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Flow and mass transfer characteristics in a falling-film extractor using hollow fiber as packing

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Abstract

To develop an efficient extractor, a falling-film contactor with polypropylene hollow fibers as packing materials was used to extract phenol and acrylic acid from an aqueous phase with kerosene containing 30% (v/v) tributyl phosphate (TBP). The two fluids were added to the top of the extractor and due to the different affinity of the two phases, the organic phase would spread to a thin film on the fiber surface and the aqueous phase would spread on the organic phase, mass transfer could occur during the falling process. The flow characteristics in the extractor were studied with a theoretical model, by which the flow status in the module can be predicted and provide a way to select suitable membrane materials for a specified extraction system. Studies on the mass transfer characteristics showed that falling-film extractor using hollow fiber as packing is highly efficient because of a very large mass transfer area and can be operated at extreme phase ratios in comparison with an extraction column.

Keywords: Hollow fiber; Mass transfer; Falling-film extractor; Phenol

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1. Introduction

Solvent extraction has been widely applied in hydrometallurgy [1], the petrochemical industry [2], environment protection [3] and for pharmaceutical manufacturing [4]. In conventional solvent extraction equipment, one phase is often firstly dispersed in another phase as droplets for increasing mass transfer area, then the droplets coalesce for phase separation [5,6]. The more fine the droplets, the more mass transfer area can be obtained, while the coalescence becomes more difficult. Furthermore, it is difficult to operate at an extreme ratio of two phases in a conventional extraction column because of flooding, low efficiency and unexpected emulsification [7]. Therefore, several new techniques such as membrane extraction and falling-film extraction have been investigated for solvent extraction.

Membrane extraction is a novel technology based upon liquid–liquid extraction in which the phase interface is immobilized by microporous membranes making it possible to operate under extreme phase ratios [8–10]. To avoid one phase breaking through the other phase, it is necessary to keep a suitable pressure drop between the two phases. However, phase breakthrough will happen in some cases even by doing this. Based on the affinity difference between the two phases, a falling-film extractor with metal fiber as packing material has been designed by American Merichem recently [11]. The two fluids are added to the extractor from the top; because of the hydrophilicity of metal fiber, the aqueous phase will spread to a thin film on the fiber surface and the organic phase will spread on the water surface; thus, mass transfer occurs during the falling process. In this process, the tiny fiber leads to a very large mass transfer area and the coalescence is easier since one phase is not dispersed as fine droplets into the other phase. The falling-film extractor applied to the removal of hydrogen sulfide and naphthenic acid from oil using NaOH solution has been reviewed by Hu [12] and Wang [13], while the flow and mass transfer characteristics in the falling-film extractor have not been comprehensively studied and the principle for selecting the packing materials has

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Nomenclature

A	mass transfer area, membrane area in module (cm^2)
b	constant in Eq. (26)
B	constant in Eqs. (13) and (14)
D	inner diameter of shell (cm)
E	mass transfer efficient, defined in Eq. (27)
F	constant in Eqs. (21)–(23)
g	acceleration of gravity ($m\,s^{-2}$)
k	individual mass transfer coefficient ($cm\,s^{-1}$)
K	overall mass transfer coefficient ($cm\,s^{-1}$)
L	flow rate ($ml\,min^{-1}$)
m	distribution coefficient, constant in Eq. (26)
M	constant in Eq. (26)
N	number of hollow fibers
p	pressure (Pa)
r	radius (cm)
R	radius (cm)
t	time (s)
u	velocity at z-direction ($cm\,s^{-1}$)
x	concentration in aqueous phase ($mg\,l^{-1}$)
y	concentration in oil phase ($mg\,l^{-1}$)
z	z-axis

Greek symbols

μ	viscosity (Pa s)
θ	direction in cylinder coordinate
ρ	density ($kg\,m^{-3}$)
Ψ	constant in Eq. (18)

Subscripts

o	oil phase
w	aqueous phase
i	the interface of two phases
in	inlet
out	outlet
1–4	consecutive numbers
z	z-direction

Superscript

*	equilibrium value

not been mentioned. In this study, hollow fibers made from polypropylene (PP) were used as packing media. Since the PP fibers are hydrophobic, the organic phase will wet the hollow fibers to form a thin layer of solvent on the fiber surface. The advantages of falling-film extraction using hollow fibers are as follows:

(1) the two phases are in contact through a thin film and the flow is laminar, avoiding strong mixing and coalescence, so the two phase will separate quickly in the settler and the loss of oil phase will be small;

(2) using the hollow fiber as packing, the specific contacting area will be up to 3000–4000 $m^2\,m^{-3}$ and the overall mass transfer coefficient $K_w \cdot a$ is 10–15 times as that of conventional sieve extraction column;

(3) it can work well at extreme phase ratios such as $L_0/L_w > 10$ or <10;

(4) the hollow fiber is much cheaper and more easily available than metal fiber;

(5) compared with membrane extraction, in the case of falling-film extraction there is no resistance in the membrane phase.

