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Process Development for the Separation of Pt, Ru, and Ir by Solvent Extraction

Ulziikhuu Otgonbayar, Lesia Sandig-Predzymirska, Alexandra Thiere, Alexandros Charitos

With the increasing demand for polymer electrolyte membrane (PEM) electrolyzers for the production of green hydrogen, the strategy to recover platinum group metals (PGMs) from the spent PEM electrolyzer should be developed simultaneously. To secure the supply of these critical raw materials in the EU, the establishment of an efficient recycling technique for PGMs is necessary. In this study, the commercial extractants along with common diluents were investigated to extract Pt, Ru, and Ir from synthetic solutions. Moreover, various stripping agents were tested for the separation of these metal ions. PGM contents in aqueous feed, raffinate, and loaded strip solutions were quantified using inductively coupled plasma optical emission spectroscopy (ICP-OES) for the determination of extraction and stripping efficiencies. The results revealed that the most effective solvent combina-

tion – Cyanex 923 in diesel – extracted Pt selectively from Ir. The extraction efficiencies of 99.2 % Pt, 87.0 % Ru, and 11.9 % Ir were achieved in a single-stage extraction process. Considering similar properties of PGMs, the following separation process was developed: 0.5 M oxalic acid in HCl enabled the selective stripping of Pt and Ir from Ru in the extract phase, while the remaining Ru was stripped using 0.5 M thiourea. A small quantity of Ir in the extract phase was completely removed using ascorbic acid in HCl. Stripping of Pt from the organic phase was attained using water in multiple stages.

Keywords:

Solvent extraction – Recycling – PGMs – Hydrometallurgy – Polymer electrolyte membrane (PEM) electrolyzers

Prozessentwicklung für die Abtrennung von Pt, Ru und Ir durch Lösungsmittelextraktion

Mit der steigenden Nachfrage nach Polymerelektrolytmembran (PEM)-Elektrolyseuren für die Produktion von grünem Wasserstoff sollte gleichzeitig eine Strategie zur Rückgewinnung von Platingruppenmetallen (PGM) aus dem verbrauchten PEM-Elektrolyseur entwickelt werden. Um die Versorgung mit diesen kritischen Rohstoffen in der EU zu sichern, ist die Entwicklung einer effizienten Recyclingtechnik für PGMs notwendig. In dieser Studie wurden kommerzielle Extraktionsmittel zusammen mit üblichen Verdünnungsmitteln untersucht, um Pt, Ru und Ir aus synthetischen Lösungen zu extrahieren. Außerdem wurden verschiedene Strippmittel für die Abtrennung dieser Metallionen getestet. Die PGM-Gehalte in den wässrigen Feed-, Raffinat- und beladenen Stripp-Lösungen wurden mit Hilfe der optischen Emissionsspektroskopie mit induktiv gekoppeltem Plasma (ICP-OES) quantifiziert, um die Extraktions- und Stripp-Effizienz zu bestimmen. Die Ergebnisse zeigten, dass die wirksamste Lösungskombination – Cyanex 923 in Diesel – Pt selektiv aus Ir extrahierte. Die Extraktionseffizienz von 99,2 % Pt, 87,0 % Ru und 11,9 % Ir wurde in einem einstufigen Extraktionsprozess erreicht. In Anbetracht der ähnlichen Eigenschaften der Platinmetalle wurde das folgende Trennverfahren entwickelt: 0,5 M Oxalsäure in HCl ermöglichte das selektive Strippen von Pt und Ir aus Ru in der Extraktphase, während das restliche Ru mit 0,5 M Thioharnstoff gestrippt wurde. Eine geringe Menge an Ir in der Extraktphase wurde mit Ascorbinsäure in HCl vollständig entfernt. Das Strippen von Pt aus der organischen Phase wurde mit Wasser in mehreren Stufen erreicht.

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Schlüsselwörter:

Lösungsmittelextraktion – Recycling – PGMs – Hydrometallurgie – Polymer-Elektrolyt-Membran (PEM)-Elektrolyseure

Développement d'un procédé de séparation du Pt, du Ru et de l'Ir par extraction au solvant

Desarrollo de procesos para la separación de Pt, Ru e Ir mediante extracción con disolventes

Paper presented on the occasion of the European Metallurgical Conference EMC 2023, June 11 to 14, 2023, in Düsseldorf, Germany.

