

[Regular Paper]

Solvent Extraction of Coal Tar Absorption Oil with Continuous Countercurrent Spray Column

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Solvent extraction of absorption oil by aqueous solution of methanol was evaluated using a continuous countercurrent spray column. The structure of the spray column is simple so that the mass transfer phenomenon in the column can be easily examined. The measured liquid-liquid equilibria between the absorption oil and aqueous solution of methanol phases required for calculation of mass transfer coefficients were found to agree with previous values. The operability and separability of extraction using the spray column were then studied. The density of the dispersed raffinate phase was sufficiently larger than that of the continuous extract phase to carry out countercurrent operation without entrainment of dispersed phase droplets into the continuous phase or flooding in the range investigated in this study. Mass transfers of components of interest could be detected in the bench scale spray column with effective height of about 0.5 m. The nitrogen heterocyclic compounds were extracted preferentially to other compounds, such as homocyclic compounds, from the absorption oil, and the separation of these compounds was governed by the liquid-liquid equilibrium. The highest yield and separation selectivity of nitrogen compounds with this column were about 0.4 and 30, respectively. Since the overall mass transfer coefficients increased with the flow rate of the continuous extract phase, some mass transfer resistance occurred in the continuous extract phase.

Keywords

Coal tar, Absorption oil, Solvent extraction, Continuous countercurrent operation, Spray column, Mass transfer rate

1. Introduction

Coal tar absorption oil, one of the coal tar distillates, contains various kinds of industrially useful compounds, such as quinolines, indoles, and naphthalenes. Isolation of these compounds from absorption oil involves separation by chemical extraction with acid and base into the basic, acidic, and neutral fractions in the first step of the series of separation operations. This chemical extraction is complicated and costly, so that a simpler and more economic method is desirable.

Conventional extraction with aqueous methanolic solution without chemical reaction is a promising and simple methods to separate absorption oil^{1)~10)}. The authors have previously studied the extraction characteristics^{4),6)}, solvent components^{4),6),10)}, additive to the solvent⁵⁾, separation of extracted and solvent components by secondary extraction⁷⁾, that by distillation⁹⁾, and so on⁶⁾. However, these studies were based only on liquid-liquid equilibrium relationships obtained from

batch runs, with no investigation of the mass transfer rate or continuous steady state operation, which is important in industrial applications.

The present study investigated the solvent extraction of absorption oil by aqueous solution of methanol in a continuous countercurrent spray column. The structure of the spray column is simple so that the mass transfer phenomenon in the column can be easily examined. The liquid-liquid equilibria between the absorption oil and aqueous solution of methanol phases were measured and compared with previous findings. The operability and separability of the extraction using spray column were then studied in terms of liquid densities, yield, separation selectivity, mass transfer coefficient, and so on.

2. Experimental

2.1. Extraction Equilibrium

Extraction equilibria between the absorption oil and the aqueous methanolic solution were measured as described previously^{4)~7),9)}. These equilibrium relationships together with those established previously⁷⁾ were

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Table 1 Material Systems and Conditions for Extraction Run with Spray Column

Feed (dispersed phase)	absorption oil
Superficial mass flow rate at inlet, R_f [$\text{kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$]	400-5000
Solvent (continuous phase)	aqueous solution of methanol
Mass fraction of water, $y_{w,b}$	0.30 (± 0.03), 0.47 (± 0.03)
Superficial mass flow rate at inlet, E_b [$\text{kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$]	900 (± 200), 2400 (± 200)
Operation temperature	293-299 (room temperature)

used to calculate the mass transfer coefficients.

2.2. Continuous Extraction

The material system and principal conditions for the continuous extraction are summarized in **Table 1**. The feed was coal tar absorption oil and the solvent was aqueous methanolic solution^{4)~7),9)}. The raffinate phases generated in some runs were reused as feeds for other runs. Superficial mass flow rates of feed, R_f , of solvent, E_b , and the mass fraction of water in the solvent, $y_{w,b}$, were varied as shown in **Table 1**.

