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REMOVAL OF PALLADIUM(II) FROM AQUEOUS CHLORIDE SOLUTIONS WITH CYPHOS PHOSPHONIUM IONIC LIQUIDS AS METAL ION CARRIERS FOR LIQUID-LIQUID EXTRACTION AND TRANSPORT ACROSS POLYMER INCLUSION MEMBRANES

**Magdalena REGEL-ROSOCKA, Martyna RZELEWSKA,
Monika BACZYNSKA, Marta JANUS, Maciej WISNIEWSKI**

Poznan University of Technology, Institute of Chemical Technology and Engineering, Berdychowo 4, 60-965 Poznan, Poland, magdalena.regel-rosocka@put.poznan.pl

Abstract: This paper presents an application of three phosphonium ionic liquids for removal of Pd(II) ions from aqueous chloride solutions with liquid-liquid extraction and transport across polymer inclusion membranes (PIM). Trihexyl(tetradecyl)phosphonium chloride and bromide (Cyphos IL 101 and 102) and bis-(2,4,4-trimethylpentyl)phosphinate (Cyphos IL 104) were investigated as carriers of Pd(II) from aqueous chloride media. Extraction of Pd(II) with Cyphos IL 102 has not been previously described in the literature. Cyphos IL 102 used for the first time as metal ion carrier, efficiently extracts palladium(II) ions both with liquid-liquid extraction and PIM. NaCl concentration does not affect Pd(II) extraction with Cyphos IL 102, while increasing HCl content in the feed aqueous phase causes decrease in extraction efficiency. Stripping of Pd(II) with 0.5 M NH₄OH is efficient (84 to 90%) and the organic phase after stripping could be reused for extraction. For PIM transport the highest values of the normalized initial flux are obtained for CTA membrane containing Cyphos IL 101 and a receiving phase: mixture 0.1 thiourea + 0.5 M HCl. It is indicated that initial flux does not determine the final result of the transport through the membranes because from the separation point of view, the most important is a complete transport of the solute to the receiving phase, described by the Pd(II) recovery factor.

Keywords: *palladium(II), phosphonium ionic liquids, polymer inclusion membranes, liquid-liquid extraction*

Introduction

Platinum group metals (PGMs) form a group of precious metals consisting of six elements: ruthenium, rhodium, palladium, platinum, osmium, and iridium. Palladium is a heavy metal, and its extraction from various industrial solid wastes is important

from an economical and environmental point of view. Palladium is a valuable element (888 \$/oz, July 2014) and has wide applications in the manufacturing of catalysts in oil refineries, petrochemical industry, jewelry and electronics (Najafi et al., 2013; Swain et al., 2010). An important way to obtain noble metals (e.g. palladium) is to recover them from sources such as used catalysts, electronic scraps, anodic mud, etc. Pyrometallurgical methods have always been used for PGMs separation. However, recently growing popularity of hydrometallurgical methods is noticed for recovery of noble metals, mainly from aqueous chloride solutions (Willner and Fornalczyk, 2012). Moreover, hydrometallurgical processes are preferred as energy-saving ones (Akita et al., 1996; Kargari et al., 2006).

Solvent extraction (SX) is a favorable method for removal of PGMs from sources containing low concentrations of these metals, because it offers a number of advantages such as high selectivity and metal purity. Among conventionally used PGMs extractants are alkyl derivatives of aliphatic amines and quaternary ammonium salts (Lee et al., 2009; Najafi et al., 2013), pyridine derivatives (e.g. ACORGA CLX 50) (Regel-Rosocka et al., 2007), phosphoroorganic extractants (e.g. Cyanex 921 or 923) (Gupta and Singh, 2013; Nowotny et al., 1997) and hydroxyoximes (Reddy et al., 2010). Moreover, some acyclic analogues of thiacalix[4]arenes (Torgov et al., 2013), malonamide derivative (e.g. DMDOHEMA) (Poirot et al., 2014), thiodiglycolamide derivative (Paiva et al., 2014) and phosphonium ionic liquids (Cieszynska and Wisniewski, 2010) were investigated. Phosphonium ionic liquids (IL) have been proposed as a new group of solvents for separation processes both in extraction and adsorption systems (Cieszynska and Wisniewski, 2011; Guibal et al., 2008; de los Rios et al., 2012).

