Non-dispersive extraction of Ge(IV) from aqueous solutions by Cyanex 923: Transport and modeling studies

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Abstract: The transport of germanium from an aqueous solution containing oxalic acid was studied using a flat sheet supported liquid membrane (FSSLM) system. Cyanex 923 immobilized in a polytetrafluoroethylene membrane was employed as a carrier. The solution chemistry and related diagrams were applied to study the transport of germanium. The effectual parameters such as oxalic acid, the carrier, and strip reagent concentrations were evaluated in this study. Based on the results, the oxalic acid concentration of 0.075 mol/L and the carrier concentration of 20 %v/v were the condition in which the efficient germanium transport occurred. Among strip reagents tested, NaOH had the best efficiency to transport germanium through the supported liquid membrane system. Furthermore, the permeation model was obtained to calculate the mass transfer resistances. According to the results, the values of 1 and 1345 s/cm were evaluated for Δm and Δf, respectively. The model curve showed that the P value reached a steady state at higher concentrations of the carrier because the viscosity governed the transport phenomenon.

Keywords: Germanium; Supported liquid membrane; Transport; Cyanex 923; Modeling

1. Introduction

Germanium is a strategic metalloid applied to a wide range of high-technology devices [1]. Germanium is found in lead and zinc ores [2] and fly ashes from coals [3]. From the view of hydrometallurgy, the leach solutions obtained from the zinc plant residues and coal fly ashes contain germanium along with other metals. The most important elements that can be found in these solutions are heavy metals such as zinc, nickel, cobalt, etc. [4,5]. The germanium separation from these solutions is a vital objective to obtain a purified germanium product.

Gasification coal fly ashes containing germanium can be leached with water to dissolve germanium as water-soluble species [6]. Several processes have been developed to recover and separate germanium from impurities such as precipitation [7], flotation [8], ion-exchange [9], distillation [3], adsorption [10], and liquid-liquid extraction (LLX) [11]. Among these techniques, the LLX processes have been extensively applied to separate germanium from aqueous solutions [12]. However, some disadvantages make this method inappropriate for treating low concentrations of ions. The supported liquid membrane (SLM) techniques have been introduced as alternative methods to overcome these disadvantages [13]. High selectivity, user-friendly operation, low consumption of carrier, and the one-step process are some advantages of SLMs [14-17].

Cyanex 923 is an extractant with solvation extraction behavior containing four trialkyl phosphine oxides, which can be used to extract neutral species. This extractant has been widely used in SLM systems. The permeation of cadmium (H(n-2)CdCln ) from wastewater containing chloride...
anions has been investigated through SLM processes using Cyanex 923 [18]. Chromium(VI) neutral species (H2CrO4) was transported across an FSSLM system using Cyanex 923 from chloride solutions [19]. Zinc(II) species in the form of HnZnCl(2+n) were permeated by a solid-SLM from chloride medium using Cyanex 923 [20]. In addition, an FSSLM system has been used to transport HFeCl4 species through a PVDF membrane with the carrier of Cyanex 923 from chloride medium using Cyanex 923 [21]. Alguacil, et al. [22] investigated the transport of Au(III) in the form of HAuCl4 through a PVDF membrane film impregnated in Cyanex 923 from HCl solution. Results showed good transport efficiency for gold species. Neutral complexes of uranium(VI) (UO2(H2PO4)2) were separated through an SLM system from phosphoric acid solutions using mobile carriers containing 2-ethyl hexyl phosphoric acidmono-2-ethyl hexyl ester (PC88A) and Cyanex 923 [23].

The presented research describes a flat-sheet supported liquid membrane (FSSLM) process with a Cyanex 923 carrier, in which the facilitated transport of germanium species is carried out from a solution containing oxalic acid. Significant parameters affecting the transport of germanium such as germanium concentration in the feed phase, carrier concentration, oxalic acid concentration, membrane type, and NaOH concentration in the strip phase have been investigated in detail. Finally, a mass transfer model was developed to find the Δaq and Δorg values, which are resistances corresponding to the species diffusion through the feed-membrane interfacial layer and membrane phase, respectively.

