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REMOVAL OF ACETIC ACID WITH AMINE EXTRACTANTS FROM FERMENTATION BROTH USING HYDROPHOBIC HOLLOW-FIBER MEMBRANE CONTACTOR

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ABSTRACT

The organic solvent with a high partition coefficient toward acid extraction must be selected to increase the efficiency of extraction of acetic acid in aqueous phase. Amine extractant plays a major role in the reactive extraction process at the interface between aqueous phase/organic phases. Therefore, it has been investigated that an effect of addition of various amines such as di-ethylamine (DEA), tri-ethylamine (TEA), and tri-n-octylamine (TOA) to organic phase could increase the extraction performance due to in-

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creasing partition coefficient. The effect of temperature with TOA content of 0 to 40 wt-% on extraction process of acetic acid was observed. The removal efficiency of acetic acid in aqueous phase increased with temperature. However, the maximum value of extraction performance was different according to experimental conditions, such as the choice of organic solvent, temperature, and TOA content. In the case of methyl isobutyl ketone (MIBK)/TOA system and chloroform/TOA used in organic phases, the maximum value of removal of acetic acid showed 64 wt-% and 53 wt-% at 40 wt-% TOA content and 25°C, respectively. In 2-octanol/TOA system, the maximum removal efficiency of acetic acid was 67 wt-% at 30 wt-% TOA and 50°C. On the basis of the energy efficiency, the optimal condition of organic phase for the extraction of acetic acid in aqueous phase was determined to be 40 wt-% MIBK/TOA at 25°C. Using the membrane extraction process with a hollow-fiber membrane contactor, the overall mass transfer coefficient was calculated by Wilson-plot method under the assumption of constant partition coefficient. These values show a significant increase in mass transfer rate with increasing the TOA content. Additionally, an improvement of extraction performance was established by multiplying the diffusion coefficient and partition coefficient of solutes.

Key Words: Liquid-Liquid extraction; Partition coefficient; Water/acetic acid separation; Porous hollow-fiber membrane; Membrane contactor.

INTRODUCTION

Increasing attention has been paid to the development of efficient methods for the separation and purification of low-molecular acids such as acetic acid, propionic acid, and lactic acid from biofermentation process bath. The use of extractants in liquid–liquid extraction has been considered a promising method for the effective extraction of low-molecular acids.

These liquid–liquid extractions can be performed using conventional equipment such as mixer/settlers and centrifugal equipment or by membrane extraction. Membrane extraction process is carried out using microporous membranes to immobilize the aqueous/organic interface within the pore structure. The solute is transported from the feed (aqueous) phase to the extractant (organic) phase, through membrane, without phase dispersion and avoiding the formation of stable emulsions that may hinder the process. The supporting membrane may also form a selective barrier to other medium constituents, thus preventing the contamination of the extractant phase.

Membrane extraction process has been applied to a variety of systems including extraction of fermentation products (1–4), pollutants (5,6), pharmaceutical products (7), metals (8,9), and in the extraction of enzymes with reversed micelles (10). The use of the hollow-fiber membrane contactor can overcome many limitations of conventional liquid extraction such as the requirement of density difference, flooding resrictions, and loading requirements, and simultaneously provide high mass transfer rate per unit volume, as hollow-fiber modules can be packed into very high surface area per unit volume. This large surface per volume, which is typically 100 times larger than in conventional equipment (11), compensates for the potential decrease in the overall mass transfer coefficient (K_a) due to an additional membrane resistance (k_m) and the usually poor hydrodynamics in the shell side of the contactor. In the liquid membrane system, two miscible liquid phases are effectively separated by immiscible phase if an adequate extractant is used. From a practical point of view, it may be important not only to extract acetic acid but also to concentrate it. For this reason, simultaneous extraction and stripping (reextraction) of acetic acid is accomplished using two hollow-fiber modules. As for the method to separate extracted acetic acid in organic phase, two kinds of reextraction process were known. One is to reextract acetic acid into the aqueous stripping phase, which is dissolved with inorganic salt such as LiCl, KCl, and $CaCl_2$ (14). The other method is to add hydrated lime to extracted acetic acid phase. One could remove the precipitant sunken down to the bottom, where the precipitant is the complex of calcium cation and acetate anion (15,16).

