



# Recycling strategy for the extraction of PGMs from spent PEM electrodes

L. Sandig-Predzymirska, T. V. Barreiros, A. Thiere, A. Weigelt, D. Vogt, M. Stelter, A. Charitos

Institute for Nonferrous Metallurgy and Purest Materials (INEMET)

TU Bergakademie Freiberg

Leipziger Straße 34

09599 Freiberg, Germany

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## Abstract

Platinum group metals (PGMs) such as iridium, ruthenium, and platinum are state-of-the-art electrocatalysts for proton exchange membrane (PEM) electrolyzers. These are of great significance concerning energy storage from renewable sources. However, the natural resources of these metals are very limited. On the contrary, worldwide consumption increases every year. Therefore, to meet future demands it becomes increasingly necessary to recycle secondary raw materials, where the PGM's concentration is usually higher compared to the natural ores. At present, the recycling of PGM-containing materials is mainly performed by hydro- and pyrometallurgical methods (a main example hereto being the secondary metallurgy of copper) or by a combination of both. Hydrometallurgical processing has attracted much attention as it requires less energy and initial investment costs compared to pyrometallurgical alternatives. However, the associated drawbacks are the generation of large amounts of waste and the use and/or generation of some toxic chemical components. Thus, the development of a high-efficiency extraction procedure for PGMs with a simultaneous minimizing amount and toxicity of the reactants is essential.

This study aims to investigate novel hydrometallurgical routes for the separation of PGMs from spent PEM electrodes. Generally, hydrometallurgical processing consists of two main stages. The first step is the transfer of metal incorporated on the material into the solution. The next step involves the separation of PGMs from the solution with subsequent treatment to recover high purity salts or metals. The leaching step is determining the efficiency of the recycling procedure. Therefore, to achieve high PGM recovery rates, the leaching of electrode materials in different lixiviant mixtures has been carried out. The influence of important operating variables (e.g. reagent concentration, additives, initial temperature of leaching solution, and leaching time) on the leaching yield of PGMs has been studied. The first results of the developed process for PGM's recovery from PEM electrodes are presented.

# 1 Introduction

Polymer electrolyte membrane electrolysers (PEMEC) have attracted much attention during the last decade due to the growing interest in the conversion of renewable energy into hydrogen. The electrochemical reactions for hydrogen and oxygen evolution are catalyzed by PGMs. Therefore, spent PEM electrodes are a rich source of PGMs compared to the mined ores. The most extensively applied metals for PEM electrolysis are Pt/Pd (at the cathode) and Ir/Ru (at the anode) [1-3]. Other PGMs are usually introduced as an alloy with Pt to reduce the costs of the electrolyser. Furthermore, due to PGM supply risk and economic importance, these metals are considered as critical raw materials (CRM) for the European Union [4]. The high price of precious metals and the constantly growing consumption require efficient recycling technologies, which also will help to reduce the environmental impact of metal supply, especially regarding climate change.

In recent years recycling technologies of spent catalysts (from automobile catalytic converters, fuel cells, electrolysers) have been considerably improved [5-7]. However, there are still some challenges in order to achieve an environmental-friendly and sustainable recycling approach with high PGMs recovery rates.

Generally, the recovery of PGMs is performed by hydro- or pyrometallurgical treatment of the secondary material. In pyrometallurgical processes, the spent catalyst is melted with the addition of metal collectors (e.g. Cu, Fe, Pb), flux components (e.g.  $\text{Al}_2\text{O}_3$ , CaO, MgO,  $\text{SiO}_2$ ), and reducing agent (e.g. powdery coke) at a high temperature above 1000 °C to concentrate PGMs in a collector phase [7, 8]. Fluxing increases the amount of waste material associated with the generation of a slag phase. These processes are high-cost and energy-intensive and require high-grade feeds. In addition, the combustion of the PEM components such as fluor-containing membranes causes corrosive and hazardous HF-containing gases, which are highly regulated in discharge [9]. Therefore, the separation of the membrane from catalysts prior to pyrometallurgical treatment is preferable.

