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Application of Crossflow Ultrafiltration to Emulsion Separation in the Extraction of Valeric Acid with Tri-*n*-butyl Phosphate

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ABSTRACT

Extractive ultrafiltration (EUF) of valeric acid from aqueous solutions with tri-*n*-butyl phosphate dissolved in kerosene, at two different phase ratios and using two ceramic membranes, was studied. The membranes used were ZrO_2 supported on α-alumina and $ZrO_2 + TiO_2$, supported on carbon with an average pore size of 20 and 45 nm, respectively. Permeation rates and degree of extraction were determined under three different modes of operation of the membrane unit: total recirculation, permeate removal, and continuous operation.

Key Words. Extractive ultrafiltration; Valeric acid; Reactive extraction; Emulsion membrane filtration

INTRODUCTION

Extractive ultrafiltration (EUF) is a separation process which combines the selectivity of solvent extraction and phase separation through an ultrafiltration unit. This technique minimizes the problems associated with conventional

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mixer-settlers such as reagent losses through entrainment, third phase formation, and difficult separations due to stable emulsions. EUF has been used for the recovery and separation of organic compounds (1–3).

Organophosphorus compounds, such as tri-*n*-butyl phosphate (TBP), have been used as extractants in hydrometallurgy and for the recovery of organic acids from aqueous solutions, including fermentation broths, wastewaters, etc. The extraction process implies solvation of the acid by donor bonds (4).

Valeric acid (*n*-pentanoic acid) is produced industrially by oxidation of amyl alcohol or by fermentation processes and can be found as a subproduct in the manufacture of adipic acid. As pure adipic acid is required when it is to be used as the monomer in polymerization processes, any valeric acid produced must be removed during the adipic acid purification step.

In previous work the extraction of valeric acid with TBP dissolved in kerosene at two different concentrations, 10 and 30 vol%, was studied, and the results were compared with those obtained by using kerosene alone (5). The removals of valeric acid using Amberlite LA-2 + toluene using EUF with a tubular ceramic membrane has also been studied (3).

The purpose of the present work is to evaluate the feasibility of the EUF technique for the recovery of valeric acid from aqueous solutions using the system TBP–kerosene. The effects of feed organic concentration, transmembrane pressure, and membrane pore size on the separation efficiency were studied in order to determine suitable process conditions.

EXPERIMENTAL SECTION

Materials

Valeric acid (Aldrich, $>$99%), tri-*n*-butyl phosphate (Fluka, $>$97%), and kerosene as the diluent (Aldrich, 64% alkanes, 26% cycloparaffins, 9.8% aromatics and sulfur) were used as supplied. Bidistilled water was used in all experiments.

Two tubular membrane modules were used, one of ZrO_2 supported on α-alumina (SCT, France) and another of $ZrO_2 + TiO_2$ supported on carbon (Carbosep, France) with average pore diameters of 20 and 45 nm, respectively. Both membranes, which had markedly hydrophilic natures, were 25 cm long, 1 cm external diameter, and 0.7 and 0.6 cm inside diameters, respectively. The filtration areas were 55 cm^2 for the ZrO_2 membrane and 47 cm^2 for the $ZrO_2 + TiO_2$ membrane.

A centrifugal pump, which has a maximum flow rate of 1500 L/h at a maximum pressure of 0.4 MPa, was used in the experiments. Feed flow and transmembrane pressure were adjusted to the desired values by using a bypass, with a globe valve and a needle valve, placed after the membrane module.

Two pressure gauges measured the inlet and outlet pressures respectively, and the feed flow was measured with a rotameter. The equipment was described in detail in an earlier publication (3).

Methods

The organic and aqueous phases were mixed in the feed tank at a volumetric ratio of 20 or 30 vol%. Experiments were carried out with kerosene alone and with TBP dissolved in kerosene at 10 and 30 vol%, respectively. The aqueous phase used was either water or a valeric acid solution with a concentration of 6 g/L. Before feeding the emulsion to the membrane, it was circulated through the bypass to mix both phases thoroughly. Once the emulsion was formed, it was pumped through the membrane circuit. A clear aqueous permeate was obtained, while the emulsion was recirculated to the feed tank.

