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Article

Permeation of $AuCl_4^-$ across a liquid membrane impregnated with $A324H^+Cl^-$ ionic liquid

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Abstract: On the system Au(III)-HCl-A324H+Cl-, liquid-liquid extraction experiments were used to define the extraction equilibrium and the corresponding extraction constant, and the facilitated transport of this precious metal, from HCl solutions, across a flat-sheet supported liquid membrane was investigated, using the ionic liquid as carrier, as a function of hydrodynamic conditions, concentration of gold(III) (0.01-0.1 g/L), and HCl (0.5-6 M) in the feed phase, and carrier concentration (0.023-0.92 M) in the membrane. An uphill transport equation was derived considering aqueous feed boundary layer diffusion and membrane diffusion as controlling steps. The aqueous diffusional resistance (Δf) and the membrane diffusional resistance (Δm) were estimated from the proposed equation, being their values 241 s/cm an 9730 s/cm, respectively. The performance of the present carrier was compared against results yielded by other ionic liquids, and also it was investigated the influence that other metals had on gold(III) transport both from binary or quaternary solutions. Gold was finally recovered from receiving solutions as zero valent gold nanoparticles.

Keywords: liquid membrane; ionic liquid; gold

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1. Introduction

The continuous growth of residues generated in cities, make of these a source for the recovery of the valuable metals contained in them, and thus, nowadays the concept of urban mining is taking more importance. Among these residues, the materials generated from the electronic and communication industries are of importance [1,2], because they contained, among other valuable metals, gold. Thus, there is an interest, from worldwide researchers, to investigate methods for the recovery of this precious metal from these residues or, in general, gold-bearing wastes [3-8].

This recovery can be executed by pyro or hydrometallurgical procedures, and in the case of the latter methodologies, and after the leaching step, a multielemental solution is generally obtained, and thus, the separation of gold (or the most valuable element) from the others is of a paramount importance. Including in the separation technologies, liquid membranes are gaining a major importance due to its operational characteristics, and the possibility of the treatment of dilute solutions, more efficiently than i.e. solvent extraction. Including in these liquid membranes technologies, supported liquid membranes combined the kinetics properties of the membrane processes, with the chemical reaction and selectivity that the organic extractants, imbibed in the micropores of the support, provided to the system, and thus resulting in the facilitated coupled transport of i.e. metal species. From the types of organic extractants, ionic liquids constituted as themselves a subcategory, of also current interest, due to the special characteristics that these reagents presented, which somewhat allowed to refer to them as green solvents. These ionic liquids are using in different fields [9-12], including the removal of metals from aqueous solutions [13-16].

The present work linked all of the mentioned above: i) the recovery of gold(III) from acidic solutions, ii) using a supported liquid membrane in flat-sheet configuration, as a previous step to its scaling up, i.e. to a hollow fiber module, and iii) the use of an ionic liquid as carrier in the membrane system.

As an ionic liquid, the quaternary ammonium salt, derived from the reaction of a tertiary amine (Hostarex A324) with hydrochloric acid, was used, and different variables affecting the metal transport were investigated, also the results were compared with that obtained with other ionic liquids, and the competitive transport of gold(III) against Cu(II), Fe(III) and Ni(II), as representative elements of which is found in electronic wastes, were investigated from binary and quaternary solutions. As a final step, gold was recovered as zero valent nanogold by precipitation of the receiving solution with sodium borohydride.

2. Materials and Methods

The tertiary amine Hostarex A324 (Hoechst AG, actually part of Sanofi) has tri-isooctyl amine as active component, and it was used without further purification, it was dissolved in Solvesso 100 (aromatic diluent, Exxon Chem. Iberia) to attain various objectives: i) to decrease the high viscosity of the extractant (amine or the derived ionic liquid), ii) being the extractants expensive, the use of a diluent allowed to adequate the concentration of the carrier for the specific purposes of the system to investigate, avoiding the presence of an unused excess of the reagent in the operation. Other chemicals used in the work were AR grade, except the ionic liquids Cyphos IL 101, Cyphos IL102 (phosphonium derivatives), and the precursor Primene JMT (aliphatic primary amine) which were obtained from Cytec (actually part of Solvay) and Rohm an Haas, respectively.

