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Yoon-Jong Yoo[a]; Hong-Soo Kim[a]; Moon-Hee Han[a]
[a] Functional Materials Research Center, Korea Institute of Energy Research, Daejeon, Korea

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Toluene and MEK Adsorption Behavior of the Adsorption System Using Honeycomb Adsorption Rotor

Yoon-Jong Yoo, Hong-Soo Kim, and Moon-Hee Han
Functional Materials Research Center, Korea Institute of Energy Research, Daejeon, Korea

Abstract: To remove toluene and methyl ethyl ketone (MEK) through adsorption, a honeycomb that is 60 cm in diameter and 40 cm in length was manufactured using ceramic paper as supporting material for adsorbents. Then, a mixture of Y type and ZSM-5 type zeolites with a ratio of 7:3 was impregnated on the surface of the honeycomb. By drying the impregnated material, the honeycomb adsorption rotor (HAR) was successfully manufactured. The amount of zeolites impregnated in the HAR was calculated to be 28 wt% and the peaks of the X-ray diffraction pattern of HAR were identical to those of the two kinds of zeolite powders. The small reduction in the BET surface area resulted from the reduction of mesopores while there was little reduction of micropores smaller than 100 nm, which plays a main role in the adsorptive separation of VOCs. Equilibrium adsorption for toluene and MEK of the HAR was measured 3.6 wt% and 3.3 wt%, respectively, at partial pressure of 0.2 mmHg. The experimental curves of equilibrium adsorption amount fitted well to the calculated curve by the Langmuir model; the interpretation of the breakthrough curve using material balances agreed with the test results. With 3 rph HAR rotation speed, 1.2 m/s face velocity, and 360 ppmv toluene and MEK inlet concentration, the rotary adsorption system showed average outlet concentrations of 18 ppmv and 14 ppmv, respectively, indicating removal efficiency was higher than 95%.

Keywords: VOC adsorption, honeycomb adsorbent, adsorption rotor, ceramic paper

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Address correspondence to Yoon-Jong Yoo, Functional Materials Research Center, Korea Institute of Energy Research, Daejeon 305-343, Korea. E-mail: yjyou@kier.re.kr

INTRODUCTION

Volatile organic compounds (VOCs) exhausted from petrochemical, coating, printing, and drying industries can cause global warming and photochemical smog as is well known. As foul-smelting carcinogens, VOCs are also typical materials that may deteriorate the indoor environment. For these reasons, there have been active attempts to remove VOCs contained in these exhaust gases through oxidation, adsorption, absorption, or condensation (1–6).

Among these various methods, adsorption techniques are being widely used, most of which consist of fixed beds packed with granule- or cylinder-type active carbons or zeolites (7–12). The adsorption process, however, with a fixed bed consumes lots of energy due to a high pressure drop within the bed. Another drawback is the necessity of supplementary adsorbents at regular intervals, which should be added to make up the loss caused by contact between particles within the bed (13, 14). The slow diffusion on the inside of the adsorbent is also a disadvantage because its surface contributes to adsorptive separation. Therefore, the rotary adsorption system using honeycomb type adsorbents is a new technology expected to overcome these disadvantages of granule- or cylinder-type adsorbents (15–18).

An extrusion method is generally used to manufacture honeycomb adsorbents; however, the resultant honeycomb could cause their porous structure to be easily damaged due to high extrusion pressure, and it is hard to form except in a limited size and form. On the other hand, the honeycomb adsorption rotor (HAR), which is being used in the rotary adsorption system, is readily formed into any type of honeycomb regardless of the size and shape by forming ceramic paper made of organic and inorganic fibers into the honeycomb shape before impregnation of adsorbents on its surface (19–22). Despite the many published articles about the rotary adsorption system, there are a few research papers on manufacturing HARs.

In this study, a HAR was manufactured by impregnating Y type and ZSM-5 type high silica zeolites, which are excellent in adsorbing toluene and methyl ethyl ketone (MEK), in honeycomb made of ceramic paper (21), its manufacturing process and adsorption were investigated. Also, the adsorptive removal characteristics of toluene and MEK by the rotary adsorption system with HAR were studied.

