ORIGINAL PAPER

Preparation and application of supported ionic liquid phases for sorption of Pt(IV) from chloride solution

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Abstract

Four ionic liquids (ILs) 1-methyl-3-tetradecylimidazolium chloride ([C_{14}MIM]Cl), 1-n-butyl-3-n-butylimidazolium chloride ([C_4BIM]Cl), 1-n-butyl-3-n-tetradecylimidazolium chloride ([C_{14}BIM]Cl), and 1-tetradecylpyridinium chloride ([C_{14}Py]Cl) were synthesized. Sorbents in nature of supported ionic liquid phases (SILPs) were prepared by impregnating ILs on solid carriers such as silica gel, XAD-7 and XAD-4 Amberlite ion exchange resins. Pt(IV) was recovered using these sorbents with the yields over 90%, however, the amounts of used ILs are much less than those of ILs used in extraction method and the process can easily be transferred to heterogeneous phase. The Pt(IV) sorption is affected by the structure of ILs and solid carriers, the IL loading, and IL/Pt molar ratio. The highest sorption yields were observed when using [C_{14}MIM]Cl-based SILPs. Moreover, the sorption isotherms of [C_{14}MIM]Cl/SiO_2 SILPs with different IL loadings and IL/Pt molar ratios were investigated and sorption mechanism was also proposed.

Keywords Ionic liquid · Sorption · Recovery of Pt(IV) · Sorption isotherms · Supported ionic liquid phase

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Introduction

Spent catalysts from oil refinery plants and catalytic converters of automobiles containing a large amount of precious metals mainly platinum while their resources are currently limited. Therefore, the recovery of platinum from these spent catalysts shows environmental and economic significance (Sun and Lee 2013). During recovering process, platinum need to be separated from its leaching solution. In this step, metals can be recovered by several methods such as liquid–liquid extraction (Rzelewska-Piekut and Regel-Rosocka 2019; Yamada et al. 2018; Yoshida et al. 2017), precipitation (Siame et al. 2013; Phetla et al. 2010), electrolytic processes (Dawson and Kelsall 2016), and solid–liquid extraction (Carausu et al. 2013); sorption (Kononova et al. 2010). Besides the advantages, these methods have some drawbacks. Precipitation method cannot fully recover Pt and only appropriates when recovering Pt from high concentration solution. Electrochemical methods need high cost and further treatment of waste solution after electrolysis process. Some liquid–liquid extraction method brings low phase distribution coefficient (Sun and Lee 2013; Lee et al. 2008). Therefore, Pt(IV) sorption method using sorbents prepared by immobilizing liquid solvents on solid carriers is received a great deal of attention (Kononova et al. 2010).

However, some sorption disadvantages such as release (loss) of hazardous and expensive extractants can be pointed out (Navarro et al. 2012).

Ionic liquids belong to an interesting group of compounds that can be used in many different fields. They are considered as green, designable solvents and catalysts for reactions and processes (Sawant et al. 2011; Bui et al. 2012; Handy 2011). Many researches focus on application of ionic liquids for separation technology such as desulfurization, CO_2 separation, gas purification (Bui et al. 2017; Ito et al. 2017; Friess et al. 2017; Anantharaj and Banerjee 2011) and ionic liquids also show an interesting potential for extraction of Co(II), Ni(II) or Zn(II) (Belhadj et al. 2019; Baczyńska et al. 2016; Wellens et al. 2013) and for platinum extraction (Yan et al. 2018; Rzelewska-Piekut and Regel-Rosocka 2019). Also, the use of supported ionic liquid phase to recover the precious metals has been widely interested. This process employs the extraction solvents capable of Pt recovery, supports them onto solid carriers to increase the exposure of solvent with the solution to be separated and therefore increase sorption performance, facilitate phase separation as well as design of a continuous, convenient recovery process in which absorbed materials are easily to be recycled.

Remarkably, ionic liquids can be immobilized on biopolymer (Guibal et al. 2009) or impregnating on solid carriers for metal sorption (Wieszczycka et al. 2020a, b). Biopolymers are more environmentally friendly but unstable and there are solvent losses resulting in the decrease in the absorption performance of Pt on polymer through the repeated extractions. A phosphonium IL (Cyphos® IL-101) was impregnated on biopolymer and on ionic exchange resin to adsorb Pt (Kononova et al. 2008; Navarro et al. 2012). Three ammonium ILs were used for Pt(IV) extraction and impregnating on solid carriers for Pt(IV) sorption from chloride solution and showed promising results (Bui et al. 2020). Several researches using mixture of [C$_6$mim]Cl, [C$_6$mim][NTf$_2$], and [C$_6$mim][DDTC] (1-Hexyl-3-methylimidazolium diethyldithiocarbamate) for separation of Pt(IV), Pd(II), Ru(III), and Rh(III), however, the recovery of ionic liquids was not mentioned (Yan et al. 2018). In order to choose selective, lower price, more common and available ionic liquids, the present work focuses on the synthesis of some imidazolium chloride ionic liquids ([C$_{14}$MIM]Cl, [C$_4$BIM]Cl, [C$_{14}$BIM]Cl) and a pyridinium chloride ionic (C$_{14}$Py]Cl liquid and on the preparation of some Pt(IV) sorbents by impregnating ILs on some solid carriers. It was the subject of this work to use only ionic liquids based on chloride anion for sorption of Pt(IV) because of their availability and moderate cost. The additional reason is all ionic liquids and SILPs could be recovered by stripping with chloride anion containing eluent systems to transfer X$_2$PtCl$_6$ complex back to chloride ionic liquids again (chloride ions are exchanged with PtCl$_6^{2-}$ ion during the stripping to recover the ILs).

Furthermore, sorbents prepared from these chloride ionic liquids are stable (chloride ions are not hydrolyzed in contact with water).

XAD-7 resin with acrylic ester nature is moderate polar and hydrophilic. It was used as carrier for impregnating phosphonium IL for sorption of Pt(IV) and gave high yield (Navarro et al. 2012). It is known that silica gel is high polar and hydrophilic while XAD-4 resin with polyaromatic nature is unpolar and hydrophobic (Sigma-Adrich and Merck product information). Therefore, the focus of this work is to use three solid carriers (silica gel, XAD-4, and XAD-7 resins) with different chemical nature and different properties in surface area, pore size, surface polarity, and hydrophilicity for preparing Pt(IV) sorbents (supported ionic liquid phase—SILPs); the obtained results would show the influence of carrier properties on Pt(IV) sorption performance. The sorption of Pt on SILPs as well as the influence of structure of ILs and solid carriers, of IL loading on carriers, and of IL/Pt molar ratio on Pt sorption ability were evaluated. In this work, the sorption isotherms with different IL loadings, and at different IL/Pt molar ratio are also systematically investigated.

Experimental

Materials

Hexachloroplatinic acid, ≥ 99.9% trace metals basis was supplied as reagent grade products by Acros Organics. Acetone (99.9%) and diethyl ether (99.9%) were purchased from Guangdong, China and used as received. Silica gel SiO$_2$-60, thin layer chromatography plate, 25DC-alufolien 20×20 cm Kiesel gel F$_{254}$, 1-methylimidazol, butylimidazol (99.5%), 1-chlorobutane (99.8%), 1-chlorotetradecane (99.8%) were purchased from Merck and used without further purification. Amberlite XAD-7 and XAD-4 ionic exchange resins (99%) were supplied by Sigma-Aldrich. The properties of silica gel and ionic exchange resins are presented in Table 1 and Fig. 1 (Sigma-Adrich and Merck product information, Deka et al. 2017). Amberlite XAD-7 and XAD-4 exchange resins (99%) were cleaned by contacting with acetone for 24 h at 25 °C then filtered under vacuum to remove acetone. The resin was washed with nitric acid (0.1 M) for 24 h and rinsed with demineralized water until the waste water becomes neutralized. Finally, the resin was contacted with ketone for 12 h, filtered and dried under reduced pressure at 50 °C.