Liquid-liquid extraction experiments were carried out in thermostatted separatory funnels provided of mechanical shaken. After the generation of the ionic liquid [17], organic solutions (25 mL) of various concentrations of the ionic liquid, were put into contact with aqueous solutions (25 mL) of 7.1x10-5 Au(III) in 1 M HCl and shaken during 15 min, enough time to achieve equilibrium, at 20º C. After phase disengagement, less than 3 min, the residual gold content in the raffinate was analyzed by Atomic Absorption Spectrometry (Perkin Elmer 1100B), and the gold concentration in the equilibrated organic phases was calculated by the mass balance.

Supported liquid membranes experiments were carried out using the same cell and procedure described elsewhere [18]. Millipore Durapore GVH4700, (polyvinylidenedifluoride, PVDF, material) was used to support the carrier phase. The characteristics of the support were: 12.5x10-3 thickness (dm), 75 % porosity (ε), 1.67 tortuosity (τ)

Gold (and metals) concentrations from both aqueous solutions, source and receiving, were analysed from samples taken at elapsed times using the same AAS method as above, and the permeability coefficient P was estimated from eq.(1) derived from the combination of gold (and metal) balance in the source phase and the Fick´s law in the membrane phase [19-21]:

$$\ln \frac{[\mathrm{Au}]_t}{[\mathrm{Au}]_0} = -\frac{AP}{V} t \tag{1}$$

where [Au]t and [Au]0 were the gold (metals) concentrations in the source phase at elapsed time and time zero, respectively, A was the membrane area (11.3 cm2), V the volume of the source phase (200 mL), P the permeability coefficient, and t the elapsed time.

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3. Results

3.1. Liquid-liquid extraction

These series of experiments used organic phases of the ionic liquid (0.012-0.0012 M) in Solvesso 100 and an aqueous solutions which composition was given in Section 2. The results from these experiments were shown in Table 1.

Table 1. Liquid-liquid extraction of gold(III) from 1 M HCl solutions by the ionic liquid $A324H^+Cl^-$ dissolved in Solvesso 100.

$[A324H^+Cl^-]$ (M)	$^aD_{Au}$
0.012	21.2
0.0058	7.3
0.0023	2.1
0.0012	1.4

^{a}The distribution coefficient D_{Au} was calculated as the ratio of gold concentrations in the organic and aqueous phases at the equilibrium.

The results were treated by a tailored computer program which compared the experimental distribution coefficient (Dexp) results with the calculated distribution coefficients (Dcal) values, minimizing the expression:

$$U = \Sigma\left(\log D_{cal} - \log D_{exp}\right)^2 \qquad (2)$$

It was shown that the extraction of gold(III) by the ionic liquid A324H+Cl- dissolved in Solvesso 100 responded to an anion exchange equilibrium as:

$$AuCl^-_{4_{aq}} + A324H^+Cl^-_{org} \Leftrightarrow A324H^+AuCl^-_{4_{org}} + Cl^-_{aq} \qquad (3)$$

with the equilibrium constant defined as:

$$K = \frac{\left[A324H^+AuCl_4^-\right]_{org}\left[Cl^-\right]_{aq}}{\left[AuCl_4^-\right]_{aq}\left[A324H^+Cl^-\right]_{org}} \qquad (4)$$

and log K= 3.11±0.18 and U= 4.5x10-2. In the above equations, the subscripts *aq* and *org* represented to the aqueous and organic phases, respectively.

3.2. Supported liquid membrane transport

3.2.1. Influence of the stirring speed

The influence of the stirring speed was investigated in order to optimize uniform mixing in the feed solution and to minimize thickness of aqueous feed boundary layer with feed and receiving conditions being maintained as: 0.01 g/L Au(III) in 1 M HCl and 0.1 M NaSCN, respectively. The extractant concentration was 0.23 M in Solvesso 100 immobilised on a Durapore microporous support. The permeability coefficient becomes virtually independent of the stirring speed from 500 min-1, indicating a first decrease in the aqueous feed boundary layer thickness, and then a minimum value of the thickness is reached from 500 min-1 (Table 2). The stirring speed of 750 min-1 was kept constant throughout further experimentation.

Table 2. Dependence of gold(III) permeabilities values on the stirring speed of the feed phase.

Stirring speed (min^{-1})	**$Px10^3$ (cm/s)**
375	2.1
425	3.0
500	3.9
750	3.9
1000	3.9

Stirring speed receiving phase: 500 min^{-1}.