EXPERIMENTAL

Preparation of HAR (Honeycomb Adsorption Rotor)

Figure 1 shows the process of manufacturing a HAR using ceramic paper. The authors have already reported the method of manufacturing ceramic paper and HARs (21, 22). Thickness of the ceramic paper was about 0.2 mm; a honeycomb 60 cm in diameter and 40 cm in width was manufactured by

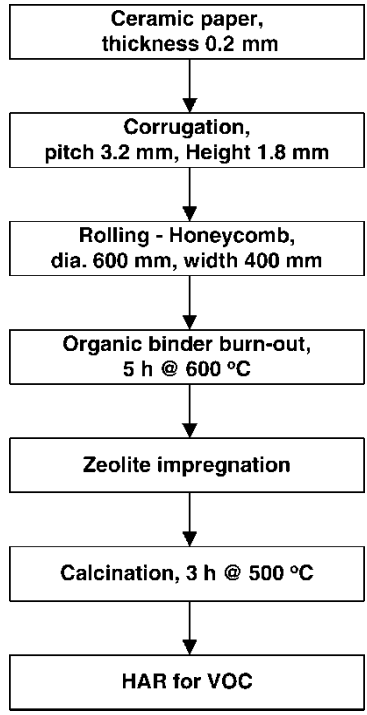


Figure 1. Manufacturing process of HAR using the ceramic paper.

rolling into a cylindrical shape after corrugating into a single-faced corrugated sheet in the wave form 3.2 mm in pitch and 1.8 mm in height. The honeycomb was heat treated at 600°C for 5 hours to remove organic binders in the honeycomb. Then, a solution was prepared for impregnating adsorbents by mixing Y type zeolite (UOP, Hisiv-1000), which has pores smaller than 0.8 nm, and ZSM-5 type zeolite (UOP, Hisiv-3000), which has pores smaller than 0.63 nm. The ratio of the Y type zeolite to the ZSM-5 type zeolite in the solution was 7:3. A silica sol (Snowtex ST-20), which was diluted to 5 wt%, was added to the solution. The heat-treated honeycomb was dipped into the solution for 10 minutes. The surplus solution was removed from the honeycomb through the forced dewatering process after natural dehydration. The preparation of the HAR was completed through dry and heat treatment.

Measurement of Equilibrium Adsorption Amount

With about 2.5 g pieces of sample detached from the HAR, the equilibrium adsorption amounts for toluene and MEK were measured with an equilibrium adsorption rate measurement system (Rubotherm, MSB-30G500P). The

sample, which was put into a sample bowl within a chemical reactor, was degassed to 10^{-3} mmHg for removing adsorbed gas completely from adsorbents with maintaining temperature at 200°C for 3 hours. Then toluene and MEK were supplied at predetermined partial pressure in the adsorption temperature of 30°C to keep it until there was no increase in weight. After that, the measurement of the equilibrium adsorption amount was continued at the different partial pressures.

Rotary Adsorption System

Figure 2 illustrates toluene and MEK adsorption process by the rotary adsorption system with the HAR. As it can be seen in Fig. 2, the HAR was divided into adsorption, regeneration, and cooling zones with a ratio of 10:1:1, respectively, and VOC removal operation was performed by rotating the HAR at 3–4 rph (rotation per hour). After normal adsorption of VOCs, a section of the HAR in the adsorption zone at room temperature was forced to rotate into the regeneration zone for desorbing the adsorbed VOCs while providing heated air at about 170°C. Usually, the desorbed VOCs were concentrated with respect to inlet concentration, and the concentrated VOCs from the regeneration zone were transferred to the catalytic combustor, where they were decomposed by catalytic combustion. The combusted air, which was discharged outside, had a VOC concentration lower than 3 ppmv. The temperature of the combusted air was higher than 500°C, and the heat was used to increase the temperature of the outside air. Through heat exchange, the outside air was heated to 170°C and supplied to the regeneration zone, as a regenerative heat source itself without auxiliary fuel. The

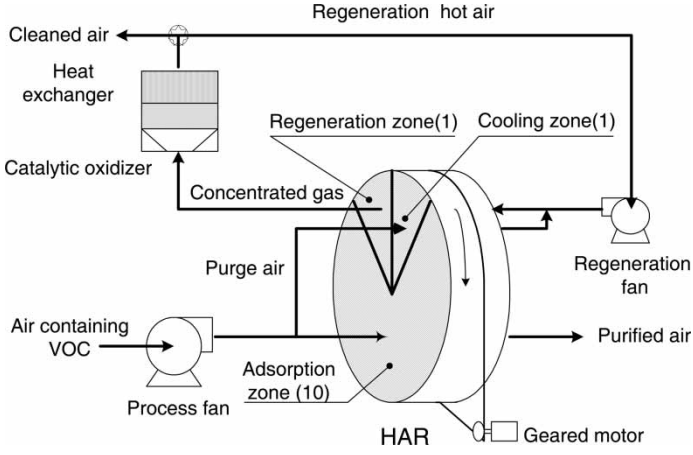


Figure 2. Schematic diagram of the rotary adsorption system and the honeycomb adsorption rotor (HAR).

section of the HAR that completed regeneration was moved into the cooling zone, where it was cooled down to recover adsorption capacity.