1 Introduction

PGMs, which are classified as critical raw materials of the EU, have increasing demand in various applications, specifically energy storage and automobile catalyst [1]. PEM electrolyzer is an electrochemical device that produces hydrogen by water electrolysis, which plays an important role to achieve a significant reduction of greenhouse gas (GHG) emissions. PEM electrolyzer is advantageous from the energy efficiency, versatility, and performance aspects compared with other electrolyzers, regardless of its higher cost [2]. The state-of-art PEM electrocatalysts typically contain Pt at the cathode [3] and IrO₂/RuO₂ at the anode [4], so spent PEM electrolyzers are a highly potential source of PGMs. However, the recycling rates of PGMs from PEM-electrolyzers are not sufficiently high, for instance, the end-of-life functional recycling rate (EoL-RR) of Ir varies between 25 and 50 % [5]. Thus, improving the recyclability of PGMs from electrocatalysts is crucial for the production growth of PEM electrolyzers.

Due to the high-energy consumption of pyrometallurgical processes and the formation of HF during thermal treatment of F-polymer membranes [6], the hydrometallurgical approach is preferred for recycling of PGMs from PEM-electrolyzers. In the first stage, the PGMs are brought into the aqueous solution by leaching [7], and then separated and purified, for example, by solvent extraction method owing to its selective separation and high-purity products. Most efforts in solvent extraction of PGMs from secondary materials are made for autocatalysts and far less for PEM electrocatalysts. Amines and organophosphorus compounds along with quaternary ammonium and phosphonium salts were adequately studied in this scope. Commercial extractants from Alamine, Aliquat, and Cyphos IL series recovered PGMs from complex solutions with high efficiencies, but the stripping and selective separation of PGMs is difficult due to their high affinities toward these metal ions [8-10]. Organic phosphorus extractants, such as trioctylphosphine oxide (TOPO), tributyl phosphate (TBP), and the Cyanex series of extractants, were moderately efficient in extracting only certain PGMs [11-14]. There are a few studies that utilized other extractants, including α -hydroxyoxime (LIX 63) [15], dialkyl sulphoxides [16], diglycoamides, and thiodiglycoamides compounds [17].

After solvent extraction of PGMs from the aqueous solution, it is essential to bring PGMs back into the aqueous phase from the organic phase, which is referred to as stripping. Although the stripping stage is as important as the extraction stage, there is a lack of data regarding the efficiencies of different stripping agents in the literature. HClO₄, NaSCN, thiourea, HCl, NH₄OH, NaOH, malonic and ascorbic acids have been reported to strip PGMs from the organic phase [18-20, 13, 14]. Some of these agents are feasible on the laboratory scale, but not practical in the industry due to their high reactivity and toxicity.

Currently, there are no studies focused on the separation of Pt, Ru, and Ir from spent PEM electrocatalysts using solvent extraction. This work aims to develop a strategy for Pt,

Ru, and Ir recovery from model solutions by investigating the performance of commercial extractants and common diluents. Moreover, eco-friendly and easily available stripping agents were examined and proposed to effectively recover PGMs from a multicomponent system.

2 Materials and methods

2.1 Materials

Synthetic solutions were prepared using 1000 μ g/mL PGM standard solutions: Pt in 20 % HCl (99.99 %, Thermo Scientific), IrCl₃ in 20 % HCl (99.99 %, Alfa Aesar), and RuCl₃ in 20 % HCl (99.99 %, Alfa Aesar). The desired concentration of PGMs and acidity was achieved by adjusting the aqueous solution with deionized water and concentrated HCl (37 wt.-%, VWR Chemicals).

The various combinations of organic solvents were obtained by mixing commercial extractants and diluents. The commercial extractants – Aliquat 336 (88.2-93 %, Alfa Aesar), Cyanex 923 (93 %, Cytec), Cyphos IL 101 supplied by Solvay (95 %, Strem Chemicals), and TBP (99 %, Sigma-Aldrich) – were used without any additional purification steps. Kerosene (low odor, Alfa Aesar), diesel (from ARAL), n-octanol (99 %, Alfa Aesar), and toluene (99.5 %, VWR Chemicals) were common diluents selected for the solvent extraction experiments.

