Desalination and Water Treatment
www.deswater.com

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doi: 10/5004/dwt.2011.2075

27 (2011) 123–129
March

Effect of liquid flow on the separation of propylene/propane mixtures with a gas/liquid membrane contactor using Ag^+-RTIL solutions

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Received 2 June 2010; Accepted 30 October 2010

ABSTRACT

Industrial olefin/paraffin separations heavily rely upon energy intensive distillation-based technologies, which represent a class of the most important and also the most costly processes in the chemical industry. The method of olefin/paraffin separations certainly holds an enormous potential for capital and energy cost savings if a more efficient technique is developed. A module containing a microporous hydrophobic membrane has been assessed for their capability in the selective removal of propylene from a gas mixture with propane. A study of mass transfer in the membrane module indicates that the overall mass transfer coefficients, $K_{overall}$, are dominated by the individual coefficients in the liquid film, k_L. A model based on the resistance-in-series model and Sherwood correlations could describe this phenomenon.

Keywords: Propylene; Propane mixtures; Silver; $BMImBF_4$; Reactive absorption; Membrane contactor

1. Introduction

Light olefins such as ethylene and propylene are important chemical precursors that play an essential role in many chemical syntheses and industrial processes. Common low-temperature distillation technology for separation of olefins from paraffin streams represents one of the most important, but also the most costly, processes in the petrochemical industry. Development of economically viable propylene/propane separation processes is becoming increasingly important, but it is extremely challenging due to the physicochemical similarities between those two molecules [1,2]. Among a number of alternatives separation processes, reactive absorption of olefins from olefin/paraffin mixtures using a silver salt solution as absorption liquid in a gas/liquid membrane contactor may be attractive in this respect [3,4].

Olefins can form reversible complexes with transition-metal cations (Ag^+) via π-bound complex formation mechanism which can easily be reversed by pressure and temperature swings [5,6]. Room temperature ionic liquids (RTIL) are proposed as potential substitutes for conventional solvents as reaction media because in addition to their renowned and remarkable properties [7–9], such as a negligible volatility, which enables a relatively easy recovery, a wide range of polarities, non-flammable compounds, and high chemical and thermal stability, they present a strong affinity for the olefinic compound compared to saturated hydrocarbons and provide stability to the metal cation dissolved or suspended in the solvent. Besides, their lack of volatility gives ionic liquids the feature that they can perform gas separations without solvent losses or gas stream pollution.

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Presented at the VII Ibero-American Conference on Membrane Science and Technology (CITEM 2010), April 11–14, 2010, Sintra, Portugal

In previous works, the physico-chemical characteristics of the system, absorption equilibrium and kinetics of the reaction between propylene and silver in 1-butyl-3-methylimidazolium tetrafluoroborate ($BMImBF_4$), required to design industrial separation processes with gas-liquid membrane contactors were studied by our research group [10,11]. In the present study, a membrane contactor was used in the propane/propylene selective absorption. The hollow fiber module contains a microporous hydrophobic membrane which offers no selectivity towards gas components in the mixture employed, but provides the module with a large surface area per unit volume. The gas mixture, containing propylene in the range of 30–70% and the balance was propane, was introduced into the hollow fiber lumen, while a silver salt-ionic liquid mixture used as an absorbing medium was fed into shell side because such a viscous liquid would cause a too high pressure drop if circulated in the tube side.

Since the solubility of propylene in the absorbing medium is much higher than that of propane, the propylene in the gas mixture may, therefore, be preferentially removed.

In a previous work [12] we found that the liquid-phase boundary-layer resistance was a significant contributing factor to the overall mass-transfer resistance. In the present work we report additional results, analyzing the influence of the liquid flow rate on the absorption rate.

2. Theory

The chemical reactions between C_3H_6 and the Ag^+ cations dissolved in ionic liquids have been described as [10]

$$Ag^+ + C_3H_6^{IL} \leftrightarrow Ag^+(C_3H_6)^{IL} \tag{1}$$

$$Ag^+(C_3H_6)^{IL} + C_3H_6^{IL} \leftrightarrow Ag^+(C_3H_6)_2^{IL} \tag{2}$$

Propylene is absorbed from the gas phase into the liquid phase following a Henry's isotherm, and subsequently it reacts with the dissolved silver ions to form the primary (Eq. (1)) and secondary complexes (Eq. (2)).