Looking for environmentally friendly separation techniques, many researchers have proposed transport across polymer inclusion membranes (PIMs) for separation of metal ions and small organic compounds as an alternative to SX (Kogelnig et al., 2011; Martak and Schlosser, 2006; Regel-Rosocka et al., 2012). PIMs are formed by casting a solution containing an extractant, a plasticizer and a base polymer (Ines et al., 2012; Nghiem et al., 2006). Up to now, PIMs are reported to be applied for separation processes including transport of precious metals, lanthanides, alkali metals, heavy metals such as Pb(II), Zn(II) or Cd(II) (Ines et al., 2012; Nghiem et al., 2006; Pospiech, 2013; Ulewicz and Radzimska-Lenarcik, 2012).

The aim of this work is to compare three phosphonium ionic liquids, for the first time tributyl(tetradecyl)phosphonium bromide (Cyphos IL 102), for the removal of Pd(II) from aqueous chloride solutions with liquid-liquid extraction and transport across polymer inclusion membranes.

Experimental

Materials

Three phosphonium ionic liquids, i.e. trihexyl(tetradecyl)phosphonium chloride and bromide (Cyphos IL 101 and 102) and bis-(2,4,4-trimethylpentyl)phosphinate (Cyphos IL 104) supplied by Cytec Industry Inc. (USA) were used as extractants or carriers for SX or PIM separations. Other organic compounds, i.e. cellulose triacetate (CTA), *o*-nitrophenyloctyl ether (NPOE), dichloromethane (DCM) were of analytical grade and used without further purification.

Liquid-liquid extraction procedure

The model aqueous solutions for extraction contained: $1 \cdot 10^{-3}$ – $5 \cdot 10^{-3}$ M Pd(II) in 0.1 M HCl with and without NaCl, $2.5 \cdot 10^{-3}$ M Pd(II) in 0.1; 1; 3 or 5 M HCl (ionic strength $I = 5$ M), $2.5 \cdot 10^{-3}$ M Pd(II) in 0.1 M HCl with 1; 3 or 5 M NaCl. $5 \cdot 10^{-3}$ M solution of extractant (Cyphos IL 102) in toluene was used as the organic phase.

Extraction has been carried out in a typical way: equimolar aqueous feeds containing metal ions added as chlorides were mechanically shaken with IL phase (volume ratio w/o = 1) for 20 min at 22 ± 2 °C in glass separation funnels and then allowed to stand for phase separation. Studies of extraction time were carried out in the time range from 1 to 60 min. Stripping of metal ions from the loaded organic phases was carried out with 0.5 M NH_4OH solution, at volume ratio w/o = 1 for 10 minutes at 22 ± 2 °C in glass separation funnels.

Transport across polymer inclusion membranes

Model aqueous feed contained $5 \cdot 10^{-3}$ M PdCl_2 in 0.1 M HCl, while 0.5 M NH_4OH , 1 and 3 M HCl or mixture of 0.1 M thiourea and 0.5 M HCl were used as the receiving phases.

PIMs were prepared from three basic substrates: polymeric matrix (CTA), carrier (phosphonium ILs), plasticizer (ONPOE). A portion of three-substrate solution in solvent (DCM) was casted on a 7.0 cm diameter Petri dish. The organic solvent was allowed to evaporate overnight and the resultant membrane (flexible, transparent, homogenous and strong thin film) was separated from a glass plate. Before membranes were fixed in the sandwich-type membrane module, presented in the previous paper (Regel-Rosocka et al., 2012), the membranes were soaked for 24 h in distilled water to achieve their homogeneity. The membrane thickness was measured by digital ultrameter (MG-401, ELMETRON, Poland) in at least 15 different sites of the membrane surface.

Pd(II) determination

Atomic absorption spectrometer (Hitachi Z-8200) was used for Pd(II) determination in the aqueous solutions before and after extraction and in the feed aqueous phase and receiving phases during PIM separation at 247.6 nm in the air-acetylene flame.

