene in the toluene-activated carbon system. Here, the relative concentration term (C/C_0) on the Y-axis implies that C is the concentration of adsorbate collected in a sampling vial and C_0 is the concentration of adsorbate in the feed during adsorption step. In Fig. 2(a), the temperature in the glass bead sector in the bed (X=0, X=thermocouple position fraction from the top of bed) took about 6 minutes to reach the input steam temperature because the bed was exposed to room temperature in the initial stage. It was expected that condensed steam might remain in the glass bead sector until the temperature reached vaporization temperature.

Compared to the temperature in the glass bead sector, the bed temperature in the adsorbent sector did not change for the first few minutes because the heat from the steam flow was used to desorb toluene, thereby condens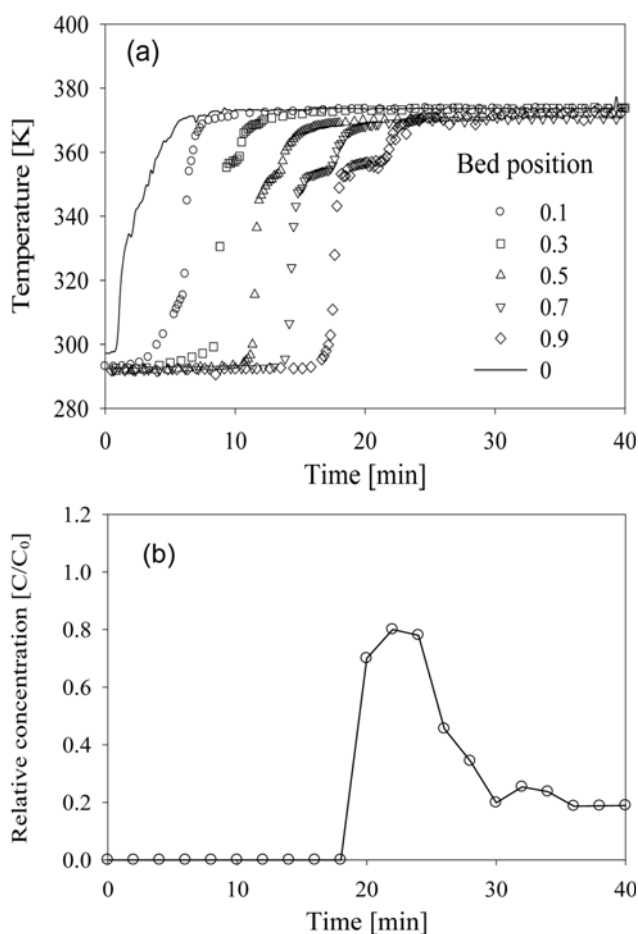ing the supplied steam. After showing a smooth increase in temperature, the bed temperature drastically increased with continuously supplied steam. Further, the slope of the temperature profile became steeper as the steam approached the product end. Then, the temperature profiles showed an inflection near 355-358 K when X=0.3, and remained almost constant for a minute. Finally, the bed temperature increased again to the inlet steam temperature.

The duration of the temperature plateau extended with approaching the bottom of the bed. At X=0.9, the temperature plateau near 355 K started at 18 minutes, and was almost the same as the desorption breakthrough time of toluene, as shown in Fig. 2(b). The desorption concentration of toluene steeply increased at that time, and the maximum desorption concentration was observed at around 20 minutes. However, it only reached 80% of the feed concentration, not 100%. The duration of the maximum desorption concentration was almost the same as the temperature plateau of 355 K at X=0.9, which is lower than the boiling temperature of toluene (383.8 K). After 23 minutes, as the temperature at the product end reached the steam temperature of 373 K, the toluene concentration decreased, but it remained near 20% until 40 minutes.

