Sustainable SoluTions FOR recycling of end-of-life Hydrogen technologies

Deliverable D1.1

Lab Scale Optimization Results on the 3 PEMFC Recycling Technologies Report

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Abbreviations

AB	Advisory Board
AD	Alcohol Dissolution
AAS	Atomic Absorption Spectroscopy
BPP	Bipolar Plate
BMIM Cl	1-Butyl-3-methylimidazolium chloride
BMIM-TFSI	1-Butyl-3-methylimidazolium bis trifluoromethyl sulfonyl imide
CCM	Catalyst Coated Membrane
CL	Catalyst layer
EoL	End-of-Life
GA	Gant Agreement
GDE	Gas Diffusion electrode
GDL	Gas Diffusion Layer
GPC	Gel Permeation Chromatography
HMT	Hydrometallurgical Process
HRD	Hensel Recycling Deutschland
HTH	Hydrothermal Treatment
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
IL	Ionic liquid
LCA	Life Cycle Assessment
LCC	Life Cycle Costing
MAS	Magic Angle Spinning
MEA	Membrane Electrode Assembly
NMR	Nuclear Magnetic Resonance
NP	Nanoparticle
PFSA	Perfluorosulfonic acid
PEMFC	Polymer Electrolyte Membrane Fuel Cell
PGMs	Platinum Group Metals
Pt	Platinum
SEM-EDX	Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy
TGA	Thermal gravimetric analysis
US bath	Ultra Sound Bath
WP	Work Package

Summary

List of Figures

List of Tables

1 Executive Summary

Within the **WP1**, which is under the leadership of Hensel Recycling Deutschland (HRD), the first Deliverable (D1.1) due on the 30th of December 2021, focuses on **Lab Scale Optimization Results on the three Polymer Electrolyte Membrane Fuel Cell (PEMFC) recycling technologies**, thus presenting the results of the first 12 months of work done.

The outcomes of WP1 are crucial to the whole project as the recovered materials will be characterized and reused in new cells as well as in a stack in WP2. The successful validation of the recovery technologies employed will be finally transferred to WP5 for the Life Cycle Analysis (LCA) and Life Cycle Costing (LCC). The partners involved in WP1 are: Environment Park, CEA, EKPO and the University of Ljubljana (UL).

According to the scope of the project, **strategic recyclable and reusable materials** and **components (Pt and ionomer)** in cells must be identified and recovered using **two existing technologies** and **one new technology**. This should also include a minimum **30%** of **recycled critical raw materials (CRMs)**, with a reduction of the overall cost of the PEMFC stack.

The goal is the recovery of 80% of Pt content via **hydrometallurgical process (HTM)**, while an **alcohol dissolution process (AD)** should recover 90% of Pt and more than 80% of ionomer. Finally, **electro-leaching** and **electrodeposition** should permit to recover Pt metal (>95% yield), spent membrane (100%) and GDL (100%) for open loop scenarios[1].

The main optimization challenges of three recycling technologies are among others to:

- Increase the platinum **recovery %** from disassembling, avoiding loss of material (CCM),
- Obtain **Pt as Pt salt (Diammonia hexachloroplatinate)** as final process product to reuse it in a closed-loop-recycling,

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[1] Billy, E.; Balva, M.; Leclerc, N.; Legeai, S.; Meuc, E. Process for the recovery of platinum, electrochemically, from a material which contain same. EP 3 263 744 B1, 2016.

- Demonstrate **high recovery capacity** (> 80%) of **ionomer** and **Pt/C** to reintroduce in closed-loop-recycling,
- Demonstrate a more **environmentally friendly** treatment route, with or without the use of organic solvents or strong acids, to avoid emissions of toxic gases.

2 Introduction

Hydrogen is becoming more and more a realistic solution contributing to the decarbonization of our economy. As demand for hydrogen technology increases, ever more devices become in use and will eventually end up in the waste stream, requiring therefore handling in a sustainable manner. The issue is not only ensuring a correct disposal path within a clear regulatory context, but also ensuring the full recovery of the critical materials used. This is crucial not only for safeguarding that the new hydrogen technologies and devices have a lower environmental impact than their counterparts over the whole life cycle but also for recirculating extremely precious and rare materials supporting the whole hydrogen economy delivery.

A correct disposal path within a clear regulatory context and the full recovery of the critical raw materials (CRM's) used is needed. This is crucial for supporting the new hydrogen systems, as lower environmental impact technologies, and the whole hydrogen economy delivery.

Currently, according to the findings of previous research initiatives, such as the HyTechCycling[2] project, there are very few examples of viable and up-scaled technologies for the recovery and recycling of CRMs from fuel cells (FCs) and electrolysers. Aside the current low volumes of arisings – a contingent factor - the recycling industry would currently be unable to take them up because of the following shortcomings: complexity and cost, also due to issues with emissions and hazardous materials, low level of technology readiness.

