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REMOVAL OF ACETONE AND METHANOL FROM GASEOUS STREAMS IN A HOLLOW FIBER ABSORBER

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ABSTRACT

Absorption removal of acetone and methanol from N_2 gas streams with liquid silicone oil was investigated in microporous hollow fiber modules. The gas flowed into the tube and silicone oil was flowed in the shell. Inlet concentrations of acetone and methanol were 1600 and 500 ppmv, respectively. All experiments were performed as a function of gas linear velocity (2.1–10.4 cm/sec), liquid linear velocity (0.6–5.8 cm/sec), and the operation time (10–60 min). The overall mass-transfer coefficients based on the gas phase were calculated and compared with the experimental results. The mass transfer mechanism was discussed and the height of a transfer unit in this device was finally determined.

Key Words: Absorption removal; Acetone; Hollow fiber module; Mass transfer; Methanol; Silicone oil

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INTRODUCTION

The discharge of volatile organic compound (VOC)-contaminated N_2 purge and air streams in chemical processes or plants would lead to serious environmental and health problems. Many methods have been used for reducing VOC emissions such as activated carbon adsorption, liquid absorption, incineration/thermal oxidation (usually without energy recovery), and catalytic oxidation (1,2). Because each method has its strengths and weaknesses, efforts are undertaken continuously to develop better techniques including membrane separation processes (3). Vapor permeation is one of the emerging methods, which uses nonporous VOC-selective polymer membranes. The partial pressure gradient, the driving force of this process, is basically small (atmospheric versus vacuum). It would be uneconomic to treat feed VOC concentration below about 100–200 ppmv because a large fraction of the feed air appears in permeate, resulting in condensation of VOCs from the feed (4–6).

Another potential technique involves the use of microporous hollow fiber modules, which resemble the shell-and-tube type heat exchangers (7,8). They can be used efficiently for removal of gaseous pollutants by either physical or reactive absorption (4–12). Taking the removal of VOCs from N_2 streams as an example, the contaminated N_2 gas flows into the tube side of the modules. An absorbent liquid having high solubility for VOCs is cross- or parallel-flow pumped through the shell side. Absorption and stripping occur at the interfaces created in the pores of microporous membranes. Such membrane-based process has many advantages over common columns/towers including: independent variations of gas and liquid flow rates without the problems of flooding, loading, and weeping; much high volumetric mass-transfer coefficient (mass transfer per unit volume); known interfacial area; high contact area per unit equipment volume compared to packed towers (10^3–10^4 vs. 10^2–10^3 ft^2/ft^3); and the versatility of modular design (7,8,13).

Although absorption removal of VOCs in hollow fiber modules has been studied extensively, the experimental data presented or the models proposed were mostly limited to the so-called "steady-state" values. It was actually reported that the time taken to attain a steady VOC concentration at the absorber outlet is about 7–8 hr in a membrane-based absorption–stripping process (4,5). Although the time taken to attain a steady state was considerably reduced when the stripper was operated at a much higher temperature (12), the time-dependent data of VOC removal may be of practical interest and significance, at least, at the early stage of the process. In this work, time profiles of acetone and methanol removal were measured. Experiments were carried out as a function of the gas and liquid linear velocities, and the operation time. The overall mass-transfer coefficients based on gas phase were obtained and the height of a transfer unit in this device was determined. This provided quantitative evidence on mass transfer mechanisms of previous models or results (4,5).