Hydrometallurgical processes are based on the selective dissolution of PGMs from the spent catalyst in alkaline (e.g. NaOH, NaCN) or acidic (e.g. HCl,  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$ ) media, with the addition of different reagents (e.g.  $\text{H}_2\text{O}_2$ , HF, NaCl, HClO) [6, 10]. The oxidation agent must ensure the oxidation of the metal to an ionic form and the acid concentration must be sufficient to activate the complexation reaction with metal ions. Despite high corrosivity, the role of aqua regia in hydrometallurgical processing is central [6]. The leached solution, which contains the PGMs complexes, is subsequently treated by precipitation, solvent extraction, ion exchange, etc. in order to concentrate the precious metals. The application of concentrated acids and the consumption of large amounts of reagents in the hydrometallurgical route is to be minimized from both a technical and environmental standpoint.

Up to now, the majority of published studies were focusing mainly on the PGM recovery from spent automotive catalysts [11]. However, very few studies are available on the recycling of catalysts in PEM electrodes [12]. In the case of PEMEC, the recycling process is more complex due to the presence of supporting materials (e.g. membrane, gas diffusion layer (GDL), carbon nanoparti-



cles). The catalytic layers in PEMEC are usually composed of Pt nanoparticles supported on the carbon (cathode side) and Ir/Ru nanoparticles (anode side), which are mixed with an ionomer to ensure ionic contact to the membrane. Thus, the PGMs particles in PEM electrodes could be less accessible to leaching agents, impairing the efficiency. Furthermore, the studies on the hydrometallurgical treatment of spent catalysts mainly focused on the leaching and separation of Pt and Ir (mainly used in catalytic converters) [5, 13], while the leaching of Ru is poorly described in the literature.

The aim of this study is to propose a hydrometallurgical method for the recovery of PGMs, such as Pt and Ru, from spent PEM electrodes by the application of less aggressive and corrosive reagents at the mildest conditions possible. The focus of the experimental work is mainly on the leaching process since it is an essential part of the whole recycling procedure. The goal was to investigate the influence of various leaching parameters on the recovery yields of Pt and Ru in the electrode material.

## 2 Methodology

### 2.1 Sample preparation

The electrode (supplied by the Danish Technological Institute (DTI), Denmark) consists of a carbon cloth with a PTFE treated microporous layer. A carbon-supported PtRu catalyst mixed with a Nafion ionomer dispersion was deposited on top using ultrasonic spray deposition. The pre-treatment of the electrode material with an isopropanol-water mixture was performed to separate the catalyst from the hydrophobic carbon substrate. The produced “dry residue” was further utilized for the leaching experiments. Table 1 shows the values provided by the supplier, which are termed as “theoretical” and the obtained values of PGMs. This is realized from the PtRu-based electrode material and the dry residue that contains the catalyst, after separation from the substrate. The alkaline fusion with a mixture of  $\text{NaNO}_3 + \text{NaOH}$  (1:5) at 600 °C was applied for the determination of ruthenium and platinum in the material. Despite the differences in the theoretical values and those resulting from analysis, the values determined in this study were confirmed by an external laboratory. Moreover, during the separation step, some parts of the PTFE layer may be detached from the substrate and contribute to the total mass of the dry residue, which results in a lower amount of PGMs compared to the theoretical value. Therefore, it is the “analysis” values respective to the dry residue that are considered for the calculation of the leaching efficiency.

In the electrode material, the total carbon content originates from carbon nanoparticles, Nafion polymer dispersion, and C-substrate. Carbon content was determined using the ELTRA CS-580 analyser. Scanning electron microscopy energy-dispersive X-ray spectroscopy (SEM-EDX, Carl Zeiss – Ultra 55) has been used to provide information about the surface material composition.

Table 1: Composition of the electrode material and the dry residue (wt.% - dry basis)

		Pt	Ru	C	Nafion	Substrate
<b>Electrode</b>	<i>Theor.</i>	22.1	11.1	11.1	14.7	41.0
	<i>Analysis</i>	20.4	10.3	(Total C-content: 47.4)		
<b>Dry residue</b>	<i>Theor.</i>	37.5	18.8	18.8	24.9	-
	<i>Analysis</i>	31.8	16.1	(Total C-content: 21.8)		