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[2] http://hytechcycling.eu/

The innovative technologies developed within the WP1 concentrate on Pt (high material value and high criticality[3]) and ionomer (PFSA, medium material value but also medium criticality[2]) for PEMFCs. In terms of recovery targets, it is known that Platinum group metals (PGMs) rich devices enter in the recycling chain for only 11% of the arisings (low value), while a good rate of recovery at end-of-life (up to 95%) is achieved for industrial catalyst and for automotive catalyst (50-60%), but to give the correct contribution it is necessary to increase the input rate. PGMs present a high value of recovery and have a high value which makes them particularly attractive for their recycling because they have a high added value both in closed loop and in open loop recycling (interesting revenues) [4].

The technologies are based on readaptation of existing methods applied for the recycling of devices with similar level of complexity and content of risky/precious/critical materials based on Hydrometallurgical Process (HMT) for Pt salt recovery for re-manufacturing the PEMFC cells. Novel technologies are also studied and developed to recover ionomer via Alcohol Dissolution (AD) process and reintegrate it in a cell; the electrochemical recovery route that couples electrochemical leaching and electrodeposition is optimized for precious metallic materials suitable in other markets and applications.

In parallel the document concentrates on how the dismantling can ensure the maximum overall recovery efficiency – therefore feeding back this information to the eco-design of the PEMFC cells. PEMFC stacks, cell package and membrane electrode assembly (MEAs) dismantling approach studies are ongoing via manual, mechanical and gaseous approach, with less than 1% losses in dismantling. This is a significant improvement to a current scenario where non-FC specific dismantling would account for a 15% loss of Pt from the EoL PEMFC entering the recycling process, which would result in an overall efficiency of the recycling of around 68%. This initial loss is untenable given the value of Pt.

The present deliverable “Lab scale optimization results on the 3 PEMFC recycling technologies report” describes the main steps to design and implement the recycling processes according to the final cell requirements in terms of purity and quality testing. The results are linked to the work done in the first year of the BEST4Hy project.

The processes below are applied and described as:

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[3] Mori, M.; Stropnik, R.; Sekavčnik, M.; Lotrič, A. Criticality and Life-Cycle Assessment of Materials Used in Fuel-Cell and Hydrogen Technologies. Sustainability 2021, 13, 3565. https://doi.org/10.3390/su13063565

[4] JRC, “Critical Raw Materials and the Circular Economy, Background Report”, 2017

1. HRD proposes a high purification process consisting of a series of steps in order to recover Pt as a solid $[NH_4]2PtCl_6$ salt, Ammonium-hexachloroplatinate. The first step consists of a leaching process. Literature data reports different leaching agents (H_2O_2/HCl and aqua regia HCl/HNO_3[5]). Aqua regia will be used as leachate. A filtration stage will be actuated to separate carbon powder by the hexachloroplatinate $PtCl_6^{-2}$. The final process consists of the precipitation of Pt with ammonium chloride/HCl solution in order to obtain the Pt salt.

HRD is optimizing each step at TRL3, to develop an efficient and selective method, the compositional and features of materials after cell operation is essential to identify the most suitable conditions: in terms of temperature, leaching pH, extraction and stripping conditions, to separate Pt as a solid $[NH_4]2PtCl_6$ salt, Ammonium- hexachloroplatinate used as precursor in the synthesis processes at CEA.

2. HRD proposes a novel way based on Alcohol Dissolution (AD) for the extraction and re-use of both the precious metals and the ionomer in PEMFC. The process consists of an AD process in a high temperature and pressure autoclave reactor. Conventional recovery PEMFCs technologies as HMT and High Temperature Treatment (HTHF, which is an incineration process) are able to recover Pt but no other valuable materials as ionomer membrane. However, the high content of fluoropolymer perfluorosulfonic acid (PFSA) generates emissions of fluorine and hydrofluoric acid processed in HTH. Hydrogen Fluoride (HF) gas is toxic to humans, animals, and plants. It is also highly corrosive.[6]

3. CEA proposes an electrochemical recovery route which is more environmentally friendly, it is performed under “soft” temperature conditions, and does not result in any gas emissions. It consists in the coupling of the electrochemical leaching of platinum in chloride-based ionic liquids (ILs), followed by its electrodeposition. Through the modulation of the composition of the ionic-liquid melts, it is possible to leach and electrodeposit the platinum from FC electrodes in a single-cell process under an inert or ambient atmosphere. ILs represent an alternative to aggressive and toxic aqueous solutions for the leaching of platinum-based catalysts. ILs are salts that melt below 100 °C and are usually composed of a large organic cation and an organic or inorganic anion. ILs are promising owing to their unique properties such as extremely low volatilities, relatively good electric conductivities,

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[5] L. Duclos, L. Svecova, V. Laforest, G.Mandil, P.-X. Thivel, “Process development and optimization for platinum recovery from PEM fuel cell catalyst”, Hydrometallurgy 160 (2016) 79–89

[6] Zuber et al, in Recycling of Precious Metals from Fuel Cell Components, Umicore AG&Co KG, Hanau, Germany

and wide electrochemical windows of usually more than 2 V[7], which could allow the electrochemical leaching of platinum by oxidation without the degradation of the ILs. The wide electrochemical window could also allow the recovery of metallic platinum by electrodeposition.

