



Non-dispersive extraction of gold(III) with ionic liquid Cyphos IL101



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ABSTRACT

The permeation of gold(III), through a supported liquid membrane (SLM), in flat-sheet operational mode, impregnated with Cyphos IL101 (ionic liquid with a quaternary phosphonium salt as active substance) in Solvesso 100 has been investigated. The transport of gold(III) has been studied under various experimental conditions: stirring speed of the aqueous phases, initial metal and carrier concentrations, HCl concentration in the source phase, etc. The overall mass transfer coefficient was calculated as 0.19 cm/min and the thickness of the aqueous boundary layer was of 3.2×10^{-3} cm. The performance of Cyphos IL101 has been studied with respect to other carriers and also the selectivity of the membrane-carrier system on various metal-bearing aqueous solutions. Gold is recovered in the strip phase as nanogold.

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1. Introduction

In hydrometallurgical terms, separation technologies achieved the obtention of streams in which a given metal (or solute) is separated, and also concentrated, from undesirable accompanying metals (or solutes) in the feed solution prior to its final recovery, recycling or dumping.

Among these separation technologies, supported liquid membranes (SLMs) are of interest [1–10].

In SLM technology, the classical extraction and stripping operations of liquid-liquid extraction are combined in a unique stage. Moreover, and from an engineering and practical stand point, SLMs are of particular interest because of their properties.

Prior to the scaling-up to a smarter SLM operational mode i.e. hollow fiber modules (HFMs) in any configuration, i.e. pseudo-emulsion hollow fiber with strip dispersion (PEHFSD), investigations about the performance of a given system are needed, and then is when SLMs in flat-sheet configuration play a decisive role to understand and to estimate the necessary main operational parameters to run successfully HFMs, except of course, that related with the hydrodynamics of the operation, i.e. stirring speed of the phases in flat-sheet operation or phases flow in fiber modules.

From a time ago, ionic liquids are being considered as “green” replacements of many conventional extractants for separation processes and due to their properties [11–16]. Thus, during last years, several studies have deal with the permeation of a number of metals through liquid membranes using different membrane configurations

and/or ionic liquids, but with no specific data about the transport of gold(III).

The present investigation, and in order to expand the knowledge of these ionic liquids compounds in metals transport, presented data about the system Au(III)-HCl-Cyphos IL101. The ionic liquid Cyphos IL101 had been investigated as potential and effective carrier, in flat-sheet membrane configuration, for gold(III)-bearing solutions. Various parameters which may influenced the transport of gold(III) were investigated, also comparing the performance of the present system with other organic carriers. Gold can be recovered, from the strip solution, as nanogold.

2. Experimental

The ionic liquid Cyphos IL101 (phosphonium derivative) was obtained from Cytec Ind. (Canada) and was used without further purification, the active substance of the reagent is trihexyl(tetradecyl)phosphonium chloride. Other carriers used in the investigation were obtained from different sources, being their main characteristics summarized in Table 1. Solvesso 100 (Exxon Chem. Iberia, Spain) is an aromatic diluent with boiling range (165–181 °C), flash point (approx. 41 °C) and density (0.88 g/cm³ at 15 °C). It was used to dilute Cyphos IL101, and thus decreasing the high viscosity of the reagent and adequate the carrier concentration to the practical experimental conditions used in this work, this diluent was also used to dilute all the others investigated carriers. All the other chemicals used in this work were of AR grade.

The supported liquid membrane was impregnated with the carrier solution by immersing it for 24 h and then leaving it to drip for 15 s before being placed in the cell. Previous experiments had

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Table 1
Others extractants used in the transport of gold(III).

Extractant	Type	Active substance	Vendor
Primene JMT	Primary amine	C ₁₂₋₂₂ branched aliphatic chains-amine	Rohm and Haas
Amberlite LA2	Secondary amine	C ₁₂ linear and branched aliphatic chains-amine	Rohm and Haas
Hostarex A324	Tertiary amine	C ₁₀ linear aliphatic chains-amine	Hoechst
Aliquat 128	Quaternary ammonium salt	Tricaprylmethylammonium chloride	Aldrich

shown that extended immersion times, i.e. 48 h, of the support in the carrier solution had not influenced the overall mass transfer coefficient values obtained using 24 h. The support used was Millipore Durapore HVHP 4700 of 125×10^{-4} cm thick microporous poly(vinylidene difluoride) film with nominal porosity of 75%, effective pore size of 4.5×10^{-5} cm and tortuosity of 1.67. In a series of experiments (Section 3.7.), Durapore GVHP 4700 support was tested for gold transport, its characteristics being the same that above, except effective pore size of 2.2×10^{-5} cm.

Transport experiments were performed in a permeation cell [17], consisting of two cubic compartments made of methacrylate and separated by the solid support. The membrane effective area was of 11.3 cm², and the volume of the source and the strip phases of 200 cm³ (each). Agitation was performed in both compartments by using impellers having a diameter of 2.8 cm. All the experiments were carried out at 20 °C.