In the case of the receiving phase, and considering that the stirrer in the cell is very close to the membrane support, the thickness of the boundary layer is minimized; thus, the resistance in the receiving side can be neglected [22], and the stirring speed of 500 min^{-1} was used in the receiving side throughout all the experiments.

3.2.2. Effect of receiving phase composition on permeability of gold(III)

Sodium thiocyanate solutions were used as receiving phase for gold(III) due to the ability of thiocyanate to form a stable complex with gold(III), Au(SCN)4-, with log β2 of 42 [23]. The feed solution had the same composition than above, whereas the receiving solution was of 0.1, 0.25 and 0.5 M sodium thiocyanate. Results derived from this experimentation indicated that the increase of the sodium thiocyanate composition, up to 0.5 M, had a negligible effect on gold permeation.

3.2.3. Effect of HCl concentration in the feed phase on permeability of gold(III)

The single gold transport across the SLM of 0.01 g/L Au(III) from aqueous feed phases of varying (0.1-6 M) HCl concentrations was investigated using 0.1 M NaSCN as receiving phase. Fig.1 shows that the time dependent fraction ln [Au]t/[Au]0 in the feed phase at the various HCl concentrations indicated a strong dependence in the permeability of gold with this variable. A maximum in permeability (Table 3) was obtained at 1 M HCl, whereas the progressive decreased in this value was attributable to one or both of the next factors: i) increase of the ionic strength, ii) shift of the next equilibrium to the right, and thus, decreasing the availability of $AuCl^{4-}$ species with the increase of the HCl concentration:

$$\mathrm{AuCl_4^-} + \mathrm{H^+} \Leftrightarrow \mathrm{HAuCl_4} \qquad (5)$$

Table 3. Gold(III) permeabilities at various HCl concentrations in the feed phase.

HCl (M)	**$Px10^3$ (cm/s)**
0.5	2.8
1	3.9
2	2.6
4	2.2
6	1.8

Experimental conditions as in Fig. 1.

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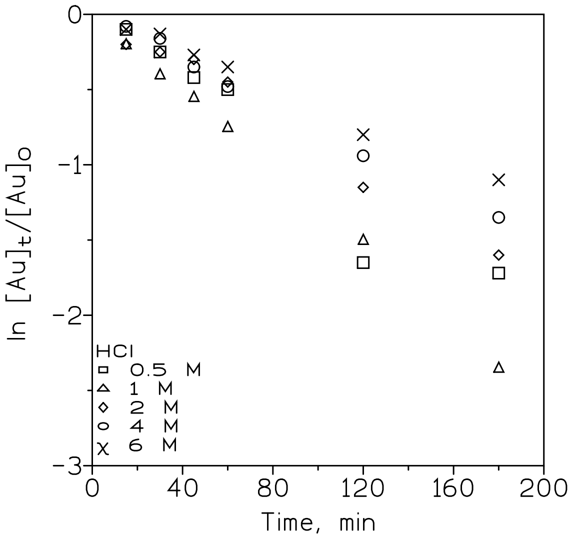

Figure 1. Influence of the HCl concentration on permeability of gold(III) as a function of ln $[Au]_t/[Au]_0$. Feed phase: 0.01 g/L Au(III) in HCl. Membrane phase: 0.23 M ionic liquid in Solvesso 100 on Durapore support. Receiving phase: 0.1 M NaSCN.

3.2.4. Effect of carrier concentration on permeability of gold(III)

The results in relation with the transport of gold(III) from a feed phase containing 0.01 g/L Au(III) in 1 M HCl and the receiving solution 0.1 M NaSCN, and varying concentrations of the ionic carrier in the range 0.023-0.92 M dissolved in Solvesso 100 revealed an increase of the gold(III) permeability up to 0.17 M, an then a maximum value of P was obtained in the 0.17-0.23 M range (Table 4). These results indicated that the transport process was first dominated by diffusion in organic membrane, and then a limiting permeability value Plim was obtained, being this situation indicative of a transport process controlled by diffusion in the stagnat film of the feed phase. In the limiting situation:

$$P_{lim} = \frac{D_{aq}}{d_{aq}} \tag{6}$$

and d_{aq} estimated as $2.6x10^{-3}$ cm, considering the average coefficient of species in the aqueous phase D_{aq} as 10-5 cm^2/s, and Plim of $3.9x10^{-3}$ cm/s. This d_{aq} represented the thickness of the feed boundary layer. The decrease in P value at higher carrier concentrations (0.46-0.92 M) in Solvesso 100 can be attributed to a gradual increase of the organic solution viscosity, with the increase of the carrier concentration, which augmented the membrane resistance to the transport.