2.1 EoL different materials incoming

During the first year of BEST4Hy project, different stacks, and cell packages of different time of operation (EoL - fresh, 200h, 2800h, 7800h) were sent by partner EKPO to HRD. Delivered PEMFC packages (see Figure 1) were disassembled and underwent recovery processes.

Figure 1 The arrival of stacks and cells at Hensel Recycling GmbH

This first delivery was composed of 4 different products:

Material incoming	Material type	CCM Type	Operating Hours	Cells
NMG-207-10	stack	B	~200	10
NMG-366-240	stack	A	~200	240
MEA-A 271-200	Cell package (MEA+BBPs)	A	~2800	70
MEA-A	Cell package (MEA+BBPs)	A	0	10

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[7] H. Matsumoto, in Electrochemical Aspects of Ionic Liquids (Ed.: H. Ohno), John Wiley & Sons, Hoboken, 2005, pp. 35– 54

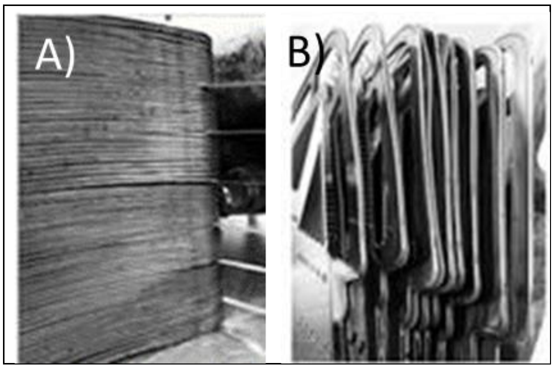


Figure 2 A) Detail of cells package with 70 cells and 2800 operating hours (MEA-A 271-200); B) Detail of cells package with 10 cells

In particular the material incoming was composed of 2 different MEAs: MEA-A and MEA-B with different membranes and Pt content.

The full-size stack NMG-366-240 was used for mobility applications, such as passenger cars, the short-stack NMG-207-10 was used to perform internal tests by EKPO. The cell package MEA-A 271-200 was used in smaller mobility applications, such as forklifts.

The second delivery was composed of two different materials after 7000 h of operation:

- MEA_61-80: 476,4 g of not damaged cells (20 cells of a full-stack)
- MEA_140-159: 475,8 g of cells with some damages (19 cells of a full-stack)

After unpacking the stacks and cell packages, the material follows the HRD internal processes:

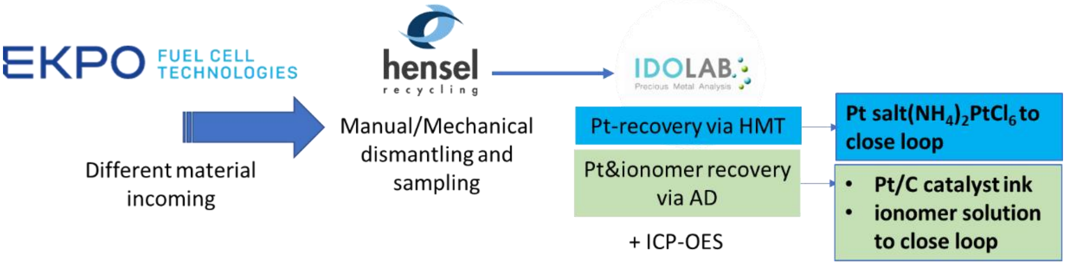


Figure 3 HRD internal processing of incoming material from EKPO

During the first year of BEST4Hy project, also different cell packages of different EoL (fresh, 200h, 2800h) were sent by EKPO to CEA; they were disassembled and underwent recovery processes:

- 21 Fresh MEAs
- 30 Aged MEAs (200h operation)
- 31 Aged MEAs (2800h operation)

The material received follows the CEA internal processes:

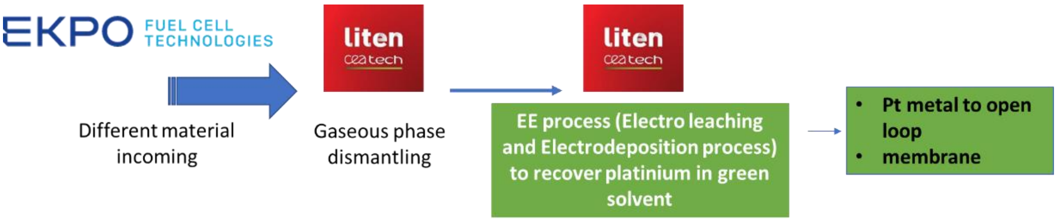


Figure 4 CEA internal processing of incoming material from EKPO

3 Disassembling

The goal of this stage of the project is to dismantle the different incoming material by EKPO: PEMFC stacks, cell packages, and MEAs in more convenient and sustainable way, avoiding the contamination of different components and elements herein contained. The disassembling procedures described below are conducted in order to reduce material (mainly Pt) losses, thus making subsequent recovery processes more efficient.