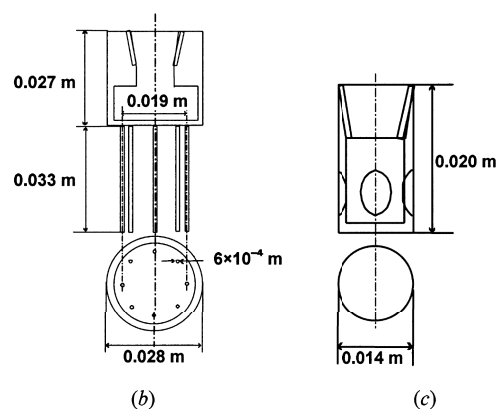
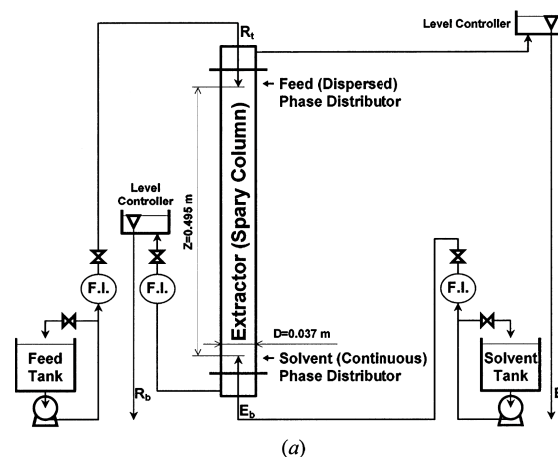
A schematic diagram of the apparatus used in continuous extraction is shown in **Fig. 1 (a)**. The spray column, the main extractor, was made of Pyrex glass so that the behavior in the column could be observed. **Figures 1 (b)** and **(c)** show details of the dispersed and continuous phase distributors, respectively. The dispersed phase distributor had 8 needle nozzles. Nozzles were arranged on a side face of the continuous phase distributor to prevent dispersed drops entering into the nozzles.

Before starting the run, adequate amounts of the feed absorption oil and aqueous methanolic solution were prepared in the respective tanks.

First, the solvent, the continuous phase, was provided into the column from the bottom. After the column was filled with the continuous phase, the feed was dispersed in the continuous phase from the top to begin the continuous operation. The feed and solvent phases were thus brought into countercurrent contact. The inlet flow rates were monitored and kept constant by flow indicators, which were calibrated beforehand. The accumulated layer of dispersed phase at the bottom was maintained at a constant level by the valve at the exit of the dispersed phase. The outlet flow rates were determined by weighing the mass of liquid phases flowing out during a specific time period.

The densities of the feed, solvent, raffinate, and extract phases were measured with a pycnometer. These phases were also analyzed with a gas chromatograph and a Karl Fischer titrator as described previously^{7),9)} to determine their compositions.

The continuous operation was carried on until the



(a) schematic flow diagram, (b) dispersed phase distributor, (c) continuous phase distributor.

Fig. 1 Apparatus for Extraction Run with Spray Column

system attained a steady state, in which the changes in the flow rates and compositions of raffinate and extract phases at the exit were negligible over time.

At the end of the run, all valves at the inlets and exit of liquid phases were closed at the same time. The increase in the volume of the accumulated layer of dispersed phase was measured to obtain the holdup of the dispersed phase in the column.

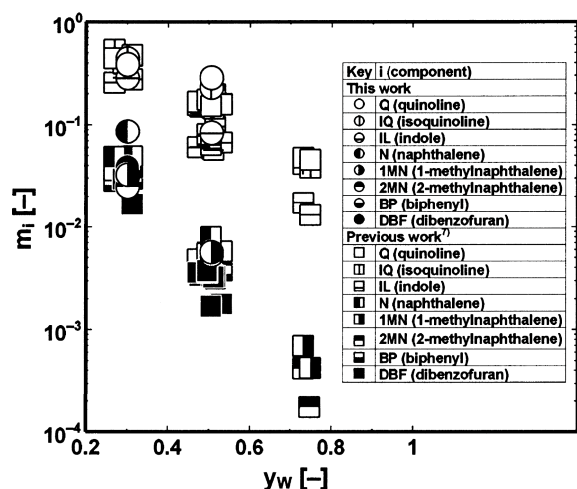
3. Results and Discussion

3.1. Composition of Feed Absorption Oil

The compositions of the feed absorption oils are shown in **Table 2**. Quinoline, isoquinoline, indole as nitrogen heterocyclic compounds, naphthalene, 1-methylnaphthalene, 2-methylnaphthalene, biphenyl, and dibenzofuran were the compounds in the absorption oil in this study. In the extraction runs, some differences were detected in the feed composition, and the solvent components, methanol and water, were detected in the feed, because of the reuse of raffinate absorption oil phase as mentioned above. The mass fractions were 0.1 to 0.2 of nitrogen heterocyclic compounds and 0.4

Table 2 Composition of Feed Absorption Oil: Mass Fractions of Studied Components

Nitrogen heterocyclic compounds	[—]	0.1-0.2
Quinoline, $x_{Q,t}$	[—]	0.05-0.09
Isoquinoline, $x_{IQ,t}$	[—]	0.02-0.03
Indole, $x_{IL,t}$	[—]	0.04-0.06
Homocyclic hydrocarbons <i>etc.</i>	[—]	0.4-0.6
Naphthalene, $x_{N,t}$	[—]	0.02-0.03
1-Methylnaphthalene, $x_{1MN,t}$	[—]	0.09-0.1
2-Methylnaphthalene, $x_{2MN,t}$	[—]	0.2-0.3
Biphenyl, $x_{BP,t}$	[—]	0.06-0.09
Dibenzofuran, $x_{DBF,t}$	[—]	0.06-0.1
Solvent components		
Methanol, $x_{M,t}$	[—]	0-0.04
Water, $x_{W,t}$	[—]	0-0.004

Fig. 2 Effects of Mass Fraction of Water in Extract, y_w , on Distribution Coefficients, m_i

to 0.6 of other components such as homocyclic compounds in the absorption oil.