Most experiments were carried out batchwise at constant concentration, i.e., returning the permeate and retentate to the feed tank. A concentration experiment (removing the permeate continuously) and a continuous operation experiment (removing the permeate and feeding a fresh valeric acid solution) were also carried out. All the experiments were performed at isothermal conditions at 298 K. Two different crossflow velocities were used, depending on the membrane installed: 6.14 m/s with the 20 nm pore-sized membrane and 6.40 m/s with the 45 nm pore-sized membrane.

Transmembrane pressure was taken as the average between the inlet and outlet values in the membrane module. The volumetric permeate flow was measured by timing and weighing the permeate with a precision balance with an error lower than $\pm 1 \times 10^{-2}$ g.

The concentration of valeric acid in the permeate was determined by titration with a standard 0.01 M NaOH solution using phenolphthalein as indicator. The concentration of valeric acid in the retentate was obtained by determining the acid content in both the aqueous and the organic phases after settling. The acid concentration in the organic phase was determined by a two-phase titration: 1 mL of the organic phase was mixed with 7 mL of ethanol and 3 mL of water and was titrated with the NaOH solution (6). A maximum error of 2% was observed in the mass balance closure.

The extraction performance was determined by the degree of extraction, *E*, defined as

$$E = \left[1 - \frac{C_{\mathrm{HA}_{(\mathrm{w})}}}{C_{\mathrm{HA}_{(\mathrm{w})\mathrm{i}}}}\right] \times 100 \qquad (1)$$

where $C_{\mathrm{HA}_{(\mathrm{w})}}$ is the analytical equilibrium concentration of the acid in all its possible forms in the permeate phase, and $C_{\mathrm{HA}_{(\mathrm{w})\mathrm{i}}}$ its initial concentration in the feed solution.

Both membranes were always rinsed after the experiment with filtered (1 μm) tap water, and then cleaned with 0.5 M NaOH and 0.1 M nitric acid solutions consecutively. A final rinse with filtered tap water was always carried out. This cleaning procedure restored the initial water flux.

RESULTS AND DISCUSSION

Experimental results at constant concentration using the 20-nm pore-sized membrane, Fig. 1, show the influence of the applied pressure on the permeate flux once steady-state is reached. Permeation fluxes increase linearly with pressure at the low pressure range, while at higher pressures they tend to reach a limiting value. Figure 1 shows that the water–kerosene system gave lower permeation rates than the water–TBP–kerosene system. This is due to the physical characteristics of the emulsion (7).

When kerosene was used as the solvent, a fine emulsion was formed; however, the addition of TBP increased the drop size of the emulsion, thus lowering the resistance on the membrane and improving the permeate flux. Photographs of these two emulsions, one with kerosene and the other with TBP dissolved in kerosene (10 vol%), are shown as Figs. 2A and 2B, respectively. In both cases the total load of the organic phase was 20 vol%. Under these conditions the size of the larger drops increased from 20 to 70 μm with the addition of TBP. These results show that the smaller drops in a feed are predominantly responsible for membrane fouling (8–10).

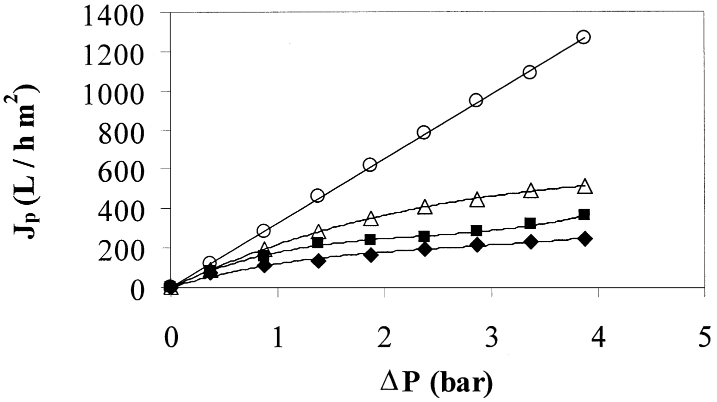

FIG. 1 Influence of transmembrane pressure on permeation flux with the 20-nm pore-sized membrane, crossflow velocity 6.14 m/s: (○) pure water, (△) valeric acid solution (6 g/L), (◆) valeric acid solution (6 g/L) with 20 vol% kerosene, (■) valeric acid solution (6 g/L) with 20 vol% (TBP + kerosene, 10 vol%).

