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Original Article

Extraction behaviors of platinum group metals in simulated high-level liquid waste by a hydrophobic ionic liquid bearing an amino moiety

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ABSTRACT

A hydrophobic ionic liquid including an amino moiety ([DiOcAPmim][NTf$_2$]) was synthesized. Its extraction behaviors towards Pd(II), Ru(III), Rh(III) were investigated in nitric acid aqueous solution as a function of contact time, effect of concentration of nitric acid, effect of temperature, and effect of co-existing metal ions. The extraction kinetics of Pd(II) was fairly fast and extraction equilibrium can be attained within only 5 min under the [HNO$_3$] = 2.05 M. When [HNO$_3$]< 1 M, the extraction percentage of Pd(II), Ru(III), Rh(III) were all above 80%. When [HNO$_3$] reached 2 M, all of the extraction percentage decreased and in an order of Pd(II)>Ru(III)>Rh(III). When [HNO$_3$]> 2 M, the extraction performance gradually recovered. The effect of temperature can slightly affect the extraction performance of Pd(II). Furthermore, in simulated high-level liquid waste, [DiOcAPmim][NTf$_2$] showed a better preference towards Pd(II) under the interference of various other co-existing metal ions.

1. Introduction

Palladium (Pd), rhodium (Rh), and ruthenium (Ru) are commonly categorized as platinum group metals (PGMs) which with a very limited natural resources on the earth and can be used in the field of automotive catalyst, batteries, etc. [1] Meanwhile, PGMs are important kinds of fission products with relatively high yields in spent fuels. For example, in a commercial light water reactor at a burn-up of 33 GWd/t, there are about 4 kg of PGMs are produced per ton of heavy metal in the fuel, and about 11 kg Pd, 13 kg Ru, 4 kg Rh in per ton spent fuel of 150 GWd/t fast breeder reactor, which could significantly fulfill the desired amount obtained naturally [2,3]. On the other hand, during the vitrification of high-level liquid waste (HLLW) into the molten glass, the PGMs with a low solubility in molten glass are partially precipitated and accumulated at the bottom of the container. Furthermore, formation of the conductive metallic sludge will cause a serious loss of an electrical current in the glass, which has a possibility of causing erosion of the container and shortening its lifetime [4,5]. Therefore, study on the separation and recovery of PGMs from spent nuclear fuel is very urgent and meaningful which can benefit not only the advanced nuclear fuel cycle but also enlarge the resources of PGMs meantime.

Many studies have been reported about separation and recovery of PGMs from HLLW in the past decade [6–8]. However, due to the complexity of HLLW, the industrialization of recovering PGMs from HLLW has not yet been realized, and the widely used methods are solvent extraction, electrochemistry precipitation, ion exchange, adsorption etc. [9,10] T. Ito et al. have used commercial available thiodiglycolamic acid functional silica gel (TDGAA-Si) to separate Ru(III), Rh(III), and Pd(II) from nitric acid solution which exhibited very fast and selective adsorption of Pd(II), while the slow uptake of Ru(III), Rh(III) were improved by temperature elevation [11]. S. Ikeda et al. have reported that microwave (MW) irradiation was useful for accelerating the extraction of the inert PGMs by using betainium bis(trifluoromethylsulfonyl)amide ([Hbet][Tf$_2$N]) as an extractant. The uptake rates of PGMs followed the order of Pd(II) > Ru(III) > Rh(III) [12]. N. Iyer et al. have introduced a novel imino diacetamide grafted styrene divinyl benzene resin for separation and recovery of palladium from simulated HLLW. Their results showed value of *D* higher than 10,000 was obtained for 0.25 M HNO$_3$, which dropped to about 890 for 3.0 M HNO$_3$ [13]. Based on

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the researches above, it was found that Pd(II) can be separated and extracted, while good results for efficient recovery of Ru(III) and Rh(III) have rarely been reported especially in a relatively strong nitric acid condition.