The objective of this study is to investigate the effect of the addition of various amines such as di-ethylamine, tri-ethylamine, and tri-n-octylamine to organic phase on the extraction performance and to study the effect of temperature with tri-n-octylamine content of 0 to 40 wt-% on the extraction process of acetic acid. Additionally, using a membrane extraction process with a hollow-fiber membrane contactor, the overall mass transfer coefficient was calculated by Wilson-plot method under the assumption of constant partition coefficient.

EXPERIMENTAL

Materials

Reagent grade glacial acetic acid produced by Aldrich (Milwankee, WI) was used as received. Then 2 wt-% aqueous solutions of acetic acid in first-distilled water, simulating the products from biofermentation, were prepared. Chloroform (Aldrich), methyl isobutyl ketone (MIBK, Yakuri Co., Osaka, Japan), and 2-octanol (Junsei Co., Tokyo, Japan) utilized as organic phases were used as received. They were immiscible with aqueous solution. To evaluate the efficiency of acid-extraction as a function of partition coefficient, the extractants such as di-ethylamine (DEA), tri-ethylamine (TEA), and tri-n-octylamine (TOA) produced by Aldrich were added in organic phase.

Hollow-fiber modules with membranes from Sambo Glove Co. (Ansan, Korea) were used in this study. Each module has 2.8 cm of internal diameter, contains 800 hydrophobic poly(propylene) hollow fibers, 15 cm in length, with a nominal internal diameter of 260 μm, nominal thickness of 30 μm, and effective pore size of 0.04 μm. Each module provides an effective membrane area of 498.8 cm^2.

Liquid–Liquid Extraction and Partition Coefficient (*H*)

Liquid–liquid extraction at equilibrium state was determined by contacting the known volume and concentration of both organic and aqueous solutions, the latter being 2 wt-% acetic acid. The total volume of the sample, adding up to 50 cm^3, was placed in a capped flask and magnetically mixed and thermostated in a water bath at 25°C. Once the two phases have settled, the concentration of acetic acid was determined in aqueous phase.

The partition coefficient (*H*) of the solute (acid) between the organic and aqueous phase assuming to be constant during the extraction process is usually described by the equilibrium conditions at the interface and defined as in following Equation (1).

$$H = \frac{M_{org}}{M_{aq}} \ (at\ equilibrium\ state) \tag{1}$$

where M_{org} and M_{aq} is the molar concentration of acid in the organic phase and in the aqueous phase, respectively. The partition coefficient is the efficiency of the transition of solute in aqueous solution into organic solution. Therefore, the high partition coefficient implied a good extraction property.

Membrane Contactor: Apparatus and Procedure

The aqueous phase of feed side was pumped through the lumen of the fibers to circulate from the respective reservoir and back into the reservoir. The organic phase of extraction side circulates cocurrently in the shell side of the module.

Mechanically driven gear pump (FMI, RP-D-2 pump) was used. To measure both aqueous and organic flow rates during the experiments, flowmeters were used. The pressure of the aqueous phase was maintained higher than the pressure of the organic phase in order to stabilize the interface within the membrane, as the

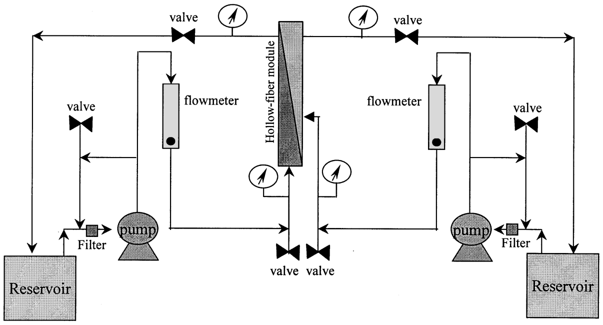

Figure 1. Experimental setup for membrane extraction. The hollow fiber module was shown in the center of the figure. It is attached to the two liquid reservoirs whose concentrations are measured as a function of time.

hydrophobic fibers are preferentially wetted by the organic phase. The pressure was adjusted using the four valves at the inlet and outlet of the modules, preventing the emulsion formation.