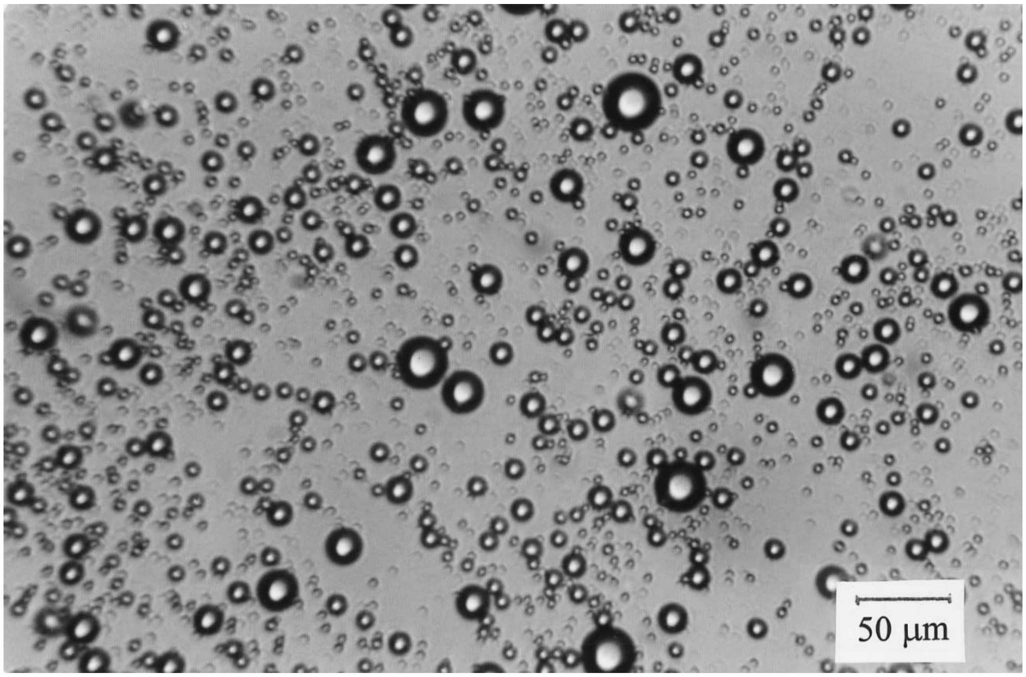

FIG. 2a Emulsion photograph of valeric acid aqueous solution, 6 g/L, and 20 vol% kerosene.

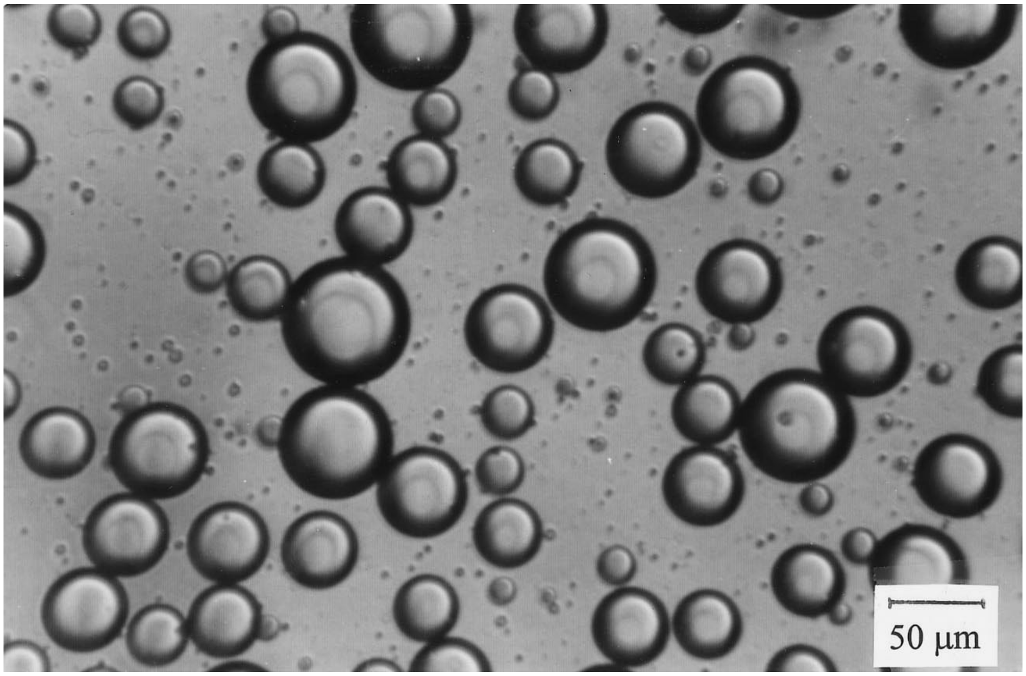

FIG. 2b Emulsion photograph of valeric acid aqueous solution, 6 g/L, and 20 vol% TBP + kerosene (10 vol%).