In this work, the flow characteristics in the falling-film extractor were studied with a theoretical model and the mass transfer characteristics were further investigated in contactors with different packing density.

2. Experimental

2.1. Experimental apparatus

The scheme of the experimental apparatus is shown in Fig. 1. The organic solvent and aqueous phase were pumped into the shell side from the top of the module with and evenly distributed to the cross-section through a liquid distribution apparatus (Fig. 1b). When the two phases fell down along the fibers, the organic solvent spread on the fiber surface to form a very large contact area for the aqueous phase. Mass

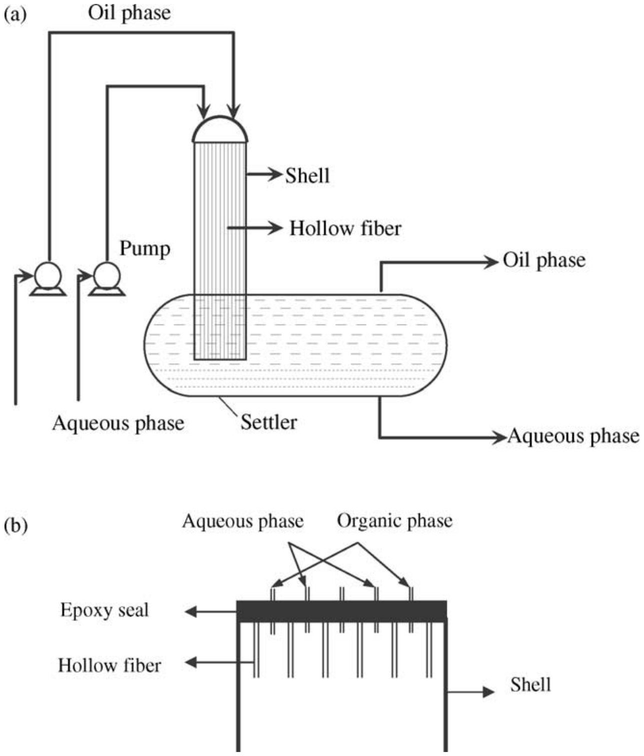

Fig. 1. Scheme of falling-film fiber extraction system: (a) the whole extraction system and (b) entrance of the module.

Table 1
The characteristics of fiber modules

No.	*D* (cm)	Fiber length (cm)	Outer diameter of fiber, R_1 (cm)	*N*	Specific surface area ($m^2\ m^{-3}$)	Packing density
Module 1	3.2	65	0.048	2100	3938	0.454
Module 2	3.2	65	0.048	2400	4500	0.540

transfer between the two phases can be varied at different flow rates. Thereafter, the organic phase and aqueous phase would separate in the settler. Because the hollow fibers used are porous and hydrophobic, a small amount of organic phase will enter and stay in the fiber pore; since it is not large enough compared to the bulk fluid, this amount of solvent is omitted during calculation.

2.2. Mass transfer in the falling-film extractor

The characteristics of the falling-film extractors are shown in Table 1. The polypropylene hollow fiber modules were manufactured by Seawater Desalting Center of National Ocean Agency, Tianjin City, China. Kerosene containing 30% (v/v) of tributyl phosphate (TBP) was used as solvent to extract phenol and acrylic acid from their aqueous phases, respectively. Kerosene was obtained from a gasoline station; the analytical reagents of TBP, phenol and acrylic acid were purchased from Beijing Chemical Factory. The pH in the initial solutions of phenol and acrylic acid was kept to the natural ones. Sampling was done at the inlet and outlet of the falling-film contactor, phenol concentration was determined with HP8452 UV spectrophotometer (Waldbronn, Germany) at 270 nm and acrylic acid was titrated with NaOH solution (1N). The loaded solvent was regenerated with NaOH solution (3%, w/v).

2.3. Determination of TBP concentration

TBP concentration in the aqueous phase was determined by measuring the phosphorus content. Four milliliters of $K_2S_2O_8$ solution ($50\ g\ l^{-1}$) was added into 25 ml aqueous sample and reacted at 120 °C for 30 min, whereby TBP was oxidized into phosphate. Afterward, 1 ml ascorbic acid solution ($100\ mg\ l^{-1}$), 2 ml $(NH_4)_6Mo_7O_{24}$ solution (4%) were added in and the optical density of the sample was measured at 700 nm.