Synthesis of ionic liquids

Ionic liquids were synthesized using modified conventional heating method of Bui et al. (2017).

Table 1 Characteristics of different carriers (Sigma-Adrich and Merck product information, Deka et al. 2017, Frederik et al. 2001) and Pt sorption yields of carriers and SILPs prepared from different carriers and [C$_{14}$MIM]Cl [T = 25 °C; t = 48 h, V_{IL}/V_{por} (IL loading) = 20%; IL/Pt = 6:1 (mol)]

Solid carrier	Chemical nature	Average pore diameter (A°)	Surface area (m² g⁻¹)	Pore volume (cm³ g⁻¹)	pH working range	Pt sorption yield (%) on carriers	Pt sorption yield (%) on SILPs
SiO₂-60	Hydrophilic	60	510	0.79	0–14	4.7	99.96
XAD-4	Hydrophobic (polystyrene-divinylbenzene, nonionic resin)	90	725	1.14	0–14	3.5	91.67
XAD-7	Hydrophilic (acrylic ester)	90	450	1.14	0–14	5.8	99.88

1-methyl 3-n-teradecyl imidazolium chloride [C$_{14}$MIM]Cl (Bui et al. 2017)

1-methylimidazole (1.232 g, 0.015 mol) and 1-chlorotetradecane (3.502 g, 0.015 mol) were added in a flask containing a magnetic stir bar. The flask was connected to a reflux condenser and immersed in an oil bath. The reaction mixture was stirred at 90 °C for 72 h. The ionic liquid was washed three times with diethyl ether to remove any unreacted material and then dried at 50 °C under vacuum (yield = 93.43%).

1-n-butyl-3-n-butylimidazolium chloride [C$_4$BIM]Cl (Bui et al. 2017)

1-butylimidazole (3.725 g, 0.03 mol) and 1-chlorobutane (3.330 g, 0.03 mol) were added in a flask containing a magnetic stir bar. The flask was connected to a reflux condenser and immersed in an oil bath. The reaction mixture was stirred at 90 °C for 72 h. The ionic liquid was washed three times with diethyl ether to remove any unreacted material and then dried at 50 °C under vacuum (yield = 84.82%).

1-n-butyl-3-n-tetradecylimidazolium chloride [C$_{14}$BIM]Cl (Bui et al. 2017)

1-butylimidazole (3.725 g, 0.03 mol) and 1-chlorotetradecane (6.975 g, 0.03 mol) were added in a flask containing a magnetic stir bar. The flask was connected to a reflux condenser and immersed in an oil bath. The reaction mixture was stirred at 90 °C for 120 h. The ionic liquid was washed three times with diethyl ether to remove any unreacted material and then dried at 50 °C under vacuum (yield = 91.04%).

1-n-tetradecylpyridinium chloride ([C$_{14}$Py]Cl) (Bui et al. 2017)

Pyridine (3.950 g, 0.05 mol) and 1-chlorotetradecane (11.625 g, 0.05 mol) were added in a flask containing a magnetic stir bar. The flask was connected to a reflux condenser and immersed in an oil bath. The reaction mixture was stirred at 90 °C for 120 h. The ionic liquid was washed three times with diethyl ether to remove any unreacted material and then dried at 50 °C under vacuum (yield = 92.03%).

To get high pure ILs, which was used as standard for analysis, thin layer chromatography method was applied.

The molecular structures of the four ionic liquids are given in Fig. 2.

Impregnation of ILs on solid carriers

In this part four ionic liquids were impregnated on three solid carriers to form 12 different kinds of SILPs. Each kind of SILP was prepared with V_{IL}/V_{por} ratios (IL loading)

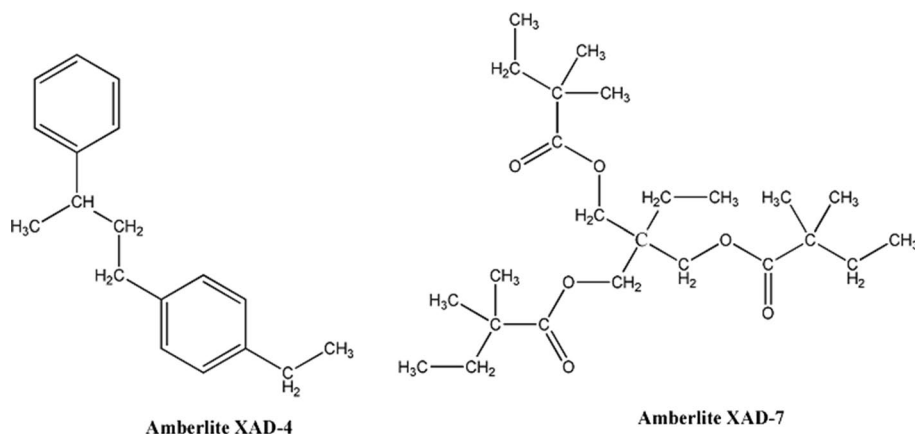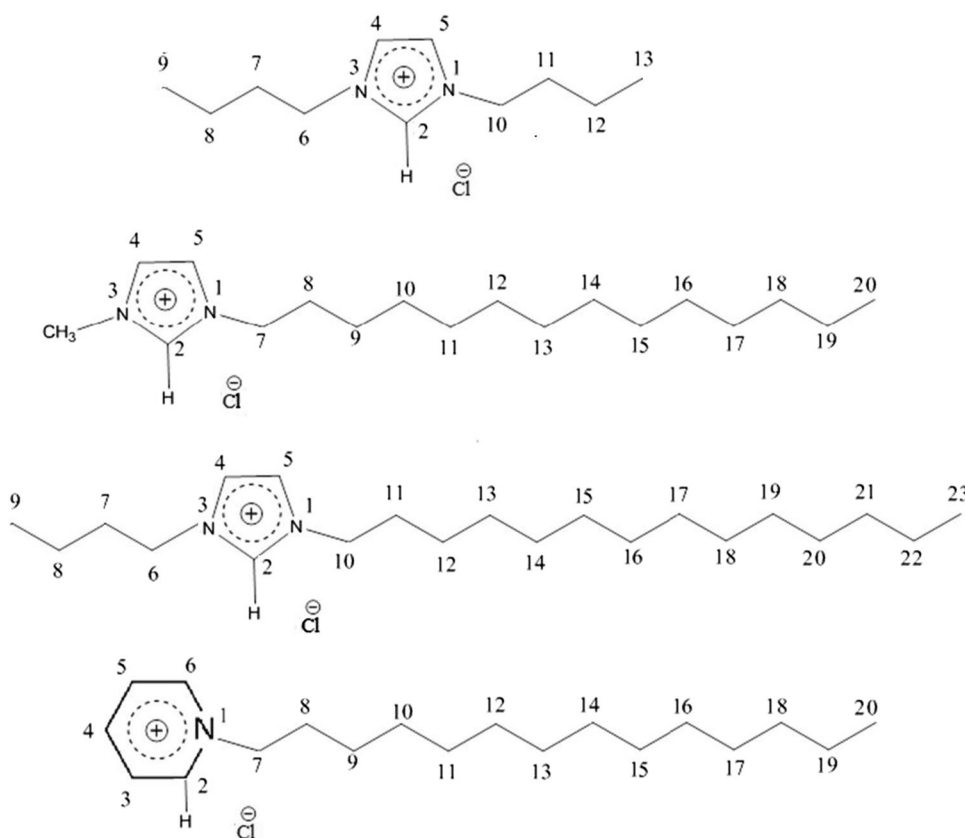**Fig. 1** Structure of Amberlite XAD-4 and XAD-7 resins (Deka et al. 2017)