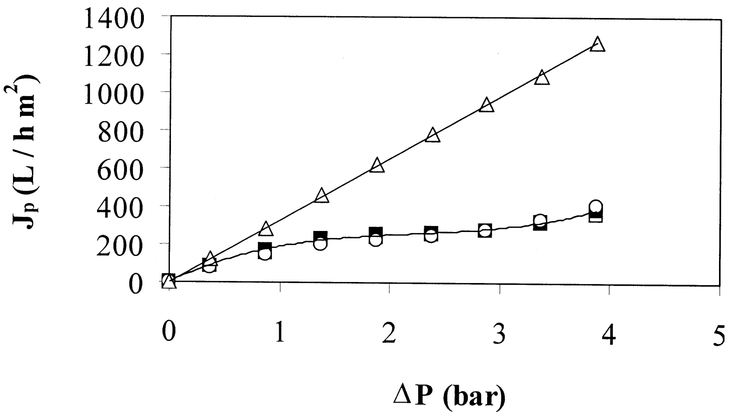

FIG. 3 Effect of the organic load on permeation flux with the 20-nm pore-sized membrane, crossflow velocity 6.14 m/s: (△) water, (□) 20 vol% organic load (TBP + kerosene, 10 vol%), (■) 30 vol% organic load (TBP + kerosene, 10 vol%), (○) 20 vol% organic load (TBP + kerosene, 30 vol%); (●) 30 vol% organic load (TBP + kerosene, 30 vol%) not shown because of overlapping symbols in the lower line.

Permeation rates were not influenced by the organic load, as shown in Fig. 3. However, the concentration of TBP had a marked effect on the degree of extraction, as shown in Fig. 4. The degree of valeric acid extraction in the organic phase increased from 66.1% for TBP 10% in kerosene at an organic load of 20%, to 91.3% for TBP 30% in kerosene at an organic load of 30%. Transmembrane pressure did not affect the degree of extraction.

As previously shown (5), temperature has almost no effect on the extraction efficiency and TBP 30% in kerosene yields the best results.

Runs at constant concentration with the 45-nm pore-sized membrane were carried out with TBP dissolved in kerosene (30 vol%) at a total organic load of

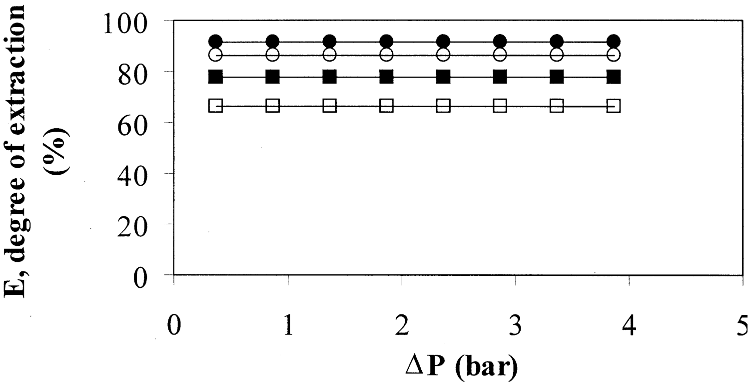

FIG. 4 Degree of extraction of valeric acid using several solvent concentrations and the 20-nm pore-sized membrane, crossflow velocity 6.14 m/s: (□) 20 vol% organic load (TBP + kerosene, 10 vol%), (■) 30 vol% organic load (TBP + kerosene, 10 vol%), (○) 20 vol% organic load (TBP + kerosene, 30 vol%), (●) 30 vol% organic load (TBP + kerosene, 30 vol%).