The propylene absorption flux, $J_{C_3H_6}$, is calculated by performing a mass balance over the contactor, based on the measured changes in the propylene composition in gas flow rate between the inlet and outlet of the membrane module at any time, assuming propane absorption negligible.

$$J_{C_3H_6} = \frac{1}{A}\left(F_{in} \bullet y_{C_3H_6 in} - F_{out} \bullet y_{C_3H_6 out}\right) \tag{3}$$

Experimentally determined mass transfer flux and observed concentration change from the feed to the outlet of the module in the gas phase can be used to calculate the experimental overall mass transfer coefficient.

$$J_{C_3H_6} = K_{overall}\,\frac{\Delta y_{C_3H_6,lm} \bullet P_T}{R \bullet T} \tag{4}$$

P_T is the total pressure in the gas phase and $\Delta y_{C_3H_6,lm}$ is the logarithmic mean of the driving force based on gas phase molar fractions and taking into account the propylene concentration at the inlet ($y_{C_3H_6(g),in}$) and outlet ($y_{C_3H_6(g),out}$) of the contactor.

For the gas-liquid membrane contacting process, the mass transfer flux ($J_{C_3H_6}$) at steady state is given by Eq. (5) based on the concentration difference driving forces [13].

$$\begin{aligned} J_{C_3H_6} &= k_g(C_{C_3H_6,g} - C_{C_3H_6,g,mem}) \\ &= k_{mg}(C_{C_3H_6,g,mem} - C_{C_3H_6,g,int}) \\ &= k_i\ (C_{C_3H_6,l,int} - C_{C_3H_6,l}) = K_{overall}\Delta C_{C_3H_6 lm} \end{aligned} \tag{5}$$

where $K_{overall}$ is the overall mass transfer coefficient (m/s), k_g, k_{mg}, and k_l are the individual mass transfer coefficients of the gas phase, membrane and liquid phase, respectively.

The resistance in series approach can be used to relate individual mass transfer resistances to the overall mass transfer resistance.

$$R_{overall} = R_g + R_{mg} + R_l \tag{6}$$

For a hollow fiber configuration with the liquid phase flowing in the shell side and the gas phase flowing through the lumen side the gas-liquid interface is located at the outer diameter of the tubes, in non-wetting mode. The resistances in series approach accounting for a chemical reaction in the liquid side (expressed by the enhancement factor, E_A) leads to,

$$\frac{1}{K_{overall}} = \frac{d_o}{k_g d_i} + \frac{d_o}{k_{mg} d_{lm}} + \frac{1}{k_l H_{adi} E_A} \tag{7}$$

where H_{adi} represents the dimensionless Henry's constant ($H_{adi} = C_{C_3H_6,L} / C_{C_3H_6,G} = H_{C_3H_6}RT$), and d_o, d_i, and d_{lm} are the inner, outer, and logarithmic mean diameters of the fibers.

Eq. (8), proposed by Hausen [14] for laminar flow and low *Gz* number, has been used for the estimation of the individual mass transfer coefficients within the fibers,

$$Sh = \frac{k_g d_i}{D_{C_3H_6,b}} = 3.66 + \frac{0.0668 \bullet Gz}{1 + 0.04 \bullet Gz^{2/3}} \tag{8}$$

For a porous membrane, the mass transfer coefficient when the pores are completely filled by the gas phase has been calculated using the following equation:

$$k_{mg} = \frac{D_{eff,g}}{\delta} = \frac{D_{C_3H_6,g} \cdot \varepsilon}{\tau \cdot \delta} \tag{9}$$

where $D_{eff,g}$ is the effective diffusion coefficient of propylene through the gas phase that fill the pores, which is a combination between the molecular diffusion coefficient, $D_{C_3H_6,b}$, and Knudsen diffusion. ε, τ, and δ are the porosity, tortuosity and thickness of the membrane respectively.

In contrast to tube side flow, the shell-side liquid flow has not been clearly understood yet. There have been a lot of reports on the experimental and analytical studies on the shell-side mass-transfer [15]. However, the results vary significantly, possibly arising from the irregularity of the fiber spacing, the polydispersity of fiber diameters, and the influence from the module wall as well as inlet and outlet effects. Despite the differences, a general empirical correlation arrives for parallel-flow hollow fiber membrane contactors:

$$Sh = \frac{k_l d_h}{D_{C_3H_6,l}} \propto f(\varphi) Re^{\alpha} Sc^{\beta} \left(\frac{d}{L}\right)^{\gamma} \tag{10}$$

where φ is the packing density; α, β, and γ are constants; *Sh* is the Sherwood number; *Sc* is the Schmidt number; and *Re* is the Reynolds number. Diffusion parameters and Henry´s law constants at 298 K needed in the correlations were obtained in previous works [10–12].