3.2. Extraction Equilibrium

The distribution coefficient, m_i , was defined by the following equation with the mass fractions in the absorption oil phase, x_i , and in the aqueous methanolic solution phase, y_i , at equilibrium:

$$m_i = \frac{y_i}{x_i} \quad (1)$$

This distribution coefficient is presented in Fig. 2 with the mass fraction of water in the aqueous solution phase, y_w . The distribution coefficients of nitrogen heterocyclic compounds were higher than those of the other components in the absorption oil. The distribution coefficients of the components in the absorption oil decreased with y_w . The previous results⁷⁾ are also given in this figure, and show good agreement.

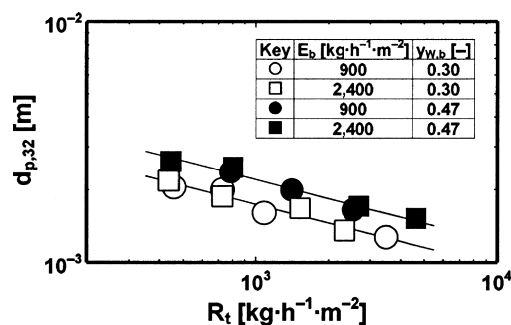
3.3. Continuous Extraction

3.3.1. Operability and Behavior of Liquid Phases in Column

The density of each liquid phase is summarized in Table 3. The density of the dispersed raffinate phase

Table 3 Densities of Liquid Phases

Phase	Density [$\text{kg} \cdot \text{m}^{-3}$]
Feed (absorption oil), R_t (dispersed phase)	1100
Solvent (aqueous solution of methanol), E_b (continuous phase) ($y_{w,b} = 0.3$)	870
Solvent (aqueous solution of methanol), E_b (continuous phase) ($y_{w,b} = 0.5$)	920
Raffinate, R_b (dispersed phase)	1100
Extract, E_t (continuous phase) ($y_{w,b} = 0.3$)	880-890
Extract, E_t (continuous phase) ($y_{w,b} = 0.5$)	920-930

Fig. 3 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Sauter Mean Diameter of Dispersed Phase Particle, $d_{p,32}$

was always higher than that of the continuous extract phase. Although the density of the extract phase was slightly higher than that of the solvent because of the mass transfer between the raffinate and extract phases, this slight change of density did not disturb the continuous countercurrent operation. Entrainment of the dispersed phase droplets into the continuous phase and flooding were not observed in the range of this study. The density of the continuous extract phase in the case of $y_{w,b} = 0.47$ was higher than that of $y_{w,b} = 0.30$ as expected from the densities of methanol and water.

Figure 3 shows the Sauter mean diameter of dispersed phase drops, $d_{p,32}$, which decreased with the flow rate of the dispersed phase, R_t . The diameters in the cases of $y_{w,b} = 0.47$ were larger than those of $y_{w,b} = 0.30$, since the density difference between the dispersed and continuous phases was smaller in the case of $y_{w,b} = 0.47$ as shown in Table 3. The flow rate of the continuous phase, E_b , did not affect the diameters. Drop coalescence and axial mixing of the dispersed phase were observed, especially at higher flow rates of the dispersed phase.

The holdup of the dispersed phase in the column, H , is shown in Fig. 4. The holdup could not be measured by the method mentioned above, if the flow rate of the dispersed phase was lower than about $500 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$. The holdup increased with increasing flow rate of dispersed phase, R_t . The holdup also increased with $y_{w,b}$ as a result of the decrease in density difference between the two liquid phases. The flow rate of the continuous phase, E_b , had no effect on the holdup, so that flow rates

of both phases resided in the range under the loading point.

Figure 5 shows the specific interfacial area between the dispersed raffinate and continuous extract phases, a , obtained from the Sauter mean diameter of dispersed phase drops, $d_{p,32}$, and the holdup, H , as,

$$a = \frac{6 \cdot H}{d_{p,32}} \tag{2}$$

The specific interfacial area increased with the flow rate

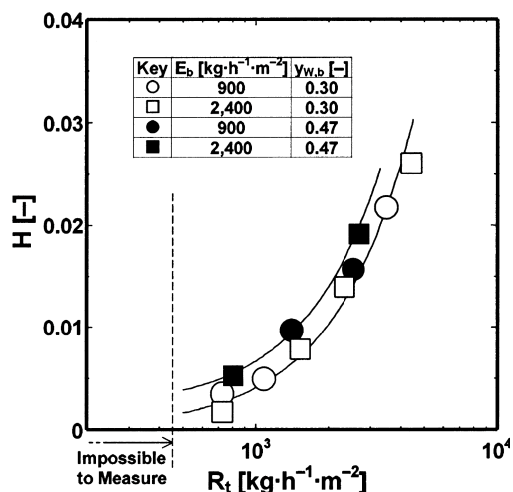


Fig. 4 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Holdup of Dispersed Phase, H