2. Experimental

2.1. Materials

Cyanex 923 consisting of four trialkyl phosphine oxides (93%) was supplied by CYTEC Inc., NJ, USA. Various concentrations of the mobile carrier (5-30 %v/v) were prepared by dissolving Cyanex 923 in kerosene (Sigma-Aldrich, MO, USA). In all experiments, purified water was used supplied using a water purifier (Siemens, Germany). The solution used in this study was a solution containing 100 mg/L of germanium prepared by dissolving GeO2 (99.99%) provided by Sigma-Aldrich. Desired amounts of oxalic acid powder from Panreac, Barcelona, Spain were added to the aforementioned solutions to prepare various oxalate concentrations in the range of 0.05-0.2 mol/L. In order to choose a proper stripping phase, some reagents provided by Sigma-Aldrich such as ammonium chloride (NH4Cl), sodium hydroxide (NaOH), catechol (C6H6O2), citric acid (C6H8O7), ammonia (NH3), sodium sulfate (Na2SO4), sulfuric acid (H2SO4) and purified water were used as stripping phases. A FHLP series of poly tetra fluoro ethylene (PTFE) flat sheet hydrophobic membranes with the characteristics being a porosity of 85%, 47 mm diameter, and pore size of 0.45 μm and a HVHP series of polyvinylidene difluoride (PVDF) hydrophobic membrane with a pore size of 0.45μm, diameter of 47 mm, and thickness of 125 μm were supplied from Millipore, KGaA, Darmstadt, Germany.

2.2. Membrane experiments

FSSLM experiments were carried out in a system including two cells attached together with a flanged chamber between the cells in which a membrane filter could be placed. The effective membrane area was calculated to be 11 cm2. The configuration of this system was introduced elsewhere [24]. The system contained two cells for the feed and strip (receiving) phases with a volume of 220 mL for each cell. Before starting the experiments, the membranes were impregnated in various concentrations of Cyanex 923 diluted in kerosene. The feed and strip phases were mixed well by means of a mechanical stirrer. Samples with volumes of 0.5 mL from both phases were taken to evaluate the concentrations of ions during the experiments. The composition of solutions was determined using an inductively coupled plasma atomic emission spectroscopy (ICP-AES Agilent, USA).
2.3. Transport equations

Transport phenomena in SLM processes principally occur in three steps including the reaction of species with the carrier at the feed-membrane interface, diffusion across the membrane and stripping at the membrane–strip interface. According to the literature, the flux of species (J) can be found by disregarding the concentration of germanium in the strip phase with respect to Eq. (1) [14]:

\[ J = P_f C_f \]  

(1)

In this equation, \( P_f \) represents the permeability coefficient at the feed–membrane interface and \( C_f \) depicts the concentration in the feed phase. Furthermore, the flux can be written according to Fick’s first law in a differential form as Eq. (2):

\[ J = \frac{V}{A} \frac{dC_f}{dt} \]  

(2)

Where \( V \) is the volume of the feed phase and \( A \) shows the membrane efficient area. With respect to Eqs. (1) and (2), the integration form of Eq. (2) and Eq. (3) can be written as:

\[ \ln\left(\frac{C_{f,t}}{C_{f,0}}\right) = -\frac{AP_f t}{V} \]  

(3)

Where \( C_{f,0} \) and \( C_{f,t} \) depict the concentration of ions in the feed phase at the initial time and time of \( t \), respectively. Hence, the permeability coefficient can be evaluated from the slope corresponding to the plot of \( \ln\left(\frac{C_{f,t}}{C_{f,0}}\right) \) against \( -\frac{t}{V} \). Furthermore, the transport efficiency (%Transport) of germanium is calculated as Eq. (4):

\[ %T = \frac{C_{f,t}}{C_{f,0}} \times 100 \]  

(4)

3. Solution chemistry

Germanium dioxide can be dissolved in water. However, this dissolution is slowly carried out as an intermediate tetra-hydroxide. These species can be converted to Germanic acid with the solubility product (Ksp) of 2.39×10^{-4.5} as Eq. (5) (McCrory-Joy, 1985):

\[ 2H_2O + GeO_2 \Leftrightarrow Ge(OH)_4 = H_2GeO_3 + H_2O \]  

(5)