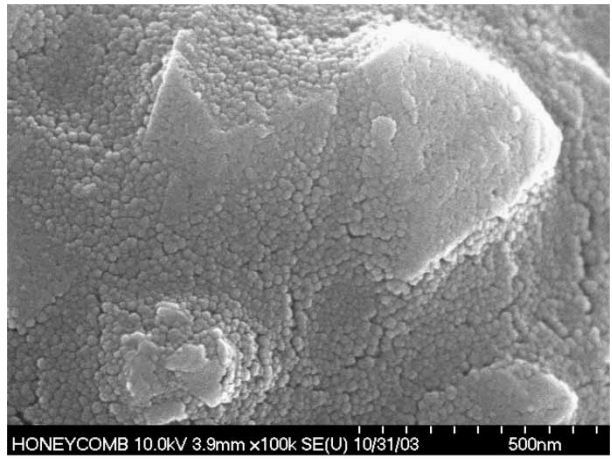
In order to operate the VOC adsorption system effectively with various VOC concentrations and inlet flow rates, the optimum rotation speed of the HAR should be determined experimentally under such conditions. For that purpose, the HAR was heated to complete desorption state, before VOC-loaded air, which had different concentrations of toluene and MEK (250 ppmv and 350 ppmv) that flowed through the HAR with a rate of 1.2 m/s for removing the VOCs by adsorption. In order to confirm that the VOCs were adsorbed and desorbed in the same part of the HAR, the HAR was not rotated during the desorption treatment by means of high-temperature regenerative heat source. During the adsorption test, the concentrations of the VOCs at the outlet side of the HAR were measured by using a total hydrocarbon analyzer (HORIBA, FIA-510). When the rotary adsorption system was operated, the VOCs were removed by the adsorption zone, and desorbed from the regeneration zone. The ratio of the VOC concentration at the outlet side to that of the inlet side of the adsorption zone was called adsorption removal efficiency, and the ratio of the VOC concentration at the outlet side of the desorption zone to that of the inlet side of the adsorption zone was called concentration ratio. The toluene and MEK adsorptive removal efficiencies and concentration ratios were measured with various HAR rotation speed, 3–4 rph.

RESULTS AND DISCUSSION

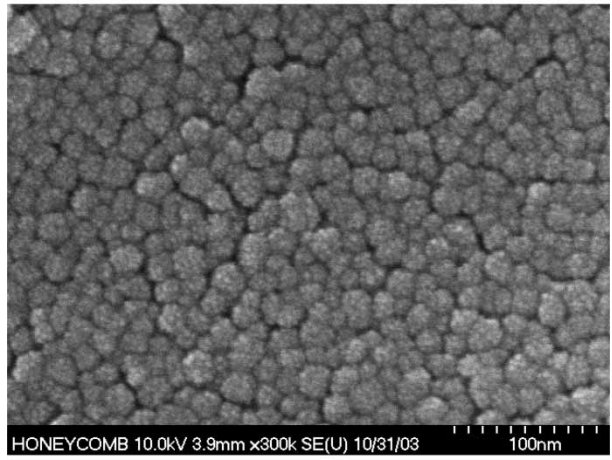
Zeolite Impregnation Characteristics of HAR

The prepared HAR was 60 cm in diameter, 40 cm in length, 17.9 kg in weight, and 0.158 g/cm^3 in bulk density. Zeolites impregnated in HAR amounted to 5.0 kg, which was approximately 28 wt% of the HAR. Figure 3 represents the SEM images of the detached sample of the surface of the HAR impregnated with zeolites. A mixture of two kinds of zeolites evenly distributed between ceramic fibers, which formed a honeycomb framework on the surface of the HAR shown in Fig. 3(a). In Fig. 3(b), zeolites were bound firmly, and surrounded by silica particles as binders. The most appropriate amount of silica was 5 wt% in consideration of binding strength intensity, impregnated amount, BET surface area, and so on.

Figure 4 shows X-ray diffraction patterns of the HAR. In this figure, ceramic paper shows an amorphous diffraction pattern; Y type zeolites showed major peaks at the diffraction angles (2θ) of 6.2°, 10.2°, 12.0°, 15.8°, and 23.9°, and ZSM-5 type zeolites at the diffraction angles of 7.8°, 8.7°, 22.9°, 23.8°, and 29.8°. When a mixture of zeolites was impregnated in the HAR, major peaks appeared at the same diffraction angles as those of two kinds of zeolites. This result implies that when zeolites were impregnated



(a)



(b)

Figure 3. SEM surface morphology of the HAR: (a) fixed zeolites by 5 wt% silica binder and (b) silica nano particles.

on the surface of the HAR using silica sol and heat-treated at 600°C, there was no reaction between binder and ceramic fiber.