HRD decided to try a manual approach – considered the laboratory level of operation during the BEST4Hy project – but also ventured a mechanical solution in view of a future, industrial scalable scenario.

CEA proposes to make the dismantling operation by the gaseous approach to separate the MEAs (fresh, aging) to gas diffusion layer (GDL) and catalyst coated membrane (CCM). The recycling method will produce a low quantity of effluents (high gas recovery efficiency > 95%) and will be a fast process (<30min). The laboratory scale reactor is useful to provide proof of concept for MEA dismantling and to optimize process parameters. A laboratory autoclave is used for these experiments. Thanks to an initial pressurization, the solvents introduced into the reactor are evaporated and injected through the MEA to achieve membrane delamination. The influence of the type of solvent, flow rate, initial pressure value and temperature is studied.

3.1 Manual Disassembling

The manual disassembling is performed on the 4 first incoming materials.

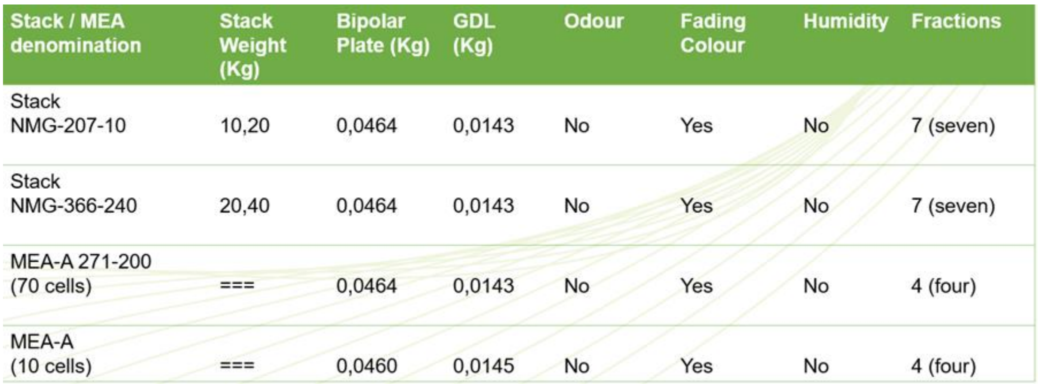
The PEMFC stack is manually disassembled via:

- Removal of tie rodes, cables and casing. The material is mainly made of high-grade steel.
- Removal of endplates
- Layers splitting & removal of sealants & MEAs

These are the 4 manual separation steps on the cell package:

1. Separation of the bipolar plate from the MEA manually
2. Cutting of rubber sealant by means of a scissor.
3. Manual separation of the CCM from the GDL
4. Cutting the CCM film into 2,5 cm x 4 cm pieces by means of a scissor.

A summary of results can be found in the table hereafter, showing respectively 4[8] and 7[9] fractions:



Stack / MEA denomination	Stack Weight (Kg)	Bipolar Plate (Kg)	GDL (Kg)	Odour	Fading Colour	Humidity	Fractions
Stack NMG-207-10	10,20	0,0464	0,0143	No	Yes	No	7 (seven)
Stack NMG-366-240	20,40	0,0464	0,0143	No	Yes	No	7 (seven)
MEA-A 271-200 (70 cells)	===	0,0464	0,0143	No	Yes	No	4 (four)
MEA-A (10 cells)	===	0,0460	0,0145	No	Yes	No	4 (four)

Table 1 Stacks and MEAs disassembling details

The manual disassembling of the 2 stacks by using non-electrical tools was respectively:

NMG-207-10 (total mass 10,2 kg)	15 minutes
NMG-366-240 (total mass 20,4 kg)	25 minutes

3.2 Mechanical disassembling

After the manual disassembling, different trials of mechanical disassembling took place. The main reason for the mechanical disassembling is in connection with HRD wish to envision an industrially scalable method to dismantle stacks and MEAs taking industrial volumes into account. The 5 trials below are performed and optimized.

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[8] 1) Bipolar Plates, 2) rubber sealants, 3) carbon fiber, 4) MEA.

[9] 1) Bipolar Plates, 2) rubber sealants, 3) carbon fiber, 4) MEA, 5) plastic plates, 6) metallic plates with screws (magnetic steel), 7) metallic plates with screws (non-magnetic iron).

3.2.1 Mechanical Trial 1 (Rotating Knives Shredder)

The first mechanical trial uses a rotating knives shredder named “ZM Granulator” (7,5 kW). The shredding of the MEAs along with bipolar plates took 7 minute-time (0,600 Kg, 10 pieces), a considerable saving with respect to manual disassembly. After the shredding through a screen with Ø25mm diameter, the total mass of the mixed-up fractions is 0,596 kg.