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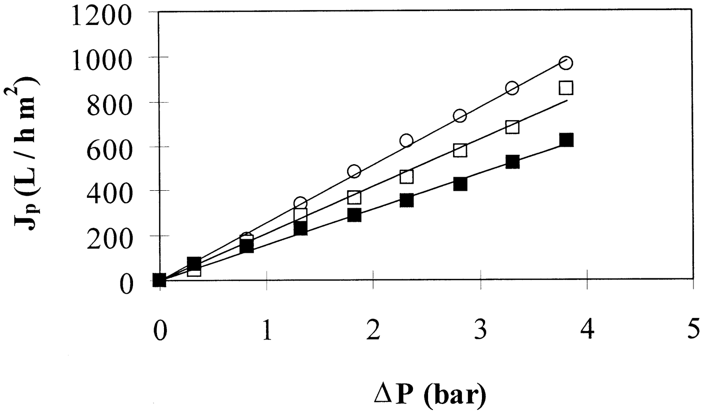

FIG. 5 Dependence of transmembrane pressure on permeation flux with the 45-nm pore-sized membrane, crossflow velocity 6.40 m/s: (○) pure water, (□) water with 30 vol% (TBP + kerosene, 30 vol%), (■) valeric acid solution (6 g/L) with 30 vol% (TBP + kerosene, 30 vol%).

30 vol%. The results showed a linear increase in the permeate flux with pressure (Fig. 5). This means that flux was in the pressure-controlled region and that permeation rates were lower when the emulsion contained valeric acid. This effect was not observed with the smaller pore-size membrane. Moreover, when valeric acid was not added and the larger pore-size membrane was used, a 100% rejection of the organic phase was not achieved under transmembrane pressures of 3.25 and 3.85 bar. The addition of valeric acid to the emulsion modifies the interfacial tension between the organic and the membrane surface, and it prevents the passage of the organic phase through the membrane.

As might be expected, permeation fluxes were higher for the larger pore-size membrane. Comparative results are shown in Fig. 6 for the system 30

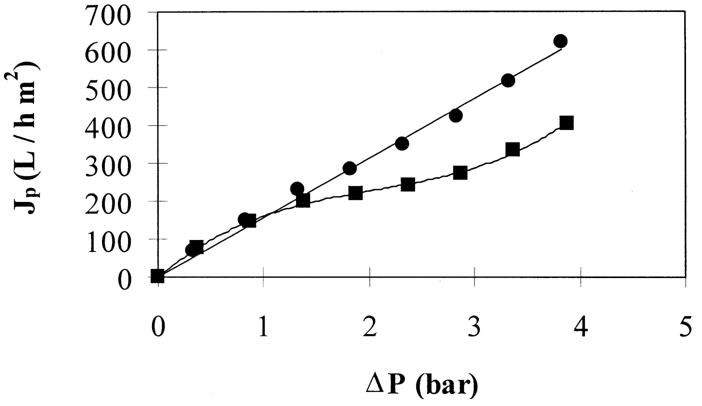

FIG. 6 Effect of the membrane pore on permeation flux for the extraction of valeric acid with 30 vol% (TBP + kerosene, 20 vol%): (●) 45 nm pore-sized membrane, crossflow velocity 6.40 m/s; (■) 20 nm pore-sized membrane, crossflow velocity 6.14 m/s.

vol% TBP dissolved in kerosene at an organic load of 30 vol%. When valeric acid was present, the organic phase was completely rejected by both membranes in the pressure range tested and, consequently, a clear aqueous permeate was obtained. The degree of extraction was similar for both membranes (91.3%) and independent of pressure.

Fouling was more severe with the larger pore-size membrane, and a longer cleaning process was needed. Fouling occurred in this case through a combination of surface and internal pore blockage (8, 11).

The concentration experiment was carried out with the 45-nm pore-sized membrane and TBP in kerosene (30 vol%) at an initial organic load of 30 vol% under a transmembrane pressure of 2.85 bar. The aqueous phase was continuously removed, and the organic concentration of the emulsion was increased from an initial value of 30 vol% to 75 vol% after 2.50 hours of operation, as shown in Fig. 7. Higher concentrations could not be reached because the organic phase began to permeate the membrane. The increase in organic load affects the interfacial tension, which allows the organic droplets collected upon the pores to coalesce and spread over the surface of the membrane, thereby causing fouling (1). At higher organic phase ratios the flux decreased, but the values were still higher than 400 L/h·m^2.