Results and discussion

Extraction and stripping of Pd(II) with Cyphos IL 102

As SX of Pd(II) with Cyphos IL 101 and 104 was presented elsewhere (Cieszynska and Wisniewski, 2011) in the paragraph referring to liquid-liquid extraction there are presented data only on Cyphos IL 102.

As shown in Fig. 1, extraction equilibrium state was established quickly with Cyphos IL 102 as an extractant. After 1 min of shaking constant values of $E_{Pd(II)}$ were obtained. This fast extraction kinetics with IL 102 is in accordance with the results obtained in our team for the other phosphonium ILs (Cieszynska et. al., 2007; Cieszynska and Wisniewski, 2011). Moreover, extraction efficiency of Pd(II) with Cyphos IL 102 was high; almost 100% Pd(II) was extracted from 0.1 M HCl which was confirmed by change in color of the aqueous phase. All feeds were orange before extraction, but after extraction from 0.1 M HCl solutions were discolored. The initial rate of metal ion transfer to the organic phase containing $5 \cdot 10^{-3}$ M Cyphos IL 102 in toluene was equal to $4 \cdot 10^{-5}$ mol/dm³·s.

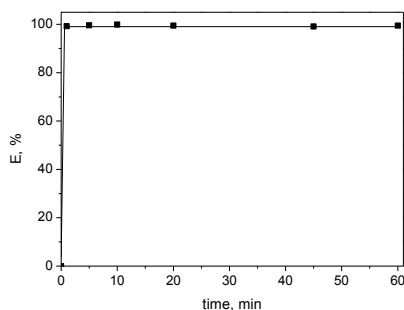


Fig. 1. Extraction of Pd(II) from 0.1 M HCl with $5 \cdot 10^{-3}$ M Cyphos IL 102 (feed: $2.5 \cdot 10^{-3}$ M Pd(II))

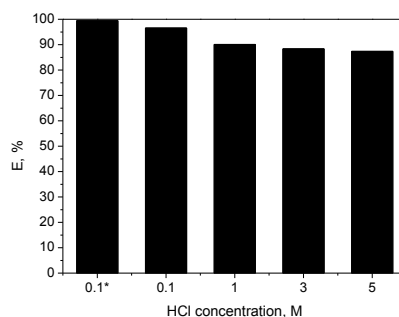


Fig. 2. Effect of HCl concentration on the efficiency of Pd(II) extraction with $5 \cdot 10^{-3}$ M Cyphos IL 102 (feed: $2.5 \cdot 10^{-3}$ M Pd(II), 0.1; 1; 3 or 5 M HCl; *without NaCl; I = 5 M)

After the organic phases were loaded with metal ions, Pd(II) was stripped with 0.5 M NH₄OH. This stripping of Pd(II) was rapid and steady state of the process was obtained in the period from 1 to 5 min. As a result of extraction the organic phase was loaded with Pd(II) up to 260 mg/dm³, and stripping with 0.5 M NH₄OH solution allowed it to recover 220 - 230 mg/dm³ Pd(II). It means that the Pd(II) transport from the organic to the stripping phase is efficient (84 to 90%) and the organic phase after stripping could be reused for extraction.

As Pd(II) forms anionic chlorocomplexes, its extraction depends on the concentration of HCl and chloride anions in the aqueous feed. Therefore, an effect of HCl concentration in the feed aqueous phase on Pd(II) extraction has been investigated and the results are shown in Fig. 2. With the increase in HCl

concentration in feed phase, the extraction efficiency slightly decreased, which was consistent with the results for Cyphos IL 101 and IL 104. The highest percentage extraction (about 100%) was achieved without NaCl addition.

The effect of ionic strength (I) on Pd(II) extraction was also verified. The Pd(II) extraction efficiency to the IL phase was equal to 99.5; 99.7; 97.5 and 98.5% from feed solutions containing 0.1; 1; 3 and 5 M NaCl, respectively ($I = 0.11$; 1.11; 3.11 and 5.11 M, respectively). Extraction efficiency with and without NaCl was almost at the same level. Percentage extraction of Pd(II) at 3 M chloride concentration was about 3% lower (which is within error limits) than at the other concentrations. It means that Pd(II) extraction with Cyphos IL 102 was not affected by NaCl concentration. NaCl presence in the aqueous phase is advantageous because of an improvement of phase separation after extraction.