A drawing of the equipment used is attached hereunder:

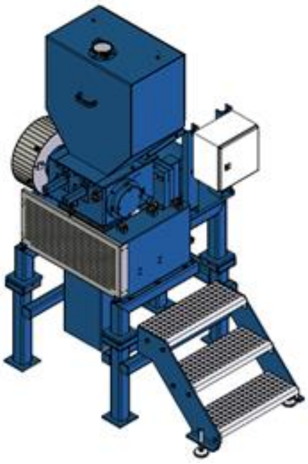


Figure 5 The ZM Granulator

The result of this first mechanical attempt of disassembling is reproduced below in figure 5; ideally, the output of the process should be a separation of the granulated material, as represented below in figure 6 which shows the materials separated manually by hand:

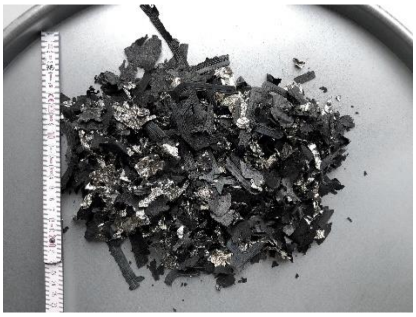


Figure 6 Mixed up fractions after shredding operations

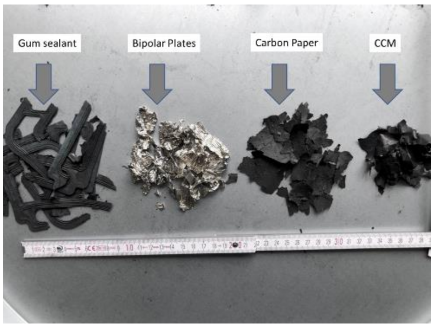


Figure 7 Manually separated fractions of the mass obtained retrospectively with the ZM Granulator shredding experiment

The **recovered product was not satisfactory**; while the different parts could be perfectly separated manually, the recovered fractions stick together **jeopardising the purity of the ink obtained in the recycling process**. Furthermore, the contamination of all other parts with Pt (2-2,5 % Pt in bipolar plates, 0,1 %Pt in sealants) was present.

3.2.2 Mechanical Trial 2 (Erdwich Equipment)

The second trial is performed using a screen with Ø20mm diameter; the shredding equipment is in Figure 8. This mechanical process follows a manual disassembling of the bipolar plates. The result of this operation is shown hereafter in figure 8 and will be evaluated with a final table at the end of this deliverable.

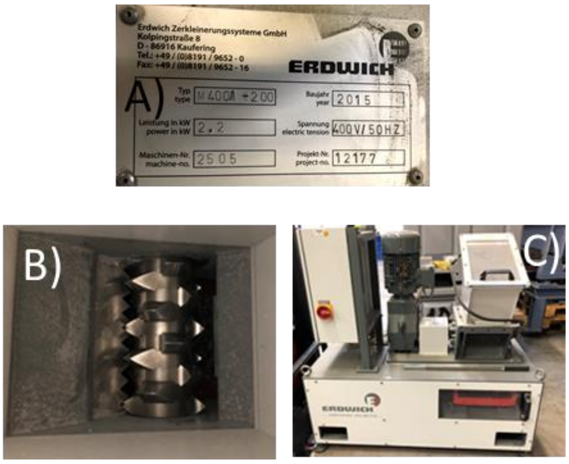


Figure 8 A) Label on the equipment; B) Details of the knives; C) The Erdwich Shredder

Two separated fractions resulted from this operation:

- Whole bipolar plates manually separated from the MEAs
- Mix of rubber sealant, GDL and MEAs

Figure 9 Heterogeneous mix of GDL and MEAs after the Erdwich shredding

While this mixture is certainly more adequate than the first one, since it does not contain any metallic parts, being the BPP already removed, it still presents the criticality of the sealant which is shredded with the MEAs and GDL, as can be seen from the picture attached (fig. 9)

3.2.3 Mechanical Trial 3 (Polar Mohr Equipment)

A third mechanical trial to separate the MEA both from sealants and carbon paper is followed, after having removed the bipolar plates manually.

This operation took place by means of a guillotine system to get CCM (delamination) and proved to be more efficient than the two previous ones. The manufacturer is Polar Mohr and the nominal power of the system is 2,4 kW. The overall cutting time to remove all sealant from 3 pieces of GDL was 3 min.

During these mechanical trials, more MEAs were stapled together (3 pieces, one on top of another) without manual separation of the layers (anode, membrane, and cathode), but rather cutting everything with the guillotine. It was observed that the thicker the staple is, the less precise the cutting edge, also considering that EKPO’s gaskets are made of thermoplastic and tend to stick to one another.