3. Experimental

3.1. Materials

Propylene and propane gases were purchased from Praxair and their purity was of 99.5%. The RTIL used in this study was 1-butyl-3-methylimidazolium tetrafluoroborate (CAS number: 174501-65-6) from Iolitec, with a minimum purity of 99% and residual halide content less than 500 ppm. Reactive media were prepared using silver tetrafluoroborate $AgBF_4$ of 99% purity (Apollo Scientific Ltd.) dissolved in RTIL at room temperature. All chemicals were used as received.

3.2. Methods

The absorption experiments were carried out in a laboratory-scale unit previously used by the authors in other studies. The experimental setup is described in detail elsewhere [12]. A membrane contactor was used in the propane/propylene selective absorption; the characteristics of the membrane module are compiled in Table 1. A propylene-containing mixture was contacted with the ionic liquid/silver salt solution, and propylene was absorbed. After the absorption step, where the outlet gas stream leave the membrane contactor rich in propane, propylene was recovered by desorption in the regeneration step. Concerning the membrane contactor unit, a hollow-fiber module (Enka-Microdyn) with hydrophobic microporous polypropylene hollow-fiber membranes, with a nominal pore size of 0.20 μm, was used. The absorbing liquid selected circulated through the shell-side.

Table 1
Characteristics of the membrane module

Membrane material	Polypropylene
Housing material Polypropylene	Polypropylene
Inner diameter of the shell (m)	0.02
Inner diameter of the hollow fibers (m)	5.5×10^{-3}
Wall thickness (m)	1.55×10^{-3}
Nominal pore diameter (m)	0.2×10^{-6}
Porosity (%)	75
Number of hollow fibers	3
Module length (m)	0.75
Effective membrane area (m^2)	0.036
Packing factor	0.55
Tortuosity (assumed as 1/ε)	1.4

The gas flow rate of 16.66 ml/min used was flowed at 1.2 bar and 298 K. (Brooks Instrument MFC 5850, Emerson Process Management, Spain). In order to prevent mixing of the phases, the liquid absorbent flows at a pressure of 0.05 bar higher than that of the gas phase. The fluid-dynamic control of the phases is easier to maintain at low pressures.

Compositions in the outlet gas streams of propylene/propane were analysed every 2 min using a HP 6890 gas chromatograph (Agilent) equipped with an auto sampling gas valve, a TCD detector and with a semicapillary column (length 50 m, nominal diameter 0.53 mm, model HP AlS). Moreover, the regeneration step of the Ag^+-BMImBF$_4$ reactive medium after the C_3H_6 absorption was carried out at room temperature through the external vacuum pump (Telstar 2P3) connected to the storage tank in the experimental setup which was used to achieve the vacuum pressure value (<2 mbar) and flowing the absorbent liquid in a closed circuit in the storage tank.

Two replicates of each experiment were performed and the experimental error was determined. The weighted standard deviation of experimental data was calculated leading to values of $\sigma_w = 1.8$ % and concluding that the experiments were replicable.

4. Results and discussion

Separation results of propane/propylene gas mixtures based on the reactive absorption of propylene in a Ag^+-BMImBF$_4$ [0.25 M] medium reported in a previous work using the hollow fiber membrane module MD020 TP 2N for the operational conditions selected (Q_L = 300 ml/min) proved that the membrane contactor presents a severe limitation to the mass transfer in the liquid side and the performance of the microporous contactor is controlled by mass transfer rate in the liquid phase.

In order to improve the mass transfer rate in the membrane contactor, the hydrodynamics of the liquid phase has been enhanced in this study. Fig. 1 shows the effect of the absorbing liquid flow rate on the change in the gas stream composition between the inlet and outlet of the membrane module versus time working with constant inlet gas flow rate (50–50 % v/v C_3H_6–C_3H_8) and Ag^+-BMImBF$_4$ [0.25 M] as reactive medium at 298 K.