The BET surface area of the HAR was 139 m^2/g. This surface area was 25% of the weight-average surface area of mixed raw zeolites (558 m^2/g), the value of which was calculated based on the surface areas of the Y type zeolite (596 m^2/g) and ZSM-5 type zeolite (469 m^2/g), and the mixing ratio of 7:3. This value was similar to the weight gain of the HAR, which was 28 wt%. Figure 5 shows relations between the pore size and the pore volume determined by argon adsorption of HAR. As seen in the figure, the

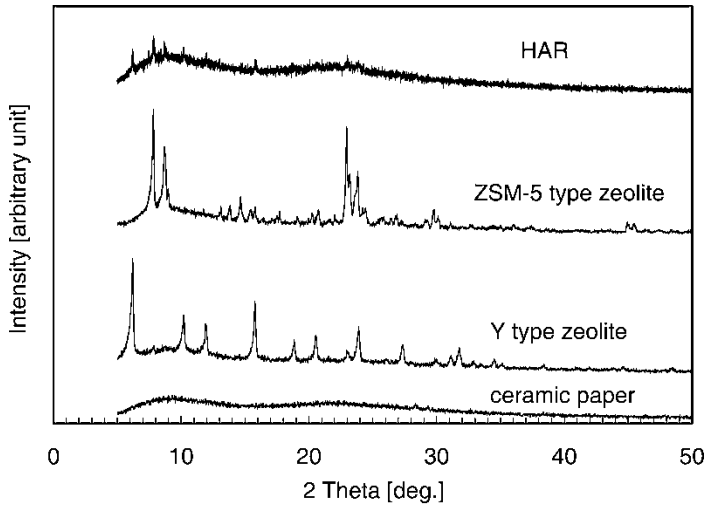


Figure 4. X-ray diffraction patterns of the ceramic paper, Y type and ZSM-5 type zeolites and HAR.

volume of the micropores, which were smaller than 1 nm, showed about 25% of the volume of micropores of the raw zeolites, and the value was very similar to the ratio of the BET surface area. Therefore, because 10–20 nm particles in silica sol were used as binder for coating 1–2 μm zeolite crystals to the surface of the honeycomb, a reduction in the volume of mesopores formed

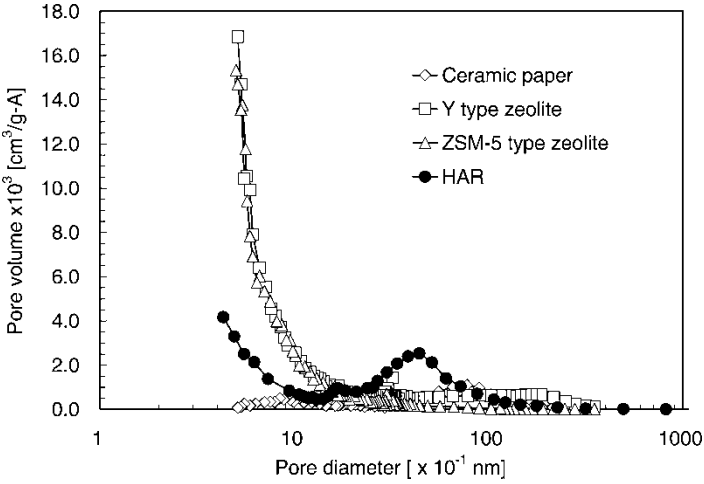


Figure 5. Pore size distribution of the Y-type, ZSM-5 type zeolites, and the HAR with 5 wt% silica content.

between zeolite crystals resulted. However, the volume of the micropores smaller than 1 nm did not change, which contributes to adsorptive separation. Consequently, the impregnation will not adversely affect the adsorption of toluene and MEK, the kinetic diameter of which are 0.59 nm and 0.43 nm, respectively.

Equilibrium Adsorption Amounts of HAR

Figure 6 shows toluene and MEK equilibrium adsorption amounts of the HAR. As can be seen in the figure, the HAR adsorbed 3.5 wt% at the toluene partial pressure of 0.2 mmHg. This corresponds to 26.7% of 13.1 wt% in the equilibrium adsorption amount of Y type zeolites on the same conditions, and it also corresponds to 95.5%, considering that the impregnated amount of zeolites is 28 wt%. However, zeolites impregnated in the HAR showed almost no decrease in adsorption because they were mixed with 30% ZSM-5 type zeolites used to increase MEK adsorption. The MEK equilibrium adsorption amount of the HAR was about 3.2 wt% at the MEK partial pressure of 0.2 mmHg, which was 25.8% of 12.4 wt%, the adsorption amount of ZSM-5 type zeolites under the same conditions. This value also corresponds to 92.2% of raw powder considering the impregnated amount of zeolites; there was almost no decrease in adsorption in consideration of the mixing ratio of zeolites and the increase in weight by the use of binders. It is judged that these results are very encouraging, considering that the pore-maintaining rate and adsorption are usually about 85% of raw