2.4. Solvent loss during extraction

The solvent loss during phase contact in the falling-film extraction was investigated by measuring the TBP concentration in aqueous phase after extraction. To compare with the conventional mixer–settler process, 40 ml solvent and 40 ml aqueous phase were mixed with a HZS-H shaker (Harbin Donglian Company) at 180 rpm for 5 min, and the obtained mixture was left at room temperature for 1 h, then the TBP concentration in aqueous phase was determined.

3. Theoretical

3.1. Hydrodynamics model in falling-film extractor

As shown in Fig. 2a, an even fiber distribution in the module can be obtained when the packing factor is relatively high. It is assumed that the hollow fibers are packed regularly in the geometry of an equilateral triangle. The flow channel corresponding to one fiber is a normal hexagon. When the two phases flow down along the fibers, each phase will occupy a certain space surrounding the fibers, the interface of two phases will locate between the fiber outer surface and the normal hexagon. Taking the axis-symmetry into consideration, the hexagon is approximated to be circular (shown in Fig. 2b). In Fig. 2c, R_1, R_i and R_2 are the radius of hollow fiber, the boundary between the two phases and the equivalent radius of hexagon, respectively. u_o and u_w represent the flow rate of oil phase and aqueous phase, respectively. R_2 can be obtained from the number of fibers, *N*, and the inner diameter of the falling-film extractor, *D*:

$$N \times \frac{\pi}{4} \times R_2^2 = \frac{\pi}{4} \times D^2 \tag{1}$$

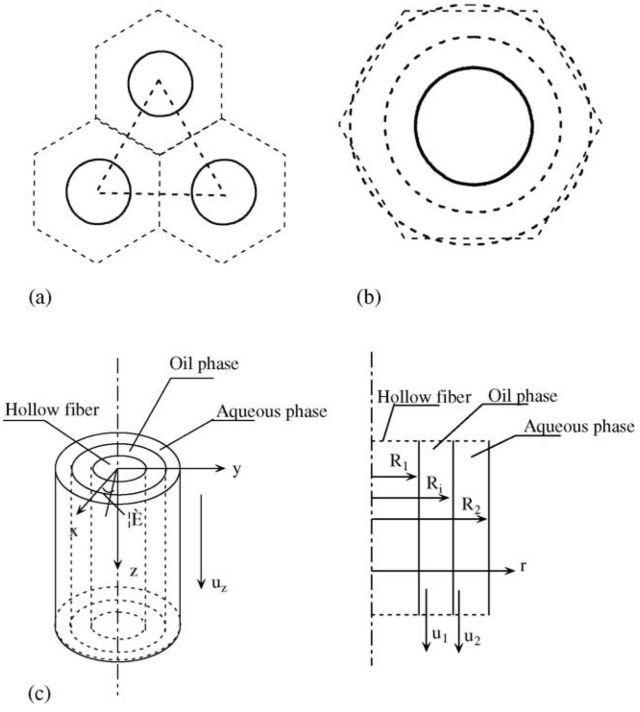

Fig. 2. Scheme of the flow paths in the module: (a) arrangement of hollow fibers, (b) the simplified process and (c) flow paths of the two phases.

$$R_2 = \sqrt{\frac{1}{N}} \times D \tag{2}$$

It is further assumed that the two phases are incompressible and will be completely developed to a laminar flow in the module. Then, the Navier–Stokes (N–S) equations can be used to predict the flow rate.

The steady flow leads to:

$$\frac{\partial u_z}{\partial t} = 0 \tag{3}$$

The flow is axis-symmetric and u_z is constant for those points with the same radius:

$$\frac{\partial u_z}{\partial \theta} = 0, \qquad \frac{\partial u_z}{\partial z} = 0 \tag{4}$$

The flow along θ and r directions is neglected:

$$u_r = u_\theta = 0 \tag{5}$$

Then, the N–S equations can be simplified as:

$$\frac{\mathrm{d}p}{\mathrm{d}z} - \rho_\mathrm{o} g = \frac{\mu_\mathrm{o}}{r}\frac{\mathrm{d}}{\mathrm{d}r}\left(r\frac{\mathrm{d}u_\mathrm{o}}{\mathrm{d}r}\right), \qquad R_1 \le r \le R_i \tag{6}$$

$$\frac{\mathrm{d}p}{\mathrm{d}z} - \rho_\mathrm{w} g = \frac{\mu_\mathrm{w}}{r}\frac{\mathrm{d}}{\mathrm{d}r}\left(r\frac{\mathrm{d}u_\mathrm{w}}{\mathrm{d}r}\right), \qquad R_i \le r \le R_2 \tag{7}$$