Amberlite XAD-4

Amberlite XAD-7

Fig. 2 Structure of [C$_4$BIM]Cl, [C$_{14}$MIM]Cl, [C$_{14}$BIM]Cl, and [C$_{14}$Py]Cl ionic liquids

of 10%, 20%, and 30% (calculated by Eq. 1). For each experiment, 5 g of solid carriers were contacted with 25 mL of acetone for 24 h to form carrier slurry. The amount of 0.5 M solution of ionic liquid used for each experiment was calculated in order to form the SILP with a certain V_{IL}/V_{por} ratio. For example, to prepare SILP based on SiO$_2$ and [C$_4$BIM]Cl (with IL loading of 20%) 4.79 mL of 0.5 M solution of this ionic liquid in acetone was added into the carrier slurry and the mixture was agitated for

24 h by HY-2 Laboratory shaker. Acetone was removed by slowly evaporation to get SILP [C$_4$BIM]Cl/SiO$_2$ 20%. The IL loading was calculated by V_{IL}/V_{por} (%) ratio as follow:

$$\frac{V_{IL}}{V_{por}}(\%) = \frac{m_{IL}}{d_{IL} \times V_{por}} \times 100\% \qquad (1)$$

where m_{IL} and d_{IL} are mass (g) and density (g mL^{-1}) of ionic liquid; V_{por} is porous volume of solid carrier (Table 2).

Table 2 BET measurements of solid carriers and SILPs (IL loading of 20%)

Properties	SiO_2	IL/SiO_2	XAD-4	IL/XAD-4
Surface area ($m^2\ g^{-1}$)	510	195	725	504
Pore volume ($cm^3\ g^{-1}$)	0.80	0.26	0.98	0.70
Pore diameter (Å)	60	24	50	24

The experimental procedure allowed the preparation of SILPs with V_{IL}/V_{por} ratios of 10%, 20%, and 30%. SILPs were stored in desiccator for sorption experiments.

Characterization

The Pt(IV) contents in samples were measured by ICP-MS PE7300DV-Perkin–Elmer. Infrared (IR) spectra of the prepared ionic liquids were obtained using a Bruker Tensor 37 spectrometer. The samples were measured using the method of a KBr pellet. Nuclear magnetic resonance spectra 1H NMR and ^{13}C-NMR were measured using Bruker AM0 FT-NMR Spectrometer (at 500 MHz for proton and at 125 MHz for ^{13}C) in $CDCl_3$. Textural properties of SILPs were investigated by NOVA 1000e Brunauer–Emmett–Teller (BET) instrument.

Sorption of Pt(IV) using SILPs

Pt in spent catalyst was leached by using H_2O_2 in 9 M HCl solution (Sun and Lee 2013). Therefore, in order to simulate the Pt recovery in industry, the model leaching solution (9 M HCl solution containing 460 ppm of Pt(IV) (H_2PtCl_6) was used for sorption experiments.

The sorption was performed by mixing the calculated amount of sorbents with the H_2PtCl_6 solution as described above for 48 h. The contact was operated on HY-2 Laboratory Shaker with an agitation speed of 150 movements per minute at constant room temperature. Pt(IV) remained in aqueous phase was analyzed by inductively coupled plasma mass spectrometry. The amount of different IL loading sorbents was varied in order to get different IL/Pt ratios in ranges of sorption experiments. The yields of Pt(IV) recovery in extraction and sorption experiments were determined by mass balance equation:

$$\text{Yield}(\%) = \frac{Vx(C_o - C_{eq})}{VxC_o} \times 100\% \tag{2}$$

where yield is Pt(IV) recovery efficiency (%), C_o is Pt(IV) concentration before sorption (mg Pt L^{-1}), and C_{eq} is the residual Pt(IV) concentration (mg Pt L^{-1}).

Sorption isotherms at different IL/Pt molar ratios

Experiments of influence of IL loading and sorption isotherms were plotted by mixing calculated amounts of SILP with a fixed volume (5 mL) of H_2PtCl_6 solutions so that the IL/Pt molar ratio varied from 1 to 10. At equilibrium (i.e. after 24 h of contact), the residual Pt(IV) concentration was used to calculate the Pt(IV) sorption yield and capacity.

Recovery of Pt(IV)

In this work, Pt(IV) was recovered from SILP (containing X_2PtCl_6 complex dissolved in IL (X is cation of IL) after sorption) using thiourea/HCl as stripping reagent.

SILP was mixed with stripping reagent at 80 °C for 20 min. After cooling down to room temperature, the mixture was transferred to a separatory funnel and left still until the phase separation is completed. The aqueous layer was separated and analyzed for the Pt(IV) content in order to calculate the Pt recovery yield. The ionic liquid was dried under 50 mmHg at 80 °C for 1 h.

$$\text{Recovery}(\%) = \frac{m_1}{m_o} \times 100\% \tag{3}$$

where m_1 is mass of stripped Pt; m_o is mass of Pt loaded in SILP phase.

The sorbent was then dried under 50 mmHg at 80 °C.

Results and discussion

To determine the reproducibility of the experiments and analysis, three experiments were performed under the same conditions. The products of each experiment were analyzed three times and the mean values were used for evaluation. The results show that the reproducibility of the experiments and analysis is acceptable. The analysis has a good reproducibility ($> 97\%$) and the Pt(IV) sorption and desorption match in a range of 97–99% reproducibility. It is therefore better to evaluate the Pt(IV) recovery by repeated experiments with a repeated analysis of the products, and thus at least three analysis were done for all experiments presented in this work.

Sorption of Pt(IV)

Influence of IL structure and IL/Pt molar ratio

In order to evaluate the Pt(IV) sorption on SILPs and the difference between the adsorption of Pt(IV) on solid carriers (silica gel, XDA-4, and XAD-7 resins) and SLIPs.

The Pt(IV) sorption of pure solid carriers was determined. As presented in Table 2, the sorbents themselves can only adsorb Pt(IV)-ion with very low yields. The very low Pt(IV) sorption yields (around 3–6%) of the four pure solid carriers confirmed that ILs bring the high Pt(IV) sorption capacity of the four SILPs.

The Pt(IV) sorption of all SILPs increased with increasing the IL/Pt molar ratio (Fig. 3). Depending on solid carrier and IL structure, the suitable IL/Pt molar ratio changes. The Pt(IV) sorption of [BBIM]Cl and [C$_{14}$MIM]Cl-based SILPs increases with the increasing length of the 3-alkyl side chain of imidazolium ring. These can be explained by the increased van der Waals volume leads to a decrease in hydrophilicity of ionic liquids and an increase in stability of the imidazolium cation and Pt(IV) efficiency. Pt(IV) recovery yields of [C$_4$BIM]Cl-based SILPs were in the range of 11.58–49.41% when the IL/Pt molar ratio is 5–10. The yield reached 45.37% when using [C$_4$BIM]Cl/XAD-4 SILPs and stood about 80% when [C$_4$BIM]Cl/XAD-7 and [C$_4$BIM]Cl/SiO$_2$ SILPs were used at IL/Pt molar ratio of 10. Pt(IV) sorption yield of [C$_{14}$BIM]Cl/SiO$_2$ was higher than 90% and of [C$_{14}$BIM]Cl/XAD-7 reached nearly 100%, especially the yields were stably high at IL/Pt ratios from 6 to 10.