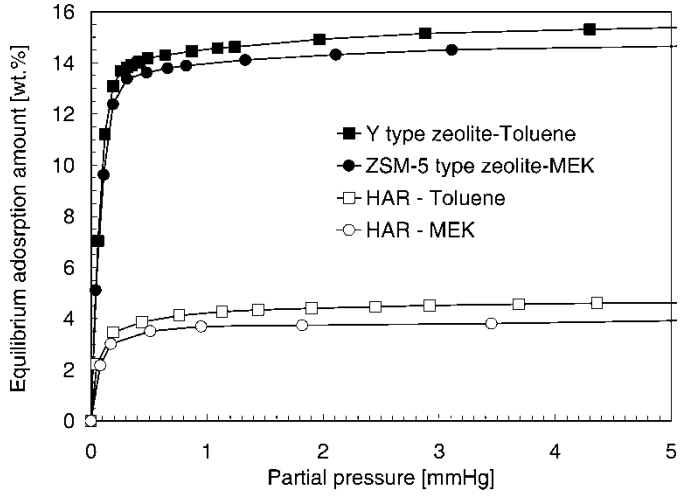


Figure 6. Equilibrium adsorption amounts of toluene and MEK into zeolite powders and the HAR.

powder when powdered zeolites are made granule- or cylinder-type along with a binder.

VOCs Breakthrough Characteristics of HAR

Figure 7 shows breakthrough curves of toluene and MEK measured at the HAR outlet when supplying toluene and MEK in 250 ppmv and 350 ppmv, respectively, with the face velocity of 1.2 m/s and the rotation of HAR stopped in order to determine HAR rotation speed of the rotary adsorption system. The following expression (1) was used to calculate adsorption amounts and adsorption efficiency.

$$q = M_w \int_{t=0}^{t=\infty} (C_1 Q_1 - C_2 Q_2)\, dt \tag{1}$$

As seen in the figure, when the concentration of each of toluene and MEK was increased from 250 ppmv to 350 ppmv at a fixed flow rate, 40-ppmv discharge time was decreased, the concentration of which is slightly lower than the allowed regulatory limit, for toluene from 22 min to 18 min and for MEK from 33 min to 19 min. Despite changed concentration of polluted air supplied to the HAR, there was little change in adsorption amounts of VOCs in the HAR with 3.3 wt% for toluene and 3.1 wt% for MEK. Particularly, when the data were compared with the equilibrium adsorption amounts of each toluene and MEK in Y type zeolite powder

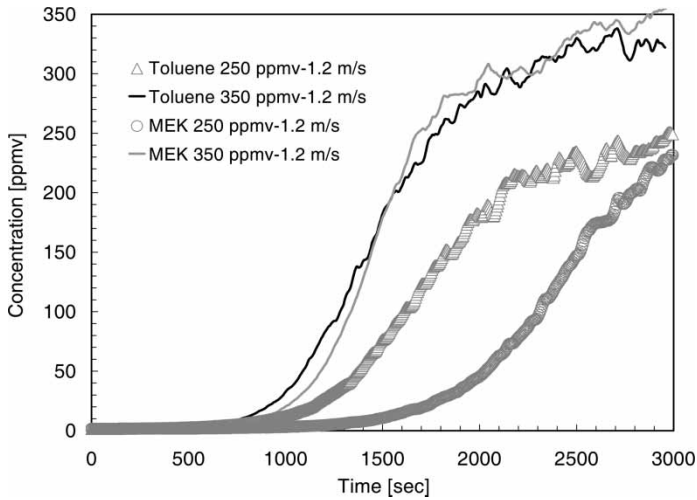


Figure 7. Toluene and MEK breakthrough curves of the HAR.

and ZSM-5 type zeolite powder at the same concentration (0.2 mmHg = 350 ppmv), which were 3.5 wt% and 3.2 wt%, respectively, toluene was adsorbed 94% while MEK was adsorbed 97% of each equilibrium adsorption amount. The adsorptive removal efficiency of each of toluene and MEK until the concentration at the HAR outlet reached 40 ppmv was very high, 94.9% and 96.2%, respectively. During the measurement of the equilibrium adsorption amount, sufficient time was allowed in measurement while the contact time of HAR with polluted air was only about 0.3 sec. It indirectly demonstrates that adsorption rate of toluene and MEK on the surface of HAR was very high.