With the following boundary conditions:

$$r = R_1, \qquad u_\mathrm{o} = 0 \tag{8}$$

$$r = R_i, \qquad u_\mathrm{o} = u_\mathrm{w} = u_i, \qquad \mu_\mathrm{o}\frac{\mathrm{d}u_\mathrm{o}}{\mathrm{d}r} = \mu_\mathrm{w}\frac{\mathrm{d}u_\mathrm{w}}{\mathrm{d}r} \tag{9}$$

$$r = R_2, \qquad \frac{\mathrm{d}u_\mathrm{w}}{\mathrm{d}r} = 0 \tag{10}$$

Integrated from R_1 to R_i for the organic phase and from R_i to R_2 for the aqueous phase, respectively, the flow velocity can be obtained:

$$u_\mathrm{o} = \frac{B_1}{4\mu_\mathrm{o}}(r^2 - R_1^2) - \frac{M}{2\mu_\mathrm{o}} \ln\frac{r}{R_1} \tag{11}$$

$$u_\mathrm{w} = \frac{B_2}{4\mu_\mathrm{w}}(r^2 - R_i^2) - \frac{B_2 R_2^2}{2\mu_\mathrm{w}} \ln\frac{r}{R_i} + \frac{B_1(R_i^2 - R_1^2)}{4\mu_\mathrm{o}} - \frac{M}{2\mu_\mathrm{o}} \ln\frac{R_i}{R_1} \tag{12}$$

where

$$B_1 = \frac{\mathrm{d}p}{\mathrm{d}z} - \rho_\mathrm{o} g \tag{13}$$

$$B_2 = \frac{\mathrm{d}p}{\mathrm{d}z} - \rho_\mathrm{w} g \tag{14}$$

$$M = B_1 R_i^2 + B_2 R_2^2 - B_2 R_i^2 \tag{15}$$

Hence, the flow rate of the two phases can be derived:

$$L_\mathrm{o} = \int_{R_1}^{R_i} 2\pi u_\mathrm{o}\,\mathrm{d}r = \frac{\pi}{8\mu_\mathrm{o}}\left[4MR_i^2 \ln\frac{R_1}{R_i} + (R_i^2 - R_1^2) \times (3B_1R_i^2 - B_1R_1^2 + 2B_2R_2^2 - 2B_2R_i^2)\right] \tag{16}$$

$$L_\mathrm{w} = \int_{R_i}^{R_2} 2\pi u_\mathrm{w}\,\mathrm{d}r = \frac{\pi}{8\mu_\mathrm{o}}\left\{\Psi B_2(R_2^4 - R_i^4) + 4\Psi B_2 R_2^4 \ln\frac{R_i}{R_2} + 2(R_2^2 - R_i^2) \times \left(B_1R_i^2 - B_1R_1^2 + \Psi B_2R_2^2 - \Psi B_2 R_i^2 - 2M \ln\frac{R_i}{R_1}\right)\right\} \tag{17}$$

where

$$\Psi = \frac{\mu_\mathrm{o}}{\mu_\mathrm{w}} \tag{18}$$

The above Eqs. (16) and (17) can be expressed in the following way:

$$\frac{8\mu_\mathrm{o}}{\pi}L_\mathrm{o} = B_1F_1 + B_2F_2 \tag{19}$$

$$\frac{8\mu_\mathrm{o}}{\pi}L_\mathrm{w} = B_1F_3 + B_2F_4 \tag{20}$$

where

$$F_1 = (R_i^4 - R_1^4) + 4R_i^4 \ln\frac{R_1}{R_i} + 2(R_i^2 - R_1^2)^2 \tag{21}$$

$$F_2 = F_3 = 4(R_i^2 - R_1^2)R_i^2 \ln\frac{R_1}{R_i} + 2(R_i^2 - R_1^2)(R_2^2 - R_i^2) \tag{22}$$

$$F_4 = \Psi(R_2^4 - R_i^4) + 2(R_2^2 - R_i^2)^2\left(\Psi + 2\ln\frac{R_1}{R_i}\right) + 4\Psi R_2^4 \ln\frac{R_i}{R_2} \tag{23}$$

$$B_1 - B_2 = (\rho_\mathrm{w} - \rho_\mathrm{o})g \tag{24}$$

Because L_w and L_o are known, R_i, B_1, B_2 can be calculated by solving Eqs. (19), (20) and (24), then from Eqs. (11) and (12), u_o and u_w can be obtained.