EXPERIMENTAL

Chemicals

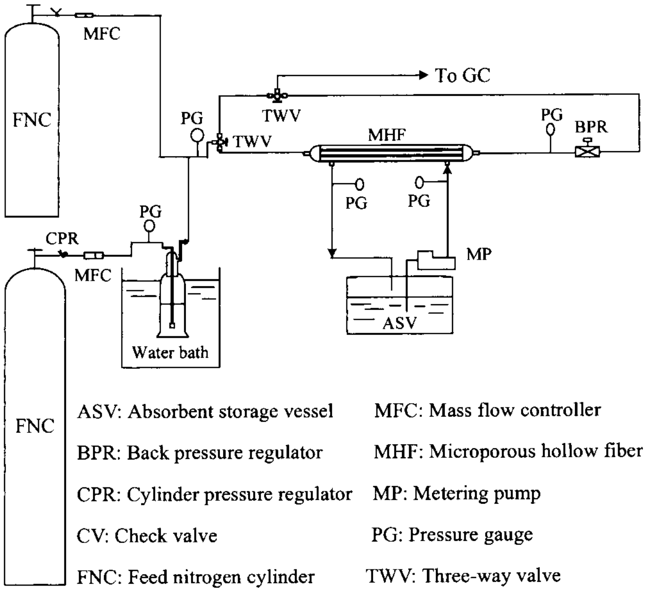
The absorbent silicone oil 200 fluid (Dow Corning, Midland, MI) was made of linear poly(dimethylsiloxane) and used as received. It had a viscosity of 50 c.p. and a density of 0.96 g/cm^3. It was inert to parts of the module, non-toxic, essentially non-volatile, and water-insoluble. Certified VOC-N_2 mixtures of acetone (1895 ppmv) and methanol (547 ppmv) were purchased in primary standard cylinders (Air Liquide, LaPorte, TX). They were used for gas chromatograph (GC) calibration.

Experimental Procedures

Figure 1 shows the experimental setup. N_2 gas first flowed through the liquid VOC solution (250 cm^3), and was then combined with another N_2 gas to control the feed VOC concentration. Finally, the gas flowed into the tube side of the polypropylene hollow fiber module. The specifications of the hydrophobic module (Microdyn LM-2P06, Microdyn Co., Wuppertal, Germany) are listed in Table 1. It was known that the membrane resistance would dominate the overall mass transfer in this case (4,5). This module was selected because the aim of this work was to provide quantitative verification on mass transfer mechanism of previous results, as stated above. Moreover, the operation seemed to be rather stable and simple when the pores were liquid-filled (8).

The inlet concentrations of acetone and methanol were fixed at about 1600 and 500 ppmv, respectively, by adjusting the temperature of water bath. The absorbent liquid (250 cm^3) was countercurrently pumped by a digital metering pump (Cole-Parmer, Masterflex 7524-00, Vernon Hills, IL) into the shell side of the module. It was completely recycled to the storage vessel during absorption. The gas outlet from the module was connected to a GC (Varian CP-3800, Varian Co., Palo Alto, CA). The exit gas was finally vented through a fume hood. This small laboratory-scale device was used for experimental purposes, making it possible to evaluate the process performance without having to prepare large amounts of absorbent and VOCs.

Prior to experiments, the shell side of the module was filled with the absorbent liquid for 30 min to wet the membrane; in the meantime, pure N_2 gas also flowed into the tube side to avoid the leakage of absorbent liquid. Then the mixture of VOC-N_2 gas flow was switched on at a preset flow rate through the tube side of the module. The time needed to replace the pure N_2 gas by the VOC-N_2 gas mixture (i.e., the residence time) was less than



ASV: Absorbent storage vessel
BPR: Back pressure regulator
CPR: Cylinder pressure regulator
CV: Check valve
FNC: Feed nitrogen cylinder
MFC: Mass flow controller
MHF: Microporous hollow fiber
MP: Metering pump
PG: Pressure gauge
TWV: Three-way valve

Figure 1. Experimental setup for removal of VOCs in hollow fiber absorbers.

0.2 min. Gas velocity was maintained by a digital mass flow controller (Sierra Instruments Inc., Monterey, CA). The gas outlet pressure was maintained at 2 psig above liquid phase pressure by adjusting the back-pressure regulator (5) and the liquid phase pressure was basically atmospheric. Such adjustment steps were completed within around 1 min. At this moment the experiment was started, and the gas outlet was sampled at preset time intervals. In all runs, the inlet concentrations of acetone and methanol were calibrated.