For stripping PGMs from the organic phase into the aqueous phase, different stripping agents were utilized, namely HNO₃ (68 wt.-%, VWR Chemicals), H₂SO₄ (98 wt. %, Merck), HCl (37 wt.-%, VWR Chemicals), ascorbic acid – C₆H₈O₆ (99.9 %, VWR Chemicals), oxalic acid – C₂H₂O₄ (99 %, Sigma-Aldrich), thiourea – CH₄N₂S (99 %, Laborchemie Apolda), NaNO₃ (99 %, VWR Chemicals), NaCl (99.5 %, Sigma-Aldrich), Na₂SO₄ (99 %, Sigma-Aldrich), Na₂CO₃ (99.9 %, Sigma-Aldrich), and NaOH (99 %, Sigma-Aldrich).

2.2 Experimental procedure

Solvent extraction experiments were carried out from the model aqueous solution containing all three PGMs (Pt, Ru, and Ir) with a PGM concentration of 100 mg/L in 2 M HCl. The organic phase contained 15 vol.-% extractant in a diluent. 10 mL of aqueous feed solution and 10 mL organic solvent were mixed for 15 min by a magnetic stirrer at 400 rpm stirring rate and room temperature. Once the mixing was completed, two phases were transferred to a separatory funnel and settled for 30 min. After filtration and dilution, PGM concentration in the raffinate was quantified using ICP-OES (Varian Agilent 725-ES). The results were then used to calculate extraction efficiency (E %) by Equation 1, in which C_{A,aq} and C_{A,raf} represent the contents of metal A in aqueous feed and raffinate. V_{A,aq} and V_{A,raf} stand for volumes of aqueous feed and raffinate, respectively. The PGM-loaded extract was not analyzed.

$$E\% = 100 \cdot \frac{C_{A,aq} V_{A,aq} - C_{A,raf} V_{A,raf}}{C_{A,aq} V_{A,aq}} \quad (1)$$

In Equation 2, the distribution coefficient of metal A (D_A) was defined by the ratio of metal concentrations in extract

($C_{A,ext}$) and raffinate ($C_{A,raf}$) phases after the solvent extraction.

$$D_A = \frac{C_{A,ext}}{C_{A,raf}} \quad (2)$$

The determined distribution coefficients of metals were used to calculate the separation factor ($SF_{A/B}$) of metal A over B in Equation 3. The separation factor is used to quantify the selectivity of the extractant.

$$SF_{A/B} = \frac{D_A}{D_B} \quad (3)$$

Stripping efficiency (S %) of stripping agents was obtained using Equation 4 in which $C_{A,str}$ and $V_{A,str}$ are contents of metal A in loaded strip and volume of loaded strip solution, respectively.

$$S\% = 100 \cdot \frac{C_{A,str} V_{A,str}}{C_{A,aq} V_{A,aq} - C_{A,raf} V_{A,raf}} \quad (4)$$

3 Results and discussions

3.1 Performance of commercial extractants

The four commercial extractants with different functional groups were studied (Table 1), specifically quaternary ammonium salt (Aliquat 336), quaternary phosphonium salt (Cyphos IL 101), phosphate extractant (TBP), and phosphine oxides (Cyanex 923). Their extraction behaviors in the various diluents (n-octanol, toluene, kerosene, and diesel) were investigated.

As depicted in Figure 1, Aliquat 336 and Cyphos IL 101 exhibited similar performances in all four diluents with a small deviation in Ir extraction. These two extractants have great affinities toward three tested PGMs in hydrocarbon diluents, namely toluene, kerosene, and diesel. This indicates poor selectivity of these solvents toward any of the PGMs by leading to a poor separation. However, Ru and Ir were not extracted at high percentages by Aliquat 336/octanol and Cyphos IL 101/octanol, thus implying an improved selectivity in octanol as a diluent. Nevertheless, Aliquat 336 and Cyphos 101 exhibited immiscibility in

some diluents forming two separate organic phases. This can greatly influence the extraction efficiencies of metals during extraction.

Dielectric constant indicates the polarity of a diluent, which decreases in the following order: octanol – 10.3, toluene – 2.4, diesel – 2.1, and kerosene – 1.8 [21, 22]. The higher dielectric constant correlate to the more polar diluent. Aliquat 336 was miscible in octanol and toluene which are more polar than the other two diluents. However, the same behavior was not observed in Cyphos IL 101 since it established a miscible solution with only toluene. This suggests that the π -electron cloud of the diluent induces interaction within quaternary phosphonium salt and promotes mixing between toluene and Cyphos IL 101 [23]. Regardless of the immiscible organic phases for Aliquat 336 and Cyphos IL 101, the extraction efficiencies of PGMs persisted high.