## 2.2 Experimental procedure

The following chemicals were used for the leaching experiments: HCl (37 %, VWR), HNO<sub>3</sub> (69 %, Merck), H<sub>2</sub>O<sub>2</sub> (30 %, Merck), NaCl (99.5 %, Merck), CuCl<sub>2</sub> (99 %, Acros organics), AlCl<sub>3</sub>·6H<sub>2</sub>O (99 %, Alfa Aesar). The samples with a mass of 0.1 g ( $\pm$  0.001 g) were used for the leaching tests. From several preliminary experiments, the leaching conditions were determined and used as a default condition. Based on the default condition, the leaching experiments were conducted with a total volume of the leaching solution (40 mL) and agitated by a magnetic stirrer (500 rpm) at 75 °C. During the experiment, samples of the solution were taken, filtrated, cooled down, diluted with water, and analysed by inductively coupled plasma optical emission spectrometer Varian (ICP-OES). The leaching efficiency for PGMs was calculated, as discussed, based on the determined percentage of PGMs in the dry residue (listed in Table 1, see “analysis” values).

## 3 Results and discussions

### 3.1 Pre-treatment procedure

The electrode material used for the tests consists of a mixture of precious metals (in the form of PtRu nanoparticles) with the ionomer and the hydrophobic substrate which is the challenge for the leaching procedure. This makes metal recovery directly from the untreated electrode material complicated because the chemical coating inhibits the contact between a lixiviant and the metal. Therefore, it is beneficial to remove the chemical coating before the leaching tests. It has been previously shown that lower alkyl-alcohols can disrupt the bond between the fluorocarbon containing membrane or GDL and the attached PGMs catalyst layers, followed by dispersal of the catalyst layers in the alcohol or alcohol-water mixtures [14]. In this work, it has been shown that by treating the electrode material with the alcohol or alcohol-water mixture the separation of the catalyst layer from the carbon substrate also takes place. The simple agitation or ultrasound treatment of the material with the solvent mixture for about 30 min was enough to separate the catalyst from the substrate. In this way, the catalyst layer is collected in the alcohol-water mixture and recovered as the “dry residue”



after evaporation of the solvent. The additional washing of the substrate with the solvent was performed to ensure the complete removal of the catalytic layer.

SEM images (Fig. 1, magnification of  $\times 100$ ) illustrate the surface topography of the electrode material (a), the pure substrate as produced (b), and the electrode material after separation from the catalytic layer with the alcohol-water mixture (c).

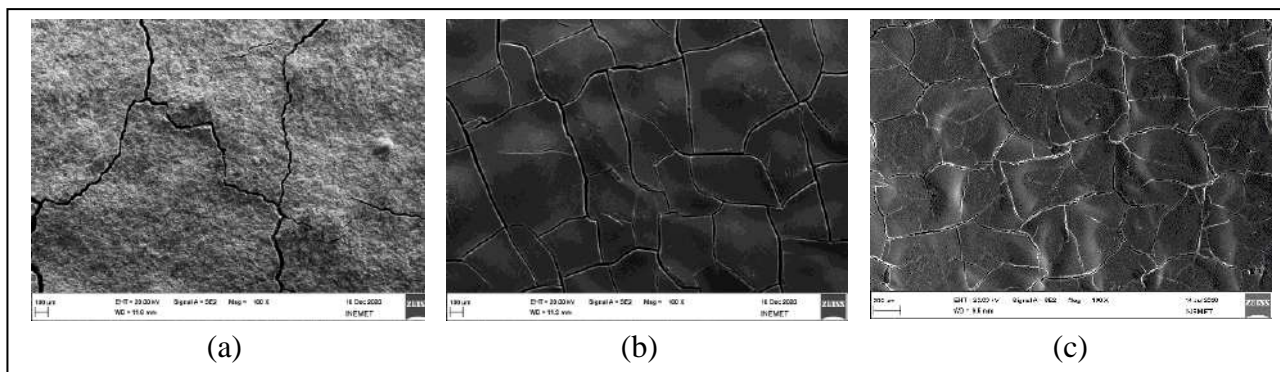


Figure 1: SEM images of PtRu-based electrode material (a,  $\times 100$ ), C-substrate (b,  $\times 100$ ), separated C-substrate from the electrode material (c,  $\times 100$ )

EDX analysis of the substrate surface (c) left after separation from the catalytic layer showed no traces of PGMs, and confirmed that the separation with the isopropanol-water mixture was efficient. The performance of isopropanol-water was found to be better in terms of separating catalyst and substrate compared to experiments using an ethanol-water mixture or pure alcohol.