Table 4. Variation of gold(III) permeation with the carrier concentration.

Carrier concentration (M)	**Px10^3 (cm/s)**
0.023	1.8
0.058	2.6
0.12	3.2
0.23	3.8
0.17	3.9
0.46	3.4
0.92	2.9

3.2.5. Influence of metal concentration on permeability of gold(III)

Examination of the effect of the initial concentration of gold(III) (0.01-0.1 g/L) in the feed phase, when the receiving phase contained no gold concentration, it was revealed that the initial metal flux:

$$J = [Au]_0 P \tag{7}$$

initially increased sharply from 0.01 to 0.05 g/L and beyond this became almost independent of the initial concentration (0.05-0.1 g/L). The results of gold flux across the membrane as a function of gold(III) concentration are shown in Fig. 2. The initial increase in gold flux is in accordance with the expected trend, as eq.(7) indicated, and since the flux varies with metal concentration. The above this rule was obeyed up to 0.05 g/L concentration of gold, beyond which the initial flux tends to a constant value, attributed to a saturation of the membrane pores, which resulted to flux maximisation, and the creation of a metal-carrier layer on the feed-membrane interface.

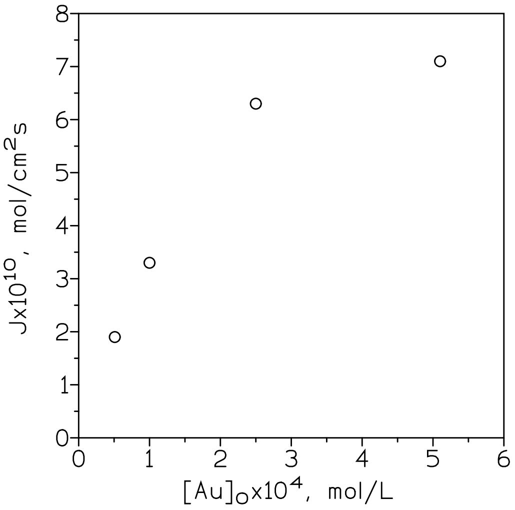

Figure 2. Influence of initial gold(III) concentration on the initial flux. Feed phase: Gold(III) in 1 M HCl. Carrier phase:0.23 M ionic liquid in Solvesso 100 immobilised on Durapore support. Receiving phase: 0.1 M NaSCN.

3.2.6. Influence of the ionic strength of the feed phase on the permeation of gold(III)

This effect was investigated by varying the source of Cl^- ions in the feed phase, with other experimental conditions as: feed phase of 0.01 g/L Au(III) in 1 M (H, Li, Na)Cl, membrane phase of 0.23 M ionic liquid in Solvesso 100, and receiving phase of 0.1 M sodium thiocyanate. From the results presented in Table 5, it can be seen that the substitution of HCl for LiCl or NaCl resulted in a decrease of gold(III) permeation, whereas the influence of changing LiCl by NaCl had a negligible effect on Au(III) transport. This variation can be attributed to the different activity of H^+ ions in the solution with respect Li^+ and Na^+, and that the driven force for gold(III) transport was the different acidity of the feed and the receiving solutions.

Table 5. Gold(III) transport varying the chloride source in the feed solution.

System	Px10^3 (cm/s)
1 M HCl	3.9
1 M LiCl	2.3
1 M NaCl	2.5

Support: Durapore GVHP4700.

3.2.7. Estimation of diffusional parameters

Figure 3 showed gold(III) concentration profile in the three phases of the system.