To prevent phase separation, phase modifiers, such as octanol are commonly added. With the addition of 10 vol.-% octanol, a miscible organic phase of Aliquat 336/kerosene/octanol was obtained. The extraction efficiencies of Ru and Ir were decreased to 78.5 % and 66.7 % with the phase modifier, while Pt extraction was not affected with the efficiency of 99.8 %.

TBP extractant was ineffective in all four diluents at the given conditions, as shown in Figure 2. The extraction efficiency of Pt did not exceed 7.2 %, and less than 6.7 % Ru and 4.2 % Ir were extracted in the organic phase. TBP was miscible with all tested diluents except octanol.

Cyanex 923/octanol resulted in poor PGM extraction. The other three diluents demonstrated similar extraction performance by leading to extraction efficiencies above 95.3 % Pt. In contrast, Ir extraction consistently remained below 18.5 %. A total of 51.3 % Ru was recovered using toluene, while around 87 % Ru was extracted from kerosene and diesel. Cyanex 923, as a neutral extractant, performed better in less polar diluents. For all tested diluents the extraction efficiency was decreased in the order Pt > Ru

Commercial name	Functional group	IUPAC nomenclature	Chemical formula
Aliquat 336	Quaternary ammonium salt	Methyltriethylammonium chloride	$C_{25}H_{54}NCl$
Cyphos IL 101	Quaternary phosphonium salt	Trihexyl(tetradecyl)phosphonium chloride	$C_{32}H_{68}P_2Cl$
TBP	Phosphate	Tributylphosphate	$C_{12}H_{27}O_4P$
Cyanex 923	Phosphine oxide	Trialkylphosphine oxides	$C_{42}H_{90}O_2P_2$

Table 1: Specifications of the commercial extractants

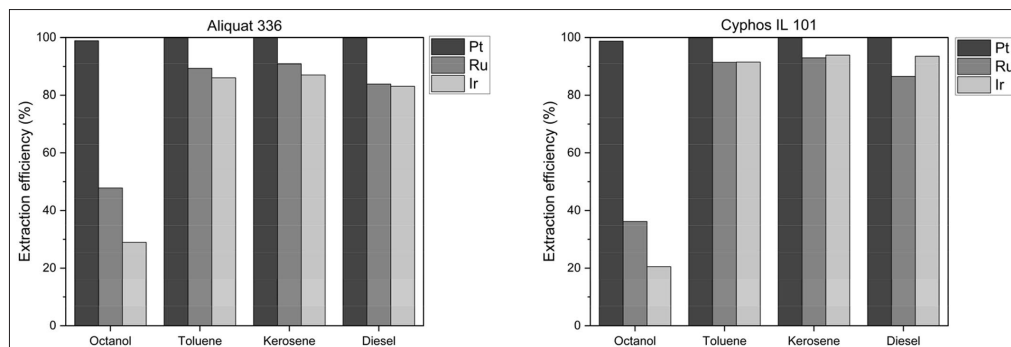


Fig. 1: Extraction efficiencies of PGMs using Aliquat 336 and Cyphos IL 101 in common diluents. Experimental conditions: aqueous feed – 100 mg/L of each PGM (Pt, Ru, and Ir) in 2 M HCl; solvent phase – 15 vol.-% extractants in diluents; O:A = 1:1, and 15 min mixing with a magnetic stirrer at 400 rpm and room temperature (25 °C).

Fig. 2:

Extraction efficiencies of PGMs using TBP and Cyanex 923 in common diluents. Experimental conditions: aqueous feed – 100 mg/L of each PGM (Pt, Ru, and Ir) in 2 M HCl; solvent phase – 15 vol.-% extractants in diluents; O:A = 1:1, and 15 min mixing with a magnetic stirrer at 400 rpm and room temperature (25 °C).

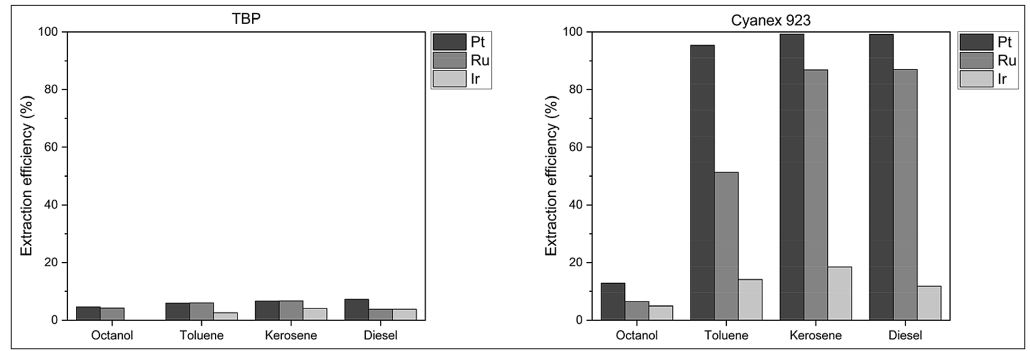


Table 2:

Separation factors of Pt/Ir and Ru/Ir in different solvents

Extractant/diluent	Octanol		Toluene		Kerosene		Diesel	
	SF _{Pt/Ir}	SF _{Ru/Ir}	SF _{Pt/Ir}	SF _{Ru/Ir}	SF _{Pt/Ir}	SF _{Ru/Ir}	SF _{Pt/Ir}	SF _{Ru/Ir}
Aliquat 336	219.9	2.3	158.9	1.4	285.9	1.5	196.2	1.1
Cyphos IL 101	304.5	2.2	77.0	1.0	79.2	0.9	130.0	0.5
TBP	0.0	0.0	2.3	2.4	1.6	1.7	1.9	1.0
Cyanex 923	2.83	1.3	124.6	6.4	628.4	29.1	932.9	49.6

> Ir, due to the labile character of Pt compared to Ru and Ir in the formation of extractable complexes with neutral ligands [24]. At the given extraction parameters, Cyanex 923 exhibited a reverse correlation between the dielectric constant and extraction efficiency of Pt which indicates a high extraction efficiency of Pt in low polarity diluents.

From the results, Aliquat 336 and Cyphos IL 101 were not suitable extractants due to their unselective extraction of PGMs from the solution. TBP was not appropriate because of poor extraction efficiencies. Cyanex 923 was effective when extracting Pt and Ru without significantly affecting Ir in the aqueous phase. The extraction efficiency of Pt was sufficiently high around 99.2 % in Cyanex 923/diesel, while only 11.9 % of Ir was extracted. The complete separation of PGMs from each other was not realizable in all solvents within a single stage, but the selective separation route of Ir from Pt and Ru is possible in several steps. As listed in Table 2, the highest Pt/Ir separation factor of 932.9 and Ru/Ir factor of 49.6 were obtained for Cyanex 923/diesel solvent. The separation factors in Aliquat 336 and Cyphos IL 101 are moderately high compared with Cyanex 923, while the selectivity of TBP was negligible. Therefore, Cyanex 923/diesel was the most selective solvent for the separation of Ir and fairly efficient for Ru extraction. Moreover, the extraction of Cyanex 923 in diesel as a solvent has not been studied for the extraction of PGMs in the literature.

3.2 Performance of various stripping agents

Cyanex 923/diesel was further investigated for stripping experiments. The PGMs in the preloaded organic phase were stripped into the aqueous solution by varying the stripping agent. Three groups of agents – mineral acids, organic compounds, and sodium salts – were studied systematically. The concentration of stripping agents in the stripping liquors was kept constant at 0.5 M, and the concentration of HCl was 0.5 M when added.

As shown in Table 3, the mineral acids were not effective in stripping PGMs from the organic phase. The highest strip-

ping efficiencies of Pt and Ir were obtained using HNO₃ which extracted the PGMs back possibly via oxidation, thus forming less extractable species with higher oxidation state. Because the stripping equilibrium of neutral extractants relies greatly on the type of anions [25], it is also probable that nitrate complexes of PGMs are more stable in water compared with sulfate or chloride complexes.

According to hard and soft acids and bases (HSAB) theory, the tested mineral acids are considered hard acids that interact strongly with hard bases [26]. However, Pt⁴⁺, Ru³⁺ and Ir³⁺ belong to the group of soft acids or sometimes Ru³⁺ and Ir³⁺ are considered borderline metals [27]. Therefore, the reaction between the PGMs and the mineral acids was not favorable.

Water demonstrated a good extraction efficiency of Pt with low selectivity of Pt to Ir. The extraction of Pt seemingly occurred via Equation 5, the same way as Pt solvating during the extraction by TBP [9]. R₃PO in the equation denotes Cyanex 923. When hydrogen ions of HCl are not sufficiently supplied to the complex formed between Pt and Cyanex 923, the reaction equilibrium shifts to the left-hand side forming pre-extraction species. Thus, the stripping by H₂O is preferably caused by the unstable organic PGM species in absence of HCl, and the production of aquated PGM species in the loaded strip is also possible.