These results imply that the desorption concentration was greatly affected by the condensed water. At the beginning of regeneration, the steam and the desorbed toluene were partially condensed in the bed. Then, after 23 minutes, the condensed steam was partially re-evaporated from 355 K to 373 K. It has been reported that the mini-
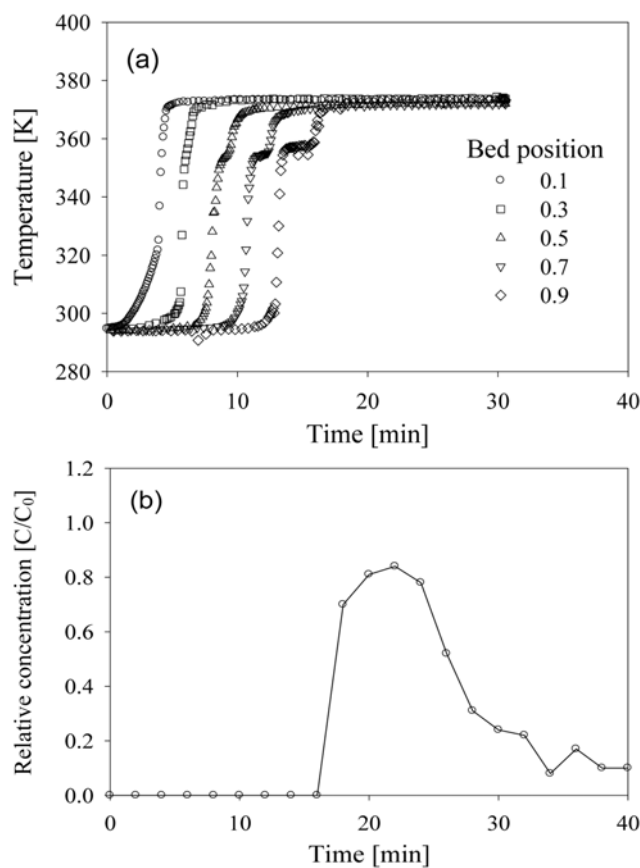

Fig. 2. Temperature (a) and Concentration (b) profiles of toluene/ activated carbon system with 0.5 g/min steam flow rate (initial condition: saturated toluene 4,020 ppm).

Fig. 3. Temperature (a) and Concentration (b) profiles of toluene/ DAY-zeolite system with 0.5 g/min steam flow rate (initial condition: saturated toluene 4,140 ppm).

mum azeotropic point of the water/toluene mixture at 1 atm is near 357 K, with 55.6% toluene mole fraction [21]. This implies that the plateau temperature of the profiles is consistent with an azeotropic temperature. As a result, the partially re-evaporated portion mixed with the remaining toluene/steam vapor mixture. Therefore, the concentration of the desorbed toluene, which is water-insoluble, may not reach the feed concentration.

2. Toluene/DAY-zeolite System

Fig. 3 shows temperature profiles and the regenerated concentration of toluene in the toluene-DAY-zeolite system. The desorption behavior of toluene on a DAY-zeolite bed was similar to that on the activated carbon bed. However, the desorption breakthrough of toluene occurred in about 15 minutes in the DAY-zeolite bed, which was earlier than in the activated carbon bed. Because the adsorption capacity of toluene on DAY-zeolite is lower than on activated carbon [22,23], a lower amount of toluene was initially saturated in the DAY-zeolite bed, leading to a short regeneration time compared to the activated carbon bed. Further, the temperature increase in the bed was faster and steeper than that in the activated carbon bed at the same regeneration conditions. It implies that the adsorption affinity was weaker than the activated carbon bed and it resulted in the faster regeneration rate.

The temperature plateau in the toluene/DAY-zeolite system was observed near 355 K at the middle of the bed (X=0.5). Then, the time from the breakthrough point to maximum concentration corresponded to the period between the temperature plateau and maximum steam temperature at X=0.9 because strong adsorption affinity of toluene leaded to faster and steeper temperature propagation. The maximum desorbed concentration was about 80% and was maintained over 5 minutes, which was almost the same as the toluene/activated carbon system. Then the regeneration curve decreased in the same manner as Fig. 2 until the concentration of toluene reached 20% during the same period with the toluene/activated carbon system. However, the desorbed concentration of toluene in the DAY-zeolite bed decreased from 20% to below 10%, because the adsorption affinity of toluene on DAY-zeolite at high temperature is weaker at low concentrations than that on activated carbon [22,23].