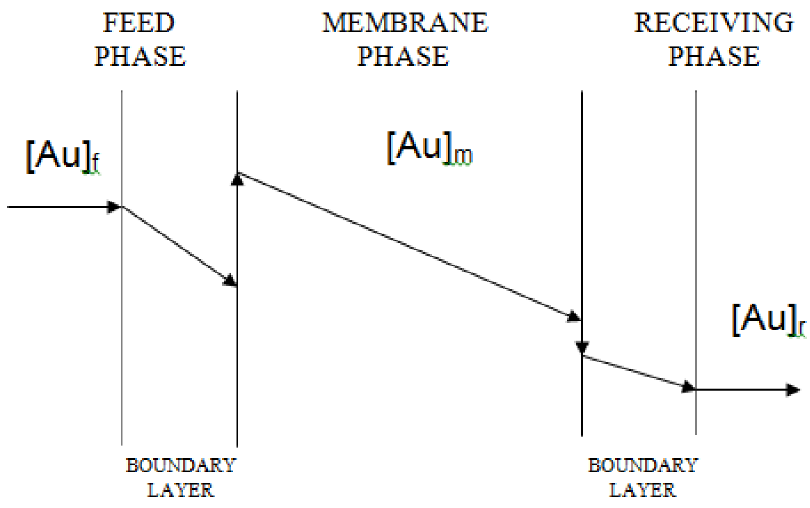

Figure 3. Gold profile in SLM

Taking into account the extraction equilibrium and constant represented in eqs.(2) and (3), respectively, and that the concentration of the $AuCl_4^-$-carrier complex in the membrane phase at the receiving side may be negligible compared with that at the feed side, a final expression for the permeability coefficient can be written as:

$$P = \frac{K[\text{carrier}][Cl^-]^{-1}}{\Delta_m + \Delta_f K[\text{carrier}][Cl^-]^{-1}} \tag{8}$$

and,

$$\frac{1}{P} = \Delta_f + \frac{\Delta_m}{K[\text{carrier}][Cl^-]^{-1}} \tag{9}$$

The above expressions combined in one equation the diffusion and equilibrium parameters involved in the Au(III) transport. In eqs.(8) and (9), Δ_f and Δ_m are the transport resistances due to diffusion in the feed and membrane, respectively, and K is the extraction equilibrium constant.

In a plot of 1/P versus $1/K[\text{carrier}][Cl^-]^{-1}$ for different carrier concentrations and chloride ion concentration of 1 M, a straight line with slope Δ_m (9730 s/cm) and ordinate Δ_f (241 s/cm) is obtained (Fig. 4).

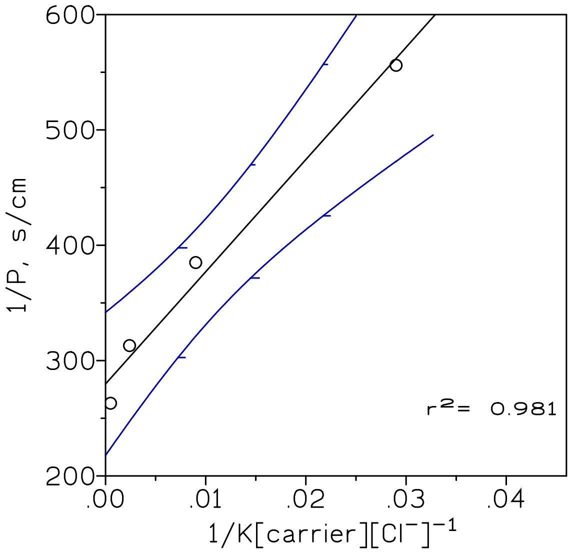

Figure 4. Plot of 1/P versus 1/K[carrier][Cl$^-$]$^{-1}$. Dotted line indicated 95% confidence interval of the regression line.

The estimated value of the membrane diffusion coefficient:

$$D_m = \frac{d_m}{\Delta_m} \tag{10}$$

was calculated as 1.3x10^{-6} cm^2/s. The diffusion coefficient of the gold-carrier complex in the bulk of the membrane phase can be estimated by:

$$D_{m,b} = D_m \frac{\tau^2}{\varepsilon} \tag{11}$$

and $D_{m,b}$ being as 4.8x10^{-6} cm^2/s. It can be seen that the value of D_m is lower than the value of $D_{m,b}$, which is attributed to the diffusional resistance caused by the microporous thin support separating the feed and receiving phases. The mass transfer coefficient in the feed phase, Δ_f^{-1} was estimated to be 4.1x10^{-3} cm/s.

3.2.8. Gold(III) permeation using different ionic liquids as carriers for gold transport

Several ionic liquids (Table 4) were used as carriers to compare their performance, in gold(III) transport, with that of A324H$^+$Cl$^-$.