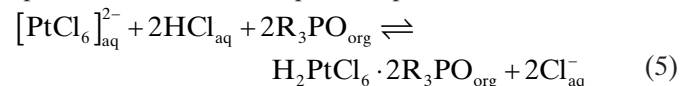


Table 3: Stripping efficiencies of PGMs using mineral acids. Experimental conditions: loaded extract – 105.7 mg/L Pt, 91.6 mg/L Ru, 15.8 mg/L Ir in 15 vol.-% Cyanex 923/diesel; stripping liquor – 0.5 M stripping agent; O:A = 1:1, and 15 min with a magnetic stirrer mixing at 400 rpm and room temperature (25 °C).

Stripping agent	S _{Pt} [%]	S _{Ru} [%]	S _{Ir} [%]
HCl	0.73	1.35	7.53
HNO ₃	14.75	1.12	16.53
H ₂ SO ₄	0.65	1.34	5.08
H ₂ O	56.95	1.30	30.58

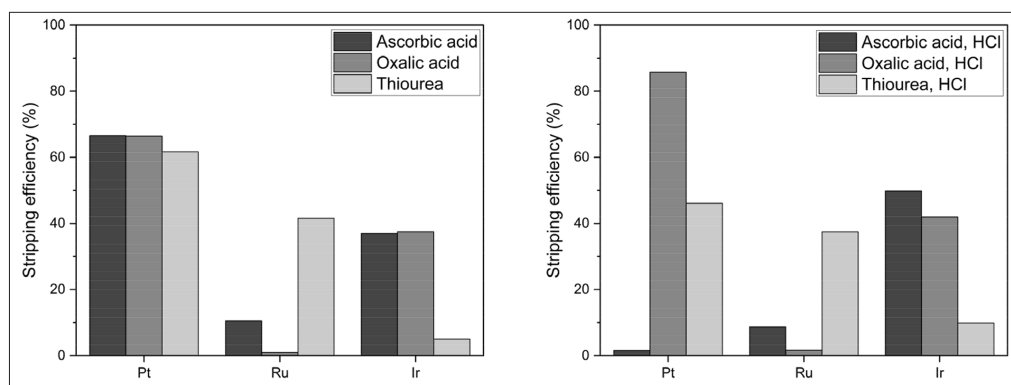


Fig. 3: Stripping efficiencies of PGMs using various organic compounds with and without the addition of HCl. Experimental conditions: loaded extract – 105.7 mg/L Pt, 91.6 mg/L Ru, 15.8 mg/L Ir in 15 vol.-% Cyanex 923/diesel; stripping liquor – 0.5 M stripping agent with/without 0.5 M HCl; O:A = 1:1, and 15 min mixing with a magnetic stirrer at 400 rpm and room temperature (25 °C).

The second group of stripping agents was organic compounds. In Figure 3, the stripping efficiencies of Pt were nearly the same between 61.6 % and 66.6 % in all three agents. Ascorbic acid and oxalic acid exhibited a similar performance when extracting Pt and Ir, but ascorbic acid was rather effective for Ru stripping with 10.5 % efficiency. Thiourea stripped a higher percentage of Pt (61.6 %) and Ru (41.6 %) and low Ir (5.0 %) from the extract phase. All three stripping agents seemingly strip PGMs via reduction.

With the addition of HCl, Pt stripping efficiency diminished in ascorbic acid to 1.6 %, while the stripping Ir was improved slightly to 49.8 %. This implies that the newly formed Pt complexes by ascorbic acid are more stable in the aqueous phase without HCl. Stripping Ru was not significantly influenced by HCl. Furthermore, stripping efficiencies of Pt and Ir were enhanced in oxalic acid/HCl to 85.7 % and 41.9 % each, while only 1.7 % Ru was stripped from the extract phase. The stripping performance of thiourea was affected slightly in the presence of HCl, as the efficiencies of Pt and Ru were decreased to 46.1 % and 37.5 %, respectively, with improved Ir extraction to 9.9 %.

The third group of agents, sodium salts were selected to provide a high stability constant of $[\text{PtCl}_6]^{2-}$ in aqueous solutions [28,29]. As illustrated in Figure 4, NaCl , Na_2SO_4 , NaNO_3 , and Na_2CO_3 were particularly effective when stripping Pt and Ir, but Ru stripping was unsuccessful below 2.2 % in all sodium salts. The addition of HCl into the stripping liquors reduced the stripping efficiencies of PGMs. This could be explained by the stabilization of PGM complexes in the organic phase in the presence of HCl according to Equation 5, and the complexes between PGM and Cyanex 923 are predominant during the stripping.

