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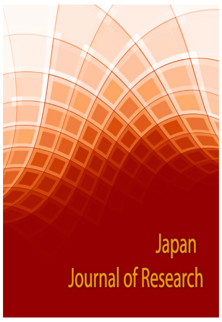

Utilizing Solid Phase Sorbents with Various Functional Groups Based on the HASAB Principle for Recovering Platinum Group Metals from Secondary Sources

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Abstract

The study investigates the recovery of platinum-group metals (PGMs), Pd, Pt, and Rh, from secondary sources - spent catalytic converters, waste from a chemical industry, and a PGM refinery. Aquaregia was used to create leaching liquors and solid-phase extraction sorbents with different functional groups - Di-Octyl Sulfide (DOS), Cyano (CN), Di-Ethylene Tetra-Amine (DETA), and strong anion exchanger (SAX) - were employed based on the "hard and soft acid and base (HASAB)" principles. The SAX-sorbent demonstrated the ability to retain PGMs in the presence of accompanying base metals selectively. In contrast, the DOS and CN sorbents were selective only for Pd, and the DETA sorbent displayed selectivity solely for Rh. Desorption experiments revealed that 3M HCl could effectively recover the PGMs from loaded solid-phase sorbents with significant HSE advantages. The study also noted the high stability of the sorbents, although improvements are needed, particularly in enhancing the reusability of the DETA-sorbent.

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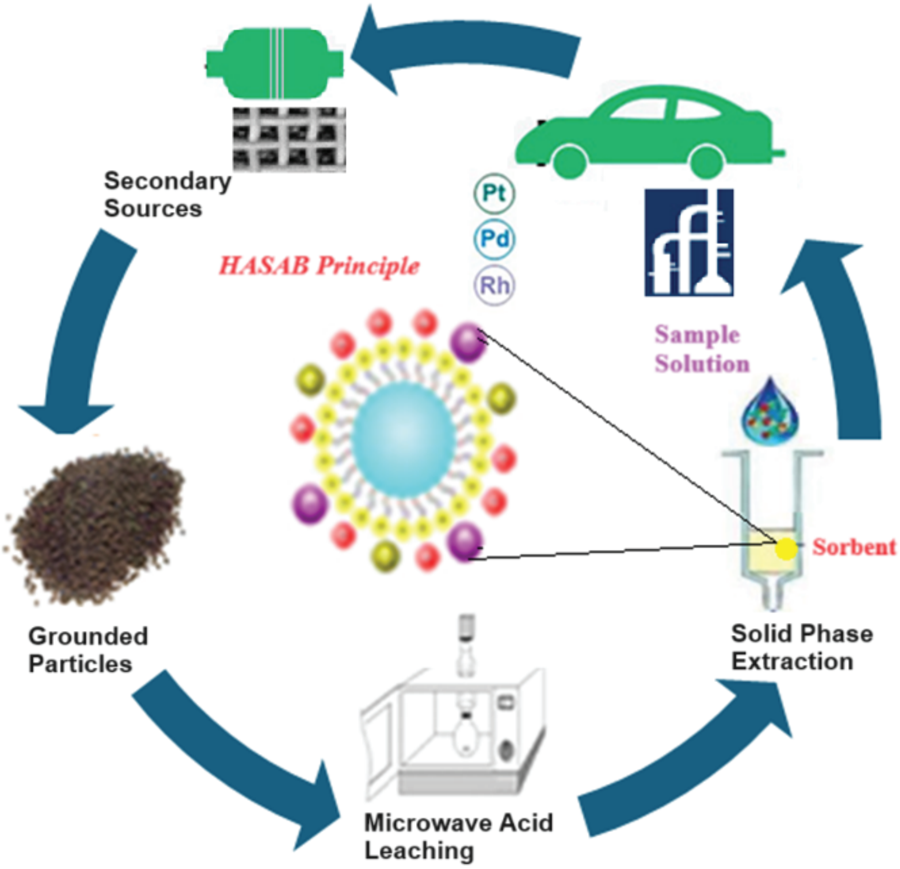
Platinum Group Metals, Secondary Sources, Solid Phase Extraction, HSAB Principle, Various Functional Groups