### 3.2 Leaching experiments

Two different oxidation agents in an HCl-based leaching system were examined in order to evaluate the leaching efficiency of PGMs recovery from the electrocatalyst. The first experimental series was carried out to evaluate the best performing leaching system. The second one studied the effect of process parameters, such as reagent concentration, leaching duration, and additives on the efficiency of PGMs recovery.

At first, the electrode and the catalyst residue were examined by using aqua regia at  $75\text{ }^{\circ}\text{C}$ . Further, the  $\text{HNO}_3$  or  $\text{H}_2\text{O}_2$  concentration was varied in the HCl-based leaching system to evaluate the influence of the oxidation agent on Pt and Ru dissolution. In the HCl media, the oxidation and dissolution of the precious metal take place and chloro-complexes are formed [15]. The dissolution of metallic ruthenium tends to be challenging because it is high resistant to dissolve in mineral acids. Therefore, the leaching efficiency for ruthenium was always lower compared to platinum.

The results of the leaching tests are presented in Figure 2. By leaching the electrode material (including substrate) with aqua regia the leaching efficiency was about 25 % lower (for both metals) compared to the dry residue (without substrate). This result is of great importance as it shows that the separation step facilitates the accessibility of PGMs for the leaching process. Figure 1 (left)

shows also the effect of the amount of HNO<sub>3</sub> addition on PGMs recovery yields from the dry residue. By varying the HNO<sub>3</sub> concentration, the leaching efficiency increases with the lowering of the HNO<sub>3</sub> amount in HCl. In the mixture of 10 vol% HNO<sub>3</sub> and 12M HCl the recovery of Ru and Pt after 3 h of leaching reaches 88 % and 97 %, respectively. Another set of experiments was carried out to study the effect of H<sub>2</sub>O<sub>2</sub> addition on the Pt and Ru leaching performance (Figure 2, right). The combination of hydrochloric acid with 10 vol% of hydrogen peroxide showed the highest extraction of PGMs (91 % and about 100 % for Ru and Pt, respectively). The higher oxidation potential of hydrogen peroxide ( $E_0$ : 1.77 V/SHE) compared to the oxidation potential of nitric acid ( $E_0$ : 0.96 V/SHE) can partly explain why HCl-H<sub>2</sub>O<sub>2</sub> mixture showed such a higher efficiency for Pt/Ru dissolution. Considering measurement errors, the leaching efficiency with HNO<sub>3</sub> at a low concentration of 10 vol% is in the same range as with H<sub>2</sub>O<sub>2</sub>. This result is important since it shows that hydrogen peroxide is an effective co-reactant and it provides the same high PGMs recovery yields as the strong mineral acid. It is worth mentioning that gas formation was observed while adding hydrogen peroxide, which can be explained by its decomposition to water and oxygen at the operating temperatures. Considering the thermal instability of hydrogen peroxide [16], which can influence the leaching reaction, the stepwise addition of hydrogen peroxide was tested. However, it did not show improved performance compared to the single addition of the oxidant at the beginning of the leaching process.