Pt(IV) sorption of SILPs based on [C$_{14}$BIM]Cl and [C$_{14}$MIM]Cl was also compared in order to investigate the influence of the second alkyl side chain of imidazolium ring (at 1-position of the ring).[C$_{14}$MIM]Cl-based SILPs gave Pt(IV) recovery yields higher than [C$_{14}$BIM]Cl-based SILPs. Pt(IV) recovery yields of SILPs based on [C$_{14}$MIM]Cl and XAD-4 resin, XAD-7 resin, silica gel-60 were 60.21%, 99.9%, and 99.64%, respectively at IL/Pt molar ratio is 5. At IL/Pt ratio of 6 Pt(IV) sorption of [C$_{14}$MIM]Cl-based SILPs reached nearly 100% and stood stably high at IL/Pt ratio from 6 to 10. These can be justified that the two long alkyl side chains hinder the access of hexachloroplatinate ions to the imidazolium ring.

In order to investigate the influence of structure of ring of cation of ionic liquids on the Pt(IV) sorption capacity, [C$_{14}$Py]Cl-based SILPs were prepared and used for Pt(IV) sorption from chloride solution. With all kinds of solid carriers, Pt(IV) sorption yields of SILPs followed the order: [C$_{14}$MIM]Cl > [C$_{14}$BIM]Cl > [C$_{14}$Py]Cl > [C$_4$BMIM]Cl. The results show that even though [C$_{14}$MIM]Cl, [C$_{14}$BIM]Cl, and [C$_{14}$Py]Cl have the similar number of carbon in the first side chain (at 3-position) of the ring, [C$_{14}$Py]Cl-based SILPs have lower Pt(IV) sorption yield. This can be due to the fact that imidazolium ion contains five-member ring with two nitrogen atoms those connect to two alkyl groups while pyridinium ion contains six-member ring with one nitrogen atom that connect to one alkyl group. Therefore, [C$_{14}$MIM]Cl is less viscous, more hydrophobic, and more stable on SiO$_2$ pore surface than [C$_{14}$Py]Cl, which leads to a higher Pt(IV) sorption of [C$_{14}$BMIM]Cl-based SILPs. Compared
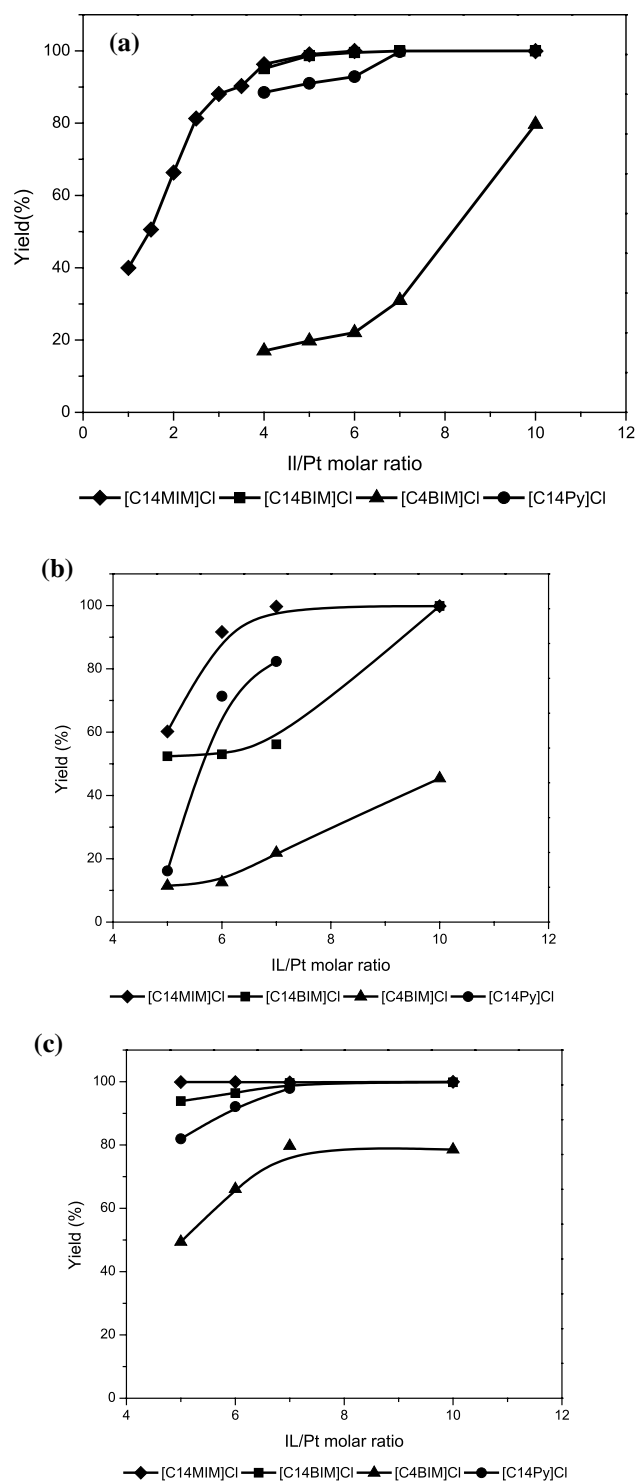

Fig. 3 Pt(IV) sorption of SILPs with V_{IL}/V_{por} (IL loading) of 20% on: **a** silica gel, **b** XAD-4 exchange resin, and **c** XAD-7 exchange resin (48 h and 25 °C)

Fig. 4 Influence of solid carriers on Pt(IV) sorption of SILPs with **a** ▶ [C$_{14}$MIM]Cl, **b** [C$_{14}$BIM]Cl, **c** [C$_4$BIM]Cl and **d** [C$_{14}$Py]Cl loading of 20% on different solid carriers (48 h and 25 °C)

to ammonium based SILPs [35], [C$_{14}$MIM]Cl and [C$_{14}$Py] Cl-based SILPs gave higher Pt(IV) sorption yields because cations [C$_{14}$MIM]$^+$ and [C$_{14}$Py]$^+$ are more stable and more easily accessible than [NR$_4$]$^+$.

Influence of solid carriers

It can be seen from the experimental results (Fig. 4) that all SILPs prepared from SiO$_2$-60 and four ILs adsorb Pt(IV) better than corresponding those from exchange resins (XAD-4, XAD-7) with the same IL/Pt molar ratio. For example, the Pt(IV) sorption yields of [C$_{14}$MIM]Cl-based SILPs at IL loading of 20% and the IL/Pt molar ratio of 4 are 96.31%; 50.63%; 94.11% and 6 are 99.96%; 91.67%; 99.73%, respectively. Similarly, the Pt(IV) sorption yields of [C$_{14}$BIM]Cl, [C$_4$BIM]Cl, and [C$_{14}$Py]Cl-based SILPs at IL loading of 20% and the IL/Pt molar ratio of 5 are 99.79%; 52.39%; 93.85% and 49.41%; 11.48%; 19.76%; and 91.02%; 16.18%; and 81.96%, respectively. Surface area and chemical nature (polarity and hydrophilicity) of the solid carriers are the important factors that affect Pt(IV) sorption. High surface area leads to a high phase contact between IL and aqueous phase containing Pt(IV), therefore, Pt(IV) sorption yields of SiO$_2$-60's SILPs are higher than those of XAD-7 resin's SILPs. The molecular interactions that occurred between ILs and solid carriers play an important role for Pt(IV) sorption. Because of Van der Waals attractive force polar solid carriers have higher affinity with polar molecules of ionic liquids. This leads to higher stability of IL in the pore of carriers and therefore, leads to a higher Pt(IV) sorption capacity. Surface area of silica gel is lower than of XAD-4 but silica gel has higher polarity and is more hydrophilic, therefore, it can better adsorb the ionic liquid molecules which are polar and hydrophilic. XAD-4 resin has higher surface area but XAD-4based SILPs led to lower Pt(IV) sorption. This could be explained by the hydrophobicity of XAD-4 resin which reduces the IL stability in the pores (small ionic liquid leaching was observed) and reduces the accessibility of aqueous phase containing Pt(IV) to surface of the resin.