Measurements of Volatile Organic Compound Concentration

The concentrations of VOCs in the gas phase were measured using GC (Varian CP-3800) equipped with a flame ionization detector (FID). The response from FID was recorded in a built-in integrator. The gas mixture was injected into the GC column through 6-port gas sampling valve (Valco, Houston, TX). A 30 m × 0.45 mm capillary column (Varian CP-624D) was used for analysis.

Table 1. Specifications of LM-2P06 Microporous Hollow Fibers from Manufacturer

1. Shell characteristics	
Material	Polycarbonate
Length	280 mm
Inner diameter, $2R_i$	14 mm
Outer diameter	18 mm
2. Fiber characteristics	
Material	Polypropylene
Number of fibers, n	85
Effective length, L	240 mm
Inner diameter, $2r_i$	0.6 mm
Outer diameter, $2r_o$	1.0 mm
Effective surface area, S	0.04 m^2
Porosity, ϵ	0.7
Average pore size	0.2 μm

RESULTS AND DISCUSSION

Time Profiles of Removal Efficiency

Figures 2–5 show the time profiles of outlet gas concentration and removal efficiency for acetone and methanol under different conditions (gas velocity u_G, liquid velocity u_L, etc.). In the present absorption process, the removal efficiency is calculated by

$$\text{Removal efficiency (\%)} = 100 \times \left(1 - \frac{\text{outlet VOC concentration}}{\text{inlet VOC concentration}}\right) \qquad (1)$$

It is seen that the outlet gas concentration increases with increasing operation time; they reach a plateau after about 50-min operation at a sufficiently high u_G (>10.4 cm/sec). In addition, the efficiency is higher at a lower u_G and larger u_L. As also indicated earlier, removal of VOCs using hollow fiber absorbers is promising (4,5). The discontinuous change in Fig. 2 when u_L increases is likely a result of the incomplete use of the total fiber area (due to bypassing by the shell side liquid) at high u_L (8). It should be noted that the flow velocity of either phase could not be extended arbitrarily because of the concern of process stability. For example, the gas outlet pressure must be always maintained at about 2–3 psig above liquid phase pressure in this work.

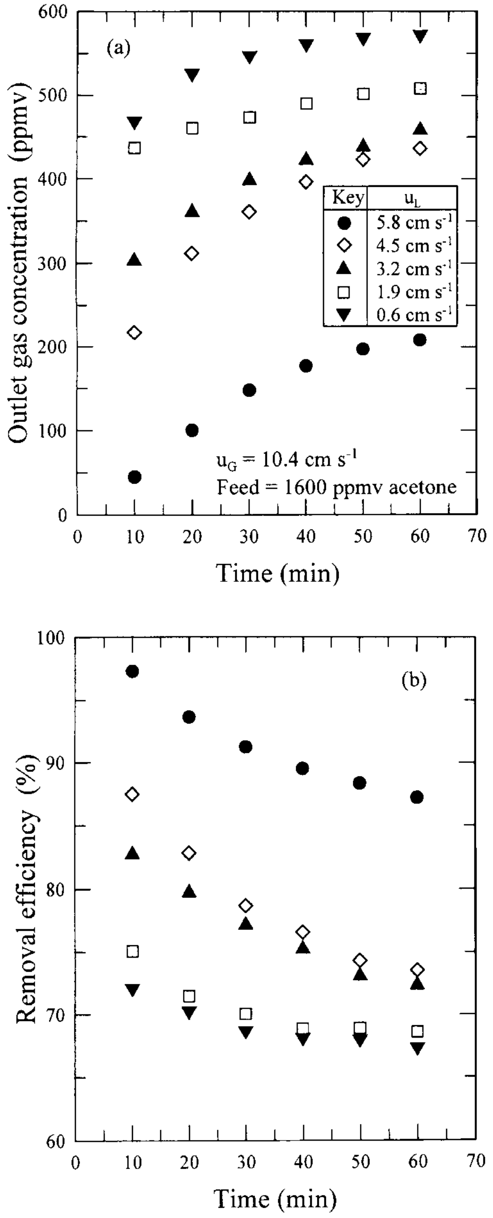


Figure 2. Time profiles of outlet gas concentration and removal efficiency of acetone (u_G = 10.4 cm/sec).