Pseudo-steady-state is obtained at initial times remaining practically constant the outlet composition in the gas phase and mass transfer flux of propylene because a large amount of liquid is continuously recycled into the process and it is very far from saturation. Table 2 collects the experimental molar fluxes of propylene absorbed and overall mass transfer coefficients for all the experiments performed at different liquid volumetric flow rate at 298 K obtained according to Eqs. (3) and (4).

Absorption rate increased with increasing the liquid flow rate. This is because an increase of liquid flow rate results in the decrease of the boundary layer thickness on the liquid side. Hence, this leads to the decrease the resistance of the liquid phase, and the increase in the mass transfer rate. For a feed composition of 50% v/v of C_3H_6/C_6H_8, the propylene flux is enhanced from 4.10 10^{-5} mol/m^2/s to 11.15 10^{-5} mol/m^2/s when the liquid flow rate increases from 300 ml/min to 900 ml/min showing a remarkable improvement in the overall mass transfer coefficient.

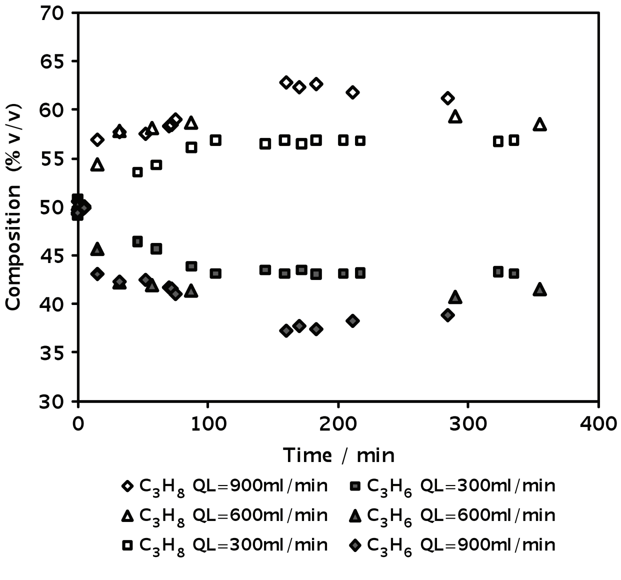

Fig. 1. Evolution of the outlet gas stream compositions in the membrane module versus time for constant inlet gas flow rate (50–50 % v/v C_3H_6–C_3H_8) at 298 K and different liquid volumetric flow rate Q_L.

The Wilson plot is the technique to experimentally determine the membrane mass transfer resistance in the gas–liquid membrane contacting process based on the resistance-in-series model [16,17]. A plot of $1/K_{overall}$ versus $1/u_L^{\alpha}$ gives a straight line. The value of the empirical parameter, α, is chosen for achieving the best straight line. In the gas–liquid membrane contacting process, if the resistance in the gas phase is much smaller than the total resistance, the term $1/k_g$ in Eq. (7) becomes negligible. Thus, the y-intercept of the Wilson plot represents the value of membrane mass transfer resistance.

Fig. 2 shows the Wilson plot of $1/K_{overall}$ versus $u_L^{-0.54}$ for Ag^+-BMImBF$_4$ [0.25 M] and propylene/propane mixtures systems using the experimental data collected in Table 2. The equation which correlated the overall mass transfer resistance with individual mass transfer resistances is as follows,

$$\frac{1}{K_{overall}} = 86583 u_L^{-0.54} + 2873 \quad (R^2 = 0.9999; S_{y/x} = 1539) \tag{11}$$

According to the above equation, the mass transfer resistance ($d_o/k_{mg}\, d_{lm}$) of the membrane used in this work can be obtained from the y-intercept value, i.e., 2873 s/m, which represents less than 1% of the overall resistance for all the liquid flow rate studied in this work, indicating that the resistances of the gas film and the membrane are negligible and that the overall mass transfer coefficient is solely controlled by the diffusional resistance in the liquid film.