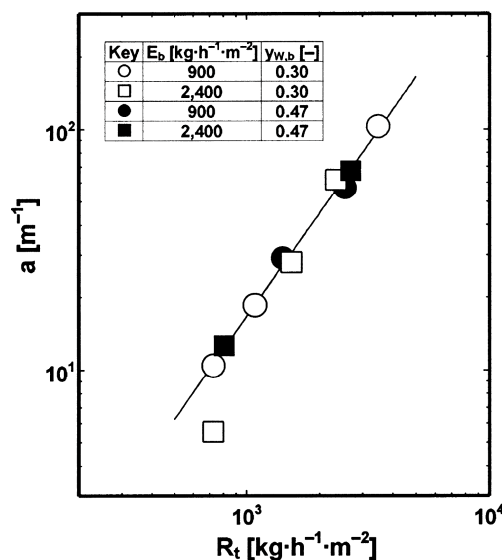
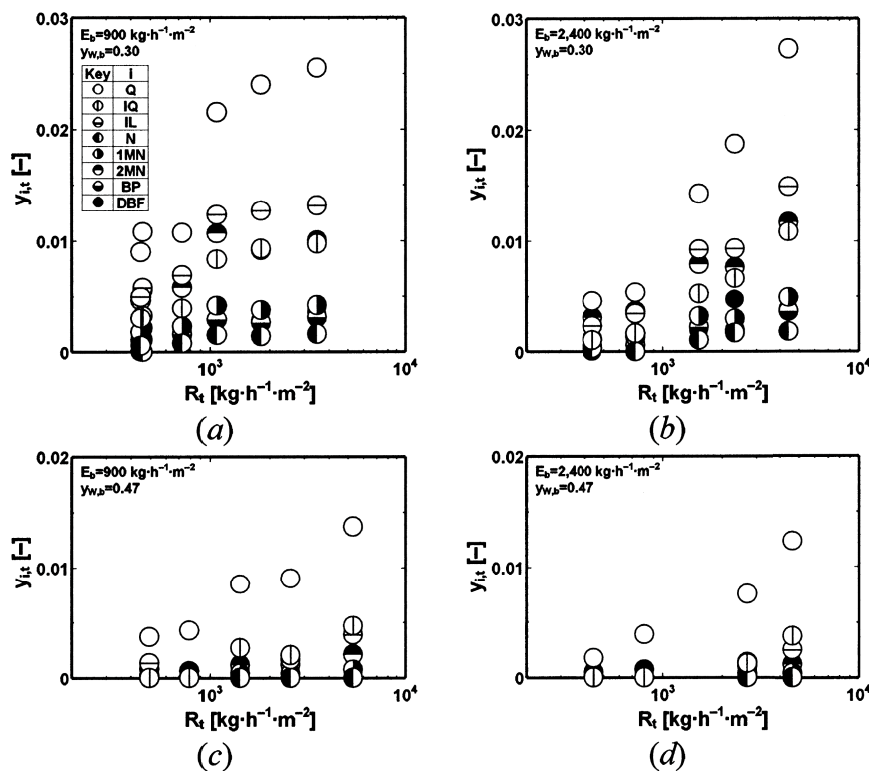
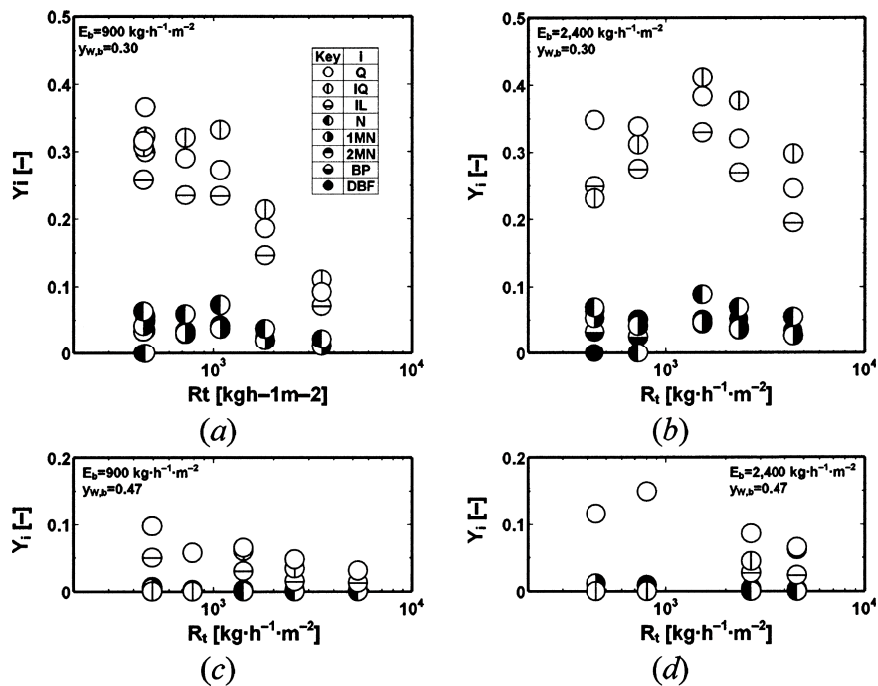