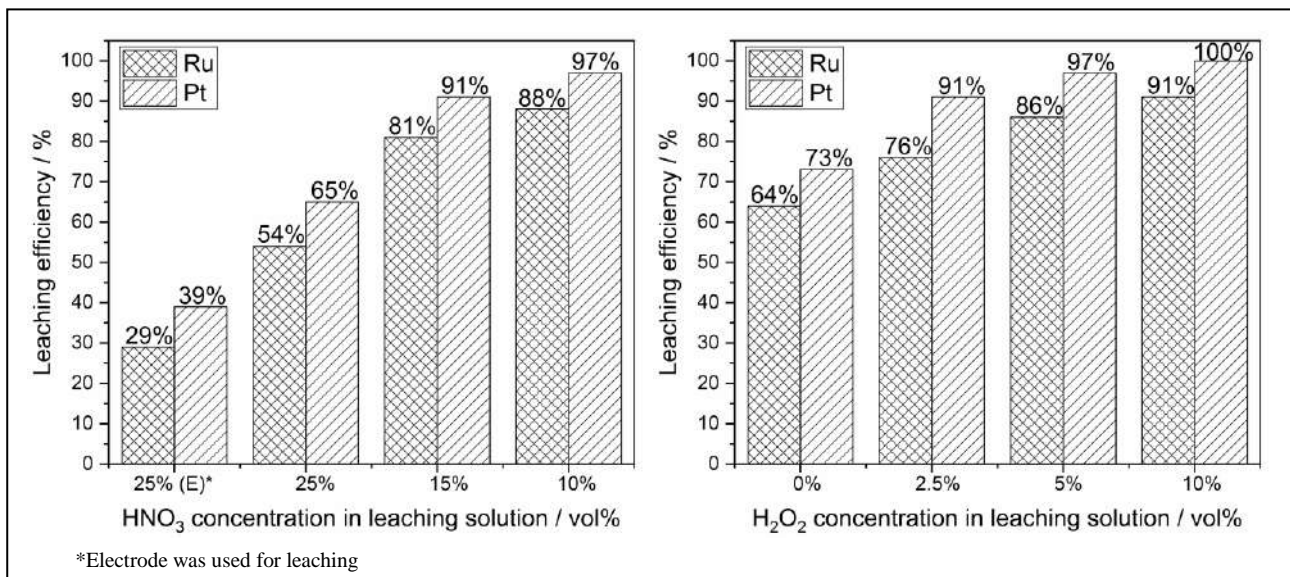


Figure 2: Influence of the lixiviant mixture on the platinum and ruthenium recovery (conditions: 12M HCl; T: 75 °C; duration: 3 h)

The HCl-H<sub>2</sub>O<sub>2</sub> mixture is less toxic and less corrosive, compared to HCl-HNO<sub>3</sub> system, and thus has advantageous properties to be applied in the hydrometallurgical process. Therefore, due to the importance of an environmentally friendly process, further research focused on the efficiency of H<sub>2</sub>O<sub>2</sub> by modifying different parameters. It is worth noting that concentrated hydrochloric acid was used in both cases in order to compare these systems. Next, several leaching tests were conducted at



75 °C for 4 h for optimizing the concentration of the hydrochloric acid. The effect of the HCl concentration on the time required for complete metal recovery from the electrocatalyst was studied. As expected, the increase of the HCl concentration from 4M to 12M significantly improved the metal dissolution, and the time required for complete metal recovery decreased (Figure 3, left). Even with the 4M HCl, the relatively high PGMs leaching yields can be achieved if the leaching time can be extended. Considering this factor, safety reasons, and reduction of corrosivity, a lower than 12M concentrated HCl was used in the subsequent tests.

Another important effect was observed when the leaching solution was pre-heated before the leaching experiment. By using 8M HCl with 5 vol% of H<sub>2</sub>O<sub>2</sub> (Figure 3, right), the leaching efficiency was more than 10 % higher (for both metals) for the pre-heated sample compared to the leaching test started at room temperature. Even though the heating to the desired temperature only takes about 13 minutes in the stirring plate applied, the influence of the temperature is significant since the leaching of the metal starts with the induction period, compared to the pre-heated solution where the reaction starts immediately after the contact of metals with the lixiviant. Therefore, further experiments were carried out by pre-heating the leaching solution before mixing it with the solid sample, in order to obtain a higher leaching efficiency in a reduced time.