In general shell side flow parallel to the fibers is not clearly understood. Several correlations have been proposed in the literature over the past few years, but none is applicable to a wide range of systems. Equations that

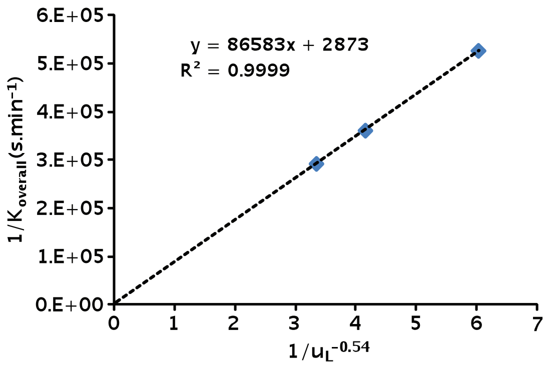

Fig. 2. Wilson plot – effect of velocity of absorbing liquid on overall mass transfer coefficient.

Table 2
Experimental propylene flux and overall mass transfer coefficients obtained in a membrane contactor at different liquid flow rate and 298 K in BMImBF$_4$ + AgBF$_4$ = 0.25M"

		$J_{C_3H_6} \times 10^5$ mol/m^2/s		
		Q$_L$ = 300 ml/min Re = 2.75	Q$_L$ = 600 ml/min Re = 5.50	Q$_L$ = 900 ml/min Re = 8.25
Feed gas	70% propane-30% propylene	2.06	–	–
Composition	50% propane-50% propylene	4.10	9.33	11.15
(% v/v)	30% propane-70% propylene	5.61	–	–
	$K_{overall} \times 10^6$ m/s	1.90	2.77	3.42

include geometrical dependence have been used successfully to describe the mass transfer coefficient outside of the fiber.

Yang and Cussler [18]:

$$Sh = \frac{k_l d_h}{D_{C_3H_6,l}} = 1.25\left(\frac{d_h^2 u}{Lv}\right)^{0.93}\left(\frac{v}{D_{C_3H_6,l}}\right)^{1/3} \quad (12)$$

Prasad and Sirkar [19]:

$$Sh = 5.8\left[(1-\phi_F)\frac{d_h}{L}\right] \bullet \left(\frac{d_h u}{v}\right)^{0.6}\left(\frac{v}{D_{C_3H_6,l}}\right)^{1/3} \quad (13)$$

Wu and Chen [20]:

$$Sh = \left(0.3045\phi_F{}^2 - 0.3421\phi_F + 0.15\right) \bullet \left(\frac{d_h u}{v}\right)^{0.9}\left(\frac{v}{D_{C_3H_6,l}}\right)^{1/3} \quad (14)$$

Costello et al. [21]:

$$Sh = (0.53 - 0.58\phi_F) \bullet \left(\frac{d_h u}{v}\right)^{0.53}\left(\frac{v}{D_{C_3H_6,l}}\right)^{1/3} \quad (15)$$

Li et al. [22]:

$$Sh = 1.164 \bullet \left(\frac{d_h^2 u}{Lv}\right)^{0.967}\left(\frac{v}{D_{C_3H_6,l}}\right)^{1/3} \quad (16)$$

Here, φ is the packing factor, which for the membrane contactor used in this work takes a value of 0.55. In order to compare the experimental results obtained with the membrane contactor and the predicted results using Eqs. (12)–(16) in Fig. 3 the liquid mass transfer coefficients, k_l, have been expressed in terms of Sherwood number against the absorbing liquid velocity in terms of Reynolds number. According to Eq. (7) mass transfer coefficients in the liquid film, k_l, can be obtained from the experimental overall mass transport coefficients through the dimensionless Henry´s constants, H_{adi}, and the enhancement factor, E_A, values.

The enhancement factors (E_A) that quantify the chemical reaction influence, have been calculated assuming instantaneous chemical reaction by Eq. (17), which was previously proved for the propylene absorption in the reactive medium Ag$^+$-BMImBF$_4$ with a silver salt concentration of 0.25 M.

$$E_{A\infty} = \left(1 + \frac{D_{Ag+}C_{Ag+}}{D_{C_3H_6}C_{C_3H_6}^{int,IL}}\right)\left(\frac{D_{C_3H_6}}{D_{Ag+}}\right)^{0.5} \quad (17)$$

where $C_{C_3H_6}^{int,IL}$ is the concentration of propylene at the interface, C_{Ag^+} is the concentration of silver ions in the liquid bulk phase, $D_{C_3H_6}$ and D_{Ag^+} are the diffusion coefficients of propylene and silver ions in the liquid phase.