Following some images of the equipment with which the third trial was performed, along with some cells and cut gaskets details:

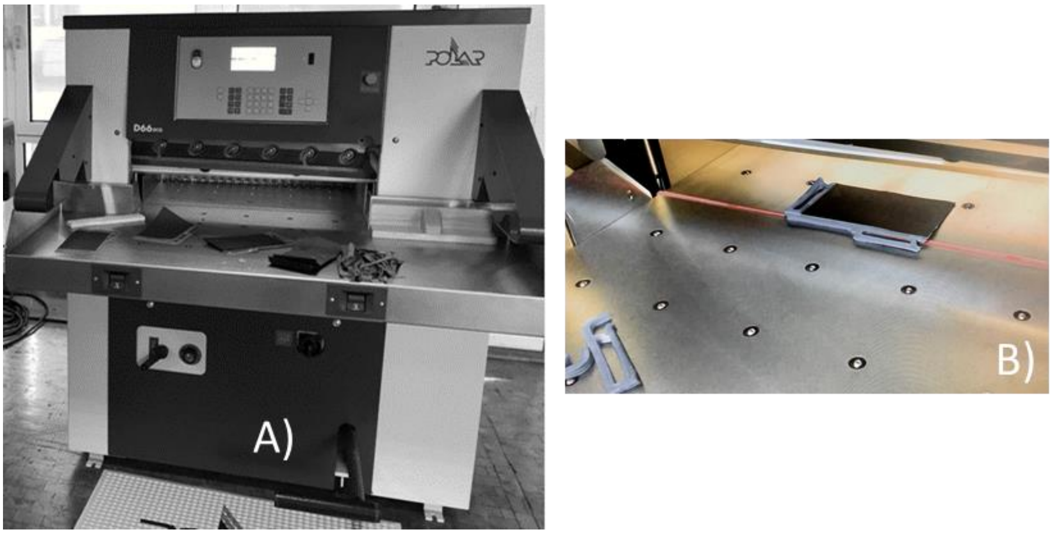


Figure 10 A) the Polar equipment used to dismantle MEA units; B) Details of MEA (GDL and gaskets). Disassembled gaskets in the lower left corner

3.2.4 Mechanical Trial 4 (Paper Shredder)

After having removed the sealants with the previous method, the whole cell unit was processed by means of a standard office paper shredder for documents disposal. The result was a clean fraction of CCM and carbon paper, with visually little or no loss of material.

The challenge here is to separate the two GDLs (carbon paper material) from the CCM. This was tried first with a vibration-sieve method and added water using the Retsch Type UR1 equipment (230V 50/60Hz) where the material is treated for 10 minutes (see figures 12 from A to E page 15), as well as using a vibrating sieve without water using the Retsch Type AS200 basic B equipment (230V50/60 Hz), again for exactly 10 minutes.

Figure 11 Mix of the 3 recovered fractions

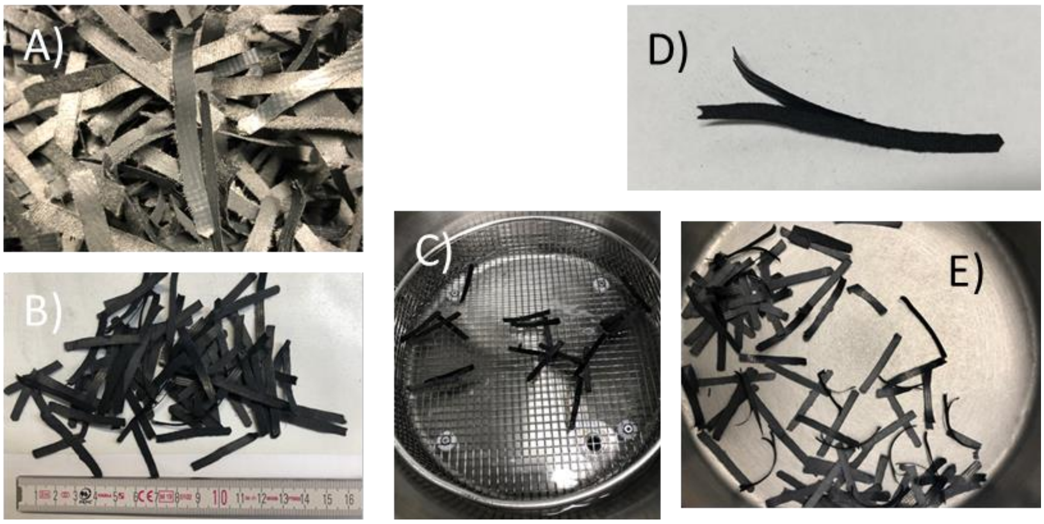


Figure 12 A) Detail of the 3 fractions- MEA structure is visible; B) Detail of the mix with fractions' dimensions; C) Detail of one single "strip" with 2 GDL layers; D) Detail of sieve and floating strips plate; E) Detail of strips inside a vibrator

Despite the a.m. actions, a definitive separation of the CCM from the GDL was only possible manually. Due to this reason, HRD went for a further disassembling trial, which is described hereafter.

3.2.5 Hybrid Trial 5 (manual disassembling of BPP and rubber sealants and mechanical shredding of CCM)

A fifth test to disassemble the MEAs is undergoing at HRD.

This fifth and last attempt is a hybrid summing up the most efficient aspects of manual and mechanical disassembling.

A short description hereunder:

- The BPP will be disassembled manually
- Shortly after the rubber sealings will be cut off by means of a professional lever cutting machine.
- Subsequently the GDLs will be "stripped off" manually
- Finally, the remaining CCM will be shredded with a regular paper shredder.