Germanium can form various complexes with organic acid such as tartaric acid, citric acid, oxalic acid, etc. Oxalic acid is a possible complexant, forming various complexes with germanium. Furthermore, it can form similar complexes with other metals. According to the literature and observations, the solubility of germanium increases in the presence of oxalic acid in aqueous solutions (Liu et al., 2017b). The formation of germanium anionic and neutral species has been reported. For instance, Pokrovski et al. (2000) described that germanium (0.02 mol/L) and oxalic acid (0.1 mol/L) form anionic species of Ge(OH)2(ox)22- (“ox” depicts oxalate) at pHs below 7. Furthermore, in a research conducted by Liu et al. (2017a) Ge(ox)32- was introduced as anionic complexes of germanium and oxalic acid extracted by tri(octyl-decyl) amine (N235). The concentrations of germanium and oxalic acid in the latter study were reported to be 0.013 and 0.67 mol/L, respectively. However, others (Kamran Haghighi et al., 2018; McCrory-Joy, 1985) reported the formation of neutral species of trisoxalatogerminates as Eq. (6), as germanium and oxalic acid concentrations were 0.11 and 1 mol/L, respectively:

\[ 3H_2C_2O_4 + GeO_2 \Leftrightarrow H_2Ge(C_2O_4)_3 + 2H_2O \]  

(6)

In the current study, since germanium could be extracted and transported by Cyanex 923, having the solvation mechanism, it can be concluded that germanium neutral soluble species in the form of trisoxalatogermanate have been extracted by the carrier.

This section may be divided by subheadings. It should provide a concise and precise description of the experimental results, their interpretation as well as the experimental conclusions that can be drawn.
4. Results and discussions

4.1. Determination of the extraction mechanism by slope analysis

With respect to the “solution chemistry” section and the literature review [18,25,26], the probable extraction reaction of germanium using Cyanex 923 can be written as Eq. (7):

$$2H^+ + nL + Ge(C_2O_4)_x^{2-} \rightleftharpoons K_{ex} \rightarrow H_2Ge(C_2O_4)_x nL$$

(7)

Where L depicts the organic extractant (Cyanex 923) and $K_{ex}$ shows the equilibrium constant.

In order to find the number of extractant molecules that participated in the reaction, a series of liquid-liquid extraction experiments was conducted. According to Eq. (7), $K_{ex}$ can be written as Eq. (8):

$$K_{ex} = \frac{[H_2Ge(C_2O_4)_x nL]}{[H^+]^n[L]^n[Ge(C_2O_4)_x^{2-}]}$$

(8)

Since $K_{ex}$ is equal to the distribution coefficient (D) at an equilibrium state, Eq. (8) can be written as Eq. (9):

$$K_{ex} = \frac{D}{[H^+]^n[L]^n}$$

(9)

By taking the logarithm of Eq. (9), Eq. (10) was rearranged:

$$\log(D) = \log K_{ex} + 2\log[H^+] + n\log[L]$$

(10)

The plot of the distribution coefficient (D) as a function of the extractant concentration was constructed to determine the number of Cyanex 923 molecules that reacted with transported germanium species. The obtained plot is illustrated in Figure 1. As seen in this figure, the slope of the linearized plot was found to be 3.60. Hence, it was concluded that 4 molecules of Cyanex 923 were associated with germanium species.

![Figure 1](image.png)

Figure 1. Liquid-liquid extraction: $\log D$ versus $\log$ [Cyanex 923] ([Ge(IV)] = 100 mg/L; [oxalic acid] = 0.1 mol/L).

4.2. Determination of appropriate concentrations for oxalic acid in the feed phase

With respect to the “solution chemistry” section, germanium was transported across FSSLM as a neutral complex (trisoxalato germanate) using Cyanex 923. The transport depended on the degree of germanium complexation with oxalates. Therefore, the concentration of oxalic acid was a significant parameter for this transportation. In this regard, a series of experiments were conducted...
at the oxalic acid concentration in the range of 0.05-0.2 mol/L with the carrier concentration of 20%v/v. The maximum transport has been achieved at the oxalic acid concentrations in the range of 0.075-0.1 mol/L with the transport efficiency of more than 88%. Figure 2a and b illustrate the obtained results. As seen in this figure, the transport efficiency and permeation coefficients descended at lower and higher than this range. The maximum permeation coefficient and transport efficiency were found at the oxalic concentration of 0.075 to be 9.97 × 10^-4 cm/s and 88%, respectively. In order to know the reason for this behavior, the speciation diagram and the extraction reaction of germanium by Cyanex 923 should be considered. As discussed in the previous section, to transport germanium, 1 molecule of germanium-oxalate that joined 2 molecules of H+ was extracted by 4 molecules of Cyanex 923. Therefore, to transport germanium species, the presence of H+ and oxalate anions (ox2-) hydrolyzed from oxalic acid (H2ox) is vital. The speciation diagram of oxalic acid has been illustrated in Figure 3. As seen in this figure, at lower oxalic acid concentration, the dissociation of oxalic acid (H2ox) is not complete, whereas by increasing the oxalic acid concentration, the concentration of ox2- enhances. On the other hand, with increasing the total oxalic acid concentration, the concentration of H+ decreases resulting in the decrease of the germanium transport at higher oxalic acid concentration. Therefore, it can be concluded that the lack of oxalate (ox2-) and H+ molecules descended the transport efficiency at the lower and higher oxalic acid concentrations, respectively.