3.2. *Mass transfer efficiency in the falling-film extractor*

Assuming that the flow status is ideal plug flow, the experimental overall mass transfer coefficient K_w for parallel flow can be obtained from the following correlation [14]:

$$K_\mathrm{w} = \frac{1}{A}\frac{L_\mathrm{w}}{1 + \frac{L_\mathrm{w}}{mL_\mathrm{o}}} \ln\frac{x_\mathrm{in} + \frac{b}{m}}{\left(1 + \frac{L_\mathrm{w}}{mL_\mathrm{o}}\right)x_\mathrm{out} - \frac{1}{m}\left(\frac{L_\mathrm{w}}{L_\mathrm{o}}x_\mathrm{in} - b\right)} \tag{25}$$

where x_{in} and x_{out} are the solute concentration at inlet and outlet, L_{w} and L_{o} the flow rate of aqueous phase and organic phase, and A is the overall outer surface area of hollow fibers, approximately representing the real mass transfer area which is difficult to measure. m and b are the two constants in the equilibrium correlation of the experimental system:

$$y = mx + b \tag{26}$$

For the two systems, b is 0, m is the distribution coefficient.

The stage efficiency of falling-film membrane contactor is then obtained:

$$E = \frac{x_{\text{in}} - x_{\text{out}}}{x_{\text{in}} - x^*} \times 100\% \tag{27}$$

where x^* is the equilibrium concentration in the aqueous phase at a phase ratio same as the flow rate ratio of the two phases. Since the flow in the falling-film contact is parallel flow, the maximum value for E is 1.

4. Results and discussions

4.1. Flow status in falling-film contactor

The flow status in Module 1 can be predicted as Fig. 3. The organic phase flows on the outer surface of the fibers and the aqueous phase flows on the outer surface of the organic phase. At the phase interface, the velocity of the organic phase is at a maximum while the velocity of the aqueous phase is minimal. The latter is always larger than the former. When the phase ratio (O/W) is 1:5 (Fig. 3a), the interface of the two phases is located at $r/R_2 = 0.82$, the holding spaces of aqueous and organic phase are reasonable; but when the phase ratio is increased to be 5:1 (Fig. 3c), the interface of the two phases is located at $r/R_2 = 0.96$, the holding space of aqueous phase is very small, the aqueous flow path among the oil paths is very narrow; therefore, the aqueous phase cannot spread well on the surface of oil phase and the mass transfer performance will decrease correspondingly. In this case, hydrophilic material should be adopted for an effective extraction. Removing