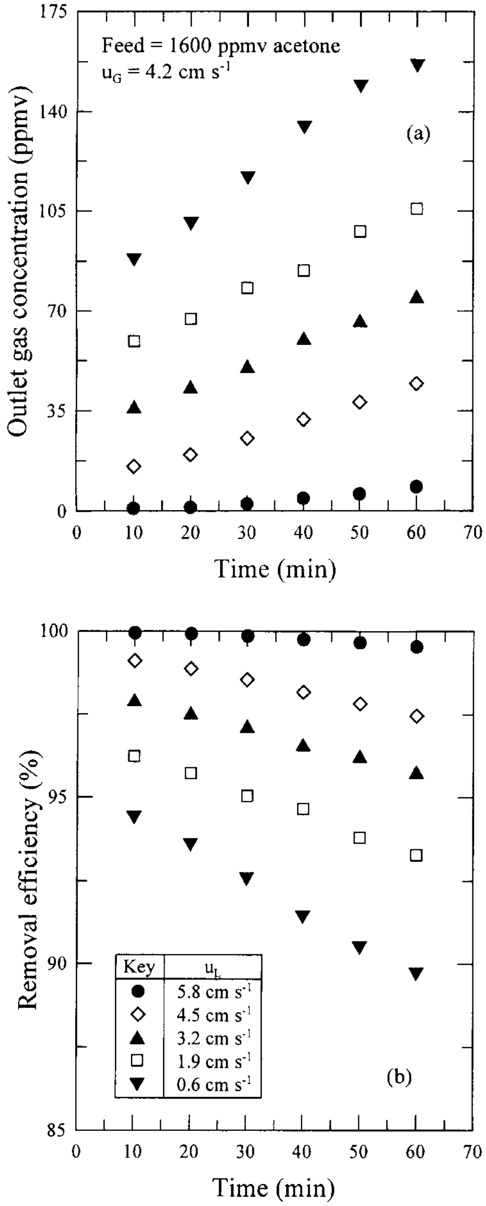


Figure 3. Time profiles of outlet gas concentration and removal efficiency of acetone (u_G = 4.2 cm/sec).