The continuous experiment was carried out adding fresh valeric acid solution to the system at the same flow rate as the permeate was removed in order to keep the volume in the feed tank constant. When the 45-nm pore-sized membrane and the extractant TBP 30 vol% dissolved in kerosene with an organic load of 30 vol% were used, the transmembrane pressure was 2.85 bar.

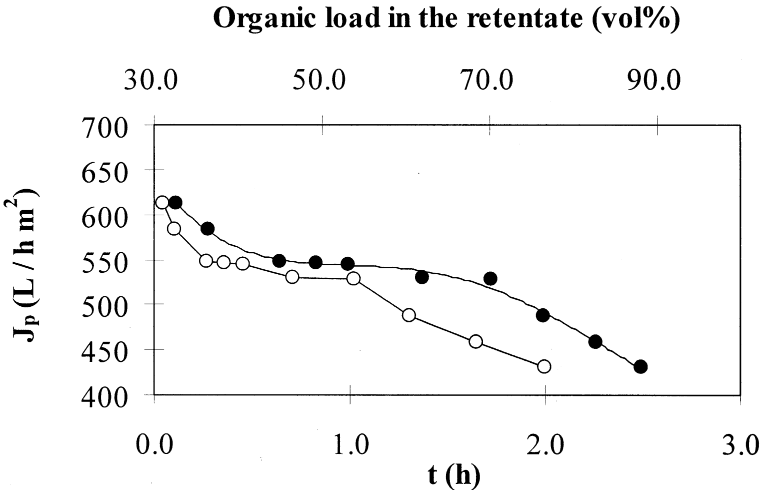

FIG. 7 Influence of time (●) and the organic load (○) in a concentration experiment with the 45-nm pore-sized membrane. Aqueous feed 6 g/L of valeric acid solution, initial organic load 30 vol% TBP + kerosene (30 vol%), pressure 2.85 bar, and crossflow velocity 6.40 m/s.

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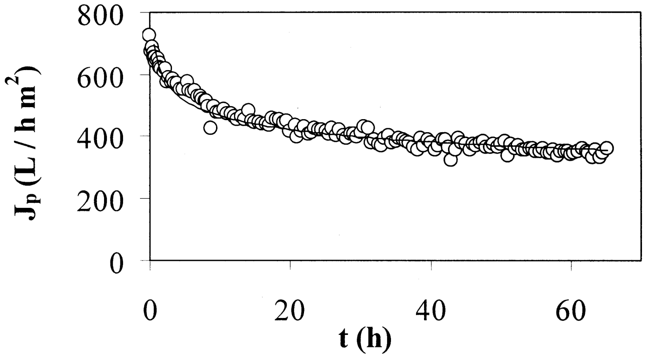

FIG. 8 Effect of time on the permeate flux in a continuous experiment with the 45-nm pore-sized membrane. Aqueous feed 4.5 g/L of valeric acid solution, organic load 30 vol% TBP + kerosene (30 vol%), pressure 2.85 bar, and crossflow velocity 6.40 m/s.

Figure 8 shows that after a continuous flux decline, an asymptotic behavior is observed.

Extraction of valeric acid decreased with time, yielding permeates with increasing concentrations of valeric acid, as shown in Fig. 9.

The concentration of valeric acid in the organic phase was calculated from the mass balance with the assumption of a well-mixed tank. Assuming a 100% rejection of the organic phase and a 0% rejection of the aqueous phase by the membrane, i.e., the permeate concentration is equal to the continuous phase concentration in the emulsion, the mass balance can be expressed by the following equation:

$$Q_f C_f - J_p A\, C_p = \frac{d[V_{(w)} C_p]}{dt} + \frac{d[V_{(o)} C_{HA_{(o)}}]}{dt} \tag{2}$$