Pd(II) extraction isotherms

Finally, the equilibrium of Pd(II) extraction has been investigated. The extraction isotherms of Pd(II) with Cyphos IL102 are presented in Fig. 3.

Pd(II) extraction isotherms shown in Fig. 3 are not linear and both reach plateau. It means that this organic phase could be loaded up to 300, 350 mg/dm³ ($2.8 - 3.3 \cdot 10^{-3}$ M) depending on the NaCl content in the feed aqueous phase. As in the previous cases, extraction occurred slightly better without the addition of NaCl than in the presence of the salt.

Positive results of Pd(II) extraction with the phosphonium ILs studied encouraged the authors to apply the phosphonium ILs as metal ion carriers in transport across polymer inclusion membranes.

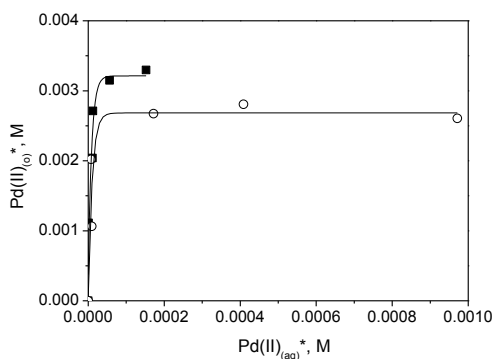


Fig. 3. Isotherms of Pd(II) extraction from the feed $1 \cdot 10^{-3} - 5 \cdot 10^{-3}$ M Pd(II) in 0.1 M HCl without NaCl (■) and with 1 M NaCl (○); the organic phase $5 \cdot 10^{-3}$ M IL102 (* denotes equilibrium concentrations)

Pd(II) transport across PIMs

Transport of metal ions through PIM can be described by initial flux (J_0 , mol/s·m²), defined in detail elsewhere (Resina et al., 2008). Depending on the amount of the membrane constituents casted on a glass dish, thickness of the membranes formed ranged from 29 to 56 μm. As the membranes differed with the thickness that might affect the transport resistance and cause decrease in metal ion flux. Therefore a normalized initial fluxes (J_N) were calculated (Regel-Rosocka et al., 2012). The thinnest CTA membrane containing Cyphos IL 102 was chosen as a reference and metal ion fluxes were calculated as follows:

$$J_N = J_0 \cdot \frac{d_M}{d_R} \quad (1)$$

where d_R was the reference membrane thickness (equal 29 μm), d_M corresponded to the thickness of the membrane which flux was normalized.