![Figure 2a](https://example.com/image1.png)

**Figure 2a.** The effect of transport and permeability coefficient of germanium across the FSSLM system ([Cyanex 923] = 20%v/v, the temperature of 22°C, and [NaOH]= 0.1 mol/L).
4.3. Evaluation of appropriate carrier concentration

The presence of mobile carriers in the membrane phase is necessary for the transport phenomena, but an excess amount enhances the viscosity, decreasing transport [27]. Therefore, in order to obtain an appropriate transport, determining an optimum concentration of the carrier is important. According to the literature, the selection of a carrier concentration range for SLM experiments depends on the type of species and other factors. For instance, Rathore, Leopold, Pabby, Fortuny, Coll, and Sastre [18] investigated the effect of Cyanex 923 concentration as a carrier on the transport of Cd(II) across an SLM system in the range of 0 to 20 %v/v. Moreover, the Cyanex 923 concentration of 20-80 %v/v was applied to transport Cd(II) from a chloride medium via an FSSLM system [28]. In the present study, the range of 0-30 %v/v for the extractant concentration was selected with respect to the liquid-liquid extraction experiments. All FSSLM experiments were conducted at the oxalic acid concentration of 0.075 mol/L, the temperature of 22°C, and the NaOH concentration of 0.1 mol/L. The result has been plotted in Figure 4a and b. As seen in this figure, the germanium transport efficiency increased up to 88% when the carrier concentration of reached 20 %v/v. As seen in this figure, the complete reaction between species and the carrier does not occur at lower concentrations of the carrier. Moreover, with increasing the carrier concentration, the curve corresponding to Cyanex 923 of 30 and 25 %v/v was shifted down, showing the decrease of the germanium transport. In Figure 4b, this behavior can be observed in the reduction of the permeability coefficient. The reduction occurred due to the enhancement of Cyanex 923 viscosity and its precipitation in pores of the membrane [29]. According to the aforementioned discussion, the concentration of 20 %v/v was selected as an optimum concentration.
4.4. Investigation on appropriate strip reagent

In order to select efficient reagent/reagents for stripping germanium from the loaded organic carrier, a series of liquid-liquid extraction experiments were conducted. In this regard, several reagents, which were predicted to be efficient in the stripping process including ammonium chloride (NH₄Cl), sodium hydroxide (NaOH), catechol (C₆H₆O₂), citric acid (C₆H₈O₇), ammonia (NH₃), sodium sulfate (Na₂SO₄), and sulfuric acid (H₂SO₄) were tested. The results have been published elsewhere [30]. Based on the results, NaOH was selected as an efficient reagent for stripping germanium from 20 %v/v Cyanex 923. Thus, FSSLM experiments were conducted to evaluate the effect of the NaOH concentration on the transport of germanium with the carrier concentration of Cyanex 923 20 %v/v in an oxalic acid concentration of 0.075 mol/L in the feed phase. The results can be seen in Figure 5a and b.

Figure 4. Effect of the carrier concentration on (a) the transport and (b) permeability coefficient of germanium across the FSSLM system ([Oxalic acid] = 0.075 mol/L, temperature of 22°C, and [NaOH] = 0.1 mol/L).
Figure 5. Effect of NaOH concentration on (a) transport and (b) permeability coefficient of germanium stripped to the receiving phase across the FSSLM system ([Oxalic acid] = 0.075 mol/L, the temperature of 22°C, and [Cyanex 923] = 20 %v/v).

As seen in this figure, the transport efficiency and permeability coefficient increase with an enhancement of NaOH concentration, as at concentrations of more than 0.08 mol/L, >88% of germanium was transferred to the strip phase. The transport efficiencies corresponding to NaOH concentrations of 0.06 and 0.04 mol/L are approximately 84%. Furthermore, according to Figure 5b, the permeability coefficient increased up to 9.20 × 10⁻⁴ cm/s at the NaOH concentration of 0.1 mol/L. This is due to the increased amount of OH⁻ anions, enhancing the de-complexation rate at the interface of the strip side [25].