Many of commonly used organic solvents in liquid-liquid solvent extraction process are considered to be toxic, flammable and volatile. In a comparison, ionic liquids (ILs) consist of only ionic species, possess many unique properties such as non-flammability, negligible vapor pressure, wide electrochemical window, high thermal stability and the ability to solubilize a wide range of solutes etc. Therefore, ILs are highly expected to be feasible as non-aqueous media in solvent extraction process instead of traditional organic solvents [14,15]. At the same time, relatively strong radiation resistance ability of ILs extraction system in most cases [16,17] benefits its application in the reprocessing of spent nuclear waste or recovery of PGMs from radioactive HLLW [18,19]. On the other hand, ILs can function not only as diluent but also as extractant in solvent extraction system, and both functions can even be combined in one IL. ILs that are used as extractant are known as functionalized ionic liquids (FILs) or task-specific ionic liquids (TSILs) [20]. From previous studies, a functionalized ionic liquid bearing an amino moiety 1-methyl-3-dioctylaminopropyl-imidazolium bis(-trifluoromethanesulfonyl)imide ([DiOcAPmim][NTf$_2$]) was used as an effective extractant to selectively separate PGMs from HCl [21]. Herein, the purpose of this study mainly focused on the elucidation of the extraction behaviors of Pd(II), Ru(III), Rh(III) from nitric acid aqueous solution to [DiOcAPmim][NTf$_2$] phase and then evaluated the possibility of efficient separation of these elements which may benefit in the field of nuclear fuel reprocessing process.

2. Experimental

2.1. Reagent and apparatus

Palladium (II) nitrate (Pd(NO$_3$)$_2$) solution with 10 wt% of Pd(II) in 10 wt% nitric acid (99.99% trace metals basis), ruthenium (III) nitrosyl nitrate (Ru(NO) (NO$_3$)$_x$ (OH)$_y$, $x + y = 3$) with 1.4 wt% of Ru(III) in dilute nitric acid, rhodium (III) nitrate solution (Rh(NO$_3$)$_3$) with ~10 wt % of Rh(III) in >5 wt % HNO$_3$ and other metal nitrates were supplied by Sigma-Aldrich Corporation etc. All the other reagents needed for the synthesis of [DiOcAPmim][NTf$_2$] [21,22] such as 1-methylimidazole, lithium bis(trifluoromethanesulfonyl)imide were of analytical grade and were used as received without any other purification. Obtained characterization result of ^{1}H NMR spectrum of [DiOcAPmim][NTf$_2$] (Fig. 1) was shown without other impurity peaks, which meant the purity of synthesized [DiOcAPmim][NTf$_2$] was high enough and can be used directly in extraction experiments.

2.2. Extraction studies

Extraction experiments were carried out using the following method. Firstly, aqueous solutions were prepared by diluting appropriate amounts of above PGMs solution in doubly deionized water with a specific resistance of 12 MΩ cm or greater. The concentration of HNO$_3$ in aqueous phase was adjusted by using concentrated nitric acid. At the same time, ionic liquid phase was prepared by directly using [DiOcAPmim][NTf$_2$] without dilution. The synthesis and purification procedures of [DiOcAPmim][NTf$_2$] were introduced from precious studies and its chemical structure was briefly as shown in Scheme 1. Both the aqueous phase and the ionic liquid phase were mixed in a small vial (volume ratio = 1 : 1) and shaken vigorously at room temperature. After centrifugation, the concentration of Pd(II), Ru(III), Rh(III) that remained in the aqueous phase was measured by ICP-AES. The extraction percentage (E%), distribution coefficient (D) were calculated by the following equations, respectively [23]:

$$E\% = 100 \times ([\mathrm{M}]_{\mathrm{ini}}\text{-}[\mathrm{M}]_{\mathrm{af}})/([\mathrm{M}]_{\mathrm{ini}}) \quad (1)$$

$$D = ([\mathrm{M}]_{\mathrm{ini}}\text{-}[\mathrm{M}]_{\mathrm{af}})/([\mathrm{M}]_{\mathrm{af}}) \quad (2)$$

where $[\mathrm{M}]_{\mathrm{ini}}$ and $[\mathrm{M}]_{\mathrm{af}}$ denote the metal ion concentration in the aqueous phase before and after extraction.