To build a mathematical model for interpreting breakthrough curves, the following were hypothesized. The flow rate of air in gas phase was fixed during a breakthrough test; all the gases in gas phase follow the ideal gas law. Pressure drop within the HAR and diffusion of gas toward an axis can be neglected, and the temperature is fixed. Adsorption and desorption rate is proportional to the difference between the equilibrium adsorption amount and the actual adsorbed amount. Also, the equilibrium adsorption amount follows the Langmuir model. By applying these hypotheses, the material balance can be given as in the following expression:

$$\frac{\partial y}{\partial t} = u\frac{\partial y}{\partial z} + \frac{\rho_b}{\varepsilon}\frac{RT}{pM_w}\frac{\partial q}{\partial t} \tag{2}$$

The Langmuir model introduced to calculate the equilibrium adsorption amount is given in the following expression:

$$\frac{q_{eq}}{q_s} = \frac{bp}{1+bp} \tag{3}$$

Since it was hypothesized that adsorption and desorption rate is proportional to the difference between the equilibrium adsorption amount and the actual adsorption amount, the adsorption and desorption rate are given by the following expression (4):

$$\frac{\partial q}{\partial t} = k(q_{eq} - q) \tag{4}$$

Figure 8 shows the experimental results of the equilibrium adsorption amount for toluene and MEK of the HAR and that of the calculated results by the Langmuir model. Table 1 shows values of parameters in the Langmuir model for the calculation, which resulted from the regressive analysis using experimental results. As can be seen in Fig. 8, the equilibrium adsorption amounts of each of toluene and MEK were well interpreted by means of the Langmuir model. Since concentration at the inlet (y_0) was

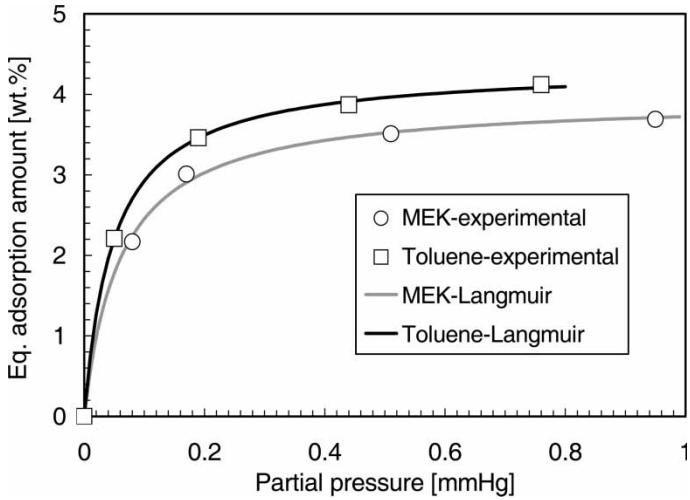


Figure 8. Adsorption isotherms of the toluene and MEK as experimental data and calculated data by Langmuir model.

fixed during the breakthrough test, the primary and boundary conditions are as follows:

$$y(0, z) = 0 \tag{5}$$

$$y(t, 0) = y_0 \tag{6}$$

Equation (2) using conditions of above Eqs. (5) and (6) was solved through differentiation into Crank-Nicolson-Upwind Scheme.

Table 2 shows input data used to calculate breakthrough curves. Figure 9 shows experimental curves and the calculated curves of the breakthrough test for toluene and MEK of the HAR. The calculated curve in the figure used the material transfer coefficient in Eq. (4), $0.003\,s^{-1}$ for toluene and $0.0025\,s^{-1}$ for MEK; this material transfer coefficient was selected to interpret an adsorptive destruction curve properly. As can be seen in Fig. 9, the model in Eq. (2) was found to interpret adsorption behaviors of the HAR properly.

Table 1. Parameters of Langmuir model

Parameters materials	q_s (g/g—Adsorbent)	b ($mmHg^{-1}$)
MEK	3.95	16.37
Toluene	4.34	20.58

Table 2. Input data used for the calculation of breakthrough curves

Items	Value
HAR length (cm)	40
HAR void fraction (ε)	0.7922
Face velocity (m/s)	1.22
Mass transfer coefficient for MEK (s^{-1})	0.0025
HAR diameter (cm)	60
HAR bulk density (g/cm^3)	0.16
Inlet concentration (ppmv)	350
Mass transfer coefficient for toluene (s^{-1})	0.003