3. Acetone/Activated Carbon System

The temperature and concentration profiles of acetone on activated carbon are shown in Fig. 4. The shapes of the temperature profiles are similar to those of toluene, but a temperature plateau is observed near 330 K and is longer than that of toluene due to more adsorption amount of acetone on activated carbon as well as the high feed concentration. The long temperature plateau implies that a large amount of acetone is adsorbed before regeneration stemming from the multi-layer adsorption of acetone on activated carbon [16] as well as the higher feed concentration.

The temperature plateau near 330 K may stem from the evaporation of acetone. Acetone is highly soluble in water, in contrast to
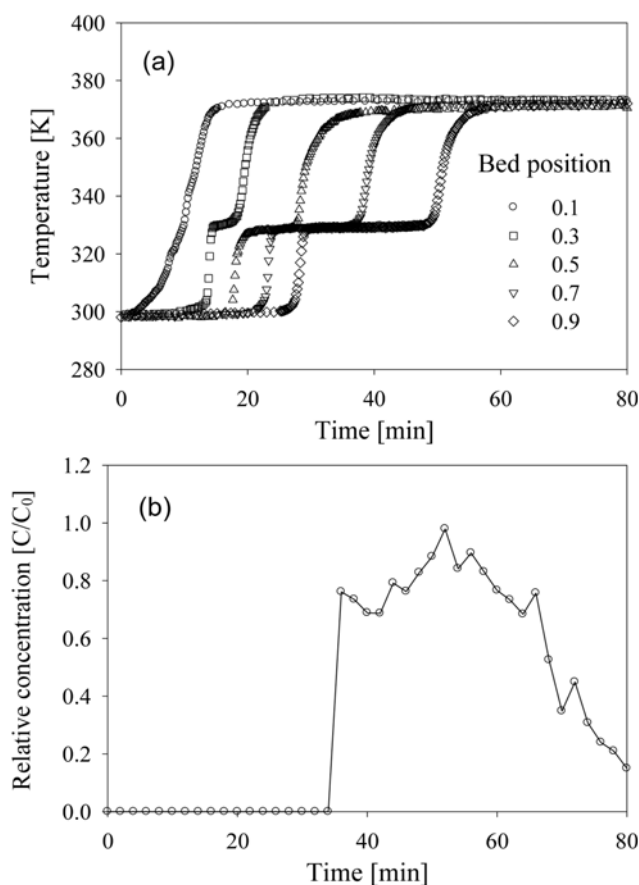

Fig. 4. Temperature (a) and Concentration (b) profiles of acetone/activated carbon system with 0.4 g/min steam flow rate (initial condition: saturated with acetone at 16,000 ppm).
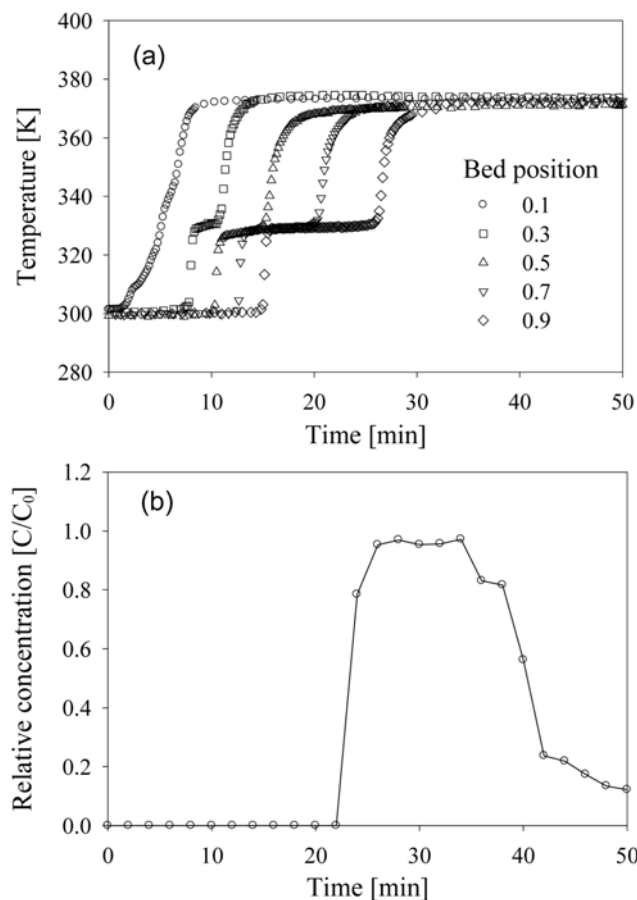