At elapse times, gold and metals concentrations were measured in the source and strip phases for analysis using a Perkin Elmer 1100B spectrophotometer.

The gold (and metals) overall mass transfer coefficient (K_{Au}) was estimated from:

$$\ln \frac{[Au]_t}{[Au]_0} = -\frac{AK_{Au}}{V}t \quad (1)$$

where $[Au]_t$ and $[Au]_0$ are gold concentrations in the source phase at an elapsed and initial time, respectively, A is the effective membrane area, V is the volume of the source phase and t is the elapsed time.

The percentage of gold recovered in the strip phase was calculated by:

$$\% \text{recovery} = \frac{[Au]_{st,t}}{[Au]_0 - [Au]_t} \times 100 \quad (2)$$

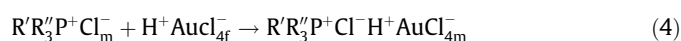
$[Au]_{st,t}$ being the gold content in the strip phase at an elapsed time, and $[Au]_0$ and $[Au]_t$ as in Eq. (1).

3. Results and discussion

Due to the chemical characteristics of Cyphos IL101 and that gold(III) is presented in low to moderate HCl concentrations as $AuCl_4^-$ species, at a first instance, one can think that gold(III) is transported to the membrane phase containing Cyphos IL101 via an anion exchange reaction:



where m and f represented to the membrane and feed phases, respectively. A similar mechanism was suggested in the literature [18] when Au(III) is extracted by Cyphos IL101. However, at higher HCl concentrations, see Section 3.3, there is still some transport of gold(III), which indicated that there is an involvement of ion-pair species:



3.1. Influence of the stirring speed of the source and strip phases

The influence of the stirring speed of the source phase was studied in order to optimise uniform mixing of both aqueous feed and

to minimise thickness of the aqueous boundary layer with the feed and strip conditions being maintained as follows: Au(III) 0.02 g/L in 0.1 M HCl and water, respectively. The Cyphos IL101 concentration was 10% v/v in Solvesso 100 immobilised on a Durapore microporous support.

The results obtained are shown in Table 2; the overall mass transfer coefficient (K_{Au}) becomes a maximum at 1000 min^{-1} , indicating a first decrease in the aqueous boundary layer thickness, and then a minimum value of thickness is reached at this stirring speed. The decrease in K_{Au} values at stirring speeds above 1000 min^{-1} is attributable to a greater turbulence in the system making the membrane unstable.

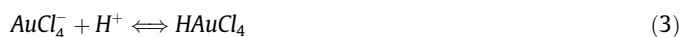
It was observed, that variation in the stirring speed in the strip phase over the $300\text{--}1000 \text{ min}^{-1}$ range has no influence on the overall mass transfer coefficient values.

3.2. Influence of stripping phase composition on transport of gold(III)

Using source phases of 0.02 g/L Au(III) in 5 M HCl and organic solutions of 10% v/v Cyphos IL101 in Solvesso 100, the effect of changing the composition of the strip solution on gold transport was evaluated. This study was carried out using water or 0.1 M sodium borohydride solution in the stripping phase for Au(III), being the K_{Au} values as 0.23 and 0.25 cm/min for the above strip-pants, respectively. These results showed that gold(III) transport (from feed to the membrane) is not greatly influenced by the stripping phase composition, however, the recovery of gold in the strip phase increases dramatically when sodium borohydride is used to strip gold (68% versus <5% for water). Furthermore, Table 3 showed the gold distribution between the feed, membrane and strip phases during a typical transport experiment; from the initial times, there is a gold flux from the feed to the membrane phase and then to the strip solution. It should be noted here that gold is recovered in the strip phase as nanogold, demonstrated by the violet or red-brown color of the solution which is indicative of this phenomena. Under vigorous stirring of this solution, nanogold agglomerates and coarse violet particles are obtained, that at a magnifier-view allowed to see the characteristic metallic yellowish color¹ of gold (Fig. 1).

3.3. Influence of HCl concentration in the source phase

To study the influence of the hydrochloric acid concentration in the source phase on gold transport, a series of experiments was carried out at various HCl concentrations, keeping other variables constant. Thus, the single gold transport through the membrane at 0.02 g/L Au(III) from aqueous source solutions of varying HCl concentrations was studied using 0.1 M sodium borohydride as strippant. Fig. 2 shows that the time-dependent fraction $\ln[Au]_t/[Au]_0$ of gold in the source phase at the various HCl concentrations indicated decreasing gold transport as the HCl concentration of the phase is increased, this behaviour can be attributed that at higher HCl concentrations, the equilibrium:



¹ For interpretation of color in Fig. 1, the reader is referred to the web version of this article.

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