The probable reaction for stripping species from the loaded carrier can be written as Eq. (11) [30]:

\[ \text{H}_2\text{Ge(O}_2\text{)}_3\text{,}4\text{L} + 2\text{OH}^- \leftrightarrow 4\text{L} + \text{Ge(O}_2\text{)}_3\text{)}^2^- + 2\text{H}_2\text{O} \]  

(11)

4.5. Effect of membrane PVDF type on the transport

In order to know the transport behavior of germanium through a polyvinylidene difluoride (PVDF) membrane, a transport experiment was conducted the condition of 100 mg/L Ge, Ni, Cd, Co and 1000 mg/L of Zn; oxalic acid of 0.075 mol/L, and the NaOH concentration of 0.1 mol/L in the strip phase. The PVDF membrane used had a pore size of 0.45 μm, the porosity of 80% and thickness of 125 μm.

As seen in Figure 6, there is not a significant difference between the transport efficiencies corresponding to the PTFE and PVDF membranes obtained from the experiments conducted under a similar condition. However, the overall transport efficiency of the PTFE membrane is higher. The germanium permeability coefficients belonging to the PVDF and PTFE membranes were obtained to be 3.14 × 10⁻⁴ and 9.28 × 10⁻⁴ cm/s, respectively. Therefore, the germanium permeability through the
PTFE membrane is approximately 3 times the PVDF permeability. Similar results have been reported by Adnan, et al. [31]. Various parameters such as higher tortuosity of PVDF membranes and thickness are resulted in reducing their permeability [32].

![Image](image1)

**Figure 6.** Transport efficiency of germanium versus time across PTFE and PVDF membranes (100 mg/L of Ge, Ni, Cd, Co and 1000 mg/L of Zn, the oxalic acid of 0.075 mol/L, and the NaOH concentration of 0.1 mol/L in the strip phase).

4.6. Permeability model

The permeation of Ge(IV) through the FSSLM (with PTFE membrane) of this study was modeled to find mass transfer resistances. To model the mentioned system, it was assumed that the species transport through SLM was performed by diffusing, and chemical reactions took place promptly [33]. Figure 7 illustrates how germanium oxalates permeate through a PTFE membrane containing Cyanex 923 based on the following steps:

(i) in the feed phase, the germanium oxalates and protons diffuse to the interface layer.
(ii) diffused species and the Cyanex 923 molecules react together at the mentioned layer.
(iii) the produced complexes permeate across the membrane toward the membrane-strip interface layer.
(iv) NaOH detaches germanium-Cyanex 923 complexes at the membrane-strip phase interface. Thus, germanium is stripped from the organic carrier.
(v) the unloaded carrier molecules permeate inversely toward the feed phase.

![Image](image2)

**Figure 7.** A schematic transport of germanium through FSSLM-Cyanex923.

The extraction equilibrium reaction of germanium by Cyanex 923 has been described in Eq. (8) with n=4. According to the experimental data, the extraction equilibrium constant of this reaction was calculated to be 2056. The germanium flux can be obtained using Fick’s first diffusion law.
Hence, the fluxes at the feed-membrane boundary layer and the membrane phase (\(J_f\) and \(J_m\), respectively) can be provided as Eqs. (12) and (13):

\[
J_f = \frac{1}{\Delta_f}([\text{Ge(IV)}]^f_{\text{bl}} - [\text{Ge(IV)}]_{\text{bl},f})
\]  
(12)

\[
J_m = \frac{1}{\Delta_m}([H_2\text{Ge(ox)}_3, 4R]_{\text{bl},f} - [H_2\text{Ge(ox)}_3, 4R]_{\text{bl},s})
\]  
(13)

Where \(\Delta_m\) and \(\Delta_f\) depict the resistances corresponding to the membrane phase and the feed phase boundary layer, respectively. Subscripts of \(f\), \(bl\), and \(s\) show feed, boundary layer, and strip, respectively. Moreover, \([\text{Ge(IV)}]^f\), \([H_2\text{Ge(ox)}_3, 4R]_{bl,f}\) and \([H_2\text{Ge(ox)}_3, 4R]_{bl,s}\) represent the germanium concentration in the feed phase, the feed-membrane boundary layer, and the strip-membrane boundary layer, respectively. It is noted that since the germanium concentration in the membrane-strip boundary layer is lower than that in the feed-membrane boundary layer, \([H_2\text{Ge(ox)}_3, 4R]_{bl,s}\) has been neglected. Hence, Eq. (13) can be rewritten as Eq. (14):

\[
J_m = \frac{1}{\Delta_m}([H_2\text{Ge(ox)}_3, 4R]_{bl,f})
\]  
(14)