Operation Characteristics of Rotary Adsorption System

Table 3 and Figure 10 show the result of operation of the rotary adsorption system using the HAR. As can be seen in Table 3, when the operation condition of the system with the HAR was 3 rph in the rotation speed, 360 ppmv in the toluene and MEK inlet concentration, and 1.2 m/s face velocity, the average outlet concentration was approximately 18 ppmv for toluene. And when adsorbed toluene was desorbed by hot air, the outlet concentration was approximately 2200 ppmv, which was about six times higher than that at the inlet. It means the 95% of toluene contained in the inlet air was removed. As for MEK, the average outlet concentration was 14 ppmv

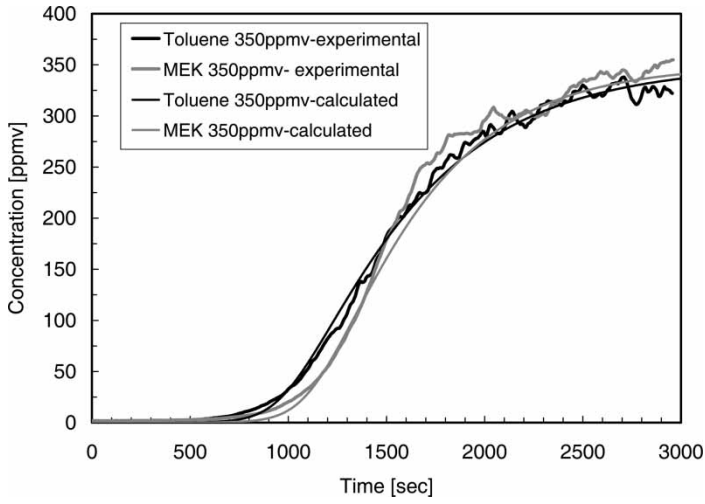


Figure 9. Experimental and calculated breakthrough curves of toluene and MEK of the HAR.

Table 3. Operation characteristics of rotary adsorption system

VOCs	Rotation speed (rph)	Face velocity (m/sec)	Average concentration (ppmv)			Removal efficiency (%)
			Inlet	Outlet	Concentrated	
Toluene	4	1.22	380	37.4	2569	90.2
	3	1.22	361	18.3	2208	94.9
MEK	4	1.22	357	9.3	2487	97.4
	3	1.22	357	14.4	2067	96.0

and the removal efficiency 96% under the same conditions. On this basis, removal efficiencies for the two kinds of VOCs were all more than 95% and outlet concentrations were less than 40 ppmv, a regulatory-allowed outlet concentration. It seems to be useful to remove VOCs through the rotary adsorption system. The HAR abated approximately 1050 Nm^3/hr VOC-polluted air when its concentration was 360 ppmv. It was possible to supply hot air for regeneration inside the system itself through catalytic combustion of concentrated VOCs.

Figure 11 shows a comparison between theoretical pressure drop calculated by Ergun equation with packed density of 0.66 g/cm^3 that is fixed-bed packed with granule- or cylinder-type adsorbents and the change in pressure drop due to the change in adsorption flow rate measured with the HAR with a bulk density of 0.21 g/cm^3. As can be seen in Fig. 11, pressure drop in the HAR is very low, about 10%, compared with the fixed bed for equal bed size and flow rate. Particularly, with bulk density of the HAR at

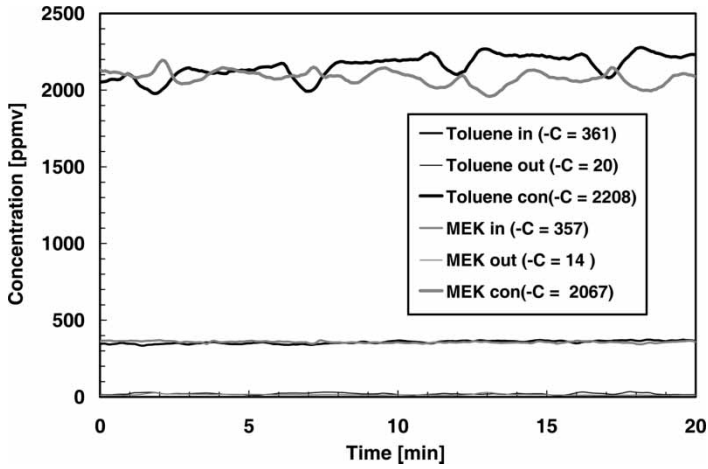


Figure 10. VOCs adsorption and concentration behaviors of the rotary adsorption system installed HAR.