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Introduction

The Platinum Group Metals (PGMs) include six chemical elements: platinum (Pt), palladium (Pd), rhodium (Rh), ruthenium (Ru), osmium (Os), and iridium (Ir) [1]. PGMs are incredibly rare, even compared to other scarce elements, such as rare earth elements. Their crustal abundance ranges from 1-10 ng/g for Pt, 0.1-3 ng/g for Pd, less than 0.5 ng/g for Rh, less than one ng/g for Ru, 0.3-5 ng/g for Ir, and less than 0.1 ng/g for Os [1-3]. Platinum group metals are highly sought after because of their exceptional physical and chemical properties. These metals show outstanding chemical and thermal resistance and excellent thermal and electrical conductivity. Additionally, they are resistant to corrosion and oxidation, making them highly valuable for a wide range of applications. Pt, Pd, and Rh are found to be extensively used in the automotive industry as crucial components of pollution-control catalytic converters and catalysts in chemical and petroleum industries [4-6]. Furthermore, PGMs have significant potential for future growth in fuel cells, electronics, and glass manufacturing [2,3]. The demand for PGMs for industrial purposes has notably increased over the past few decades due to the enactment of more stringent emission regulations in most countries [6]. Consequently, the prices of these PGMs have risen over the last five years [6]. Due to the critical nature of PGMs for the economy and the precarious supply situation, the U.S. and the European Commission have classified PGMs as critical elements [7,8].

Natural ores, such as sulfide minerals and, to a lesser extent, tellurides, selenides, arsenides, and antimonides, are the primary sources of PGMs [3]. However, the extraction and processing of PGM ores are highly energy-intensive and contribute to environmental damage [6,8]. Consequently, a growing interest is in recovering PGMs from unconventional sources such as spent automotive catalytic converters, obsolete electronic scraps, spent batteries, used electric lamps, and jewelry scraps. At present, only about 25% of the global supply of PGMs comes from secondary resources, with the remaining 75% sourced from primary resources concentrated in a limited number of geographical areas, often associated with high environmental and societal impacts from mining activities [9]. Therefore, there is a clear need to prioritize recycling PGMs from secondary materials to conserve the already scarce primary resources and stabilize their market prices [10,11].

Recent advancements in the extraction and recovery of PGMs from secondary sources are reviewed, focusing on hydrometallurgy and pyrometallurgy methods [12–14]. Hydrometallurgy, or "wet methods," is preferred over pyrometallurgical methods because it generates less waste and requires less energy. Precipitation [15], ion exchange chromatography [16-19], solvent extraction [20–23], and solid phase extraction [24–29] are important hydrometallurgy methods for recovering PGMs. Solvent extraction is widely used in commercial PGM refineries but generates much organic waste, raising environmental concerns [2]. Solid phase extraction has recently gained popularity due to its simplicity, speed, versatility, ease of automation, and low solvent usage [30], which is the focus of this study.

Understanding the retention behavior of PGMs on different sorbents is essential for developing efficient and environmentally friendly processes. The research delves into various sorbents using the “hard and soft acid and base (HASAB)” concept [31]. This principle categorizes acids and bases as hard or soft based on their size, charge, and electronegativity. Hard acids and bases are small and have high charge and low electronegativity, favoring ion exchange interactions. On the other hand, soft acids and bases are large, have low charge, and high electronegativity, favoring covalent interactions. Most PGM ions belong to “soft acids” and own a strong affinity for “soft base” donor atoms in the order of $S \sim C > I > Br > Cl \sim N > F$ [32]. Thus, it is logical to assume that functional groups with these elements can be used as a sorbent to extract and recover PGMs. This work uses a theoretical framework to examine the ability of various solid-phase sorbents to recover and separate PGMs from secondary sources, such as spent catalytic converters (SCC), spent catalytic gauze (SCG) from an ammonia oxidation reactor, and waste from a precious metals refinery company known as "Cu-black." The sorbents under examination encompass Di-octyl Sulfide (DOS), Cyano (CN), Di-Ethylene Tetra-Amine (DETA), and a commercial strong anion exchanger (SAX).