Considering the formation of turbid organic phases, Na_2CO_3 was not preferred regardless of the high stripping efficiencies. The same behavior was observed when testing NaOH as a stripping agent. Two phases formed a milky emulsion upon mixing, mostly caused by the precipitation of PGM, such as $\text{Ir}(\text{OH})_4$ [30, 31], at the high pH of the solution. The separation of the aqueous and organic phases was not achieved when using NaOH even after 24 h but the two phases remained emulsified.

To sum up, ascorbic acid in HCl can be used for selective extraction of Ir from the loaded extracts containing only Pt and Ir. Because oxalic acid/HCl poorly stripped Ru from the extract, the stripping agent can be used to selectively separate Pt and Ir from Ru in complex solutions. Oxalic acid/HCl and NaNO_3 were the most efficient stripping agents for Pt separation while water exhibited reasonably good performance. In this work, water was selected as the suitable stripping agent for Pt to maintain the number of chemicals low so that no additional steps are required to separate the chemicals from the final products. Thiourea can recover Pt and Ru selectively from Ir, and it was the only agent that stripped a considerable amount of Ru into the aqueous strip, which is related to strong complexation with Ru due to S- and N-donor atoms [32].

3.3 Separation strategy of PGMs

Upon careful analysis of the results, the separation strategy for Pt, Ru, and Ir from spent PEM electrocatalyst was proposed. After pre-treatment processes, including size reduction and leaching of the PEM electrocatalyst, and removal of the impurities, the purified PGM-containing leachate is produced. This solution is then directed to the first solvent extraction process using Cyanex 923/diesel as

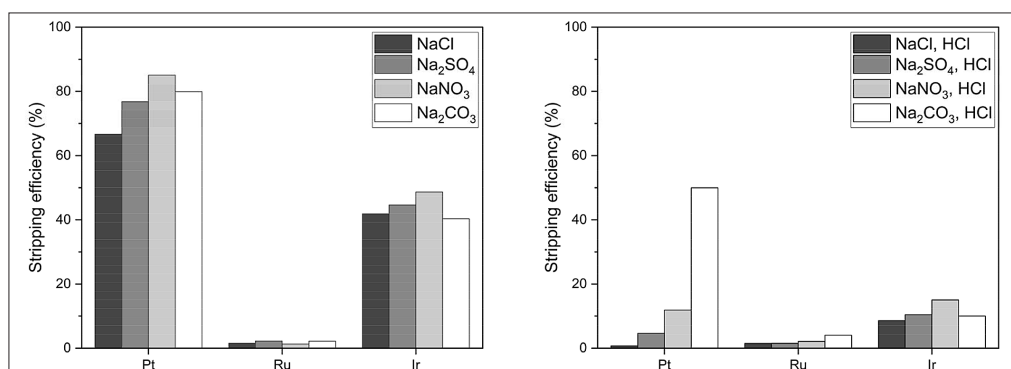


Fig. 4: Stripping efficiencies of PGMs using 0.5 M various sodium salt solutions with and without the addition of 0.5 M HCl. Experimental conditions: loaded extract – 105.7 mg/L Pt, 91.6 mg/L Ru, 15.8 mg/L Ir in 15 vol.-% Cyanex 923/diesel; stripping liquor – 0.5 M stripping agent with/without 0.5 M HCl; O:A = 1:1, and 15 min mixing with a magnetic stirrer at 400 rpm and room temperature (25 °C).