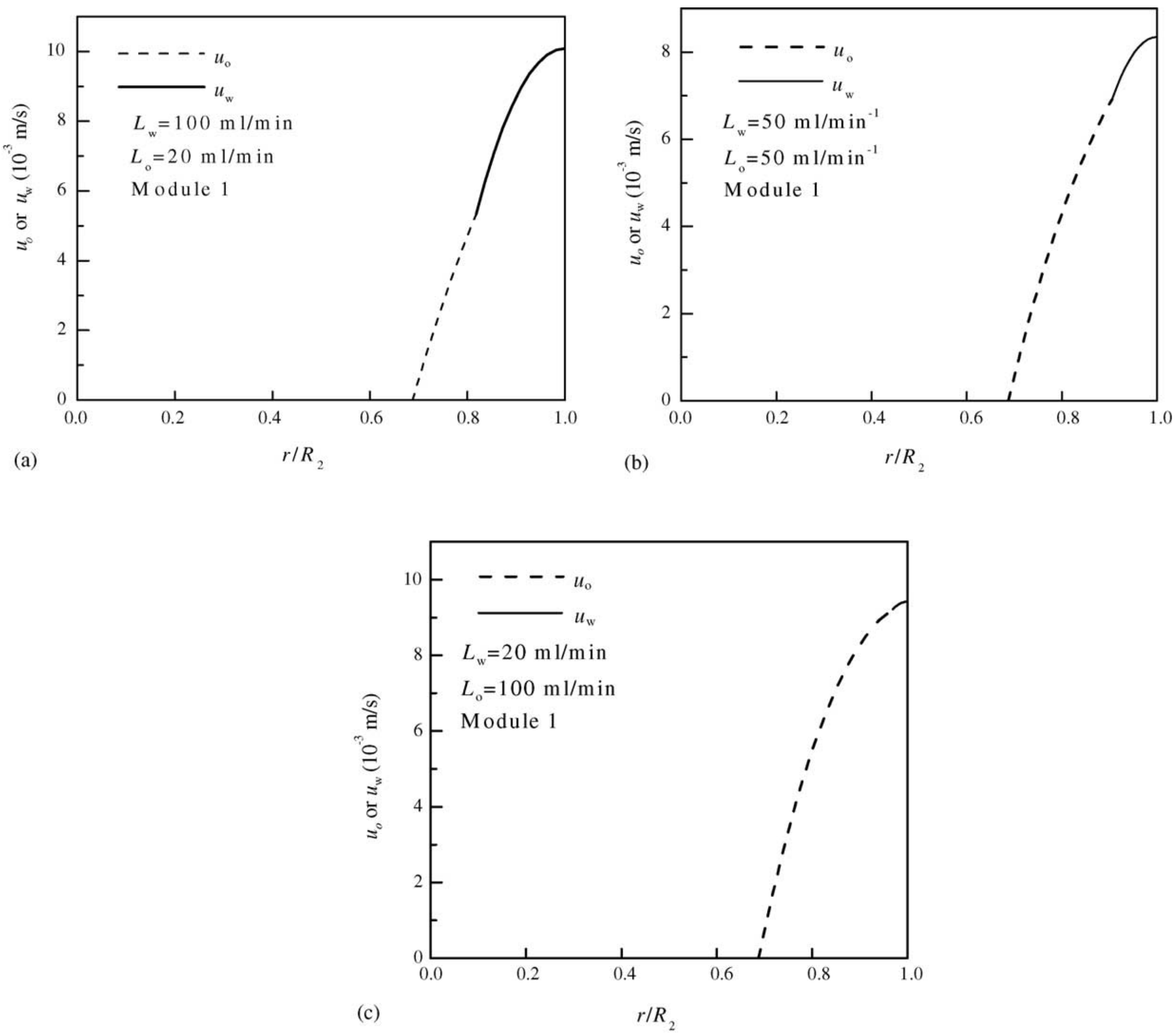

Fig. 3. The velocity distribution of the two phases in Module 1.

naphthenic acid from oil is always operated with a large flow rate of oil phase and small flow rate of aqueous phase, so the metal fiber is preferable.

4.2. Effect of flow rate on mass transfer coefficient in Module 1

The effects of flow rate of aqueous phase on overall mass transfer coefficient K_w are shown in Fig. 4. It was found that K_w increased quickly with the flow rate of aqueous phase. In falling-film extraction process, when the flow rate of aqueous phase increases, the mean velocities of both phases will increase accordingly and the boundary layers of the two phases become thin; as a result, the K_w increases rapidly. This process is very different from membrane extraction using a hollow fiber module [15], in which the interface of two phases is immobilized in the membrane pores and the two phases flow through the tube side or shell side, respectively, so the mean velocity of one phase does not influence that of another phase.

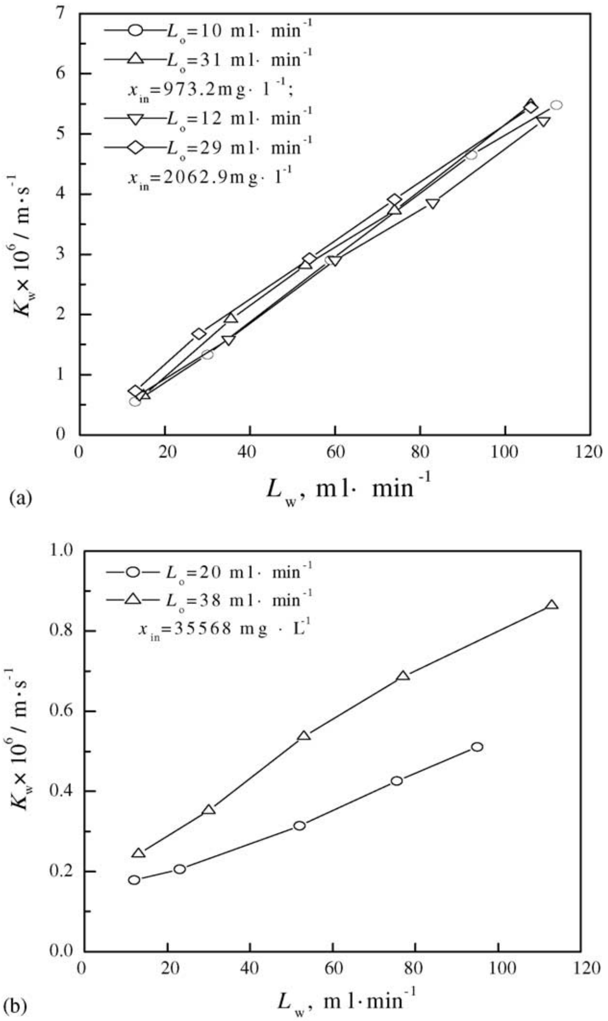

Fig. 4. Effect of the flow rate of aqueous phase on mass transfer coefficients for: (a) phenol and (b) acrylic acid.