Experimental

Chemicals and Sorbents

Ultra-pure and deionized water from a Millipore Milli-Q system (Bedford, USA) was used throughout. All reagents used in the test were of analytical grade unless otherwise said. Suprapur 30 % (w/w) HCl and 65 % (w/w) HNO_3 were from TCI-America (USA). Single-element stock solutions of Pd, Pt, and Rh, (1,000 µg/mL in 10%v/v HCl) and base metals: Al, Cu, Fe, Mg, Ni, Pb, and Zn (50 µg/mL in 4.9 % HCl) were obtained from Scientific-Fisher (USA). Dioctyl sulfide (DOS) and Di-Ethylene Tetra-Amine (DETA) reagents were bought from Sigma-Aldrich (Germany). Polymeric-C18 SPE, Cyano (CN) SPE, and Strong Anion Exchanger (SAX) SPE sorbents packed in syringe barrels were bought from Phenomenex (Phenomenex, USA) and were used as they are. Each barrel has a reservoir volume of 6 mL and holds 500 mg sorbent with an average particle size of 55 µm and pore diameter of 70 Å. The DOS and DETA sorbents were prepared by impregnating respective polymeric-C18 SPEs with the ligand. .

Material

The materials used in this study are a honeycomb spent three-way gasoline catalytic converter, a spent catalytic gauze (SCG) from an ammonia oxidation reactor, and waste from a PGM refinery company known as "Cu-black." The honeycomb unit is obtained from Cohen Recycling Company in Sharonville, Cincinnati, Ohio. The spent catalytic gauze and “Cu-black’ are supplied by K. A. Rasmussen AS, Hammar, Norway.

The honeycomb unit was crushed and ground using a grinding machine until the powder size reached 0.3 mm. The ground sample was homogenized before preconditioning in a 2 M NaOH solution at 90oC for 60 min, followed by hot water washing and overnight drying in a warm air oven at 80oC. The spent catalytic gauze and the Cu-black are reduced by cutting to ensure they fit into the leaching vessels.

Instrumentation

Temperature and pressure feedback Milestone ETHOS 1600 microwave oven (Sorisole, Italy) equipped with 10 x 100 mL TFM vessels was used for microwave acid leaching of the PGMs from the secondary materials. An Agilent 5800 Inductively coupled atomic emission spectrometer (ICP-AES), Agilent (USA), was used for the elemental analysis of the samples. A peristaltic pump (Minipuls 3, Gilson, Paris) was used for SPE sorption and desorption tests.

Microwave Leaching

While different protocols for the digestion of PGM-containing materials exist in the literature, the aqua-regia system, i.e., the combination of hydrochloric acid and nitric acid, is particularly effective and employed in this study. Three replicates and 5 g each of SACC powder, spent wire mesh catalyst, and Cu-black samples were weighed accurately, and each was placed in 100 mL PFTE vessels with 25 mL aqua-regia (solid/liquid ratio 1:5). The decomposition vessels were sealed and heated following a stepwise digestion program. Vessel No. 10, having 1.7 g for each of the three sample materials (5.1 g in total) and 25 mL of Aquaregia, was fitted with temperature and pressure sensors and used to control the leaching process. The microwave program consists of a 5min ramp to reach 200°C, a 15-minute holding step at 200°C, a 5min ramp to 240°C and a 10min holding step at 240°C, which allowed near complete digestion of SCG and Cu-black samples, and partial dissolution of the SCC sample. The solid residue left in the SCC vessels is from undissolved silica material. After cooling, the digests were quantitatively transferred to 100 mL beakers, and the residue was filtered off using a 0.45 µm filter membrane before being transferred to 100 mL volumetric flasks and brought to volume with water. The resulting solution was diluted one hundred times using 0.15M HCl solution and ready to use. A small part of the digest solutions was analyzed using ICP-OES to verify the content of PGMs and base metals concentrations in the sample materials after proper sample dilution. The remaining solution was used for the extraction test.