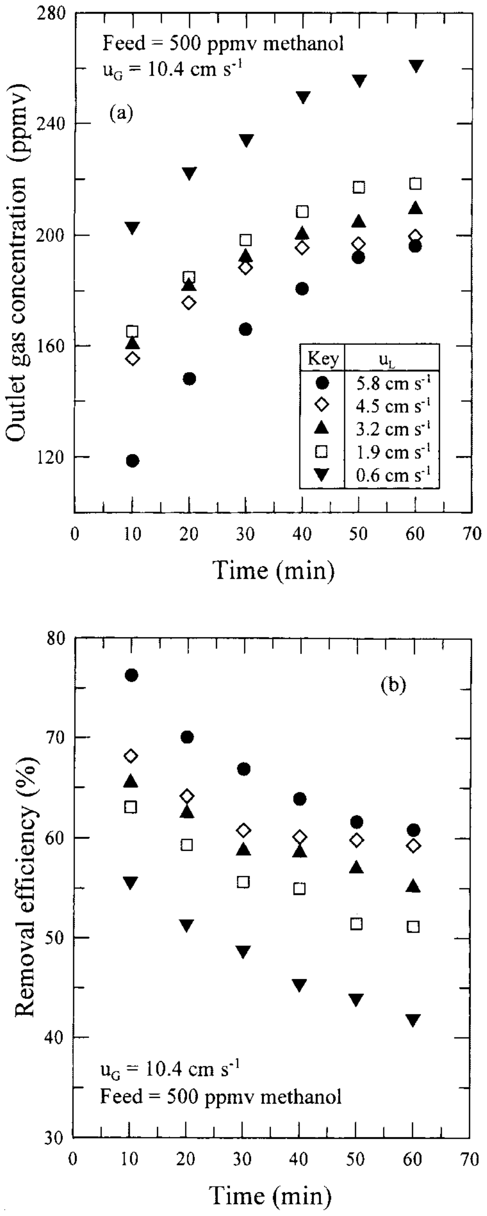


Figure 4. Time profiles of outlet gas concentration and removal efficiency of methanol (u_G = 10.4 cm/sec).

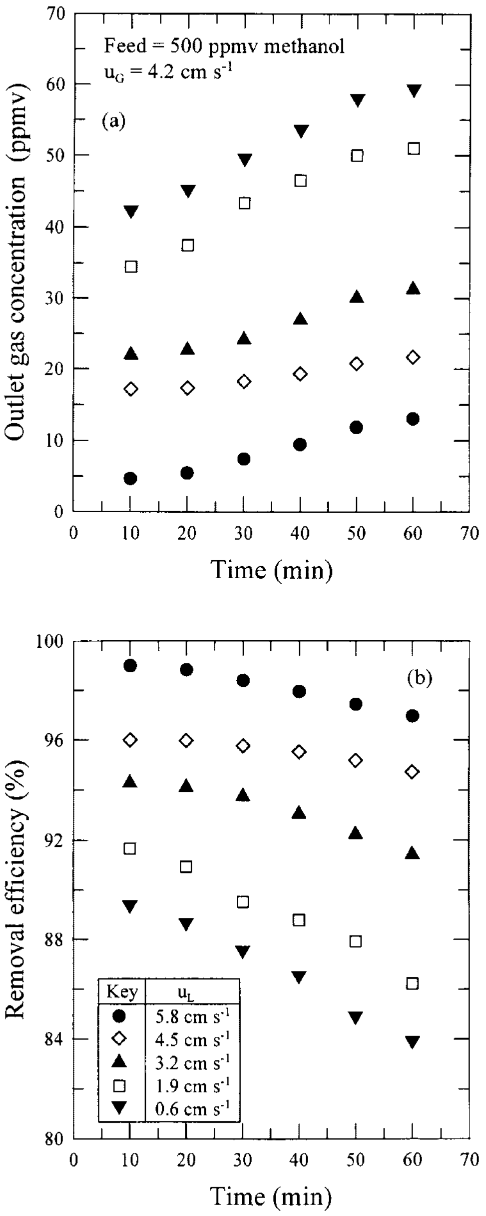


Figure 5. Time profiles of outlet gas concentration and removal efficiency of methanol (u_G = 4.2 cm/sec).

Determination of Mass Transfer Coefficients

At the pseudo steady state of absorption process (after 60-min operation), the overall mass transfer coefficient based on gas phase, K_G, can be calculated from Figs. 2–5 as follows (14):

$$K_G = \frac{Q_G}{n\pi r_i^2 RT} \ln\left(\frac{\text{inlet VOC concentration}}{\text{outlet VOC concentration}}\right) \tag{2}$$

where Q_G is the volumetric gas flow rate, n the number of the fibers, and r_i the inner radius of the fibers. The measured results of K_G are listed in Table 2.