Fig. 5 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Specific Interfacial Area between Dispersed and Continuous Phases, a



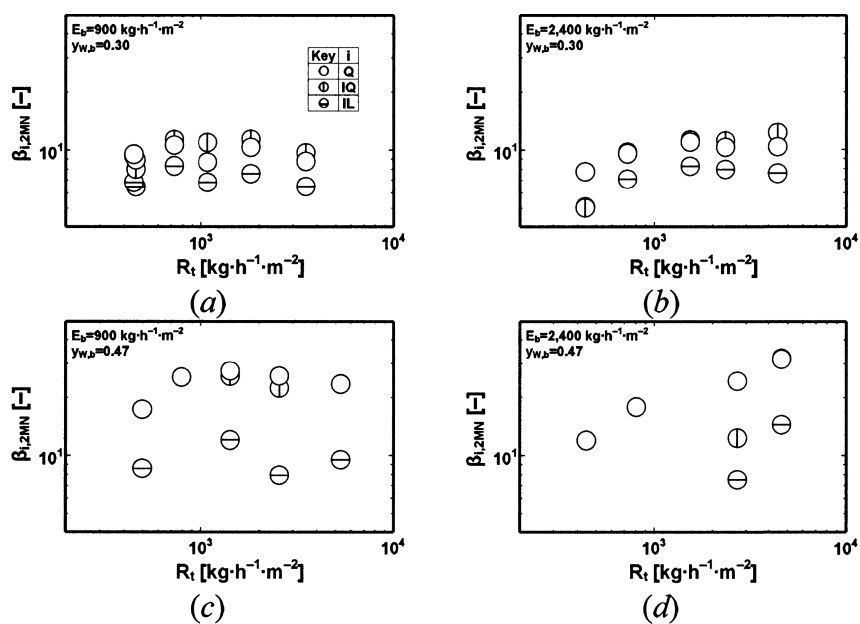
(a) superficial mass flow rate of solvent, E_b , of $900 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and mass fraction of water in solvent at bottom, $y_{w,b}$, of 0.30; (b) E_b of $2,400 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.30; (c) E_b of $900 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.47; (d) E_b of $2,400 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.47.

Fig. 6 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Composition of Extract Phase at Exit, y_{it}



(a) superficial mass flow rate of solvent, E_b , of $900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and mass fraction of water in solvent at bottom, $y_{w,b}$, of 0.30; (b) E_b of $2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.30; (c) E_b of $900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.47; (d) E_b of $2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.47.

Fig. 7 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Fractional Yield, Y_i



(a) superficial mass flow rate of solvent, E_b , of $900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and mass fraction of water in solvent at bottom, $y_{w,b}$, of 0.30; (b) E_b of $2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.30; (c) E_b of $900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.47; (d) E_b of $2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ and $y_{w,b}$ of 0.47.

Fig. 8 Effect of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Separation Selectivity Relative to 2-Methylnaphthalene, $\beta_{i,2MN}$