3. Results and discussion

3.1. Dependence of contact time

Fig. 2 shows the extraction percentage of Pd(II), Ru(III), Rh(III) as a function of contact time at the concentration of HNO$_3$ in 0.55 and 2.04 M, respectively. From the results, when [HNO$_3$] = 0.55 M, we can find that the extraction percentage of Pd(II), Ru(III), Rh(III) increased sharply to more than 60% at the very beginning of the extraction. However, the extraction kinetics slowed down gradually and finally extraction equilibrium was achieved around 2 h. On the other hand, when [HNO$_3$] = 2.04 M, for Pd(II), the extraction kinetics was fairly fast and extraction equilibrium can be attained within 5 min. The extraction percentage of Pd(II) was more than 80%. However, the extraction equilibrium of Ru(III) were not attained more than 2 h. The extraction percentage of Ru(III) still increased slowly with increasing the extraction time. Any significant extraction ability towards Rh(III) was not observed when [HNO$_3$] = 2.04 M. The difference in extraction kinetics of Pd(II), Ru(III) and Rh(III) can be derived from the following several aspects: Firstly, in the water-immiscible ionic liquid extraction system, cation exchange was considered to be a major extraction reaction in acidic solution from low up to middle H$^+$ concentration range [24]. A part of the [DiOcAPmim]$^+$ with amino moiety which was protonated will form a divalent cation. And the formed [DiOcAPmim]$^{2+}$ and non-protonated [DiOcAPmim]$^+$ in the IL phase was considered to exchange with Pd(II), Ru(III) and Rh(III) in the aqueous phase [25]. Secondly, based on the soft-hard acid-base (*HSAB*) principle, it was considered that the affinity of IL towards PGMs was developed by soft-soft affinity between soft N donor and soft PGMs [26]. Moreover, based on previous studies, the slow kinetics and the following low extraction efficiency of Ru(III), Rh(III) in relatively more acidic 2.04 M HNO$_3$ condition can be explained as: with increasing the concentration of HNO$_3$, water molecules and nitrate ions competed for places in the first coordination sphere of Ru(III) and Rh(III). The equilibrium of this water exchange reaction (τ_{H2O}) exhibited extremely long lifetime even up to several days or even years, while that of Pd(II) was only 10^{-3} s. Thus, the slow kinetics and low extractability of Ru(III) and Rh(III) could be ascribed to their inertness in the ligand substitution reactions [27].

Therefore, under the experimental condition, the extraction kinetics for PGMs from nitric acid aqueous solution could be caused by several reactions introduced above.

3.2. Dependence of HNO$_3$ concentration

The results of extraction percentage of Pd(II), Ru(III), Rh(III) with the variation of the [HNO$_3$] in aqueous phase was shown in Fig. 3. It can be briefly divided into three stages: Firstly, [HNO$_3$] from 0.1 to 1. It was found that a high extraction percentage of Pd(II), Ru(III), Rh(III) under moderately acidic conditions was observed. For instance, more than 80% of Pd(II), Ru(III), Rh(III) were extracted from aqueous phase into ionic liquid phase when the [HNO$_3$] values varied from 0.1 to 1 M. Cation exchange reaction between the cationic constituent of IL ([DiOcAPmim]$^+$) with metal ions was considered as the main