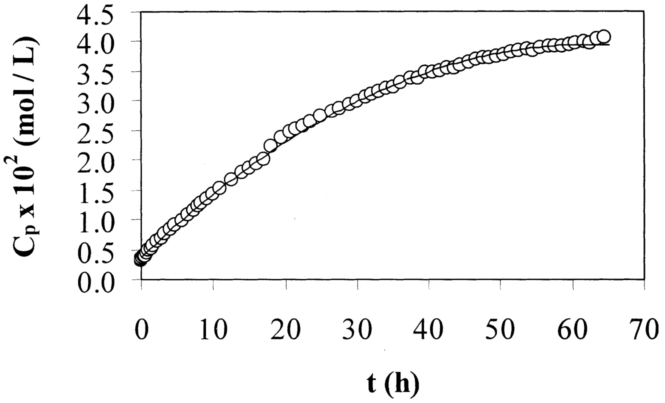

FIG. 9 Valeric acid concentration in the permeate, C_p, as a function of time in a continuous experiment with the 45-nm pore-sized membrane. Aqueous feed 4.5 g/L of valeric acid solution, organic load 30 vol% TBP + kerosene (30 vol%), pressure 2.85 bar, and crossflow velocity 6.40 m/s.

where Q_f is the feed flux, J_p is the permeate flux, A is the area of the membrane, C_f is the feed concentration (0.044 mol-g/L), C_p is the permeate concentration, $C_{HA_{(o)}}$ is the organic concentration, t is the operation time, and $V_{(w)}$ and $V_{(o)}$ are the aqueous and organic volumes in the tank, respectively.

Volume change due to extraction solvent solubility in the aqueous phase ($S = 0.00035$ L/L) was considered to be given by the following equations:

$$d[V_{(o)}]/dt = -J_p A S \qquad (3)$$

$$d[V_{(w)}]/dt = Q_f - J_p A(1 - S) \qquad (4)$$

The experiment was started under total recirculation conditions where a 4.5-g/L solution was mixed with the organic phase. In this case the initial conditions were $C_p = 0.0038$ mol-g/L and $C_{HA_{(o)}} = 0.123$ mol-g/L, which correspond to $E = 91.3\%$, $V_{(o)} = 3$ L, and $V_{(w)} = 9.19$ L. Equation (2) was solved numerically and the results are shown in Fig. 10.

In a previous paper (5), a model for the equilibrium extraction of valeric acid from aqueous solutions with TBP dissolved in kerosene was given by the expression

$$C_{HA_{(o)}} = P[\mathrm{HA}]_{(w)} + 2K_d P^2[\mathrm{HA}]^2_{(w)} + 3K_d K_t P^3[\mathrm{HA}]^3_{(w)} + \frac{K_c[\mathrm{HA}]_{(w)}[\mathrm{TBP}]_{(o)T}}{(1 + K_c[\mathrm{HA}]_{(w)})} \qquad (5)$$

where $C_{HA_{(o)}}$ is the total equilibrium concentration of valeric acid in the organic phase, $[\mathrm{HA}]_{(w)}$ is the equilibrium concentration of undissociated acid in the aqueous phase, $[\mathrm{TBP}]_{(o)T}$ is the total concentration of TBP in the organic phase, P is the partition constant of the undissociated acid between the organic

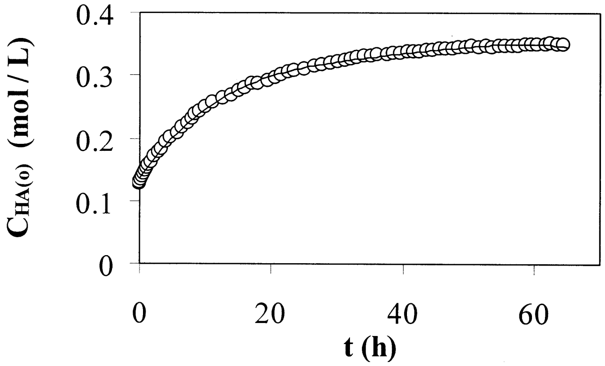

FIG. 10 Estimated valeric acid concentration in the organic phase, $C_{HA(o)}$, as a function of time in a continuous experiment with the 45-nm pore-sized membrane. Aqueous feed 4.5 g/L of valeric acid solution, organic load 30 vol% (TBP + kerosene, 30 vol%), pressure 2.85 bar, and crossflow velocity 6.40 m/s.

phase and the aqueous phase, K_d and K_t are the dimerization and trimerization constants of the valeric acid in the organic phase, and K_c is the equilibrium constant for the formation of the acid–TBP complex. All concentrations are expressed as mol-g/L.