Sorption and Desorption Procedures

Before the sorption, the sorbents were pre-conditioned with three column volumes of Milli-Q water followed by the same amount of 0.15 M HCl solution to activate the sorbent's functional group and remove air in the void volume. Three replicates and 5 mL aliquot of each of the sample solutions were manually loaded to CN, DOS, DETA, and SAX sorbents, and a peristaltic pump connected to the outlet of the barrel was used to percolate the solution through the sorbent at a flow rate of 0.25 mL/min. The desorption of retained PGM ions was conducted by applying 3 mL of 3 M HCl solution onto loaded sorbents as eluent. The eluent was allowed to soak the sorbent for 2 minutes before the pump was started, with a flow rate of 1mL/min. The SPE effluents from both sorption and desorption tests were collected in respective glasses. The concentration of metal ions in the effluents was analyzed using ICP-OES without further dilution. The retention efficiency was calculated from the concentration of metal ions in the sample solution $[M]_{Sample}$ and column effluent $[M]_{Effluent}$ using Equation 1.

$$\% \text{ Retained} = ([M]_{Sample} - [M]_{Effluent}/[M]_{Sample}) \times 100 \text{ .... Eq. 1.}$$

The elution efficiency was calculated using Equation 2, based on the concentration of metal ions recovered from the sorbent and metal ions retained by the sorbent.

$$\% \text{Recovered} = ([M]_{Recovered}/[M]_{Retained}) \times 100 \text{ .... Eq. 2.}$$

The polypropylene tubes and barrels used in the study were tolerable in highly acidic environments. Unless otherwise mentioned, all the experiments were conducted in triplicates and at room temperature.

Results and Discussion

Compositional Analysis of Materials

Figure 1 shows the ICP-OES elemental analysis of the SCC unit, spent catalytic gauze (SCG), and "Cu-black" that were studied. The SCC part has three PGMs: platinum, palladium, and rhodium. On the other hand, the spent catalytic gauze (SCG) and Cu-black materials only have two PGMs, Pt and Rh. All three materials also have various base metals in different amounts. Please refer to the supplementary document for detailed information about the results of the ICP-OES analysis.

Sorption Studies

Figures 1 - 3 show the sorption profiles of PGMs (Pt, Pd, and Rh), along with accompanying base metals like Al, Cu, Fe, Mg, Ni, and Sn on SAX, DOS, CN, and DETA sorbents from the SCC, spent catalytic gauze, and Cu-black leachate solutions. These profiles were obtained using the conditions described in the experimental section. Each bar represents the percentage of the retained metal ion calculated using equation 1 (section 2.5) and averages two extractions. The relative error falls within the range of 1.8% to 4.8%.

The observed sorption profiles may be explained by the solution chemistry of the PGMs and their interaction with the functional groups of the sorbents. As mentioned in the introduction section, in high chloride environments where the sample materials are digested, PGMs, which are soft acids, exist not as simple cationic forms but as anionic chloro-complexes [33,34]. In contrast, most base metals are hard acids and exist in their cationic form.

As shown in Figure 1, the SAX sorbent selectively and quantitatively retained Pd and Pt and a large amount of Rh. It would make sense to assume that the retention of the PGMs on the SAX sorbent is due to the ion exchange mechanism between the anionic chloro-complexes of the PGMs and the amine functional group of the SAX sorbent. The non-quantitative retention of Rh (70% - 76%) suggests that a small part of Rh exists as a neutral species without being captured by the SAX sorbent. Rh is susceptible to forming neutral hydroxy and hydrated species at lower acidity, which may explain the phenomenon [2,3,34]. As expected, base metals in their cationic form are not retained by the similarly charged functional group of the SAX sorbent in the solution.