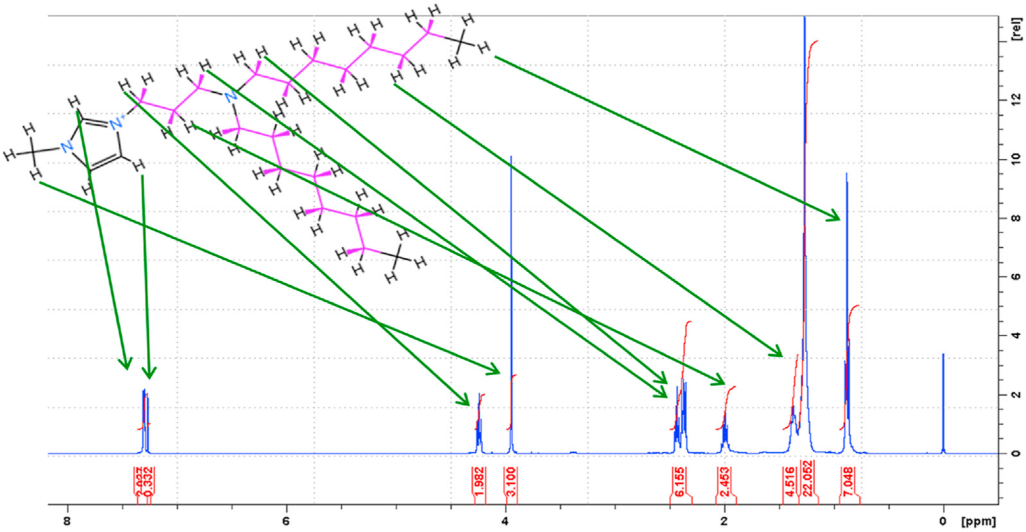
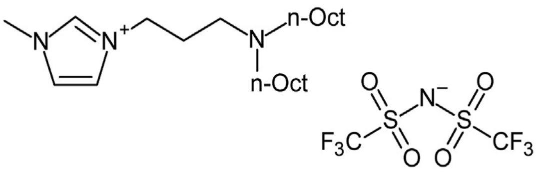


Fig. 1. ^{1}H NMR spectrum of synthesized [DiOcAPmim][NTf$_2$].



Scheme 1. Chemical structure of [DiOcAPmim][NTf$_2$].

reaction in this stage (equation (3)) [28]. On the other hand, [DiOcAPmim][NTf$_2$] was clarified as a soft N donor ligand which also exhibited affinity towards soft acids (equation (4)).

$$n[\text{DiOcAPmim}]^{+}{}_{\text{IL}} + \text{M}^{n+}{}_{\text{aq}} \leftrightharpoons n[\text{DiOcAPmim}]^{+}{}_{\text{aq}} + \text{M}^{n+}{}_{\text{IL}} \tag{3}$$

$$\text{M}^{n+}{}_{\text{aq}} + [\text{DiOcAPmim}]^{+}{}_{\text{IL}} \leftrightharpoons [\text{M}][\text{DiOcAPmim}]^{n+1+}{}_{\text{IL}} \tag{4}$$

where, M represented metal ions in aqueous phase;

In the second stage, with increasing [HNO$_3$] to an intermediate level (1–2 M), [DiOcAPmim]$^+$ was gradually protonated. The extraction percentage of Pd(II) was slightly decreased when [HNO$_3$] increased from 0.1 to 2 M. However, the extraction percentage Ru(III), Rh(III) decreased drastically when the [HNO$_3$] varied from 1 to 2 M. Rh(III) even showed no extraction ability by [DiOcAPmim][NTf$_2$]. And the possible major extraction mechanism in this stage can be temporarily explained as competing reactions between PGMs with proton (equation (5)) [29].

$$2[\text{DiOcAPmim}]^{+}{}_{\text{IL}} + \text{M}^{n+}{}_{\text{aq}} + \text{H}^{+}{}_{\text{aq}} \leftrightharpoons [\text{H}][\text{DiOcAPmim}]^{2+}{}_{\text{IL}} + [\text{M}][\text{DiOcAPmim}]^{n+1+}{}_{\text{IL}} \tag{5}$$

where, M represented metal ions in aqueous phase;