The steps and results of this last test will be in the next deliverable, giving account of the number of cells tested and the results obtained.

3.3 MEA Gaseous Phase Dismantling

The goal of this method is to develop **novel EoL technologies** based on vapor phase and separate the different components of MEA (GDL and CCM)

The initial process is based on moderate vacuum (~5000 Pa). Preliminary tests are conducted using an existing test bench in CEA's lab. The concept consists in vaporizing and fully saturating a stirred tank initially under vacuum by injecting a preheated solvent.

An efficient vaporization has been obtained with ethanol, but due to its bad wettability with GDL, no dismantling was observed.

The new process is based on high pressure to force the penetration of the solvent through the GDL to reach the membrane, promoting the swelling to complete the MEA's dismantling.

This new concept, based on high pressure conditions, can be carried out under both liquid and vapor phases. (See Figure below).

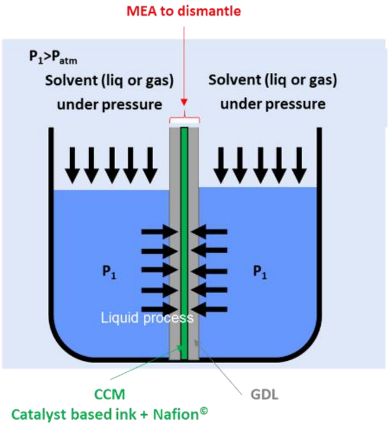


Figure 13 High pressure-based process

<u>In 1st tests, the MEA dismantling</u> has been conducted in liquid phase with MEAs provided by CEA (MEAs assembled at hot pressure). These first tests are useful to optimize the processes by not using directly the BEST4Hy incoming material.

Different solvents have been tested:

- Pure ethanol
- Mix water/ethanol (50/50)
- Mix ethanol/acetone (70/30)
- Pure isopropyl alcohol
- Pure acetone
- Pure water

Dismantling has been only obtained by considering ethanol or isopropyl alcohol-based solvent. During process, the major part of catalyst is delaminated from the membrane and is brought into the solvent. The remaining catalyst will be recovered during the post-processing.

Filtration can be performed on solvent (after liquid dismantling and post-processing) to recover catalyst.

Figure 14 shows membranes once tests have been performed.

			
Water/Ethanol 50%/50% vol	**Ethanol/Acetone 70%/30% vol**	**Pure Isopropyl alcohol**	**Pure Ethanol**

Figure 14 Membranes after dismantling performed with high pressure-based process

The final membrane aspect directly depends on the solvent composition. Only pure ethanol seems to enable a complete separation of the membranes.

Tests will be reproduced on different sources of MEA's to check the final visual aspect.

In post processing, the 2 post-processes have been evaluated:

- Rinsing in water
- Treating in ultrasonic bath

Experiments have been performed on CEA's MEAs (Hot pressure-based process) with pure ethanol. Figure below summarizes the results.

				
	High Pressure process	✓	✓	✗
Post-Process	**US Bath**	✗	✓	✓
	Rinsing	✓	✗	✓

Figure 15 Post Process efficiency studied on membrane from CEA's MEAs and dismantled with high pressure process performed with ethanol

A high-pressure process is required to disassemble and clean the membrane properly. The ultrasonic bath-based post process appears to be more effective in removing the catalyst from the membrane.

Similar tests have been performed on EKPO's MEAs received (Figure 16)

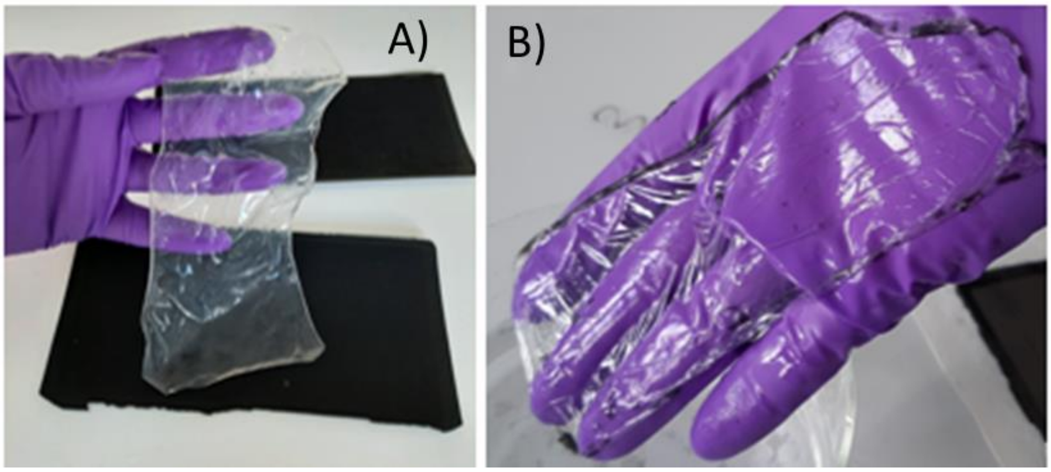


Figure 16 GDL and membranes dismantled thanks to high pressure process. On the left, MEA provided by CEA, on the right, MEA from EKPO.