Table 4. Ionic liquids used in the transport of gold(III).

Name and acronysm	Active group
Cyphos IL101	quaternary phosphonium chloride salt[a]
Cyphos IL 102	quaternary phosphonium bromide salt[a]
Aliquat 336	quaternary ammonium chloride salt[b]
Primene JMT, PJMTH$^+$Cl$^-$	quaternary ammonium chloride salt[c]

[a] Same organic radicals, the only difference is the anion. [b] Ammonium group derived from a tertiary amine. [c] Ammonium group derived from a primary amine.

In these series of experiments, the feed solution contained 0.01 g/L Au(III) in 1 M HCl, and the membrane phase was of 0.17 M of the corresponding ionic liquid in Solvesso 100 supported in

Durapore GVHP4700 support, as receiving phase, a 0.1 M sodium thiocyanate was used throughout the tests. Results from this investigation were shown in Fig. 5.

Practically the same permeabilities coefficients were obtained when A324H$^+$Cl$^-$ (3.8x10^{-3} cm/s) and Cyphos IL101 (3.4x10^{-3} cm/s) were used as carriers for gold(III) transport, and then the sequence was Cyphos IL102>Aliquat 336>PJMTH$^+$Cl$^-$, with permeabilities coefficients of 3.1x10^{-3}, 2.3x10^{-3} and 1.8x10^{-3} cm/s, respectively.

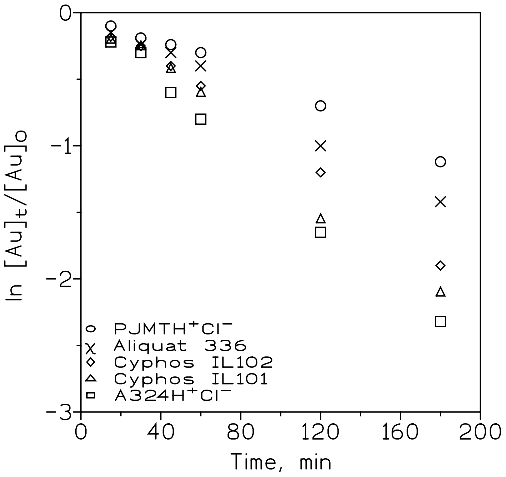

Figure 5. Permeability behaviour of gold(III) using various ionic liquids as carriers.

3.2.9. Gold(III) permeation from multielemental feed solutions

The permeation of gold(III) in the presence of other metal ions, generally accompanying the precious metal, was investigated using binary and quaternary-metal solutions. In the case of the binary solutions, these contained gold(III) in an equimolar concentration (5.1x10^{-3} M) with that of the corresponding ion (Cu(II), Fe(III) or Ni(II)) in 1 M HCl, the organic solutions were of 0.23 M carrier in Solvesso 100 immobilised in the support, and the receiving solution was of 0.1 M NaSCN. The results from these set of experiments, in the form of the separation factors, were shown in Table 6. The separation factors were calculated as:

$$\beta_{Au/M} = \frac{P_{Au}}{P_M} \qquad (12)$$

Thus, this carrier offered a good selectivity for the transport of gold(III) from these metals. This investigation were also done using a quaternary metal-bearing solution, and with the experimental conditions fixed as above. Fig. 6 presented the results of Au(III) and metals transport, the clean permeation of gold(III) was observed, and thus, the selectivity of this ionic liquid with respect to gold(III) (Table 5). It was worth to mention here, that the presence of these metal ions in the feed phase, did not appreciably influence the permeation of gold(III) with respect to what was obtained when the feed solution only contained the precious metal.

Table 6. Separation factors for the transport of gold(III) in the presence of other metals.