Jacketed reservoirs were used to maintain a constant temperature (25°C). The solution was magnetically stirred to prepare the homogeneous solutions. The instrument was designed to measure the acid concentration in the feed reservoir as a function of time. The experimental setup is schematically shown in Fig. 1. The experiments were carried out with cocurrent flow of aqueous and organic solution at a feed flow rate of 8.2 cm^3/sec.

Analytical Method

To determine the concentration of acid transferred in the liquid–liquid extraction equilibrium and membrane contactor experiments, the concentration of the acid in aqueous phase was determined by conductivity measurement. The conductivity meter (Orion Instrument, model 162) used was calibrated with standard solutions. The conductivity of pure water (first-distilled water, *p*Ka: 15.7) was about 0 μS/cm, whereas that of the 2 wt-% acetic acid and propionic acid in aqueous phase indicated 2.77×10^3 μS/cm and 2.34×10^3 μS/cm, respectively. This result was in accordance with their respective trend of *p*Ka values. The *p*Ka value of acetic acid and propionic acid is 2.75 and 2.89, respectively. Therefore, as *p*Ka value increases, the conductivity decreases. The acid concentration of organic phase was determined by two-phase titration (1 cm^3 of the organic solvent mixed with 7 cm^3 of ethanol and 3 cm^3 of distilled water) with NaOH solution, using phenolphthalein as an indicator (12).

RESULTS AND DISCUSSION

Effect of Organic Solvent on Partition Coefficient

The organic solvent having a high partition coefficient must be selected to increase the efficiency of extraction of low-molecular-weight acid in aqueous phase. Experiments were carried out using three solvents such as methyl isobutyl ketone (MIBK), chloroform, and 2-octanol. These solvents are immiscible with an aqueous phase.

Then 50 g of 2 wt-% aqueous acetic acid solutions were prepared by adding 16.6 mmol acetic acid to 367 mmol first-distilled water. Each 50 g of organic phases, extractant phase, containing various tri-octylamine (TOA) content of 0, 10, 20, 30, and 40 wt-%, respectively, were contacted with aqueous acid solution prepared above. The mixed solutions, which were observed at the interface between aqueous and organic phases formed in the middle of the mixed solution without emulsion, were kept to reach an equilibrium state within 30 min. Then, the concentration of acid both in aqueous and in organic phases were simultaneously measured. Partition coefficient calculated according to Equation (1) is presented in Fig. 2. It could be seen from this figure that the partition coefficients of methyl isobutyl ketone (MIBK) and chloroform increased with TOA content to attain a maximum at 40 wt-% TOA. Thereafter it did not increase despite of further increase of TOA content. However, when 2-octanol was used as an organic phase,

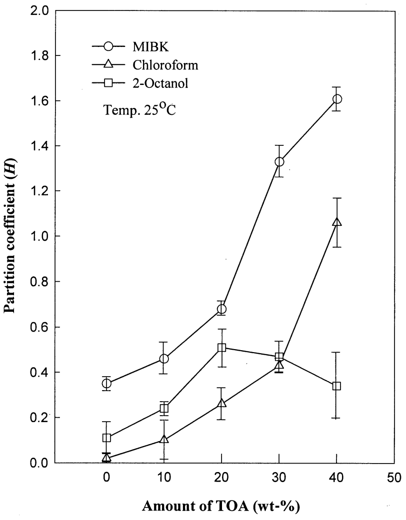

Figure 2. The effects of various organic phases on the partition coefficient; solid line denotes a regression.