On the other hand, as depicted in Figure 2, only Pd was selectively captured by the DOS and CN sorbents. In contrast, the other PGMs (Pt and Rh) and the accompanying base metals passed through the sorbents without being retained. Unlike the SAX sorbent, the retention mechanism of PGMs by both CN and DOS sorbents could involve a ligand substitution mechanism rather than an ion exchange mechanism. The selective retention

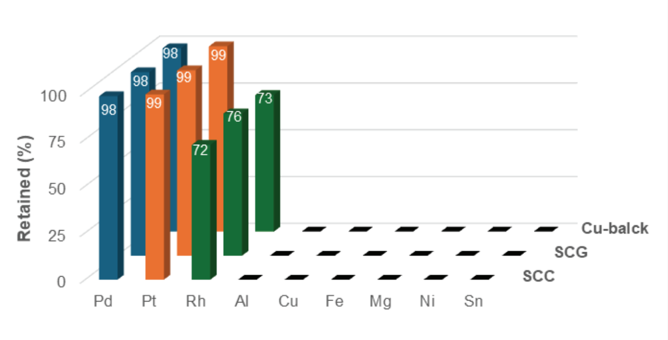

Figure 1. *The percentage of Pd, Pt, and Rh retained on the SAX sorbent from leachate liquor of SCC powder, spent catalytic gauze (SCG), and "Cu-black" samples in the presence of different base metals. Each bar averages two extractions, and the relative error is ≤ 5 %.*

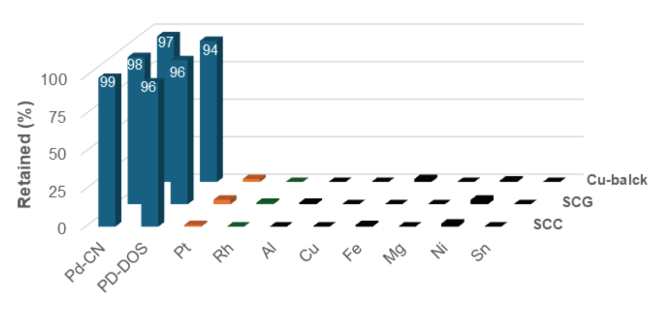

Figure 2. *The percentage of Pd retained on CN and DOS sorbents from leachate liquor of SCC powder, spent catalytic gauze (SCG), and "Cu-black" samples in the presence of different base metals. Each bar averages two extractions, and the relative error is ≤ 5 %. (Please note that the retention profiles of the PGMs and base metals on CN and DOS sorbents are combined in a single figure).*

of Pd by these sorbets may be attributed to the kinetic factor, where the rate of ligand exchange plays a significant role. Low oxidation state PGMs such as Pd (II) are tetra-chloro-complexes with square planar geometry, while high oxidation state PGMs such as Pt (IV) and Rh (III) exist as hexa-chloro-complexes with octahedral geometry [33,34]. Due to the steric effects of the atoms involved, it is faster for ligand substitution reactions to happen in square planar complexes compared to coordinationally saturated octahedral complexes [34,35]. For example, Reedijk [36] found that the rate of ligand exchange for Pd (II) is about five orders of magnitude faster than that of Pt (IV). This difference might explain why both CN and DOS sorbents selectively retain Pd.

The DETA sorbent selectively and quantitatively (> 91%) retained Rh while showing no affinity towards the other PGMs (Pd and Pt) and accompanying base metals (Fig. 3). The retention mechanism may occur through ligand substitution instead of ion exchange. As seen in Figure 5, around 9% - 10% of Rh is unretained by the DETA sorbent. The reason for this is the existence of neutral and hydrated Rh chloro-complexes, which are not reactive and, therefore, cannot be extracted by DETA.

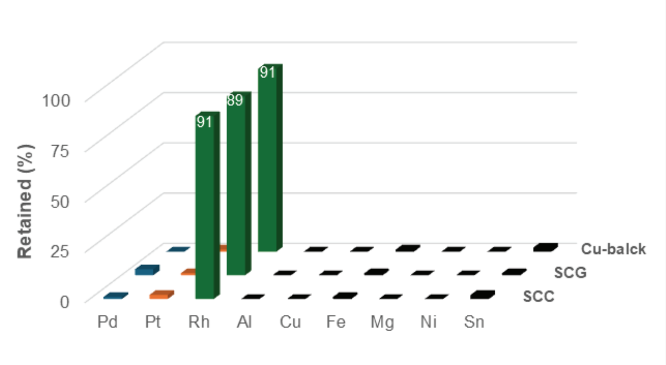

Figure 2. *The percentage of Rh retained on DETA sorbent from leachate liquor of SCC powder, spent catalytic gauze (SCG), and Cu-black samples in the presence of different base metals. Each bar averages two extractions, and the relative error is ≤ 5 %.*