Mass transfer coefficients of species in bulk phase can be estimated simply according to the geometry of the cell and the physical properties of the solutions (15,16). In the case of gas absorption in hollow fiber modules, the gas flow through the fibers in the modules will be laminar ($Re<400$). Within this region, the individual mass transfer coefficient in the tube side is dependent on the flow

Table 2. Mass Transfer Parameters Estimated or Measured in This Work

Parameters	Value	Reference
Diffusion coefficient (solute/medium)		
Acetone/silicone oil	$D_A = 2.2 \times 10^{-10}$ m^2/sec	(18)
Methanol/silicone oil	$D_A = 5.1 \times 10^{-10}$ m^2/sec	(18)
Acetone/N_2 gas	$D_A = 5.2 \times 10^{-5}$ m^2/sec	(18)
Methanol/N_2 gas	$D_A = 7.6 \times 10^{-5}$ m^2/sec	(18)
Individual mass transfer coefficient[a]		
Acetone/silicone oil	$k_L = 8.4 \times 10^{-7}$ m/sec	
Acetone/N_2 gas	$k_G = 8.3 \times 10^{-2}$ mol/(atm m^2 sec)	
Acetone/membrane (liquid-filled)	$k_M = 3.9 \times 10^{-7}$ m/sec	
Methanol/silicone oil	$k_L = 2.6 \times 10^{-6}$ m/sec	
Methanol/N_2 gas	$k_G = 2.7 \times 10^{-2}$ mol/(atm m^2 sec)	
Methanol/membrane (liquid-filled)	$k_M = 8.9 \times 10^{-7}$ m/sec	
Overall mass-transfer coefficient[a]		
Acetone system	$K_G = 1.6 \times 10^{-3}$ mol/(atm m^2 sec)	
Acetone system (measured)	$K_G = 1.5 \times 10^{-3}$ mol/(atm m^2 sec)	This work
Methanol system	$K_G = 6.3 \times 10^{-3}$ mol/(atm m^2 sec)	
Methanol system (measured)	$K_G = 5.9 \times 10^{-3}$ mol/(atm m^2 sec)	This work

[a] Conditions: $u_G = 2.1$ cm/sec, $u_L = 5.8$ cm/sec.

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velocity u_G according to Ref. (8)

$$\left(\frac{2\mathrm{r_i} k_G}{D_A}\right) = 1.62\left(\frac{4r_i^2 u_G}{D_A L}\right)^{1/3} \tag{3}$$

where D_A is the diffusivity of solute A in the bulk phase. Equation (3) is a limiting case of the more general Graetz solution, which predicts tube side mass transfer coefficients with reasonable accuracy (8).

For the shell side, the mass transfer correlation in the liquid phase is (17)

$$\left(\frac{k_L d_h}{D_A}\right) = 5.8(1-\varphi)\left(\frac{d_h}{L}\right)\left(\frac{d_h u_L}{\nu}\right)^{0.6}\left(\frac{\nu}{D_A}\right)^{1/3} \tag{4}$$

where φ is the fiber packing fraction within the module, which equals 0.16 in this case (17). The hydraulic diameter, d_h, is defined as

$$d_h = \frac{4\,(\text{volume of voids filled with fluid})}{\text{wetted surface area in the shell side}} = \frac{2\left(R_i^2 - nr_o^2\right)}{nr_o} \tag{5}$$

The value of ν for liquid silicone oil is taken as $5.21\times10^{-5}\,\mathrm{m^2/sec}$.

The diffusion through the membrane pores can be approximated by diffusion through a cylindrical wall. That is, the individual mass-transfer coefficient within the liquid-filled membrane can now be expressed as (8,17)

$$k_M = \frac{\varepsilon D_A}{\tau(r_o - r_i)} \tag{6}$$

where ϵ and τ are the porosity and tortuosity of the membrane. For membranes with a high porosity, a tortuosity of 2.0 is a good approximation (16).

The diffusivity of acetone and methanol in liquid silicone oil is estimated to be 2.2×10^{-10} and $5.1\times10^{-10}\,\mathrm{m^2/sec}$, respectively, by the Hayduk and Minhas equation (18). The value of ν for VOC-N_2 gas is taken as the pure N_2 gas, which is $1.55\times10^{-5}\,\mathrm{m^2/sec}$. The molar volumes of acetone and methanol at their normal boiling points are 73.5 and 40.5 $\mathrm{cm^3/mol}$, respectively (19). On the other hand, the diffusivities of acetone and methanol in N_2 gas are found to be 5.2×10^{-5} and $7.6\times10^{-5}\,\mathrm{m^2/sec}$ at 298K and 1 atm, respectively, using the known Chapman–Enskog equation (18). The estimated individual mass transfer coefficients, together with the diffusivities, are listed in Table 2. The values of k_L and k_G are typically given at specific gas and liquid velocities.