Influence of IL loading

The experiment results show that the SILPs prepared from [C$_{14}$MIM]Cl and silica gel have the highest Pt(IV) recovery efficiency among investigated sorbents (Fig. 4). Based on the results, the influence of IL loading on Pt(IV) sorption yield using [C$_{14}$MIM]Cl/SiO$_2$ system was investigated (Fig. 5).

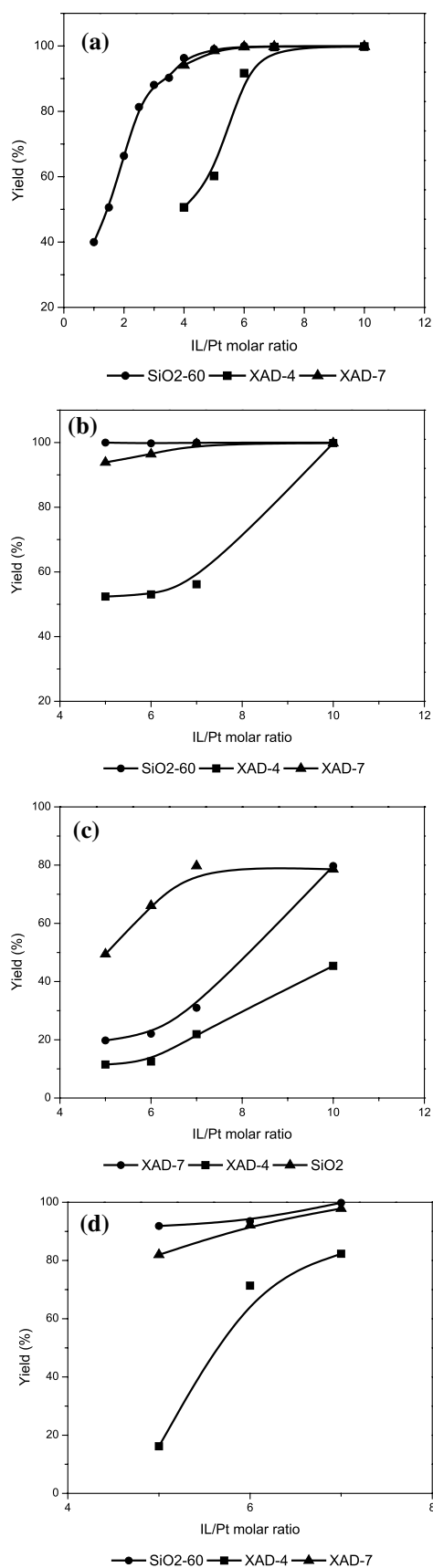Figure 5a shows that in investigated IL loading range Pt(IV) sorption yield increased with the decreasing ionic

liquid loading at given IL/Pt molar ratios. These results can be explained by a decrease in the phase contact between PtCl$_6^{2-}$ ion and IL when IL loading increases. Indeed, at a given IL/Pt molar ratio, the higher IL loading (or the lower amount of carrier) the thicker layer of IL on the sorbent surface. In addition, when a high amount of IL enters into the solid carrier pores (ionic loading of 30%), the pores can be occluded by ionic liquid, which in turn prevents the PtCl$_6^{2-}$ ion from entering the pore to get into contact with IL. The higher IL/Pt molar ratio the less difference in Pt(IV) sorption yield when using different IL loadings because in these cases IL is used in excess amounts compared to PtCl$_6^{2-}$ ion. At lower IL loadings, ionic liquid forms a thinner layer on the silica gel surface that leads to a higher contact area between IL and PtCl$_6^{2-}$ ion, therefore, the Pt(IV) sorption capacity of ionic liquid (mg Pt g^{-1} IL) increases (Fig. 5b). However, a lower IL loading results in a higher amount of silica gel for a given IL/Pt molar ratio, therefore, the Pt(IV) sorption capacity of SILP (mg Pt g^{-1} SILP) decreases (Fig. 5c).

To look insight the reaction between IL and H$_2$PtCl$_6$ (PtCl$_6^{2-}$), the variation of sorption capacity (mmol Pt g^{-1} SILP) with increasing the IL loading (mmol IL g^{-1} SILP) at different IL/Pt molar ratios is exhibited in Fig. 4d. The sorption capacity increased with increasing the amount of IL and approximately follows a linear trend with the slope steadily decreased when the IL/Pt molar ratio increased from 1 to 10. This means that the higher IL/Pt molar ratios, the slighter variation of Pt sorption capacity. At low IL/Pt molar ratios (1–2) the sorbents were nearly saturated by PtCl$_6^{2-}$ ion, whatever IL loading the slopes were close to 0.4. This value is close to the expected stoichiometric ratio corresponding to one Pt(IV) ion for two ionic liquid molecules (Pt/IL molar ratio of 0.5 or 2) (see "Sorption isotherms"). At higher IL/Pt molar ratios (2.5–10) the sorbents were not saturated by PtCl$_6^{2-}$ ion because these ratios are farther from the stoichiometric ratio between IL and hexachloroplatinic acid. The sorption capacity decreased with increasing the ratio of IL/Pt in this case.