Feed phase	$\beta_{Au/M}$
Au(III)-Cu(II)	19
Au(III)-Fe(III)	31
Au(III)-Ni(II)	nickel was not transported
Au(III)-Cu(II)-Fe(III)-Ni(II)	23 (Cu(II)), 57 (Fe(III)), no transport of Ni(II)

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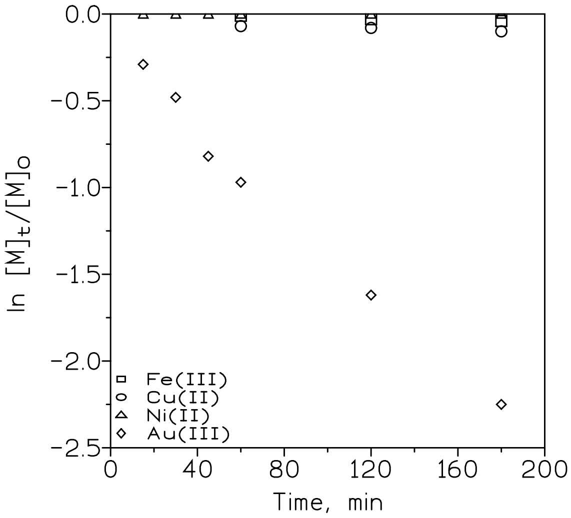

Figure 6. Transport of gold(III) in the presence of Fe(III), Cu(II) and Ni(II)

3.2.10. Precipitation of nanogold particles

Once gold(III) was released to the receiving phase, it was recovered from it by precipitation as zero valent gold nanoparticles with sodium borohydride. This salt produced hydrogen when added to the aqueous solution:

$$BH_4^- + 4H_2O \rightarrow B(OH)_4^- + 4H_2 \tag{13}$$

and this hydrogen reacted with the gold(III)-thiocyanate complex to produced zero valent gold:

$$2Au(SCN)_4^- + 3H_2 \rightarrow 2Au^0 + 6H^+ + 8SCN^- \tag{14}$$

This zero valent gold was released as gold nanoparticles (Figure 7).

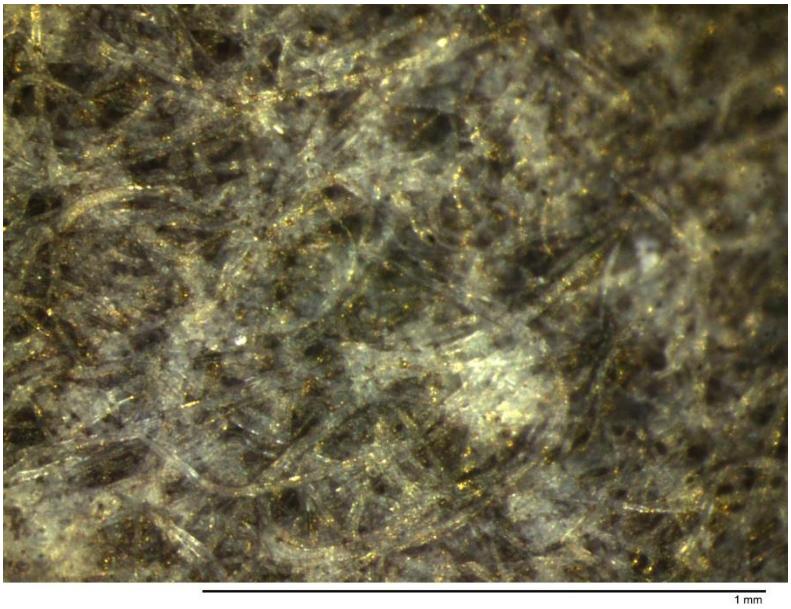

Figure 7. Gold nanoparticles after precipitation with sodium borohydride of the gold(III)-thiocyanate solution.

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4. Conclusions

It is estimated the value of the extraction equilibrium constant as 1.3x103, for gold(III) extraction, from 1 M HCl solutions, with the ionic liquid A324H+Cl- dissolved in Solvesso 100. This ionic liquid effectively transported gold(III) from HCl solutions, however, a maximum in gold(III) permeation is obtained at a HCl concentration of 1 M in the feed phase. Gold transport is controlled by the diffusion across the membrane at low carrier concentrations, then at medium carrier concentrations, limiting conditions, the transport process is controlled by diffusion in the feed phase boundary layer, finally at very high carrier concentrations in the membrane phase, gold transport decreased due to an increase of the membrane resistance in relation with the increase of the viscosity of the carrier solution. An equation, which included both diffusional and equilibrium parameters, is derived, allowing to the estimation of mass transfer coefficients as 4.1x10-3 cm/s and 1.0x10-4 cm/s for the feed and the membrane phases, respectively. Au(III) can be separated from Cu(II), Fe(III) or Ni(II). Gold is recovered, from the receiving solution, as zero valent gold nanoparticles.

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