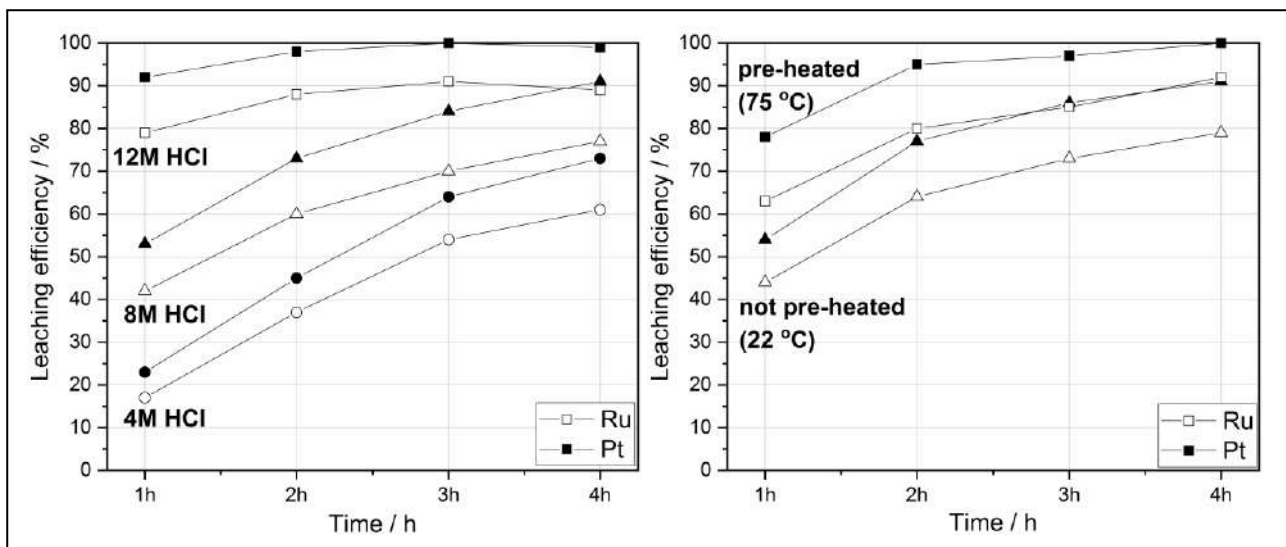


Figure 3: PGMs leaching efficiencies obtained with different HCl concentrations and 10 vol% H<sub>2</sub>O<sub>2</sub> at T: 75 °C (left), and with different initial temperatures of starting solutions of 8M HCl and 5 vol% H<sub>2</sub>O<sub>2</sub> at T: 22 °C and T: 75 °C (right)

It has been previously reported that the use of chloride salts provides the required Cl<sup>-</sup> concentration for the PGMs chloro-complexes formation under mild leaching conditions [11]. Therefore, in the next step, chloride salts were introduced in the 4M HCl leaching solution (Table 2) to substitute HCl as chloride supplier, and thus to reduce the acidity of the leaching system. The concentration of the salts was varied to investigate the influence of the cation and the chloride content on the leaching performance.

Table 2: PGMs leaching efficiencies obtained when using different chloride source and their concentrations (conditions: 4M HCl (pre-heated to T: 75 °C), 5 vol% H<sub>2</sub>O<sub>2</sub>, duration: 4 h)

<b>AlCl<sub>3</sub></b>	<b>CuCl<sub>2</sub></b>	<b>NaCl</b>	<b>Total [Cl<sup>-</sup>]</b>	<b>Ru, %</b>	<b>Pt, %</b>
0.5M	-	-	5.2M	72	81
-	0.75M	-	5.2M	56	67
-	-	1.5M	5.2M	73	82
-	1.5M	-	6.7M	79	90
1.5M	-	-	8.1M	84	96
-	-	-	3.8M	49	60

In general, AlCl<sub>3</sub> and NaCl based leaching solutions, with the same Cl<sup>-</sup>-concentration, lead to the dissolution of PGMs with the same leaching rate considering the experimental error. However, with the same salt concentration (1.5M), higher yields are achieved when using AlCl<sub>3</sub>, since the chloride ion concentration is higher compared to the other systems. As it was mentioned before, the use of hydrogen peroxide is generally associated with some challenges due to its thermal instability. In case of the HCl-H<sub>2</sub>O<sub>2</sub>-CuCl<sub>2</sub> system, it can be assumed that copper ions catalyse the decomposition of hydrogen peroxide [17], and thus lead to the decrease of the leaching efficiency, compared to NaCl or AlCl<sub>3</sub> used with the same Cl<sup>-</sup>-concentration.

Especially for Ru, good recovery rates must be noted, when using AlCl<sub>3</sub>, because leaching of Ru from catalyst materials is often less effective than leaching of Pt. This result is of great importance since it clearly shows that the ternary mixture (HCl-H<sub>2</sub>O<sub>2</sub>-AlCl<sub>3</sub>) provides high leaching yields for Pt and Ru recovery at the low HCl concentration. The future work will be focused on the improvement of this leaching mixture to develop a highly efficient and cost-effective recycling process for PEM electrodes.