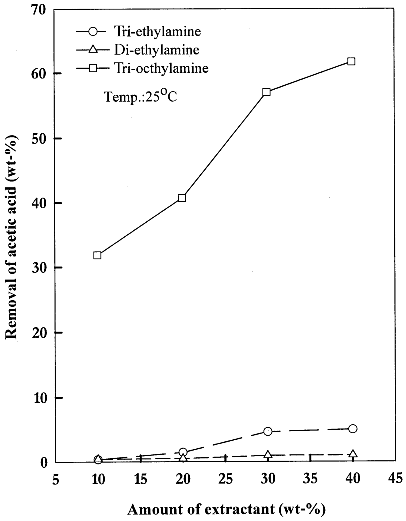

Figure 3. Removal of acetic acid with various amines.

partition coefficient reached a maximum at 20 wt-% TOA and then slightly decreased thereafter. Figure 2 also showed that MIBK has excellent acid-extraction properties compared with other two solvents.

Effect of Amine Extractant on the Removal of Acetic Acid

The selection of extractant is very important at the reactive extraction process. It has been investigated that the addition of amine extractant to organic phase could increase the extraction performance due to the increase of the partition coefficient (13).

In general, tertiary amine and quaternary ammonium salt were extensively utilized. In this study, organic phase (MIBK) containing various tertiary amines such DEA, TEA, and TOA was used to investigate the effect of the degree of alkylation of amine and that of alkyl group on extraction performance. The experimental results for the acetic acid concentration removed in aqueous phase vs. the amount of tertiary amine is presented in Fig. 3. As can be noticed, amine exhibits a better extraction performance than secondary amine. Also, as the number of carbon in alkyl group of tertiary amine increases, the extraction performance of acetic acid in aqueous phase is enhanced. Because hydrocarbon acts as an electron donor, it has generally been well known that the increase of the number of either carbon in alkyl group or alkyl group itself makes a strong non-

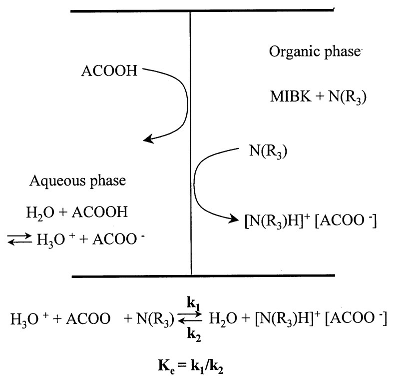

Figure 4. The schematics of reactive extraction at interface between aqueous and organic phase. Ke is the equilibrium constant of the interfacial reaction.

pair electron charge density of nitrogen atom in amine. Therefore, at the interface of an aqueous phase/organic phase, the complex between tertiary amine and acetic acid was easily formed, compared with that of other amines, to transfer toward organic phase.

A schematic of extraction mechanism was presented in Fig. 4. Under the steady-state condition, the mass transfer for a facilitation factor with reaction in the interface between the two phases was derived by the reaction rate expression for one-dimensional, steady-state diffusion with reaction in a homogeneous medium. Ke $(= k_1/k_2)$, is the rate constant for the formation of the acid-base complex $[N(R_3)H^+][ACOO^-]$.

Removal of Acetic Acid with Temperature

The effect of temperature with tri-n-octylamine content of 0–40 wt-% on the removal efficiency of acetic acid is shown in Figs. 5–7. Examining these figures, the removal efficiency of acetic acid in aqueous phase generally increased with temperature and the content of TOA. TOA in organic phase plays a role as a carrier, causing acetic acid to facilitate transfer to organic phase. Therefore, the removal of acetic acid increased with the content of amine. However, the maximum value of extraction performance was different according to the experimental conditions such as organic solvent, temperature, and TOA content. In the case of MIBK/TOA system and chloroform/TOA used as organic phases, a maximum value for the removal of acetic acid was 64 wt-% and 53 wt-%, respectively, under the condition of 40 wt-% TOA content at 25°C. However, when 2-octanol/TOA system was used as an organic phase (see Fig. 7), the extraction performance