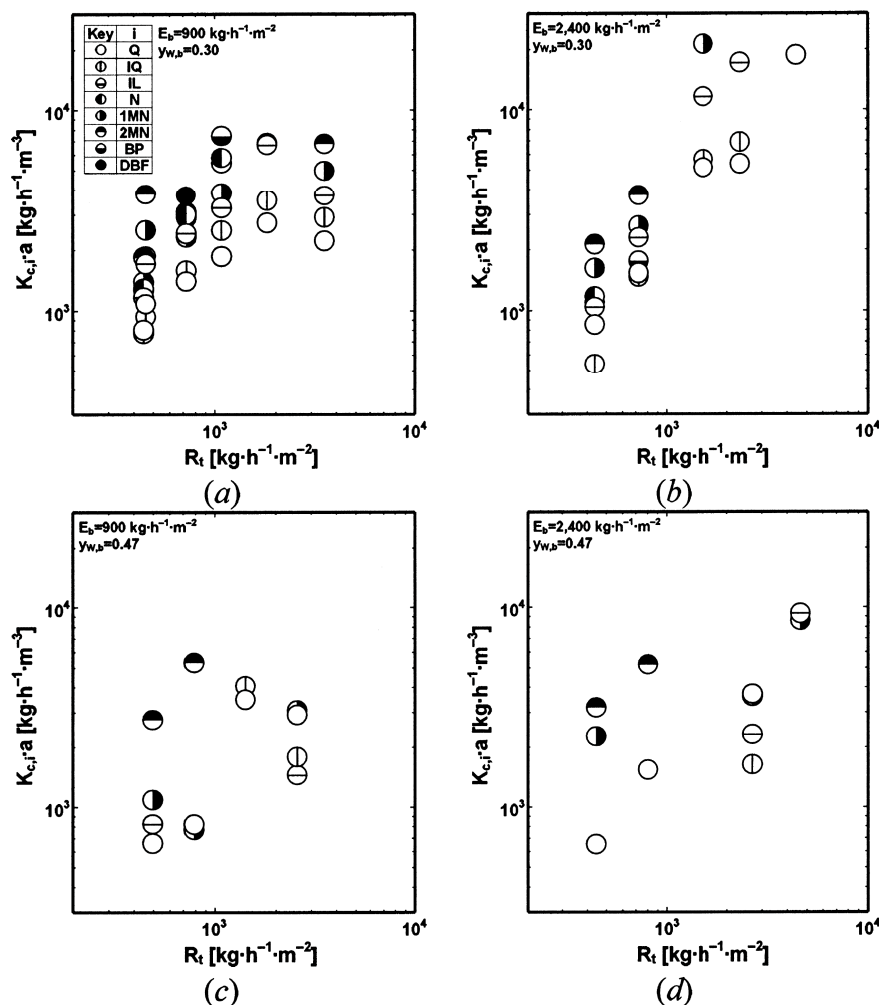


Fig. 9 Effects of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Overall Volumetric Mass Transfer Coefficients Based on Composition of Continuous Phase, $K_{c,i,a}$

of the dispersed phase, R_t , because of the decrease in $d_{p,32}$ and was not affected by $y_{w,b}$ and the flow rate of the continuous phase, E_b .