Fig. 5. Temperature (a) and Concentration (b) profiles of acetone/activated carbon system with 0.8 g/min steam flow rate (initial condition: saturated acetone 16,000 ppm).

toluene. The adsorbed acetone is desorbed by steam, but the steam/acetone mixture can condense as it propagates through the bed due to low bed temperature. In addition, although some portion of acetone vapor propagates to the bed, it can be re-adsorbed to the adsorbents. Then, the bed temperature can be increased by continuously supplied steam. When the temperature reaches the boiling point of acetone (329.3 K), acetone from the condensed acetone/water mixture is evaporated and the bed temperature remains constant because the steam energy is consumed to evaporate acetone. The duration of the temperature plateau increased near the product end because more acetone was accumulated at the product end. After the temperature plateau, the bed temperature increased to 373 K.

A steep discharge of acetone was observed near 40 minutes and the maximum relative concentration was almost 100%, which was obtained near 45 minutes, higher than that of toluene. This time duration corresponded to the later part of the temperature plateau near 330 K at X=0.9 in the temperature profile. Then, the maximum desorbed concentration was maintained until the temperature profile at X=0.9 reached the maximum. The acetone concentration decreased steeply after 60 minutes because the acetone/steam mixture did not undergo any condensation. Desorption tailing was shorter than that of toluene because the adsorption affinity of acetone is relatively weak.

The effect of steam flow rate on regeneration, which is presented in Fig. 5, was twice as fast (0.8 g/min) as in Fig. 4. Due to the increased flow rate, the profiles of temperature and concentration progressed faster and the duration of the temperature plateau was much shorter than in Fig. 4. The increased steam flow rate improved the heat transfer rate and accelerated the desorption and vaporization of acetone. In the concentration profile, after the relative concentration reached 80%, it approached 100%. The maximum desorbed concentration of acetone started from temperature plateau (355 K) to 373 K at X=0.9. After reaching the inlet steam temperature, the desorption concentration decreased steeply and desorption was completed faster than shown in Fig. 4.

4. Acetone/DAY-zeolite System

The desorption of acetone in a DAY-zeolite bed is presented in Fig. 6. The shape and inflection of the temperature profile are similar to those of the acetone/activated carbon system in Fig. 4. However, temperature propagation is slightly faster and the duration of the temperature plateau is longer than in the activated carbon bed. Further, after the temperature plateau, it takes 3 minutes to reach 100 °C where it takes 5 minutes in case of activated carbon bed. The temperature wave front is related to the mass transfer zone, which indicates that the adsorption affinity of acetone on DAY-zeolite is relatively weak [16] and acetone is re-vaporized from the acetone/water mixture. Therefore, the regeneration rate was faster in the DAY-zeolite bed than in activated carbon.