Thirdly, [HNO$_3$] from 2 to 6 M. The extraction percentage of Pd(II), Ru(III), Rh(III) recovered gradually. This extraction results revealed that Pd(II) could be well-extracted with a high extraction percentage in a wide [HNO$_3$] range till 6 M. The extraction percentage of Ru(III) increased from 35% to 83%. The possible extraction mechanism in this stage was described as: under the higher concentrated acid condition, the protonated amino groups worked as an anion exchanger by their protonation reactions. Pd(II) and Ru(III) formed the complex anion with nitric acid, and the number of nitric acid formed complex with these platinum metal ions increased with increasing the nitric acid concentration [30]. The tendency of *E*% was consistent with the difference of strengths of complexes. Thus, this phenomenon was deduced to be due to anion exchange mechanism [31].

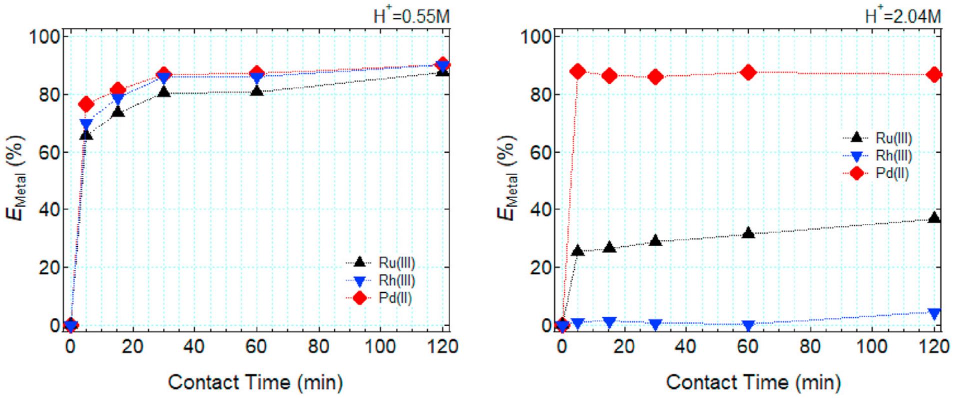


Fig. 2. Variation of the extraction percentage of Pd(II), Ru(III), Rh(III) as a function of contact time. Ionic liquid phase: $V_{[\text{DiOcAPmim}][\text{NTf2}]} = 0.5$ mL; aqueous phase: [Pd(II)], [Ru(III)], [Rh(III)] = 5 mM, [HNO$_3$] = 0.55, 2.04 M, respectively. V = 0.5 mL; T = 298 K.

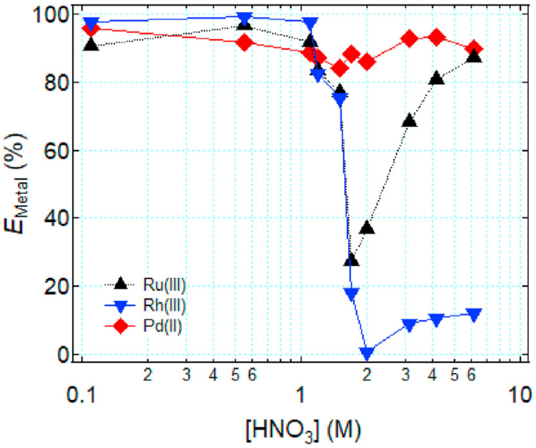


Fig. 3. Variation of the extraction percentage of Pd(II), Ru(III), Rh(III) as a function of $[HNO_3]$. Ionic liquid phase: $V_{[DiOcAPmim][NTf2]} = 0.5$ mL; aqueous phase: [Pd(II)], [Ru(III)], [Rh(III)] = 5 mM, $[HNO_3] = 0.1–6$ M, respectively. V = 0.5 mL; T = 298 K.