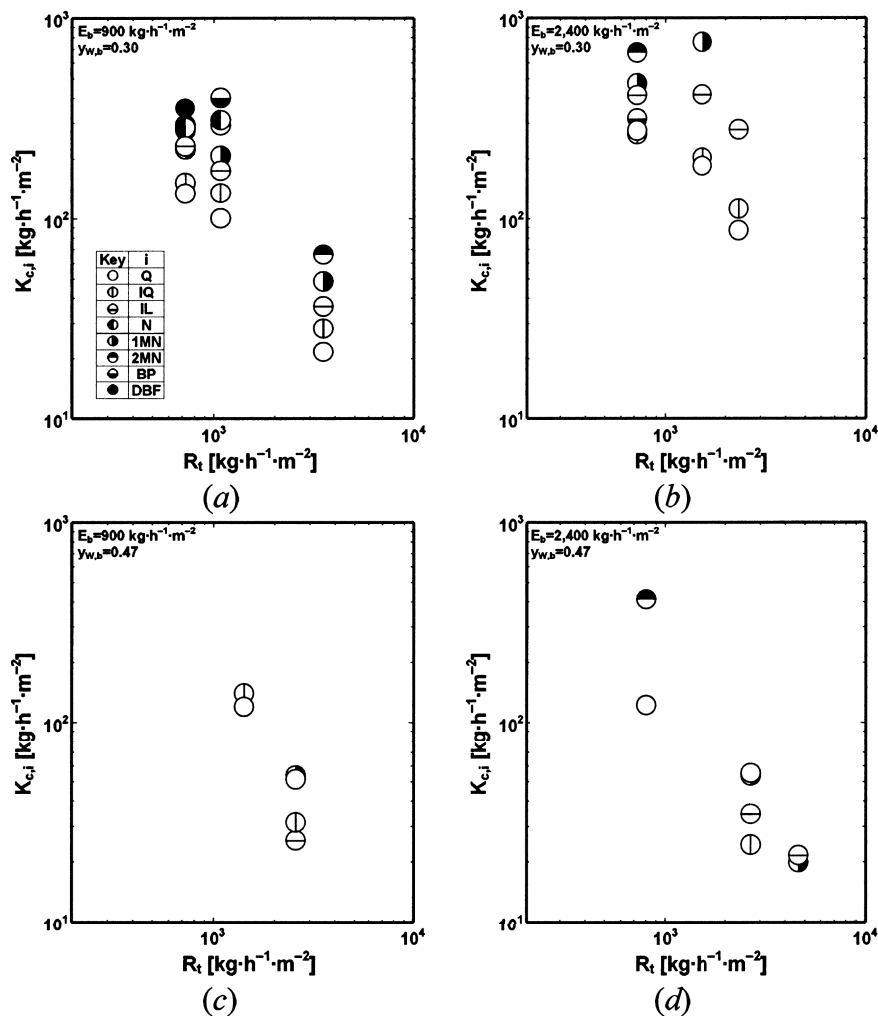
3.3.2. Separability

Figure 6 shows the composition, *i.e.*, the mass fractions of the studied components, in the extract phase at the top, the exit, $y_{i,t}$. Substantial mass transfers of the components could be detected with this bench scale column with effective contact height of 0.495 m. In all runs, $y_{i,t}$ increased with increasing flow rate of dispersed raffinate phase, R_t . Higher $y_{i,t}$ was observed if the flow rate of the continuous solvent phase, E_b , was $900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ compared to $E_b = 2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$. The $y_{i,t}$ decreased with increasing mass fraction of water in the solvent, $y_{w,b}$, because of the decreased distribution coefficients of the components with mass fraction of water in aqueous phase as shown in Fig. 2.

The fractional yield, Y_i , was defined by,

$$Y_i = \frac{E_t \cdot y_{i,t}}{R_t \cdot x_{i,t}} \quad (3)$$

with the flow rate, composition of extract phase at the top, the exit, E_t , $y_{i,t}$, the flow rate, and composition of raffinate phase at the top, the inlet, R_t , and $x_{i,t}$. Figure 7 shows this yield, Y_i . In all cases, the Y_i of nitrogen heterocyclic compounds was higher than those of the other components and separation of these compounds could be achieved by continuous solvent extraction with the spray column. Y_i decreased with increasing R_t . Y_i for $E_b = 2400 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$ were greater than those with $E_b = 900 \text{ kg}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$. Y_i was lower with higher $y_{w,b}$. In the range of this study, Y_i of the nitrogen compounds was about 0.4 at maximum with this column, whereas Y_i of the other components was less than 0.1.



(a) superficial mass flow rate of solvent, E_b , of $900 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and mass fraction of water in solvent at bottom, $y_{w,b}$, of 0.30; (b) E_b of $2400 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.30; (c) E_b of $900 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.47; (d) E_b of $2400 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ and $y_{w,b}$ of 0.47.

Fig. 10 Effects of Superficial Mass Flow Rate of Dispersed Phase, R_t , on Overall Mass Transfer Coefficients Based on Composition of Continuous Phase, $K_{c,i}$

The separation selectivity of component i relative to 2-methylnaphthalene, $\beta_{i,2MN}$, was defined as,

$$\beta_{i,2MN} = \frac{y_{i,t}/y_{2MN,t}}{x_{i,b}/x_{2MN,b}} \quad (4)$$

using the compositions of both liquid phases at the respective exits, $y_{i,t}$, $y_{2MN,t}$, $x_{i,b}$, and $x_{2MN,b}$. **Figure 8** shows the separation selectivities of the nitrogen compounds, $\beta_{i,2MN}$, against R_t . There was no obvious effect of R_t or E_b on the separation selectivity. The separation selectivities in the runs with $y_{w,b} = 0.47$ were larger than those with 0.30 and were about 30 at maximum.

Assuming plug flows of both liquid phases in the column, the material balance and mass transfer rate of component i per unit sectional area in the element of infinitesimal height at z in the column are given by,

$$\frac{d(R \cdot x_i)}{dz} = \frac{d(E \cdot y_i)}{dz} \quad (5)$$

$$\frac{d(E \cdot y_i)}{dz} = K_{c,i} \cdot a \cdot (m_i \cdot x_i - y_i) \quad (6)$$

where $K_{c,i}$ is the overall mass transfer coefficient based on the concentration in the continuous extract phase, y_i . The overall volumetric mass transfer coefficient averaged over the whole column, $K_{c,i} \cdot a$, was calculated by solving the simultaneous differential equations of Eqs. (5) and (6) with the distribution coefficients, m_i , presented in **Fig. 2** and the following boundary conditions at the bottom and top of the column:

$$\text{at } z = 0, \quad R = R_b \quad (7)$$

$$x_i = x_{i,b} \quad (8)$$

$$E = E_b \quad (9)$$

$$y_i = y_{i,b} \quad (10)$$

$$\text{at } z = Z, \quad R = R_t \quad (11)$$

$$x_i = x_{i,t} \quad (12)$$

$$E = E_t \quad (13)$$

$$y_i = y_{i,t} \quad (14)$$

The overall volumetric mass transfer coefficients, $K_{c,i} \cdot a$, are plotted against R_t in **Fig. 9**. $K_{c,i} \cdot a$ increased with R_t , mainly due to the increase in the specific interfacial area, a , with decreasing diameter of the dispersed raffinate phase drop, $d_{p,32}$.