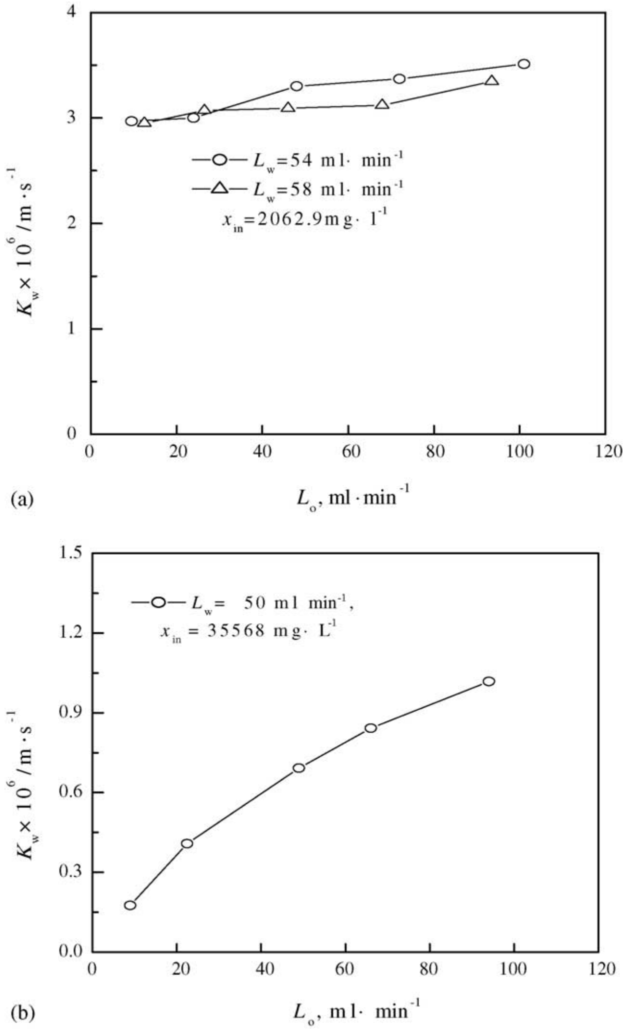

Fig. 5. Effect of the flow rate of organic phase on mass transfer coefficients for: (a) phenol and (b) acrylic acid.

The effects of flow rate of organic phase on K_w are shown in Fig. 5. For phenol, K_w increased slowly with the increasing L_o, while for acrylic acid, there was a significant impact from L_o on K_w. Higher L_o gave a higher K_w. The difference between the two systems resulted from the different distribution coefficients. For the falling-film membrane contactor, the overall mass transfer resistance equals the sum of the individual resistance:

$$\frac{1}{K_w} = \frac{1}{K_w} + \frac{1}{mk_o} \tag{28}$$

For phenol extraction, m is 173, $1/mk_o \ll k_w$, so the mass transfer resistance in the organic phase can be ignored, i.e., K_w is approximately the same as k_w; for acrylic acid extraction, m is 2.4, neither of the mass transfer resistances in the oil phase and the aqueous phase can be omitted.

4.3. Influence of the flow rate of aqueous phase on E

As shown in Fig. 6, the efficiency for phenol and acrylic acid in Module 1 was near 100% and higher than 90%, re-

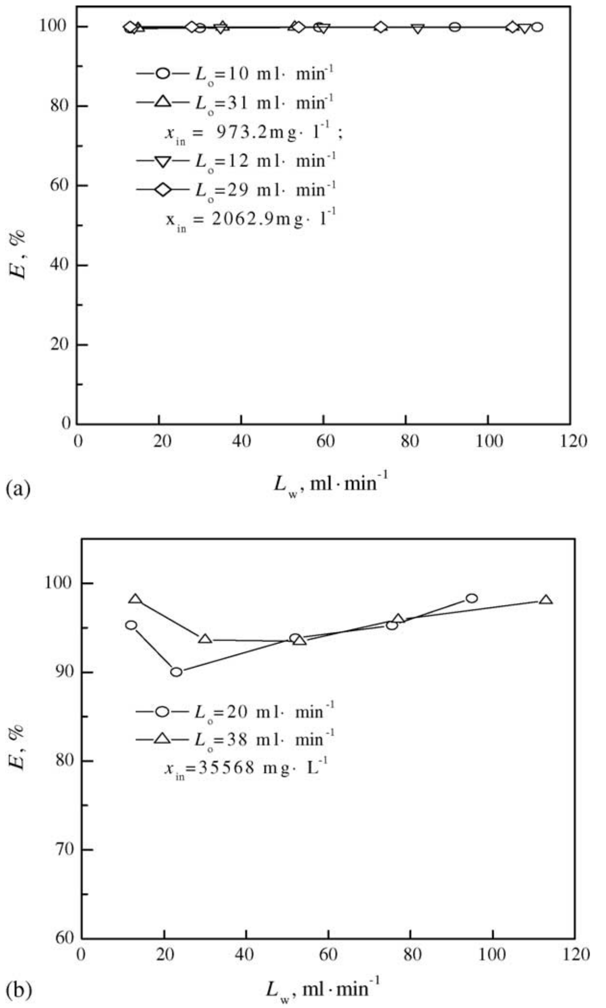

Fig. 6. Effect of the flow rate of aqueous phase on stage efficiency of mass transfer in Module 1: (a) phenol and (b) acrylic acid.