In the DAY-zeolite bed, the tailing of the acetone desorption curve was shorter than that of toluene. The isotherm of acetone varies from a Type-II isotherm to a Type-III isotherm with a temperature increase,
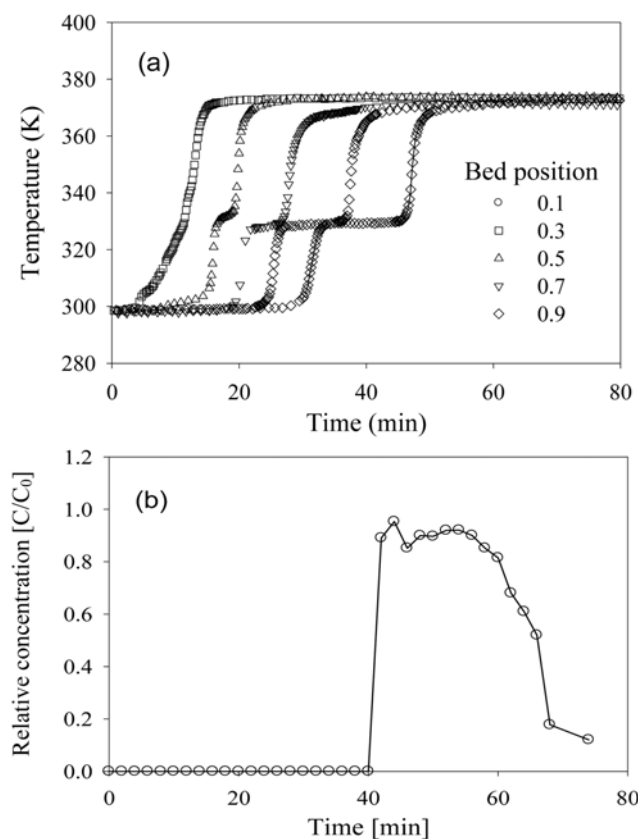

Fig. 6. Temperature (a) and Concentration (b) profiles of acetone/DAY-zeolite system with 0.4 g/min steam flow rate (initial condition: saturated acetone 16,000 ppm).
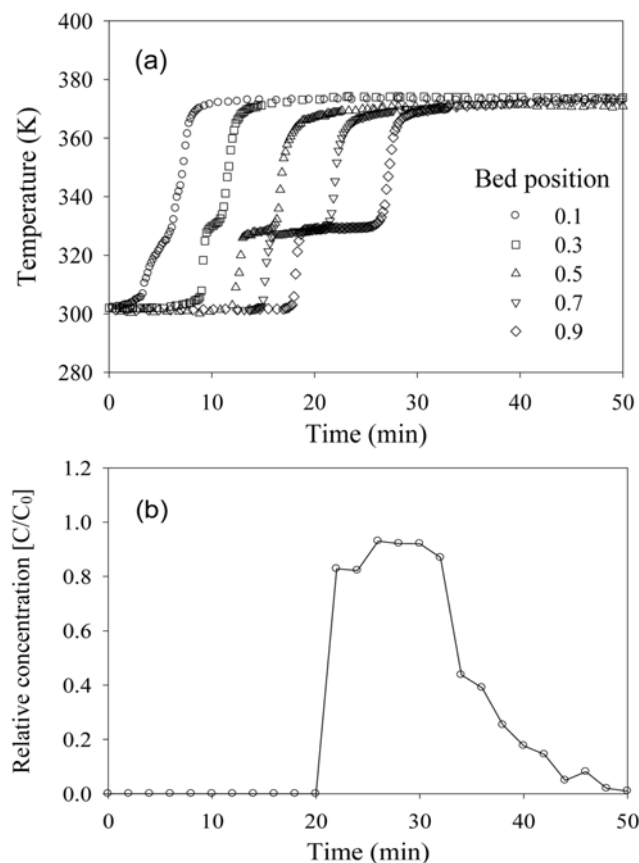

Fig. 7. Temperature (a) and Concentration (b) profiles of acetone/DAY-zeolite system with 0.8 g/min steam flow rate (initial condition: saturated acetone 16,000 ppm).

whereas toluene has a simple Type-I isotherm [16,17]. Thus, toluene has a strong adsorbate-adsorbent interaction, while acetone has a strong adsorbate-adsorbate interaction but a weak adsorbate-adsorbent interaction. Therefore, regardless of adsorbent type, a lower desorption tailing effect was observed in the regeneration of the acetone-saturated bed.