Comparison of the Measured and Estimated Results

The validity of these estimations can be checked from the measured "overall" mass-transfer coefficients. The concentration profiles that occur in and around a hollow fiber are shown in Fig. 6. It is noticed that the pores in the microporous membranes are liquid-filled. If the diffusion process can be described by Fick's equation, the rate of mass transfer (diffusion) step of solute A, J_A, within the gas-layer, membrane, and liquid-layer per unit length of the fibers is given as follows (20).

$$J_A = 2\pi r_i k_G (P_{AG} - P_{Ai}) \tag{7}$$

$$J_A = \pi d_{lm} k_M \left(C_{Ai}^{m} - C_{Ai}\right) \tag{8}$$

$$J_A = 2\pi r_o k_L (C_{Ai} - C_{AL}) \tag{9}$$

where d_{lm} is the logarithmic mean diameter of the fiber, defined by

$$d_{lm} = \frac{2(r_o - r_i)}{\ln\left(r_o/r_i\right)} \tag{10}$$

The concentration of species at the membrane–liquid interface is assumed to be identical because the interface is homogeneous due to high porosity of the membrane (13).

At pseudo steady state (after 60-min operation), the overall mass transfer resistance can be expressed by the summation of each resistance (8,20).

$$\frac{1}{2r_o K_G} = \frac{1}{2r_i k_G} + \frac{1}{d_{lm} k_M H_A} + \frac{1}{2r_o k_L H_A} \tag{11}$$

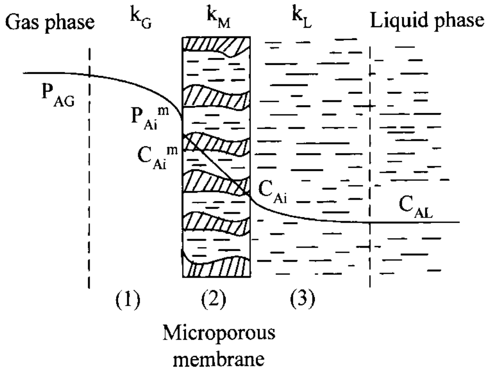

Figure 6. Concentration profiles of VOCs in a hollow fiber absorber.

where H_A is Henry's law constant of solute between N_2 and silicone oil ($H_A = C_A/P_A$). It is 2.04×10^4 and 1.06×10^5 mol/(atm m^3) for acetone and methanol, respectively at 298K and 1 atm (21). Here, the overall mass transfer coefficient K_G is defined by

$$J_A = 2\pi r_o k_G (P_{AG} - C_{AL}/H_A) \tag{12}$$

Table 2 compares the estimated (based on Eq. 11) and measured K_G values. It is found that the agreement is reasonably good. The slightly smaller K_G value obtained from experiments is likely due to the incomplete utilization of the fiber area in hollow fiber modules, e.g., by-pass by the shell-side fluid (22).

Actually, the three terms on the left-hand side of Eq. (11) in sequence represent the resistances of gas-layer diffusion, membrane diffusion, and liquid-layer diffusion. Under the conditions shown in Table 2, it is justified that the present process was controlled by membrane diffusion. The fractional resistance of membrane diffusion is 0.89 and 0.71 for acetone and methanol systems, respectively.

Finally, the height of a transfer unit (HTU) in hollow fiber absorbers is calculated as follows (23,24).

$$\mathrm{HTU} = \frac{n r_i^2 u_G}{\alpha R_i^2 (K_G RT)} \tag{13}$$

where α equals $(4nr_i/R_i^2)$.