There is slight difference in Pt sorption capacity of sorbents when using different IL loadings at high IL/Pt molar ratios (6–10) because the nearly completely sorption takes place. Therefore, the increasing of sorbent or ionic liquid amount do not results in the higher sorption yield.
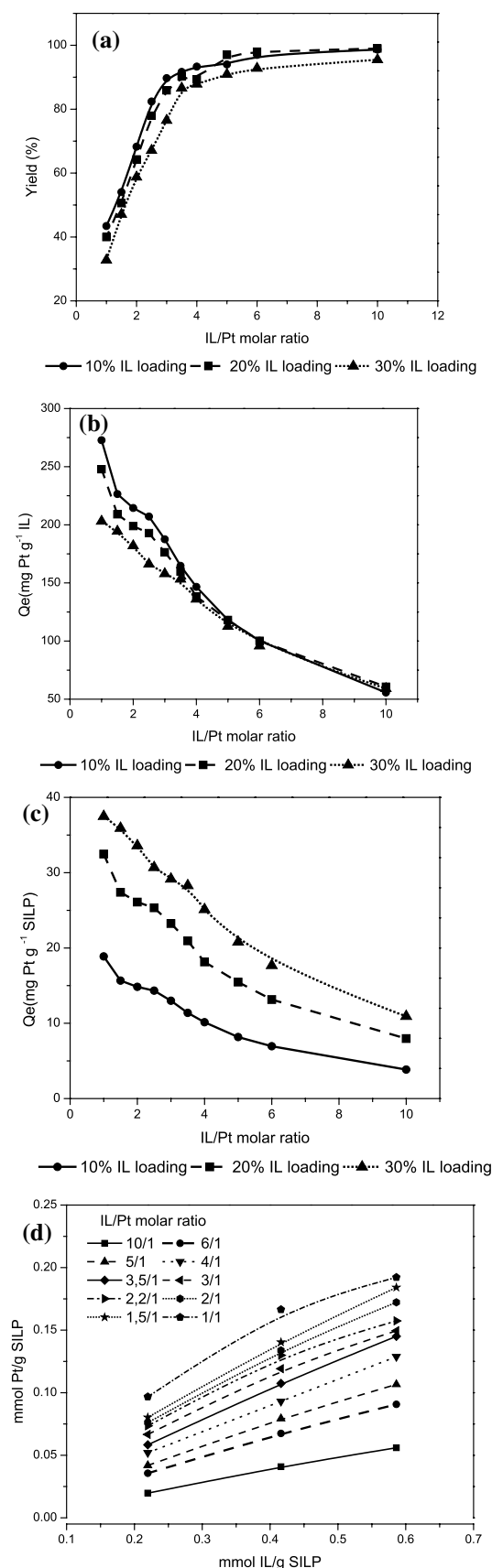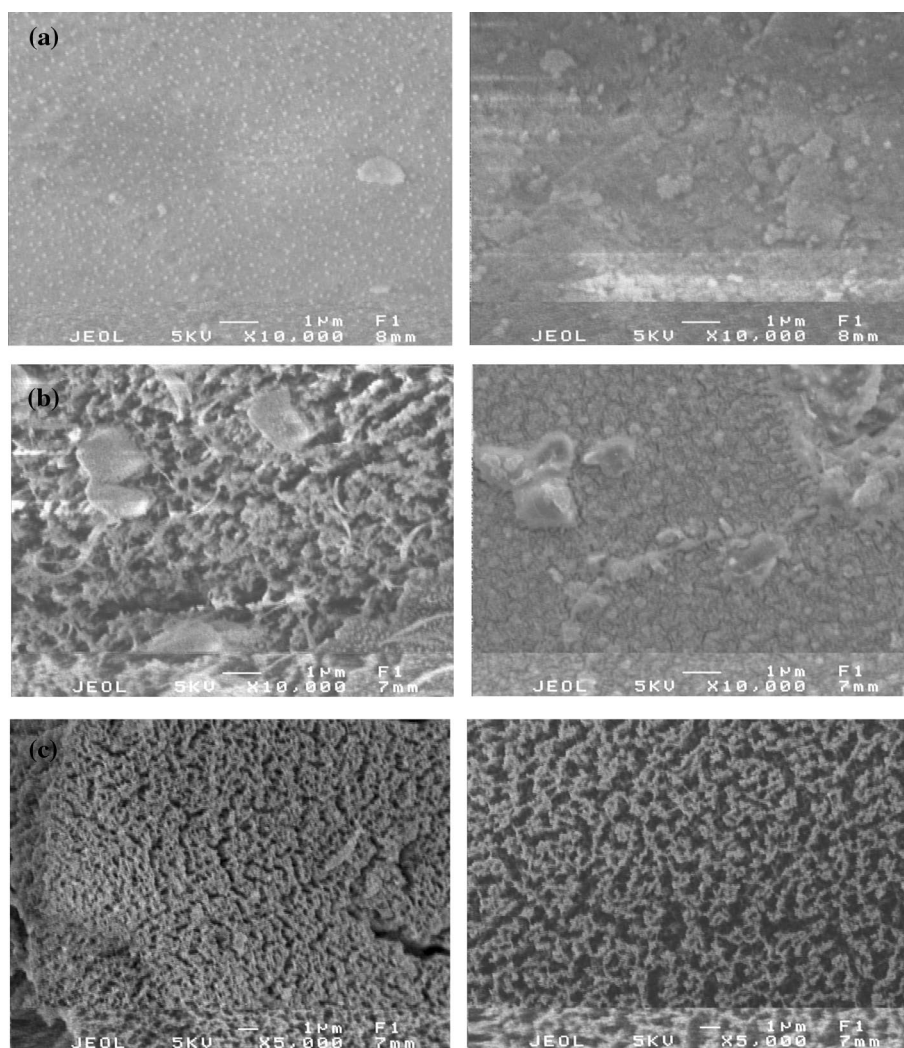
Fig. 6 SEM images of **a** SiO2, **b** XDA-4 and **c** XAD-7 before and after impregnating [C14MIM]Cl

Sorption isotherms

The surface area, pore volume, and pore diameter of the two solid carriers SiO_2 and XAD-4 decreased after immobilizing IL [C_{14}BIM]Cl (Table 2). The SEM images (Fig. 6) show that the regular surface of solid carriers was covered by [C_{14}BIM]Cl ionic liquid.

Theoretically, the IL loadings have influence on the Pt(IV) sorption efficiency, the amount of used ionic liquids, and also the sorbent preparation process. Therefore, sorption isotherms at different IL/Pt molar ratios were performed at different IL loadings. The sorption isotherms are characterized, in most cases, by a Langmuir-type shape (sharp initial slope and appearance of a saturation plateau; i.e., asymptotic trend):

$$q_e = \frac{q_m b C_e}{1 + b C_e} \text{ or } \frac{C_e}{q_e} = \frac{1}{b q_m} + \frac{C_e}{q_m} \tag{4}$$

where q_m (mg Pt g^{-1} IL or mg Pt g^{-1} SILP or mmol Pt g^{-1} SILP) (Fig. 5) is the maximum sorption capacity reached at saturation of the monolayer and b is the affinity coefficient (L mg^{-1} Pt or L mmol^{-1} Pt), q_e is the sorption capacity (mg Pt g^{-1} SILP or mmol Pt g^{-1} SILP) in equilibrium with the residual concentration C_e (mg Pt L^{-1} or mmol Pt L^{-1}). It was determined by the mass balance equation: $q = V(C_o - C_e)/m$, where V is the volume of solution (L) and m is the mass of SILP (or IL) (g). The sorption isotherms are shown in Fig. 7 and Table 3. In order to look insight the Pt(IV) sorption, the maximum sorption capacity was expressed by three ways (mg Pt g^{-1} IL or mg Pt g^{-1} SILP or mmol Pt mmol^{-1} IL). The first value is always higher than the second; the third was calculated from the first value and shows the conversion of IL to complex of IL and Pt (Table 4; Fig. 7).