Since chemical reactions promptly took place, the flux values in the feed-membrane layer and the membrane phase are equal (\(J_f = J_m = J\)). Therefore, overall \(J\) can be found as Eq. (15):

\[
J = \frac{K[H^+]^4[R]^{4+}_{\text{org}}[\text{Ge(IV)}]^f}{\Delta_m + \Delta_f(K[H^+]^4[R]^{4+}_{\text{org}})}
\]  
(15)

Regarding Eq. (1), the permeability coefficient is also written as Eq. (16):

\[
P = \frac{K[H^+]^4[R]^{4+}_{\text{org}}}{\Delta_m + \Delta_f(K[H^+]^4[R]^{4+}_{\text{org}})}
\]  
(16)

By arranging Eq. (16), Eq. (17) was found to obtain mass transfer resistances:

\[
\frac{1}{P} = \Delta_f + \frac{\Delta_m}{(K[H^+]^4[R]^{4+}_{\text{org}})}
\]  
(17)

By plotting \((K[H^+]^4[R]^{4+}_{\text{org}})\) versus \(1/P\), and the corresponding trend line, the intercept and the slope of the trend line can be found which are \(\Delta_f\) and \(\Delta_m\), respectively. The mentioned plot was shown in Figure 8a. According to this plot, the values of 1 and 1345 s/cm were found for \(\Delta_m\) and \(\Delta_f\) respectively. Also, the permeation coefficient was found according to Eq. (16). The plot of calculated \(P\) (Pcal) and experimental \(P\) (Pexp) as a function of the Cyanex 923 concentration was constructed as Figure 8b. As seen in this figure, the Pexp value increased up to the concentration of 20 %v/v followed by a decrease due to the enhancement of viscosity. On the other hand, the model points constructed a curve showing the trend of the permeation enhancement. However, a sudden up and down at the carrier concentration of 20 %v/v, could not be calculated by the model. The model curve showed the regular trend of the permeation, as the P value increased with an enhancement of the carrier concentration up to 20 %v/v, but above this concentration, the value reached a plateau because the viscosity governed the transport process.
5. Conclusion

In this study, the transport of germanium was investigated across an FSSLM system from aqueous solutions. The main achievement of this study was that germanium oxalate neutral species were effective through an FSSLM system containing Cyanex 923. The oxalic acid concentration is one of the parameters having a vital effect on the transport. As a result, an optimum concentration of oxalic acid for the complete transport of germanium was found at a concentration range of 0.075-0.1 mol/L. The investigation on the effect of the Cyanex 923 concentration in the membrane phase showed that the concentration of 20 %v/v was appropriate for the complete germanium transportation. Several stripping reagents were classified based on liquid-liquid extraction experiments to select appropriate reagents used in the FSSLM system. Accordingly, NaOH was chosen as an efficient stripping reagent for the FSSLM system. In the FSSLM experiments with higher concentrations of NaOH in the strip phase, the higher germanium transport was observed. The NaOH concentration range of 1-3 mol/dm3 was obtained in this process. The permeation model was developed to find the mass transfer resistances. As a result, the values of 1 and 1345 s/cm were found for $\Delta m$ and $\Delta f$, respectively. The model curve showed the regular trend of the permeation, as the P value reached a plateau at higher carrier concentrations because the viscosity governed the transport process.
Acknowledgments

This study was carried out in the Department of Chemical Engineering, Universitat Politècnica de Catalunya, Vilanova i la Geltrú Campus, Spain. The authors wish to acknowledge Dr. Agustin Fortuny and Dr. Maria Teresa Coll for their assistance and scientific consultation.

Author Contributions: H.K.H made a significant contribution to every stage of this paper, such as the investigation (with the essential assistance of A.M.S) and analysis and preparing the paper. M.I. contributed to the conception of the paper. H.K.H designed the tests and presented a model for this paper. M.I. and H.K.H finalized the paper by a critical revision of the paper.

Acknowledgments: This research was implemented in the Department of Chemical Engineering, Universitat Politècnica de Catalunya (Barcelona-Tec), Spain. The authors acknowledge Dr. Agustin Fortuny for his help.

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