The range of highly desorbed concentration corresponded to the time from when the temperature was at a plateau to when it reached the steam temperature. Like the activated carbon bed, after reaching the steam temperature, the desorption concentration steeply decreased. However, concentration fluctuation was more significant than in the activated carbon bed because of increased condensation-revaporization in the DAY-zeolite bed. High acetone solubility in water might lead to the concentration propagation of the condensed mixture. Due to the weak adsorption of acetone on DAY-zeolite, the desorption concentration in the range of maximum concentration was higher than that in the activated carbon bed.

Profiles of temperature and concentration are presented in Fig. 7 for an increase in steam flow rate from 0.4 to 0.8 g/min. Due to the improved heat transfer rate, the propagation of the temperature and concentration profile was faster than in Fig. 6. However, the maximum desorption concentration started from the later part of the temperature plateau to the steam temperature at X=0.9. Further, the duration was shorter, but the maximum concentration was higher than the result in the low steam flow rate. Breakthrough time in the DAY-zeolite bed changed from 40 to 20 min as the steam flow rate changed from 0.4 to 0.8 g/min. On the other hand, in the activated carbon bed, the breakthrough time changed from 34 to 22 min for the same change in steam flow rate. Therefore, it can be concluded that the effect of steam flow rate on breakthrough time was stronger in the DAY-zeolite bed compared to the activated carbon bed. Moreover, the duration of the temperature plateau at 330 K was not greatly affected by the steam flow rate in the case of DAY-zeolite bed.

Desorption phenomena of acetone can be classified as desorption, condensation and vaporization steps. The difference in the breakthrough time between activated carbon and DAY zeolite bed is related to desorption of acetone. In the case of activated carbon, the improved vaporization caused by the doubled steam flow rate resulted in 35% earlier breakthrough of adsorbate. On the other hand, in the case of zeolites, an opposite phenomenon was observed. Because the desorption worked as a rate determining step in the DAY-zeolite bed instead of vaporization, the doubled steam flow rate resulted in an earlier breakthrough time.

CONCLUSIONS

In view of the problem of greenhouse gas emissions, the recovery of massive amounts of VOCs emitted from effluent gases by petrochemical industries is important to reuse resources through energy-saving processes. The dynamics of steam regeneration in activated carbon and DAY-zeolite beds were studied to recover acetone and toluene from effluent gas.

The desorption breakthrough of toluene was faster than that of acetone due to the higher adsorption capacity of acetone on both adsorbents. However, the breakthrough shape of acetone was steeper than that of toluene and its tailing effect was shorter than the toluene

result. The steam regeneration of toluene progressed faster in the DAY-zeolite than in activated carbon, while the difference in breakthrough time between both adsorbent beds was relatively smaller in acetone than in toluene. That is, the desorption behaviors were highly affected by the wave front of the water because acetone is water-miscible and toluene is water-immiscible. Since the increase in steam flow rate led to the increased heat transfer rate, it resulted in faster regeneration and a reduced temperature plateau. The breakthrough time in the DAY-zeolite bed was linearly improved by the change in steam flow rate because the isotherm is changed from a Type-II to Type-III isotherm with increased temperature. However, the improvement due to the steam flow rate was smaller in the activated carbon bed.