Parameters in Eq. (5) were estimated using the Levenberg–Marquardt algorithm. These parameters depend on the extractant/diluent ratio, and for a solvent 30 vol% of TBP in kerosene, $[\mathrm{TBP}]_{(o)T} = 1.1$ mol/L, the following values were obtained (5): $P = 2.014$, $K_d = 4.561\ \mathrm{L{\cdot}mol^{-1}}$, $K_t = 0.217\ \mathrm{L{\cdot}mol^{-1}}$, and $K_c = 24.508\ \mathrm{L{\cdot}mol^{-1}}$.

Figure 11 shows a comparison between the valeric acid concentration in the organic phase as estimated by Eq. (2) and the theoretical equilibrium concentration in the organic phase as calculated by Eq. (5) with respect to the concentrations of the undissociated valeric acid in the permeate, all expressed as mol-g/L.

The concentrations of undissociated valeric acid in the permeate were calculated by taking into account the partial dissociation of the acid in the aqueous phase ($\mathrm{p}K_a = 4.86$) and pH values. Permeate pHs ranged from 3.67 to 3.09 over the 65-hour test. Figure 11 shows that the concentration of valeric acid in the organic phase in the first hours was the equilibrium concentration. The organic concentration moves away from equilibrium conditions with time, indicating that the residence time is not long enough for the extraction process to be completed due to a slower extraction process because the organic phase is already loaded. The extraction performance would be higher by reducing the permeate flow.

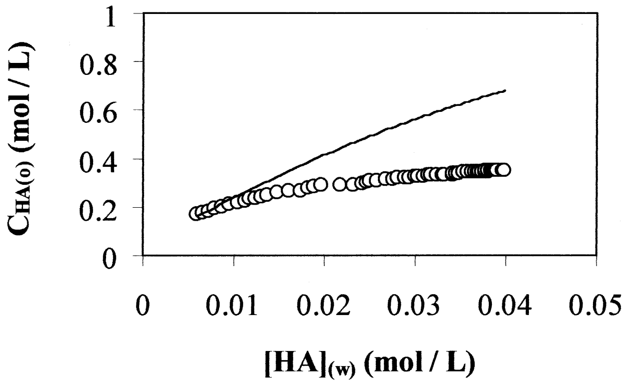

FIG. 11 Comparison between the estimated valeric acid concentrations in the organic phase and the simulated equilibrium concentrations, $C_{\mathrm{HA}_{(o)}}$, as a function of the undissociated valeric acid in the permeate, $[\mathrm{HA}]_{(w)}$, in a continuous experiment with the 45-nm pore-sized membrane. Aqueous feed 4.5 g/L of valeric acid solution, organic load 30 vol% (TBP + kerosene, 30 vol%), pressure 2.85 bar, and crossflow velocity 6.40 m/s. (○): Values estimated by mass balance, Eqs. (2)–(4). (Solid curve): Equilibrium concentrations simulated by Eq. (5).

CONCLUSIONS

For the recovery of valeric acid from aqueous solution with TBP–kerosene, EUF permeation rates were higher with the 45 nm pore-sized membrane than with that of 20 nm under identical conditions of temperature, feed concentration, and transmembrane pressure.

The addition of TBP to the organic phase increases the oil drop size of the emulsion, lowering the resistance on the membrane and improving the permeate flux.

The concentration of TBP has a marked effect on the degree of valeric acid extraction. The extraction of valeric acid in the organic phase reached a value of 91.26% when the solvent was 30 vol% of TBP dissolved in kerosene at an organic load of 30 vol%. Transmembrane pressure did not affect the degree of extraction in any case.

There was a total organic phase rejection by both membranes when the emulsion contained valeric acid and, consequently, a clear aqueous permeate was obtained in the range of pressures tested. However, when valeric acid was not present, a 100% rejection of the organic phase was not achieved with the 45 nm pore-sized membrane under transmembrane pressures of 3.25 and 3.85 bar. This implies that valeric acid modifies the interfacial tension between oil and the membrane surface, preventing the oil droplets from passing through the membrane pores.

In the concentration experiment the aqueous phase was recovered and the organic phase was concentrated up to 75 vol% at a permeation rate close to 430 $L/h{\cdot}m^2$.

In the continuous process with the 45-nm pore-sized membrane, the permeation flux remained constant over a 65-hour test. Neither fouling nor saturation was achieved after this time. Valeric acid concentration in the organic phase, estimated by a mass balance, was 51.6% of the equilibrium value after running the process for 65 hours.

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