The volumetric coefficient, $K_{c,i} \cdot a$, was divided by a given by **Fig. 5** to obtain the overall mass transfer coefficient, $K_{c,i}$. The effects of R_t on $K_{c,i}$, are presented in **Fig. 10**. $K_{c,i}$ decreased with increasing R_t , contrary to $K_{c,i} \cdot a$. This decrease in $K_{c,i}$ may be mainly attributed to the axial mixing of dispersed raffinate phase observed in the range of higher R_t as stated above. The relationships between the magnitudes of different $K_{c,i}$ of the studied components are not clear. However, the separation was confirmed to be governed by equilibrium in this extraction method. Since the $K_{c,i}$ at $E_b = 2400 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$ was larger than at $900 \text{ kg} \cdot \text{h}^{-1} \cdot \text{m}^{-2}$, there was some mass transfer resistance in the continuous extract phase. $K_{c,i}$ with $y_{w,b} = 0.30$ was high compared to that with 0.47. According to the equation of Wilke and Chang, which is widely used to estimate diffusivity of solute in dilute solution, the diffusivity increases with increasing molar mass and with decreasing viscosity of solvent. Therefore, as the average molar mass and viscosity of continuous extract phase increased and decreased, respectively, with decreasing $y_{w,b}$, the diffusivities of studied components increased in the continuous extract phase.

4. Conclusion

The liquid-liquid equilibrium relationships obtained here agreed with the previous findings.

The density of the dispersed raffinate phase was sufficiently larger than that of the continuous extract phase to allow countercurrent operation without entrainment of dispersed phase droplets into the continuous phase or flooding in the range of this study. Mass transfers of components necessary to study the phenomena could be detected with this bench scale spray column, with effective height of about 0.5 m. The nitrogen heterocyclic compounds were extracted preferentially to other compounds, such as homocyclic compounds, from the absorption oil and then the separation of these compounds could be achieved governed by the liquid-liquid equilibrium. The highest yield was about 0.4 and separation selectivity was about 30 for nitrogen compounds with this column. Since the overall mass transfer coefficients increased with the flow rate of the continuous extract phase, there was some mass transfer resistance in the continuous extract phase.

Acknowledgment

Coal tar absorption oil was provided by JFE Chemical Corp.

Nomenclatures

a	: specific interfacial area between dispersed and continuous phase	[m ⁻¹]
D	: inner diameter of spray column	[m]
$d_{p,32}$: Sauter mean diameter of dispersed phase drop	[m]
E	: superficial mass flow rate of extract phase	[kg · h ⁻¹ · m ⁻²]
H	: holdup of dispersed phase in spray column	[—]
$K_{c,i}$: overall mass transfer coefficient of component i based on concentration in continuous phase	[kg · h ⁻¹ · m ⁻²]
m_i	: distribution coefficient of component i	[—]
R	: superficial mass flow rate of raffinate phase	[kg · h ⁻¹ · m ⁻²]
x_i	: mass fraction of component i in raffinate phase	[—]
Y_i	: fractional yield of component i	[—]
y_i	: mass fraction of component i in extract phase	[—]
Z	: effective contact height of spray column	[m]
z	: distance from bottom of spray column	[m]
<Greeks>		
$\beta_{i,2MN}$: separation selectivity of nitrogen heterocyclic compound i relative to 2-methylnaphthalene	[—]
<Subscripts>		
1MN	: 1-methylnaphthalene	
2MN	: 2-methylnaphthalene	
BP	: biphenyl	
b	: at bottom of spray column	
DBF	: dibenzofuran	
IL	: indole	
IQ	: isoquinoline	
i	: component i	
M	: methanol	
N	: naphthalene	
Q	: quinoline	
t	: at top of spray column	
W	: water	

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要 旨

連続式向流接触スプレー塔によるコールタール吸収油の溶媒抽出

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メタノール水溶液を溶媒とした吸収油の液液抽出に対して、連続式向流接触スプレー塔型の抽出装置を適用した。スプレー塔は構造が簡素であることから、塔内の物質移動現象を検討するのに適している。まず、物質移動係数の算出に必要となる吸収油-メタノール水溶液間の液液平衡を実測し既往の結果と比較した。本報で得られた液液平衡関係と既往の結果とは良好に一致し結果の再現性、信頼性を確認した。ついで、スプレー塔による液液抽出における操作性ならびに分離性について検討した。連続（抽出）相に比較して分散（抽残）相の密度は十分大きく良好な向流接触操作が可能であり、本報の範囲においては

分散相の飛まつ同伴やフラッディングは観察されなかった。0.5 m ほどの研究室規模のスプレー塔により十分な物質移動が検出され、塔内の物質移動現象の検討が可能であった。同素環化合物など吸収油中の他の成分に比較して含窒素複素環式化合物が選択的に抽出され、これらの成分の分離が可能であった。また、この分離は平衡関係に基づく分離であった。本スプレー塔において、含窒素複素環式化合物の収率および分離の選択性は、最高でそれぞれ0.4および30程度であった。連続（抽出）相流量の増加とともに、総括物質移動係数は増加し、連続相側に物質移動抵抗が存在した。

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