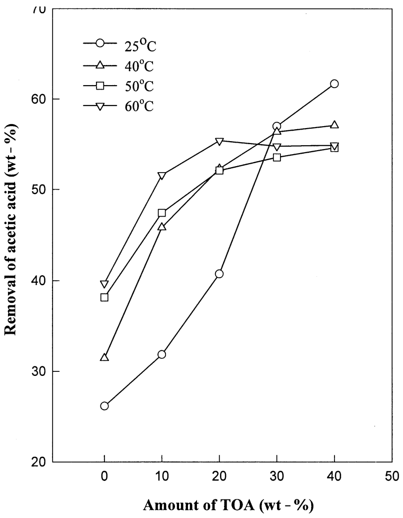

Figure 5. The effects of temperature on removal of acetic acid in aqueous phase using MIBK/TOA as organic extraction phase.

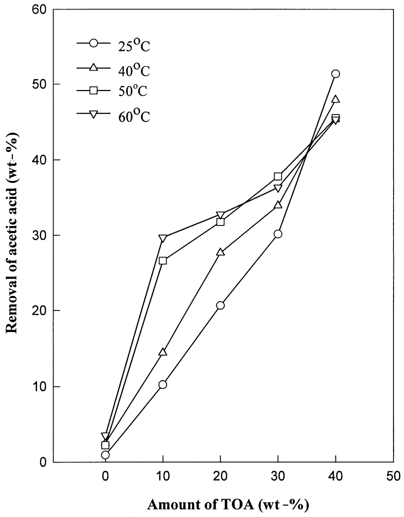

Figure 6. The effects of temperature on removal of acetic acid in aqueous phase using Chloroform/TOA as organic extraction phase.

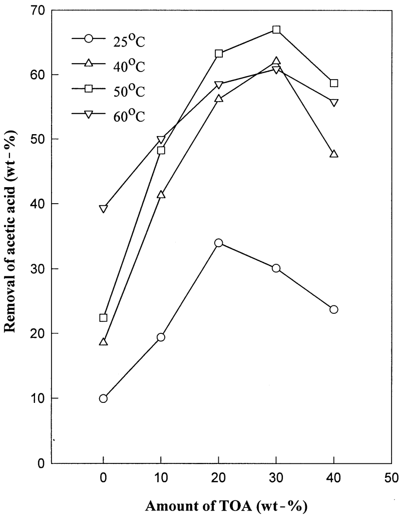

Figure 7. The effects of temperature on removal of acetic acid in aqueous phase using 2-octanol/TOA as organic extraction phase.

had a convex trend with TOA content in the temperature range of 25–60°C. Also a maximum point moved into 20 to 30 wt-% TOA content. Notice that the maximum removal efficiency of acetic acid is 67 wt-% under condition of 30 wt-% TOA at 50°C. Based on the energy efficiency, it may be deduced that the optimum condition of organic phase for the extraction of acetic acid in aqueous phase was 40 wt-% TOA/MIBK at 25°C. This condition will be later used to calculate the mass-transfer rate.

Calculation of the Overall Mass-Transfer Coefficient

Considering the resistance in series model, the reciprocal of the overall mass-transfer coefficient is the total resistance (R_a) to mass transfer and can be described as the sum of the mass-transfer resistance inside fiber (aqueous feed side, R_{aq}), across the fiber wall (membrane resistance, R_m), and outside the fibers (organic extractant side, R_{org}).

$$R_a = R_{aq} + R_m + R_{org} \tag{2}$$

Equation (2) can be converted into the form of

$$\frac{1}{K_a} = \frac{1}{k_{aq}} + \frac{d_i}{H \cdot k_m \cdot d_{lm}} + \frac{d_i}{H \cdot k_{org} \cdot d_e} \tag{3}$$

where K_a represents the overall mass-transfer coefficient, k_{aq}, k_m, k_{org} are the local mass-transfer coefficients on the fiber inside, membrane, and fiber shell side, respectively. H is the solute partition coefficient between the two contacting phases, and d_i, d_e, d_{lm} are the internal, external, and logarithmic length of fiber, respectively.