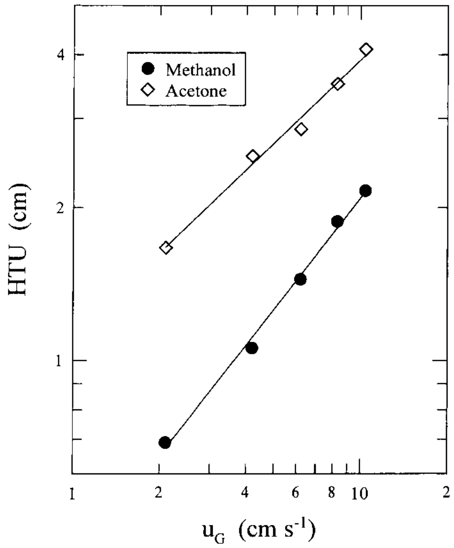

Figure 7. Effect of gas velocity on the height of a transfer unit.

Because u_G plays a large role on mass transfer rate, the effect of u_G on HTU is studied (Fig. 7). The magnitude of HTU reveals the same order in nature as that obtained in the membrane extraction of chloroform from wastewater in hollow fiber modules (23). Under the conditions examined, it is evident that HTU varies about 0.5–0.7 power of u_G, which is larger than those found in the range 0.3–0.4 for gas absorption in packed towers using Raschig rings (24). Furthermore, the HTU values obtained here are around one order of magnitude smaller than those reported in conventional packed columns (24). This confirms the promising feature of very high volumetric mass transfer coefficients in such hollow fiber absorbers.

CONCLUSIONS

Absorption removal of acetone and methanol from N_2 streams with liquid silicone oil in microporous hollow fiber modules has been studied. The VOC-N_2 streams flowed in the tube side and the liquid silicone oil was cyclically pumped across the shell side of the module. The removal efficiency was higher at a lower gas velocity u_G and a larger liquid velocity u_L, and reached above 99% under optimal conditions. Compared to the estimated overall mass-transfer coefficients, the slightly smaller measured ones were likely due to incomplete utilization of the fiber area, e.g., by-passing of shell-side fluid. Comparing the fractional resistance of each mass transfer step, it was shown that the present process was controlled by membrane diffusion, e.g., the fractional resistance of membrane diffusion was 0.89 and 0.71 for acetone and methanol systems, respectively, at $u_G = 2.1$ cm/sec, $u_L = 5.8$ cm/sec. Under the conditions investigated, HTU varied from 0.5 to 0.7 power of u_G. This work provided quantitative verification of mass transfer in such hollow fiber absorbers.

NOTATION

C_A	concentration of solute A in the liquid phase (mol/m^3)
D_A	diffusivity of solute A (m^2/sec)
d_h	hydraulic diameter (m)
H_A	Henry's law constant of solute A (mol/(atm m^3))
J_A	mass transfer rate of solute A per unit length of the fiber (mol/(m sec))
k_G	individual mass-transfer coefficient in the gas phase (mol/(atm m^2 sec))
K_G	overall mass-transfer coefficient based on gas phase (mol/(atm m^2 sec))
k_L	individual mass-transfer coefficient in liquid phase (m/sec)
k_M	individual mass-transfer coefficient within the membrane (m/sec)
L	effective length of the fiber (m)
N	number of fibers

P_A	partial pressure of species A in the gas phase (atm)
R_i	inner radii of the shell (m)
r_i, r_o	inner and outer radii of the fiber, respectively (m)
t	time (sec)
u_G, u_L	linear velocities of gas and liquid phases, respectively (cm/sec)

Greek letters

ν	kinematic viscosity of the medium (m^2/sec)
ϵ	porosity of the membrane support (−)
τ	tortuosity of the membrane support (−)

Superscript

m	membrane

Subscript

i	gas- or liquid-membrane interface

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