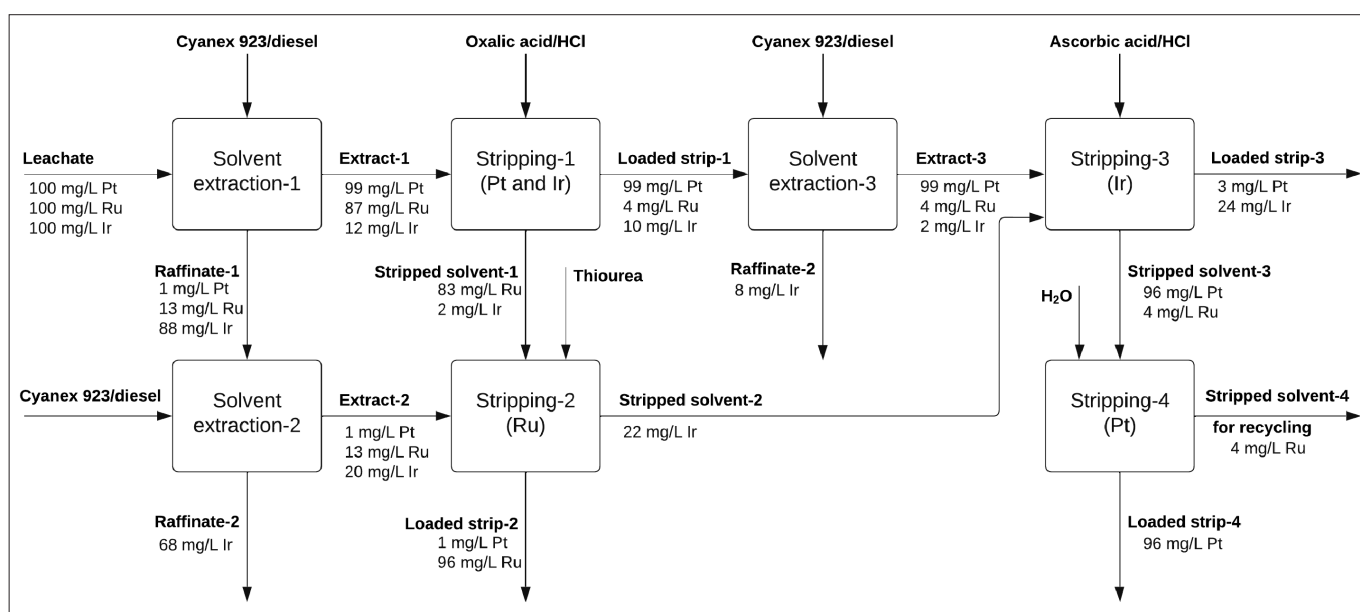


Fig. 5: Suggested process flowsheet for Pt, Ru, and Ir separation from multicomponent solution by the solvent extraction method

illustrated in Figure 5. The raffinate of the first extraction contains Ru and Ir, and it undergoes the second solvent extraction in two stages to separate Ir from Ru. The extract phase from the first extraction is transferred to selective stripping of Pt and Ir in three stages with oxalic acid/HCl by leaving Ru in the stripped solvent phase. The remaining solvent and the organic phase from the second extraction are mixed and stripped using thiourea in several stages to produce a high-purity Ru-containing solution.

The third solvent extraction is carried out in two stages from the aqueous solution containing Pt and Ir, in which Pt is majorly extracted in the organic phase, as Ir remains in the raffinate. The extract phase is purified by stripping with ascorbic acid/HCl in two stages, thus loading the Ir in the loaded strip and leaving Pt in the stripped organic phase. The resulting strip solution is sent to the third solvent extraction for the separation of Pt. Once Pt is isolated in the extract phase, it is effectively stripped using water in a number of stages. Finally, the separated PGMs can be used for the production of pure metals or salts that can serve as precursors for the new PEM electrocatalyst fabrication.

4 Summary

Solvent extraction of Pt, Ru, and Ir from a multicomponent mixture was investigated in order to develop a processing route for spent PEM electrocatalyst. The model solutions were used for extraction experiments with commercial extractants (Aliquat 336, Cyphos IL 101, TBP, and Cyanex 923) in various diluents (n-octanol, toluene, kerosene, and diesel). Aliquat 336 and Cyphos IL 101 were highly efficient when extracting PGMs but not sufficiently selective, while TBP extracted an insignificant amount of PGMs from the chloride solution. The results revealed that the mixture of Cyanex 923 and diesel was the most effective solvent for high Pt and Ru extraction efficiencies and high separation factors (Pt/Ir and Ru/Ir).

The stripping of PGMs was studied using widely available stripping agents. The selective stripping of Pt and Ir by oxalic acid/HCl enables the separation of Ru from the organic phase. A complete recovery of Ru is expected using thiourea in several stages. This study also revealed that ascorbic acid/HCl was the most selective agent for stripping Ir from an aqueous solution containing Pt and Ir. Then, Pt in the Ir-depleted extract phase can be successfully stripped using water in a few stages. To increase the efficiency of the proposed extraction procedure, further study on the optimization of both extraction and stripping is under investigation.

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