spectively. It is suggested that the falling-film membrane contactor with the hollow fiber is highly efficient at phase ratios in the range of 1:1–1:11. Since the mass transfer coefficient will increase rapidly with the increasing flow rate of aqueous phase, there will be no decrease in the mass transfer efficiency at high flow rate.

4.4. Effect of packing density and packing materials on E

As shown in Fig. 7, the extraction efficiency in Module 2 was lower than that in Module 1 especially at lower flow rate of aqueous phase, although the fiber surface of Module 2 is larger than the surface area of Module 1. When the packing density was high, the fluid had to flow across many fibers to distribute to the whole cross-section. If the flow rate of aqueous phase was low, its small momentum made it difficult to cross the fibers, a narrow flow path of aqueous phase was then obtained. With the increasing flow rate of aqueous phase, its flow path became broader, so the mass transfer efficiency increased. For hydrophobic materials, a relatively high flow rate of aqueous phase and low flow rate of oil phase are preferable for mass transfer. In environmental protection, when extracting hazard materials in very dilute solution with large flow rate, falling-film membrane made from polypropylene is suitable since an efficient extraction can be achieved with a small amount of organic phase, therefore, the solute can be concentrated in the organic phase.

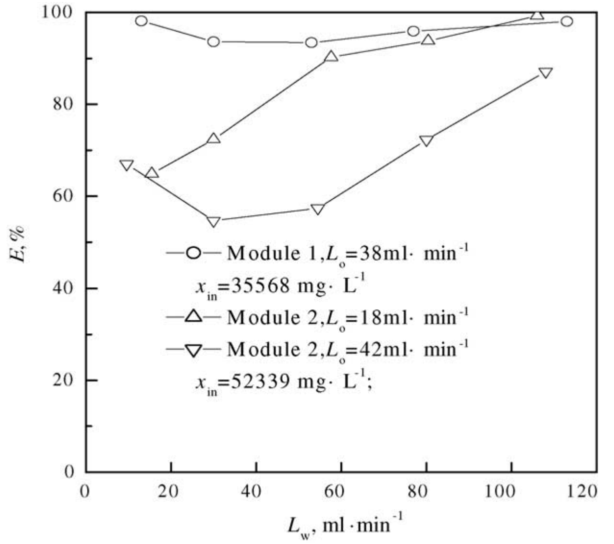

Fig. 7. Influence of packing factor on mass transfer efficiency for acrylic acid extraction.

Table 2
The loss of extractant

Process	L_w (ml min^{-1})	L_o (ml min^{-1})	TBP concentration (mg l^{-1})
Falling-film extractor	28	28	94.3
	60	60	100.1
	115	115	98.0
	115	10	97.2
Shaker–settler			182.8

4.5. Extractant loss in falling-film extraction process

As shown in Table 2, the extractant loss in falling-film extraction process was 50% of that occurring in shaker–settler process and did not change with the flow rate of the two phases, indicating there was no strong dispersion–coalescence process in falling-film extraction.

5. Conclusions

Hollow fibers were used as packing media in a falling-film extractor. The predicted flow status in the module with the theoretical model provided a way to select the suitable membrane materials for a specified extraction system. The falling-film extractor packed with polypropylene hollow fibers was used for extracting phenol and acrylic acid from their aqueous phase by kerosene containing 30% (v/v) TBP. The mass transfer experiments showed that falling-film membrane contactor is an efficient device, especially suitable for systems with high distribution coefficient and extremely phase ratio. With increase in flow rate of the aqueous phase, the mass transfer coefficient K_w increased rapidly. The flow rate of organic phase gave a different effect on the two systems. For phenol, K_w increased slowly with the increasing L_o; for acrylic acid, K_w increased rapidly with the increasing L_o. Very high extraction efficiency was obtained both for phenol and for acrylic acid at a phase ratio from 1:1 to 1:11. The experimental results showed that lower packing density gave relatively high extraction efficiency. For hydrophobic materials like polypropylene, a relatively high flow rate of aqueous phase is preferable for mass transfer. The small solvent loss in falling-film extraction process indicated there was no strong dispersion–coalescence process.

Acknowledgements

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