### 3.3 Hydrometallurgical processing of PEM electrodes

The objective of this part is to propose the hydrometallurgical process for the recovery of PGMs from PEMEC. The flow-sheet diagram for the efficient separation of Ru and Pt from the electrode material is shown in Figure 4.

The proposed hydrometallurgical process for the electrodes consists of three main steps: pre-treatment, leaching, and refining. First, the separation of the electrocatalyst from the substrate needs to be performed. The proposed method involves the extraction of the catalytic layer from the substrate by the alcohol-water mixture. The pre-treatment step is of great importance not only for the hydrometallurgical process but also for pyrometallurgical treatment, as it eliminates the issue of the emission of hazardous fluorine gases. Solvents from the separation step can be reused by evaporation from the catalyst suspension and condensation as a clean solution, thus, minimizing the waste of the alcohol in the recycling scheme. After separation, the solid residue will be subsequently





treated with the leaching solution. In the case of a bimetallic catalyst, a single leaching step with HCl-H<sub>2</sub>O<sub>2</sub> medium is required. It will be of interest in further work to test how multi-element electrodes will behave in the proposed leaching system. Finally, the refining steps (e.g. precipitation, solvent extraction, or ion exchange) are necessary to obtain high purity metals or the corresponding PGMs salts.

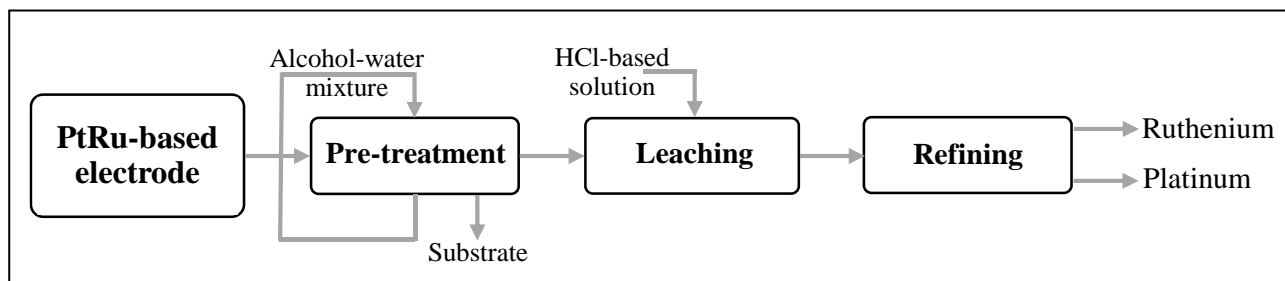


Figure 4: Proposed flow-sheet diagram for the hydrometallurgical processing of the PtRu-based electrode material

## 4 Summary

The performed study showed that the pre-treatment of the electrode material with the solvent-water mixture, in order to separate the catalytic layer from the substrate, is an essential step for the effective leaching process. In addition, the effects of two different oxidation agents (HNO<sub>3</sub> and H<sub>2</sub>O<sub>2</sub>) for the leaching efficiency of PtRu-based electro-catalyst in HCl medium were investigated. The HCl-H<sub>2</sub>O<sub>2</sub> based leaching system does not produce harmful emissions, compared to commonly used aqua regia, and confirmed to be useful for high recovery rates of Pt and Ru even at mild conditions. The maximum leaching yields (91 % for Ru and about 100 % for Pt) were achieved in one leaching step by using concentrated HCl (12M) and H<sub>2</sub>O<sub>2</sub> (10 vol%) at 75 °C for 3 h. Furthermore, PGMs were leached with high leaching rates by using a low concentration of the acid, namely 4M HCl, and widely accessible additives, such as NaCl or AlCl<sub>3</sub>. It has been shown that pre-heating of the solution before leaching significantly improves Ru and Pt recovery yields. Finally, a simplified hydrometallurgical flowsheet consisting of three steps, namely: pre-treatment (substrate separation), leaching, and refining has been presented based on the aforementioned experimental findings.

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