In steam regeneration, the stepwise temperature profile required to reach the inlet steam temperature was observed in both beds because desorption, condensation and vaporization occurred during steam regeneration. A temperature plateau was observed in the steam regeneration of both acetone and toluene, but the regeneration principles involved were totally different. The temperature plateau of toluene was observed near 355 K, which is lower than the boiling temperature of toluene and can be azeotropic. On the other hand, the temperature plateau of acetone occurred near 330 K, which is similar to its boiling temperature. The duration of the temperature plateau in the bed end corresponded to the time period of high desorption concentration in acetone desorption. However, in toluene desorption, it was shorter than the duration of high desorption concentration. Therefore, the maximum effluent concentration of toluene reached only 80% of feed concentration under experimental conditions, while that of acetone was almost 100% of feed concentration. The steam regeneration efficiency of the VOC adsorption process is significantly affected by adsorbents. To evaluate the process efficiency, the drying and cooling steps should be considered; however, the water-miscibility of the adsorbate is an important factor in steam regeneration.

REFERENCES

1. P. Monneyron, M. H. Manero and J. N. Foussard, *Environ. Sci. Technol.*, **37**, 2410 (2003).
2. M. A. Lillo-Rodenas, J. Carratala-Abril, D. Cazurla-Amoros and A. Linares-Solano, *Fuel Process. Technol.*, **77-78**, 331 (2002).
3. M. Kang, Y. S. Bae and C. H. Lee, *Carbon*, **43**, 1512 (2005).
4. S. Brosillon, M. H. Manero and J. N. Foussard, *Environ. Sci. Technol.*, **35**, 3571 (2001).
5. M. Kang and C. H. Lee, *Appl. Catal. A: Gen.*, **266**, 163 (2004).
6. D. Das, V. Gaur and N. Verma, *Carbon*, **42**, 2949 (2004).
7. X. Hu, S. Qiao, X. Zhao and G. Lu, *Ind. Eng. Chem. Res.*, **40**, 862 (2001).
8. J. Adolphs and J. M. Setzer, *J. Colloid Interface Sci.*, **180**, 70 (1996).
9. M. Guillemot, J. Mijoin, S. Mignard and P. Magnoux, *Ind. Eng. Chem. Res.*, **46**, 4614 (2007).
10. D. P. Serrano, G. Calleja, J. A. Botas and F. J. Gutierrez, *Ind. Eng. Chem. Res.*, **43**, 7010 (2004).
11. H. Ahn and C. H. Lee, *AIChE J.*, **49**, 1601 (2003).
12. J. J. Lee, M. K. Kim, D. G. Lee, M. J. Kim, H. Ahn and C. H. Lee, *AIChE J.*, **54**, 2054 (2008).
13. S. J. Lee, J. J. Hwan, J. H. Moon, J. G. Jee and C. H. Lee, *Ind. Eng.*

Chem. Res., **46**, 3720 (2007).

14. S. D. Manjare and A. K. Ghoshal, *Ind. Eng. Chem. Res.*, **45**, 6563 (2006).

15. S. W. Blocki, *Environ. Prog.*, **12**, 226 (1993).

16. J. H. Kim S. J. Lee, M. B. Kim, J. J. Lee and C. H. Lee, *Ind. Eng. Chem. Res.*, **46**, 4584 (2007).

17. D. G. Lee, J. H. Kim and C. H. Lee, *Sep. Purif. Technol.*, **77**, 312 (2011).

18. J. H. Yun, D. K. Choi and H. Moon, *Chem. Eng. Sci.*, **55**, 5857 (2000).

19. K. J. Oh, D. W. Park, S. S. Kim and S. W. Park, *Korean J. Chem. Eng.*, **27**, 632 (2010).

20. C. A. Grande, J. Gascon, F. Kapteijn and A. E. Rodrigues, *Chem. Eng. J.*, **160**, 207 (2010).

21. R. H. Perry, D. W. Green and J. O. Maloney, *Perry's chemical engineers' handbook*, 6th Ed, McGraw-Hill, New York (1984).

22. Y. K. Ryu, J. W. Chang, S. Y. Jung and C. H. Lee, *J. Chem. Eng. Data*, **47**, 363 (2002).

23. Y. K. Ryu, H. J. Lee, H. K. Yoo and C. H. Lee, *J. Chem. Eng. Data*, **47**, 1222 (2002).