Using the membrane extraction process with a hollow-fiber membrane contactor, the overall mass-transfer coefficient was calculated by Wilson-plot method under the assumption of constant partition coefficient. In the case of nonconstant H, another approach for mass balance should be considered over the extraction process (12). The concentration of the acid in the aqueous solution was measured as a function of time to determine the overall mass-transfer coefficient (K_a) defined by

$$J_{\mathrm{sol}} = Q_{sol}/A_m\,(C_{(a)} - C^*_{(a)}) = K_a\,(C_{(a)} - C^*_{(a)}) \tag{4}$$

where J_{sol} was the solute flux, A_m was effective membrane area, $C_{(a)}$ was the concentration in aqueous solution at time (t), and $C^*_{(a)}$ was the equilibrium acid concentration in the aqueous phase. An exponential dependency of the concentration of the solute in aqueous phase with time can be established. The main equations for the cocurrent flow are

$$C_{(a)} = \frac{V\cdot C_{(a)0}}{1+V} + \frac{C_{(a)0}}{1+V}\,e^{-At} \tag{5}$$

$$\begin{gathered} A = \frac{Q_{(a)}}{V_{(a)}}\cdot\frac{1+V}{1+Q}\cdot\left\{1 - \exp\left[-\frac{A_m\cdot K_a}{Q_a}\,(1+Q)\right]\right\} \\ V = \frac{V_a}{V_0 H},\; Q = \frac{Q_a}{Q_0\,H} \end{gathered} \tag{6}$$

where $C_{(a)0}$ is the initial acid concentration in the aqueous phase, $Q_{(a)}$ and Q_0, the aqueous feed and organic feed flowrates, respectively. V_a and V_0 are the volume of the aqueous and organic phases, respectively. A_m is the effective membrane area provided by the module, and H is the partition coefficient determined at the equilibrium state. According to Eq. (5), the concentration of the acid in the aqueous phase varies with time in the form of an exponential equation.

$$C_{(a)} = \alpha + \beta\,\exp(-\gamma t) \tag{7}$$

where α and β depend only on known parameters, and their values can be determined in advance. By fitting the experimental data to this exponential equation, optimal values for γ are obtained. The experimental results obtained for acetic acid concentration in feed phase ($C_{(a)}$) vs. time for one of the experiments is presented in Fig. 8. K_a is determined by substituting the value of the other parameters in Eq. (6).

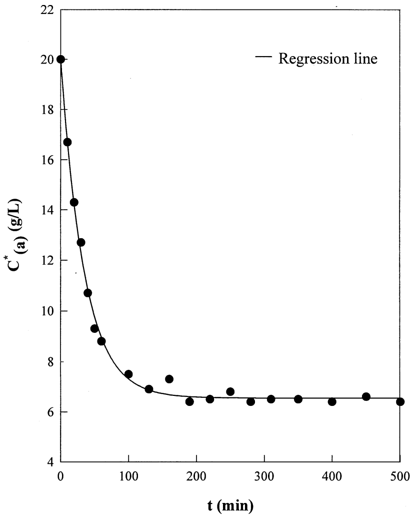

Figure 8. Acetic acid concentration as a function of time. This experiment was carried out under condition of constant aqueous velocity of 8.2 cm^3/sec at 25°C.

$$K_a = \frac{-Q_{(a)}}{(1 + Q_{(a)})\cdot A_m} \ln\left(1 - \gamma\left(\frac{V_a}{Q_a}\right)\left(\frac{1+Q}{1+V}\right)\right) \qquad (8)$$

Table 1 shows the overall mass-transfer coefficients calculated for the extraction process. These values indicate a significant increase in the mass-transfer coefficient with increasing TOA content. Coelhoso et al. (14) reported that in-

Table 1. Calculation of the Overall Mass Transfer Coefficient for the Extraction Process; Effect of TOA Concentration in MIBK; This Experiment Was Carried Out Under Operating Condition of Co-current Flow at 25°C

TOA Content (wt-%)	H	K_a (10^{-6}m/sec)
0	0.35	15.1
10	0.46	18.5
20	0.68	21.3
30	1.33	24.7
40	1.61	26.4