Pt(IV) was almost completely recovered at high IL/Pt molar ratio (6–10) because these IL/Pt molar ratios are much higher than stoichiometry of ion exchange reaction between PtCl$_6^{2-}$ and [$C_n C_m$IM]Cl. Therefore, in this range of IL/Pt molar ratio, there was a small difference in sorption capacities when using different IL loading.
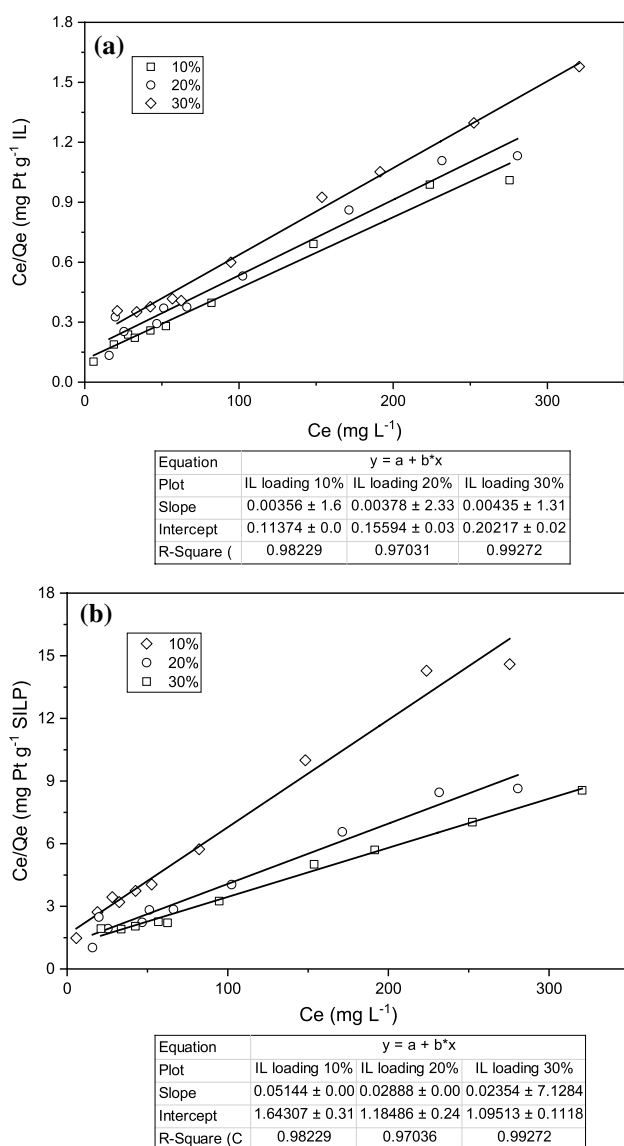
Equation	y = a + b*x		
Plot	IL loading 10%	IL loading 20%	IL loading 30%
Slope	0.00356 ± 1.6	0.00378 ± 2.33	0.00435 ± 1.31
Intercept	0.11374 ± 0.0	0.15594 ± 0.03	0.20217 ± 0.02
R-Square (	0.98229	0.97031	0.99272

Equation		y = a + b*x	
Plot	IL loading 10%	IL loading 20%	IL loading 30%
Slope	0.05144 ± 0.00	0.02888 ± 0.00	0.02354 ± 7.1284
Intercept	1.64307 ± 0.31	1.18486 ± 0.24	1.09513 ± 0.1118
R-Square (C	0.98229	0.97036	0.99272

Fig. 7 Pt(IV) sorption isotherms using [C_{14}MIM]Cl impregnated silica gel at different IL loading ([C_{14}BIM]Cl IL, SiO_2-60 solid carrier, 48 h and 25 °C)

However, the maximum sorption capacities depend on IL loading in silica gel; the maximum capacity reached 281.02 mg Pt g^{-1} IL (0.454 mmol Pt $mmol^{-1}$ IL) and 19.44 mg Pt g^{-1} supported ionic liquid phase (SILP) when

IL loading was 10% while they were 230.12 mg Pt g^{-1} IL (0.372 mmol Pt $mmol^{-1}$ IL) and 40.48 mg Pt/g^{-1} SILP when IL loading was 30% (Table 4; Fig. 5).

Vincent et al. (Vincent et al. 2008) used Cyphos IL-101 immobilized biopolymer capsules for Pt sorption and reported that the equilibrium was very affected by the concentration of HCl and chloride ions, the maximum sorption capacities were 142 mg Pt g^{-1} for dry resin (i.e. 0.73 mmol Pt g^{-1} Cyphos, or 0.57 mmol Pt $mmol^{-1}$ Cyphos). Ion exchange reactions between $PtCl_6^{2-}$ and [$R_3R'P^+$][Cl^-] was proposed in Vincent's work. The sorption capacity is 0.56 mmol Pt $mmol^{-1}$ Cyphos® IL-101; this is close to the theoretical stoichiometric ratio, based on the suggestion of a sorption by formation of ion pairs between $PtCl_6^{2-}$ and 2 $R_3R'P^+$:

$$PtCl_6^{2-}{}_{(w)} + 2[R'_3RP^+][Cl^-]_{(o)} \leftrightarrow [R'_3RP^+]_2[PtCl_6^{2-}]_{(o)} + 2Cl^-_{(w)} \tag{5}$$
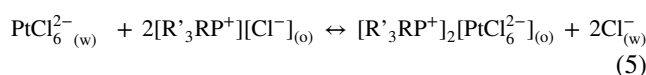
In present work the sorption experiments were performed at high HCl concentration, therefore, the maximum sorption capacities were about 0.454 mmol Pt $mmol^{-1}$ IL also suggested that ion exchange reaction (Pt/IL stoichiometric ratio is 0.5) is shown as below:

$$PtCl_6^{2-} + 2[C_nC_mIM]Cl \rightarrow [C_nC_mIM]_2PtCl_6 + 2Cl^- \tag{6}$$

$$PtCl_6^{2-} + 2[C_{14}Py]Cl \rightarrow [C_{14}Py]_2PtCl_6 + 2Cl^- \tag{7}$$
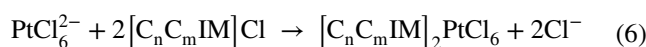
The maximum conversion of ionic liquid to complex containing Pt ([$C_nC_mIM]_2PtCl_6$) is about 90.8% when IL loading was 10% and a thin layer of IL was covered on the surface of silica gel. However, the excess amounts of IL (IL/Pt molar ratios greater than unit) were always used in experiments so that the conversion of Pt can reach nearly 100%. When IL loading increased from 10 to 30%, the maximum conversion of ionic liquid reduced from 90.8 (equivalent to maximum Pt sorption capacity 281.02 mg Pt g^{-1} IL) to 74.4% (equivalent to maximum Pt sorption capacity 230.13 mg Pt IL^{-1}), whereas the maximum sorption capacities calculated by mass of SILP increased from 19.44 to 42.48 mg Pt g^{-1} SILP. This means that the mechanisms are consistent with the ion exchange reaction proposed for Pt(IV) recovery and with discussion in Section "Influence of IL loading"

Table 3 Parameters of the model for Pt sorption from HCl solution ([C_{14}BIM]Cl IL, SiO_2-60 solid carrier, 48 h and 25 °C)

IL loading (%)	q_m (mg Pt g^{-1} IL) (from Fig. 5)	q_m (mg Pt g^{-1} SILP) (from Fig. 5)	q_m (mmol Pt $mmol^{-1}$ IL)*	b (L/mg Pt)	R^2
10	281.02	19.44	0.45*	0.03	0.98
20	235.54	30.86	0.38*	0.06	0.97
30	230.13	42.48	0.37*	0.02	0.99

*Calculated from the q_m (mg Pt g^{-1} IL) value

Table 4 Recycling capacity of SILP using thiourea/HCl (Sorption: 2.3020 g of SILP, 40.65 mL of model leaching solution (460 ppm Pt), and 40 °C; Desorption: loaded SILP, 11.60 mL of eluent (thiourea 2 M/ HCl 5 M), and 80 °C)

No.	Process	SILP	Volume of eluent (mL)	Volume of eluent after stripping (mL)	Pt content in eluent after stripping (ppm)	Sorption yield (%)	Stripping yield (%)
1	Sorption 1st	Fresh SILP				99.9	
1′	Desorption 1st		11.60	11.50	1592		98.0
2	Sorption 2nd	Recycled SILP				99.6	
2′	Desorption 2nd		11.60	11.30	1614		97.9
3	Sorption 3rd	Recycled SILP				99.2	
3′	Desorption 3rd		11.60	11.40	1591		97.8
10	Sorption 10th	Recycled SILP				98.1	
10′	Desorption 10th		11.60	11.50	1544		96.8