Desorption Studies

It is equally essential that the retained PGMs are quantitatively recovered from loaded sorbents with a suitable solvent compatible with HES and cost considerations. Figure 4 illustrates the desorption profiles of Pt, Pd, and Rh from the loaded SAX sorbent, Pd from CN and DOS sorbents, and Rh from the DETA sorbent using the procedure outlined in the experimental section 2.5. Each bar is a percentage of the recovered PGM ions obtained using equation 2 (also in section 2.5) and an average of three extractions. The relative error is in the range of 2.1 to 3.8 %.

The study revealed that the PGMs loaded on all four sorbents can be quantitatively recovered using 3 M HCl at room temperature. In most other similar works, desorption is done using hot mineral acid mixtures, prolonged shaking, or destructive conditions [5,6,22,25,26]. Using only mineral acid and ambient conditions to separate and enrich PGMs has advantages, including the process's speed and the adsorbent's reusability.

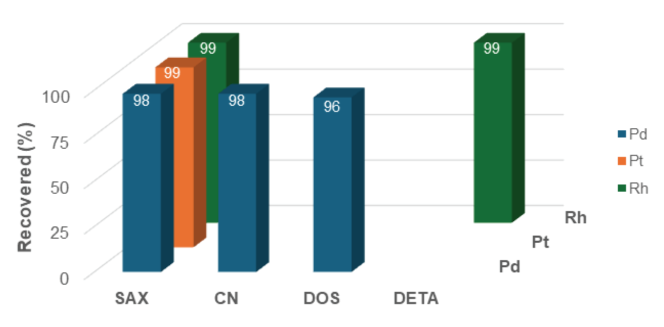

Figure 4. *The percentage of Pd recovered from loaded SAX, CN, and DOS sorbents and Rh from loaded DETA sorbent using 3 mL of 3 M HCl at ambient temperature. Each bar averages two replicates, and the relative error is ≤ 5 %.*

Repeated Use of Sorbents

The reusability of SAX, CN, DOS, and DETA sorbents was evaluated through multiple sorption and desorption cycles using synthetic PGM feed solutions and by comparing the metal's retention and elution efficiency at each step, as depicted in Figure 5.

The test solution for the SAX sorbent has 5 μg/mL of Pt, while the test solutions for CN and DOS sorbents have 5 μg/mL of Pd. The DETA sorbent test solution holds 5 μg/mL of Rh. The synthetic test solutions were kept at an acid concentration of 0.15 M HCl, and 3 M HCl was used as an eluent.

The study revealed that SAX, CN, and DOS sorbents can be used for at least six retentions and elution cycles without losing retention efficiency. In contrast, the DETA sorbent showed a consistent decrease in retention efficiency over the repeated retention/elution cycle, as illustrated in Figure 9. The primary reason for the increasing decrease in retention efficiency is the significant loss of DETA from the C18 substrate due to its high solubility in the aqueous solution. This shows that further improvements are needed. Our future studies will concentrate on resolving this issue.

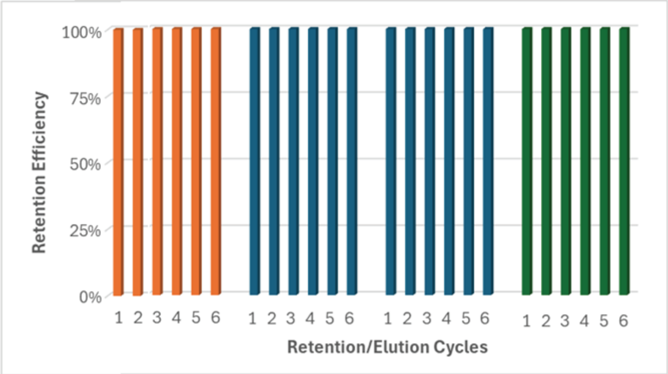

Figure 5. *Retention efficiencies of SAX, CN, DOS, and DETA sorbents after six successive retention and elution cycles using a synthetic solution having 5µg/mL Pt, Pd, and Rh.*

Proposed Scheme of PGM SPE Recovery.