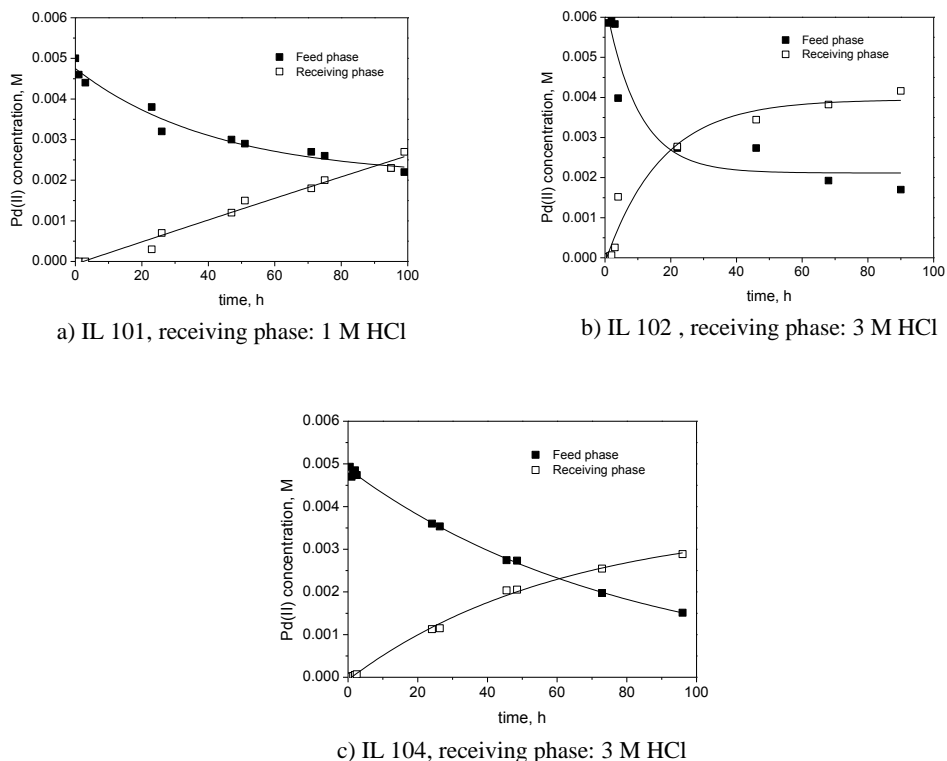


Fig. 4. Pd(II) concentration profiles in the feed aqueous phase and receiving phases vs. time of PIM process

Transport of Pd(II) profiles through PIMs containing CTA, and various Cyphos ILs (that differ with an anion) are shown in Fig. 4. For membrane containing Cyphos IL 101 1 M HCl was used as a receiving phase because 3 M HCl solution was too acidic, and caused damage of the membrane. It should be noticed that the mass transfer across membranes was rather slow due to small membrane area. Thus, significant increase in Pd(II) concentration in the receiving phase was reported after 60 - 70 h of transport process.

Pd(II) concentration in the feed aqueous phase decreased faster for membrane containing Cyphos IL 102 as a carrier and 3 M HCl as receiving phase, and finally reached lower values compared to membrane containing Cyphos IL 104 and the same receiving phase.

Extraction efficiency of palladium(II) was calculated using Eq. 2:

$$E = \frac{c_0 - c}{c_0} \cdot 100\% \quad (2)$$

where c_0 and c are Pd(II) concentrations in the feed aqueous phase and in the aqueous phase after 90 h and normalized initial flux of palladium(II) are compared in Fig. 5 for different receiving phases.

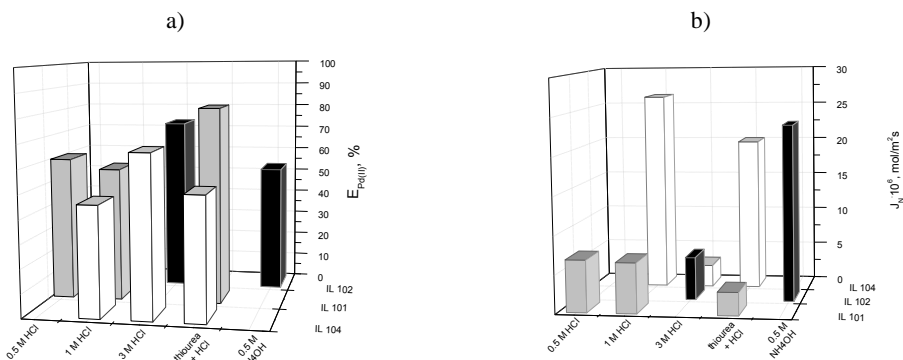


Fig. 5. Percentage extraction of Pd(II): (a) in the end of the process and normalized initial flux of Pd(II) (b) from the feed aqueous phase to the various receiving phases

It is noticeable that with increasing HCl concentration for membranes with IL 101 $E_{Pd(II)}$ decreased, only mixture of thiourea and HCl was almost twice as efficient as HCl solutions. On the other hand, Pd(II) transport through membranes containing IL 104 increased with increasing HCl content in the receiving phase. It is also noteworthy that initial fluxes for two processes with IL 104 carrier were high, while finally $E_{Pd(II)}$ values were not so significant. On the contrary, initial flux for IL 101 carrier and 0.1 M thiourea + 0.5 M HCl receiving phase was rather low, while Pd(II) extraction at the end of the process amounted to 80%. The J_N values were low for Cyphos IL 104 and 3 M HCl as receiving phase and Cyphos IL 101 (receiving phase mixture thiourea and

HCl, Fig. 5b). It can be stated that initial flux did not determine the final result of the transport through the membranes. The highest values of the normalized initial flux were obtained for CTA membrane containing Cyphos IL 101 and the receiving phase: mixture 0.1 thiourea + 0.5 M HCl. Since receiving phase containing thiourea and HCl caused formation of thin layer of precipitation at the end of the process, for Cyphos IL 102 membranes ammonium solution was applied to strip Pd(II) from the membrane and was found to be effective. However, from the point of view of further processing of the receiving phase, chloride receiving phase is more advantageous. Thus, ammonium solution was not used as stripping phase for other membranes.