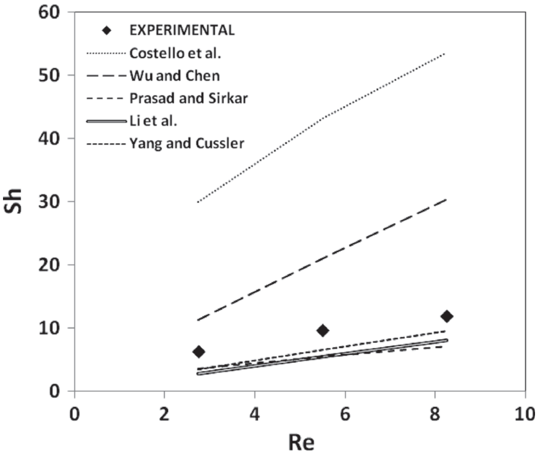

Fig. 3. Comparison between experimental results and reported correlations.

The enhancement factor calculated for the membrane contactor system at the working experimental conditions using Eq. (17) is $E_A = 7.8$.

Fig. 3 shows the observed data obtained respect the estimated values from the literature correlations. Experimental values of the Sherwood number for the Reynolds number range studied give close results that Eqs. (12), (13) and (16) predict. Sherwood numbers calculated with the Wu and Chen correlation deviate significantly from the experimental values when the Reynolds number increases whereas the Costello et al. prediction doesn´t match with the experimental values. The reason for such a deviation may be due to different range of Reynolds number used in this study, with respect to that used in the other works for obtaining the proposed correlations.

Although turbulent flow can also be achieved in a membrane contactor, unfortunately, in practice, it may not be economically and operationally viable. In the first place, to maintain a turbulent flow, a larger power-consumption is needed. Secondly, under high liquid pressures the membrane might be wetted by the liquid. In this case, the mass-transfer will be greatly reduced because of the presence of a stagnant liquid film in the membrane pores. Further work by our research group will deal with the use of cross-flow membrane contactors with improved design to reduce boundary layer resistance.

5. Conclusions

The main mass transfer resistance in the gas-liquid membrane contacting process for the separation of propane/propylene gas mixtures based on the reactive absorption of propylene in Ag^+-BMImBF$_4$ medium was analyzed. The rate-controlling step was dominated by the diffusional resistance on the liquid film. The effect of the liquid flow rate on the absorption rate has been studied in order to improve the hydrodynamics of the liquid phase and reduce the mass transfer limitations. The absorption rates of propylene increase significantly with the increasing of liquid flow rates. Hence the overall mass transfer coefficient is enhanced from $1.9\ 10^{-6}$ m/s to $3.42\ 10^{-6}$ m/s when the liquid flow rate increases from 300 ml/min to 900 ml/min. Mass transfer resistances have been quantitatively determined by the resistance-in-series approach and the Wilson-plot method. Assuming that the mass transfer resistance in gas phase is negligible, it was observed that the membrane mass transfer resistance represents less than 1% of the total. The observed mass transfer coefficients were found to be adequately described by some correlations which include geometrical parameters in the appropiate Reynolds number range.

Acknowledgements

This research has been funded by the Spanish Ministry of Science and Innovation (Projects CTQ2008-00690/PPQ and CTM2006-00317). Marcos Fallanza thanks MICINN for the FPI fellowship.

Symbols

A — effective membrane area (m^2)
C — concentration (mol/l)
D_i — diffusion coefficient of species i (m^2/s)
d_h — hydraulic diameter (m)
d_i — inside diameter of the fiber (m)
F — gas molar flow (mol/s)
H — Henry’s law constants (mol/l/bar)
J — molar flux (mol m^2/s)
k_g — mass transfer coefficient in the gas phase (m/s)
k_l — liquid mass transfer coefficient (m/s)
k_{mg} — mass transfer coefficient on the membrane filled with gas (m/s)
$K_{overall}$ — overall mass transfer coefficient (m/s)
L — fibre length (m)
Q_L — liquid flow rate (ml/min)
R — ideal gas constant
R_g — resistance in the gas side (s/m)
R_l — resistance in the liquid side (s/m)
R_{mg} — gas filled membrane resistance (s/m)
$R_{overall}$ — overall resistance to mass transfer (s/m)
$S_{y/x}$ — standard error
T — feed temperature (K)
u_L — liquid velocity (m/s)
y — gas molar fraction
υ — cinematic viscosity (m^2/s)

Superscripts / Subscript

Ag^+ — silver
C_3H_6 — propylene
IL — ionic liquid

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