Figure 6 proposes an integrated process for recovering PGMs, particularly Pt, Pd, and Rh, from secondary sources. The process involves multiple stages. Initially, shredded and pulverized secondary PGM-bearing material is microwave-leached with Aquaregia. In the second extraction stage, the solution is directed to CN or DOS sorbent for selective separation of Pd from Pt and Rh and the accompanying base metals. Rh from Pt and the base metals can be separated using DETA sorbent at the third extraction stage. Subsequently, in the fourth extraction stage, Pt is separated from base metals using SAX sorbent. Once the extraction is completed, the retained PGMs should be recovered from the loaded sorbents using 3 M HCl, and the sorbents can then be regenerated and reused. The obtained stripping solutions may undergo further treatment, such as selective reduction of Pd (II), Rh (III), or Pt (IV) to get the metallic form of PGMs.

Conclusion

SPE sorbets of different functional groups were evaluated and compared regarding platinum group metals' recovery and separation performance from several secondary sources. Strong anion exchanger (SAX) sorbent showed high affinity towards PGMs (Pd, Pt) and, to a lesser extent, for Rh over accompanying base metals, including Al, Cu, Fe, Mg, Ni, and Sn. Selective retention of Pd can be achieved from the same solution with CN and DOS sorbents, which showed no retention of the other PGMs (Pt and Rh) and the base metals. The DETA sorbent showed high selectivity for Rh while showing no affinity towards the other PGMs (Pd and Pt) and accompanying base metals. Recovery of retained Pt, Pd, and Rh ions from loaded SAX, Pd from CN and DOS sorbents, and Rh from DETA sorbent can be achieved with 3 M HCl with significant safety and environmental advantages.

The SAX, CN, and DOS sorbents showed high stability and reusability. However, the reusability of the DETA sorbent is limited due to the loss of the ligand during repeated use, attributed to its higher solubility in an aqueous solution. This suggests that further improvements are necessary. Our future studies will focus on enhancing the recovery of the Rh procedure.

The study also showed that conditions can be fine-tuned for group or individual separation of Pd, Pt, and Rh in the presence of accompanying base metal ions. The developed separation method is efficient, fast, and simple, and we expect its application to various PGM-containing matrices.

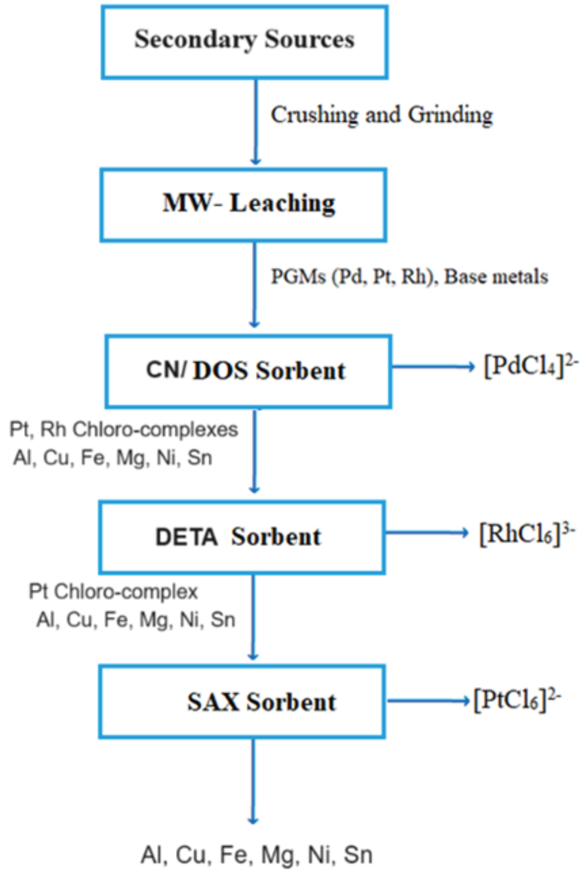

Figure 6. *Simplified flow sheet of PGM leaching and recovery process.*

Disclosure statement

The authors report there are no competing interests to declare.

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