The highest values of extraction efficiency were obtained for CTA membranes containing Cyphos IL 102, Cyphos IL 104 and 3 M HCl as receiving phase and also Cyphos IL 101 and mixture 0.1 M thiourea + 0.5 M HCl (Fig. 5a).

Another issue to be emphasized is the difference between $E_{Pd(II)}$ and recovery factor (RF) values. RF was defined, following equation 3, as the ratio of Pd(II) transferred to the receiving phase (c_s) after 90 h of the process and the initial Pd(II) content in the feed aqueous phase (c_0):

$$RF = \frac{c_s}{c_0} \cdot 100\% . \quad (3)$$

The RF values for two different receiving phases are shown in Fig. 6.

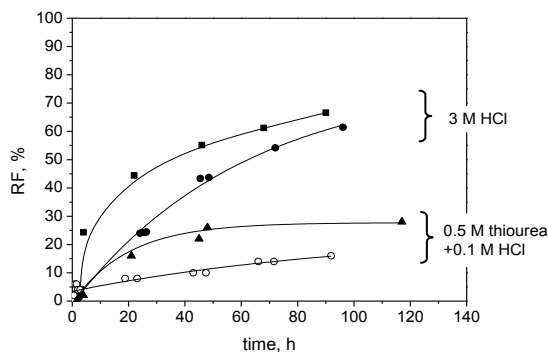


Fig. 6. Pd(II) recovery factor vs. time of transport through PIMs containing Cyphos IL 102 (■), Cyphos IL 104 (●, ○), Cyphos IL 101 (▲) as the ion carriers

From the separation point of view, the most important is a complete transport of the solute to the receiving phase, described by RF . However, not always RF of Pd(II) was equal to percentage extraction. In most cases more Pd(II) was extracted to the membrane (e.g. 73% for IL 102) than was recovered to the receiving phase (66% for IL 102). The values of $E_{Pd(II)}$ and RF strongly depended on the type of carrier and the receiving phase. For instance, the recovery factor values of Pd(II) were significantly higher when 3 M HCl was used as the receiving phase than thiourea + HCl mixture.

Conclusions

Generally, Cyphos IL 102 is an efficient extractant of Pd(II) from aqueous chloride solutions. Addition of NaCl and increasing HCl content in the feed aqueous phase slightly reduces the efficiency of Pd(II) extraction. Comparing with previous research of our team it can be stated that anion type of the phosphonium ILs studied has no influence on liquid-liquid extraction efficiency.

The results of the studies have also shown that the efficiency of PIMs containing phosphonium ILs depends mainly on the type of receiving phase. Phosphonium ILs studied, especially Cyphos IL 102, used for the first time as a metal ion carrier – efficiently extract Pd(II) both in liquid-liquid extraction and transports across polymer inclusion membranes. Transport of Pd(II) ions is better for carriers with small anion (Cyphos IL 101 and IL 102) than with carrier with big anion (Cyphos IL 104). The highest extraction efficiency was obtained for Cyphos IL 101 carrier and receiving phase composed of thiourea + HCl. 3 M HCl efficiently strips Pd(II) from PIMs containing Cyphos IL 102 and Cyphos IL 104. Though the greatest initial normalized flux values are obtained for the membranes containing Cyphos IL 101 (receiving phase: mixture 0.1 M thiourea + 0.5 M HCl) and Cyphos IL 102 (receiving phase: 0.5 M NH₄OH) Pd(II) recovery factors obtain the highest values (above 60%) for Cyphos IL 102 and 104 and 3 M HCl receiving phase.

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