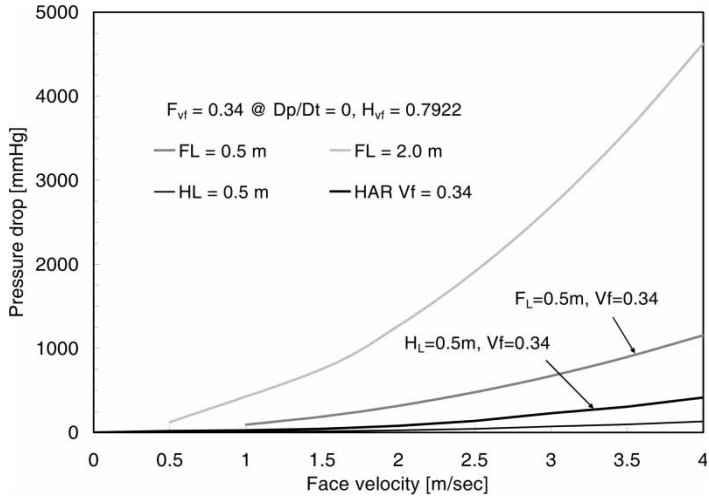


Figure 11. Comparison of pressure drops between the fixed bed and honeycomb adsorption rotor.

$0.66\,g/cm^3$, which was identical to the fixed bed, the pressure drop of the HAR was about 25% of the fixed bed; thus, it also seemed to be very useful in energy consumption necessary to operating the system.

CONCLUSIONS

The HAR, 60 cm in diameter and 40 cm in length, was successfully manufactured through mixed impregnation of Y type and ZSM-5 type zeolites with a ratio of 7:3 into honeycomb-shaped ceramic paper. Those zeolites impregnated on the surface of the HAR were evenly dispersed and bound firmly by silica. Since the X-ray diffraction pattern agreed with the diffraction pattern of the two kinds of zeolites, properties specific to zeolites were maintained without any adverse effect of heat treatment and silica used to fix them.

The amount of zeolites impregnated in HAR was 28 wt% of the weight of the honeycomb; BET surface area was 25% of that raw powder, which was almost the same value with the impregnated amount of zeolites. Minute reduction in BET surface area resulted from the reduction of mesopores more than 5 nm, which were distributed between crystals of zeolites, while there was little effect on the pore size distribution of micropores smaller than 1 nm, which were essential in adsorbing toluene and MEK molecules. The proper amount of silica was 5 wt% to minimize the reduction of BET surface area and bind zeolites firmly on the surface of HAR.

The equilibrium adsorption amounts for toluene and MEK of HAR were 26.7% and 25.8% of raw-powder zeolites, respectively, which showed similar

tendencies with the results of the BET surface area. The measured results of the equilibrium adsorption amount and the calculated results by the Langmuir model coincided very well. The material balance corresponding to the result of the breakthrough test could be used to optimize operation of the rotary adsorption system using the HAR by interpreting adsorption behaviors of the HAR properly.

As to the removal characteristics of toluene and MEK by the rotary adsorption system, when the HAR was 3 rph in the rotation speed, 360 ppmv inlet concentration, and 1.2 m/s face velocity, average outlet concentrations of adsorptives were 18 ppmv and 14 ppmv, respectively, with very high removal efficiency over 95%. Also, the outlet concentration of toluene and MEK was about 2200 ppmv, which was more than six times higher than that at the inlet. The temperature of the air recovered from the heat obtained through catalytic combustion of concentrated VOCs was sufficient to regenerate the HAR. Experimental HAR pressure drop was about 25% of pressure drop of the fixed bed calculated by the Ergun equation under the same conditions.

NOMENCLATURE

b	parameter of the Langmuir model
C_1	concentration at HAR inlet (mol/liter)
C_2	concentration at HAR outlet (mol/liter)
C	average concentration (ppmv)
con	concentrated VOC
D_p	particle diameter of adsorbents (m)
D_t	tower diameter of fixed bed (m)
F_L	length of fixed bed (m)
F_{vf}	void fraction of fixed bed
H_L	HAR length (m)
H_{vf}	void fraction of HAR
in	inlet of HAR
k	material transfer coefficient (s^{-1})
M_w	molecular weight of adsorbent (g/mol)
out	outlet of HAR
p	adsorption pressure (mmHg)
Q_1	flow rate on inlet conditions (liter/sec—P_1, T_1)
Q_2	flow rate on outlet conditions (liter/sec—P_2, T_2)
q	adsorption amount of HAR (g)
q_{eq}	equilibrium adsorption amount (g)
q_s	parameter of the Langmuir model
T	absolute temperature (°K)
t	adsorption time
u	flow rate within HAR (liter/sec)

Vf	void fraction
y	mole fraction of an adsorbent in gas phase
z	coordinate of HAR in the direction of rotation axis

Greek Letters

ε	porosity of HAR
ρ_b	density of HAR

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