Weight of fresh SILP = 2.3020 g (2 g of SiO$_2$-60 and 0.3020 g of [C$_{14}$MIM]Cl, IL loading 20%) and of SILP after the 10th desorption = 2.2903 g

Tetradecyl(trihexyl)phosphonium chloride was impregnated on XAD-7 resin (Navarro et al. 2012) for Pt(IV) sorption from HCl solutions with different concentration (from 0.01 to 8 M). The results showed that the maximum sorption capacities were 74.6 mg Pt g^{-1} extractant impregnated resin (EIR) (256 mg Pt g^{-1} IL) with the IL content of 291 mg IL g^{-1} EIR. That phosphonium IL was also immobilized on biopolymer (Vincent et al. 2008) to form the capsules for Pt(IV) sorption. Equilibrium was very affected by the concentration of HCl and chloride ions, the maximum sorption capacities was 142 mg Pt g^{-1} for dry resin (i.e. 0.57 mmol Pt mmol^{-1} Cyphos) with the IL content of 291 mg IL g^{-1} EIR. Sorption capacities calculated in mg Pt g^{-1} IL of both previous studies were similar to those of this work but the numbers in mg Pt g^{-1} EIR or mg Pt g^{-1} capsules were higher than those of this work. However, while the sorbents used in this work showed high potential of recovery of Pt from 9 M HCl solution which is obtained after leaching Pt(IV) from spent catalysts, the sorption capacities in both reported cases decreased fast (down to nearly zero) when the HCl concentration increases from 0.01 to 8 M and the higher IL loadings (equivalent to 40.23% and 65.61%) were used in both cases (Tong et al. 2015; Navarro et al. 2012). Furthermore, in this work the lower IL loading (20%) used could help ILs to stay stable on the pore of solid and, therefore, easy to reuse as presented in the next section.

Pt(IV) stripping

Pt(IV) stripping was performed using aqueous solution containing thiourea 2 M and HCl 5 M as stripping eluent, the process of Pt(IV) sorption/SILPs stripping was repeated ten cycles. A slight decrease in the sorption yields after 10 cycles showed that [C$_{14}$MIM]Cl/SiO$_2$ SILPs are stable and have a high reusability. The sorption yield reduced from 99.96 for fresh SILP to 98.08% for tenth recycled SILP. Thiourea/HCl is suitable eluent for Pt(IV) stripping from SILP [C$_{14}$MIM]Cl/SiO$_2$ with the high efficiency of 98.04% for fresh SILP (run 1′ in Table 3) and 96.80% for tenth recycled SILP (run 10′ in Table 3).

A slight decrease in Pt(IV) sorption and stripping efficiency when reusing SILP after ten cycles also shows that other parameters, which were used in the stripping process, are suitable. No ionic liquid leaching from SILPs was observed during ten successive sorption and desorption cycles. In addition, the weights of dried SILPs were determined before the first sorption and after the tenth desorption (Table 5) showed that there is a slightly change in SILP weights after ten cycles. Finally, only minor decrease in the sorption and desorption yields of recycled SILP was visually observed within ten cycles. All these results showed the advantage of using SILPs as sorbents for Pt(IV) recovery from chloride solution. After platinum desorption [C$_{14}$MIM]$^+$ ion combines with Cl$^-$ ion and transfers back to original ionic liquid [C$_{14}$MIM]Cl which remained in IL phase. The ionic liquid has high affinity with solid carriers and, therefore, stays in the pores of carriers.

Table 5 Pt(IV) recovery using some imidazolium ionic liquid

IL/SILP	Method	Molar ratio IL/Pt	Yield (%)	References
[OMIM]NTf$_2$	Extraction	500	90	Yang et al. (2014)
[CnMIM]Cl	Extraction	150	93.6	Yang et al. (2015)
[C$_{14}$BIM]Cl IL/SiO$_2$-60	Adsorption	6–10	99.9	This work

The FT-IR of $[C_{14}MIM]Cl/SiO_2$ before adsorption and $[C_{14}MIM]Cl/SiO_2$ after tenth desorption in Figs. S13 and S14 also showed that sorbents do not changed during ten successive sorption and desorption cycles.

Conclusion

In the work, four ionic liquids $[C_4BIM]Cl$, $[C_{14}MIM]Cl$, $[C_{14}BIM]Cl$, and $[C_{14}Py]Cl$ were successfully synthesized, characterized and impregnated on silica gel, XAD-4 and XAD-7 exchange resins by directly impregnation method to form twelve kinds of SILPs. Pt(IV) was recovered by sorption method and most SILPs have high affinity with Pt in HCl solutions. The influence of factors such as structure of ionic liquids and solid carriers, the IL loading, and molar ratio IL/Pt on Pt(IV) sorption is investigated. The results revealed that the higher the IL/Pt molar ratio is the higher Pt(IV) recovery yield. With all kinds of solid carriers Pt(IV) sorption yields of SILPs followed the order: $[C_{14}MIM]Cl > [C_{14}BIM]Cl > [C_{14}Py]Cl > [C_4BMIM]Cl$. Pt sorption yield increased with increasing the length of the 3-alkyl side chain of imidazolium ring (comparison of $[C_4BIM]Cl$ and $[C_{14}BIM]Cl$). However, if the length of alkyl side chain at 3-position was kept constant and alkyl side chain at 1-position of imidazolium ring ($[C_{14}MIM]Cl$) was shortened then the better Pt sorption performance was observed. The SILPs from $[C_{14}MIM]Cl$ showed the highest Pt(IV) sorption yield among SILPs from three imidazolium ILs.$[C_{14}Py]Cl$-based SILPs gave lower Pt(IV) sorption yield compared to $[C_{14}MIM]Cl$-based SILPs even though the two ionic liquids have a similar number of carbon in 3-alkyl side chain of the ring. This can be due to the lower stability of pyridinium ring on SiO_2 surface compared to that of imidazolium ring.

Among three solid carriers, silica gel based SILPs shows a higher sorption capacity and was proposed to use as carriers for Pt(IV) recovery ($SiO2 > XAD-7$ resin $> XAD-4$ resin). In IL loading range 10–30%, Pt(IV) sorption yield increased while sorption capacity decreased with the decreasing of ionic liquid loading at given IL/Pt molar ratios. At low IL/Pt molar ratios (1–2) the sorbents were nearly saturated by $PtCl_6^{2-}$ ion whatever IL loading was used and the slopes were close to 0.4, close to the expected stoichiometric ratio corresponding to one Pt(IV) ion for two ionic liquid molecules (Pt/IL = 0.5 or IL/Pt = 2). The maximum sorption capacities was 281.02 mg Pt g^{-1} IL (IL loading 30%) and 42.48 mg Pt g^{-1} SILP (IL loading 10%), equivalent to about 0.454 mmol Pt $mmol^{-1}$ IL also suggested that ion exchange reaction between hexachloroplatinate species $PtCl_6^{2-}$ and imidazolium ($[C_nC_mIM]^+$) or pyridinium $(C_{14}Py)^+$ ions with formation of ion pair; That also can explain the stoichiometric ratios obtained at saturation of the SILPs. According to our work, the sorption method using

SILPs presents advantages over the reactive extraction with IL such as lower IL consumption, simpler and easy handling procedure (Table 5). Interestingly, Pt(IV) can be stripped with thiourea/HCl eluent and recycled SILP can be reused with stably high sorption capacity.

Even a slight high temperature was used in desorption process but high speed and high Pt(IV) recovery yield (at low IL/Pt ratio) is still advantage of this method.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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