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creasing extractant concentration (Aliquat 336) improved the mass-transfer process because the use of a higher amine concentration had an effect on increasing partition coefficient, whereas the diffusion coefficient of solute decreased with increasing amine concentration due to the increase of the viscosity in organic phase. Consequently, the overall mass-transfer rate decreased with increasing amine extractant, which was regarded as a combined effect of the partition coefficient and diffusion coefficient of solute. Therefore, the membrane resistance is identical to the limiting step on the mass-transfer process. Assuming that the limiting step is the membrane resistance, the overall mass-transfer coefficient may be expressed as Eq. (9).

$$K_a = const.\ H \cdot D_{org} \tag{9}$$

Equation (9) indicates that the overall mass-transfer coefficient is proportional to the product of the partition coefficient and the diffusion coefficient of solute. In the TOA/MIBK organic system, the use of higher TOA content in organic phase made a contribution toward increasing partition coefficient and diffusion coefficient of solute at the same time. This means that viscosity does not considerably increase despite of increasing TOA content. Therefore, it was concluded that the high extraction performance of the present system was caused by a significant increase in diffusion coefficient of a solute.

CONCLUSIONS

The extraction of acetic acid through the interface between aqueous and organic phases was highly dependent on the partition coefficient of acid-extractants. It was observed that the partition coefficient of MIBK used as an organic phase increased with TOA content to attain a maximum value, 1.61, at 40 wt-% TOA. The organic solvent with high partition coefficient increased the efficiency of extraction of acetic acid in aqueous phase. The addition of various tertiary amines such as DEA, TEA, and TOA to organic phase (MIBK) increased the extraction performance in the order of TOA > TEA > DEA. The removal efficiency of acetic acid in an aqueous phase increased with temperature in the range of 25–60°C. In the case of MIBK/TOA system and chloroform/TOA used as organic phases, the maximum removal efficiency of acetic acid showed 64 wt-% and 53 wt-%, respectively, under the condition of 40 wt-% TOA content at 25°C. In 2-octanol/TOA system, note that the maximum removal efficiency of acetic acid is 67 wt-% at 30 wt-% TOA and 50°C.

Using a membrane extraction process with a hollow-fiber membrane contactor, the overall mass-transfer coefficient (K_a) was calculated with TOA content in MIBK by Wilson-plot method under the assumption of constant partition coefficient. When pure MIBK without any amine-extractant was used as an organic phase, the overall mass-transfer coefficient was calculated to be 15.1×10^{-6} m/sec. However, the overall mass-transfer coefficient was significantly increased with increasing amine-extractant (TOA) content in organic phase (MIBK). In particular, K_a became 26.4×10^{-6} m/sec when 40 wt-% TOA was incorporated in MIBK. In conclusion, the extraction performance was believed to be enhanced by diffusion coefficient.

NOMENCLATURE

J_{sol}	the solute flux ($M/L^2 \cdot T$)
$C_{(a)}$	the concentration in the aqueous solution at the time (M/L^3)
$C^*_{(a)}$	the equilibrium acid concentration at the time in the aqueous solution (M/L^3)
A_m	membrane area (L^2)
K_a	the overall mass-transfer coefficient (L/T)
k_{aq}	the mass-transfer coefficient in the aqueous solution (L/T)
k_{org}	the mass-transfer coefficient in the organic solution (L/T)
k_m	the mass-transfer coefficient in membrane (L/T)
d_i	the internal diameter of hollow fiber (L)
d_e	the external diameter of hollow fiber (L)
d_{lm}	the logarithmic mean diameter of hollow fiber (L)
δ_m	the effective membrane thickness (L)
R_a	total resistance (T/L)
R_{aq}	resistance at the aqueous solution (T/L)
R_m	membrane resistance
R_{org}	resistance organic solution (T/L)
L	length of hollow-fiber membrane (L)
H	partition coefficient
M_{org}	the molar concentration of acid in the organic phase
M_{aq}	the molar concentration of acid in the aqueous phase
D_{org}	the diffusion coefficient of solute in organic phase (L^2/T)

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