3.3. Dependence of temperature

The effect of temperature on extraction ability of $[DiOcAPmim][NTf_2]$ towards Pd(II), Ru(III), Rh(III) were studied and the results were summarized as shown in Fig. 4. It found that the extraction percentages of Pd(II) decreased with increasing temperature, indicating that higher temperature was less favorable for the extraction process. The extraction percentages of Ru(III), Rh(III) was independent with the variation of temperature. Considering the small amount of extraction percentage change upon increasing the temperature, the actual application of $[DiOcAPmim][NTf_2]$ was preferred at 298 K. From previous studies, it has been demonstrated that the liquid–liquid extraction of Ru(III) and Rh(III) was significantly accelerated under heating. However, it was not still clearly understood what drives the extraction of Ru(III) and Rh(III). The experimental results can be speculated as with increasing temperature, the exchange reaction between NO_3^- and water molecular was facilitated in the first coordination sphere of Ru(III) and Rh(III), resulting the number of NO_3^- needed for the formation of Pd/[DiOcAPmim] complex decreased and finally shown as the extraction percentage of Pd(II) decreased [32].

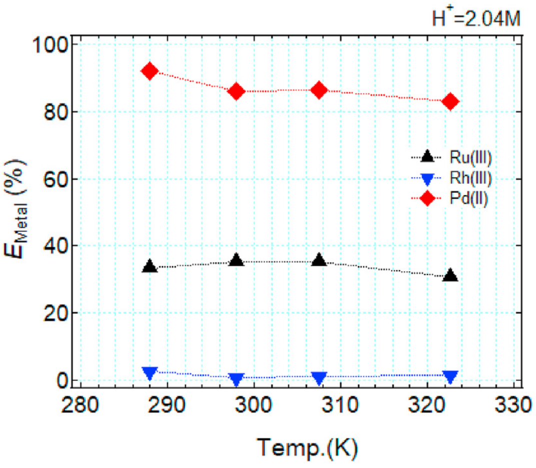


Fig. 4. Variation of the extraction percentage of Pd(II), Ru(III), Rh(III) as a function of temperature. Ionic liquid phase: $V_{[DiOcAPmim][NTf2]} = 0.5$ mL; aqueous phase: [Pd(II)], [Ru(III)], [Rh(III)] = 5 mM, $[HNO_3] = 2.04$ M, respectively. V = 0.5 mL; T = 288, 298, 308, 325 K.

3.4. Effect of co-existing metal ions

Furthermore, in order to understand the extraction performance of Pd(II), Ru(III), Rh(III) when using this $[DiOcAPmim][NTf_2]$ extraction system under the effect of co-existing metal ions. Aqueous solution was prepared by dissolving a mixture of different representative metal nitrate salts to prepare the simulated HLLW (sHLLW). The composition of sHLLW was summarized as shown in Table 1 [33].

The resluts obtained in this experiment was shown in Fig. 5. It can be seen that the extraction percentages of Pd(II), Ru(III), Rh(III) were 100%, 52%, 13%, respectively. The *D* value of Pd(II) was more than 10^3, which meant $[DiOcAPmim][NTf_2]$ showed a better preference to Pd(II) without the interference of other co-existing metal ions. On the other hand, the total extraction percentages of Pd(II), Ru(III), Rh(III) in sHLLW were higher when compared with the situation only containing Pd(II), Ru(III), Rh(III) as shown in Fig. 2. The reason can be explained as high concentration of NO_3^- was indispensable in the extraction process to promote the formation of ion pair between $[DiOcAPmim][NTf_2]$ with metals ions, which was consistent with the phenomenon obtained in the second stage of Fig. 3 [34]. In addition, a third phase was not observed under this experimental condition, fortunately. The reason was thought to correspond to the increased ionic strength in aqueous solution. The overall chemical reaction of metal ions was briefly described as shown in equation (6). This result also revealed that $[DiOcAPmim][NTf_2]$ as an effective extractant, has a possibility to be used in the separation of Pd(II) in the real practical solution.

$$M^{n+}{}_{aq} + [DiOcAPmim]^{+}{}_{IL} + nNO_3^-{}_{aq} \leftrightharpoons [M](NO_3)_n[DiOcAPmim]^{+} \qquad (6)$$

4. Conclusion

A hydrophobic functionized ionic liquid bearing an amino moiety $[DiOcAPmim][NTf_2]$ was synthezied and its extraction performances of Pd(II), Ru(III), Rh(III) ions were systematically investigated in nitric acid solution. The extraction speed of Pd(II) was fairly fast and can reach equilibrium only in 5 min when the $[HNO_3] = 2.05$ M. On the other hand, The effect of $[HNO_3]$ values in the aqueous phase was as follows: when the $[HNO_3] < 1$ M, the extraction percentage of all the Pd(II), Ru(III), Rh(III) can be maintained above 80%; when the $[HNO_3]$ reached 2 M, the extraction percentage was suddenly decreased and in an order of Pd(II) >Ru(III)>Rh(III). When the $[HNO_3]$ increased above 2 M, the

Table 1
Composition of simulated high-level liquid waste (s-HLLW) used in the present work; acidity: 2 M HNO_3.

Element	Concentration (mM)	Element	Concentration (mM)
Na	889.1	Gd	36.0
K	3.2	Ru	21.5
Ag	1.3	Rh	6.0
Cs	32.7	Pd	16.5
Rb	3.9	Zr	31.1
Sr	12.4	Mo	8.5
Ba	16.4	Cr	5.9
Y	7.3	Mn	18.8
La	22.3	Fe	31.1
Ce	34.7	Co	1.9
Pr	18.3	Ni	9.7
Nd	53.3	Zn	0.4
Sm	10.7		

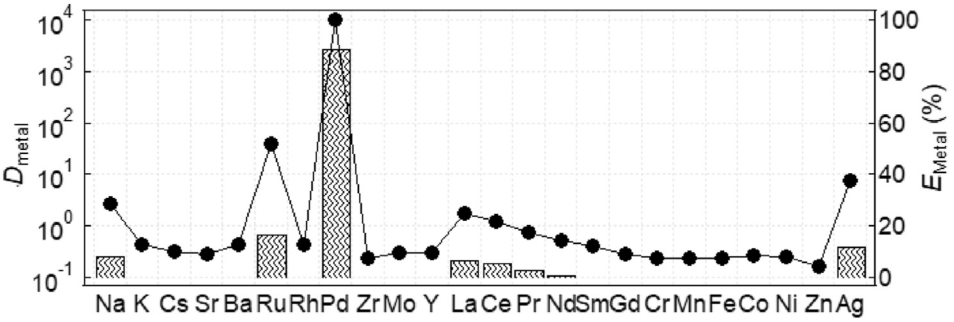


Fig. 5. Extraction performances of Pd(II), Ru(III), Rh(III) under the effect of other coexisting metal ions. Ionic liquid phase: $V_{[DiOcAPmim][NTf2]} = 0.5$ mL; , V = 1.5 mL; aqueous phase: as prepared in Tables 1 and V = 1.5 mL; T = 298 K.

extraction ability of Pd(II), Ru(III), Rh(III) recovered gradually. The effect of temperature revealed that the extraction performances of Ru(III), Rh(III) were not affected much by varying the temperature. Furthermore, The co-existing element experiments in sHLLW solution revealed that [DiOcAPmim][NTf$_2$] showed a higher preference for Pd(II) even under the interference of various metal ions in the aqueous phase, but the extraction performances of Ru(III) and Rh(III) were found to be frustrating. Based on the experimental results introduced in this study, further modification of [DiOcAPmim][NTf$_2$] should be considered to meet the requirement of extraction of PGMs from sHLLW.

Declaration of competing interest

The authors whose names are listed in the manuscript certify that they have NO confilctwith or involvement in any organization or entity with any financial interest or non-financial interest.

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