The different pictures show that the high-pressure process supplemented by an ultra-sonic post-process leads to interesting results for both MEAs: CEA and EKPO.

The high-pressure process in vapor phase is also studied using a complete stack of MEAs. The ethanol is used as solvent and the vapor is injected through inlets of bipolar plates. After dismantling, catalyst ink remains stuck on the membrane. This way of dismantling enables an easier treatment of CCM to recover catalyst and ionomer during hydrometallurgical process

Main findings and results:

- MEA recovery after vapor phase exposure.
- Very easy GDL and CCM disassembling
- Very clean aspect for GDL and membrane
- Membrane exposition to ethanol seems more homogeneous and smoother with vapor compared to liquid exposition
- Catalyst ink recovery can be easier using vapor phase process.

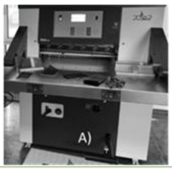
Disassembly	Results	Comments
Manual Dismantling	☺ 	Ideal sampling. though not scalable
Mechanical Trial 1 (Rotating Knives Shredder)	☹ 	Contamination inhomogeneous reactive in acid
Mechanical Trial 2 (Erdwich Equipment)	😐 	Contamination inhomogeneous
Mechanical Trial 3 (Polar Mohr Equipment)	😐 	Inefficient disassembling process
Mechanical Trial 4 (Paper Shredder)	😐 	Seem to be promising, but GDL and CCM are difficult to separate
Hybrid Trial 5 (manual dismantling of BPP and rubber sealants and mechanical shredding of CCM)	Not yet performed	Not yet performed, seem to be promising
MEA Gaseous Phase Dismantling	☺ 	Good results on GDL and membranes dismantled thanks to high pressure process

Table 2 Dismantling results: Comparison between Manual and Mechanical Trials for Hydrometallurgical Treatment at HRD; Gaseous phase dismantling at CEA.

4 Hydrometallurgical process to recover Pt-salt

The first year of the project is based on the optimization at TRL3 of reliable, cost-efficient and overall efficient operating procedures, varying different parameters to obtain the best recovery: i) the %vol oxidants influence the leaching and the separation efficiency, ii) time of the processes and iii) optimization of Pt separation. A quality analysis on Pt solution with Inductively Coupled Plasma Optical Emission (ICP-OES) is also conducted, useful to detect the grades of various streams, for the construction of mass balances: evaluation of leaching and filtration and evaluation of the efficiency of resins with inlet/outlet analysis.

The goal of this task is to recover Pt using a known and tested technology (existing EoL).

After the subs-sequences of the manual/mechanical dismantling, the original seven layers will be split into the GDL (two layers), the sealing (which needs to be cut off) and the CCM/electrodes.

The next step is the MEA or CCM dissolution using solvent, in BEST4Hy case aqua regia. HRD used a high purification process consisting of a series of steps to recover Pt as a solid $[NH_4]_2PtCl_6$ salt, Ammonium-hexachloroplatinate.

The Figure 17 below shows the workflow of the laboratory scale platinum recovery process at HRD. The filtrate/sewage contains liquid waste.

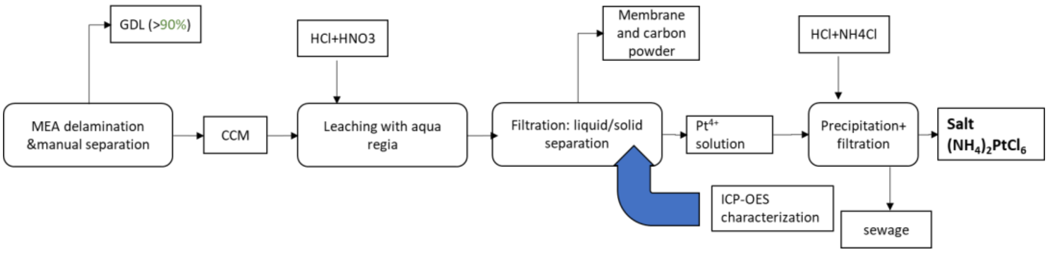


Figure 17 HMT process at IDO-lab

This is a short description of the HMT process adopted by IDO-Lab:

1. **Leaching.** The leaching agent is a mixture of a strong acid and an oxidant. Aqua regia is typically used for the leaching of spent catalysts in the recovery of PGMs (on carbon support). It is a solution of hydrochloric acid and nitric acid with a molar ratio of 3:1, and its use as a leaching agent allows Pt-recovery percentages above 95%. The leaching process generates very harsh working conditions due to high acid concentration with pH<1. Although different acid/oxidant solutions are possible, aqua regia is still used in most cases due to its high recovery efficiency.
   Leaching process:
   - Initial weight per batch of 10-20 g of CCM foil or MEA after being disassembled in 1 beaker glass.
   - The content of the beaker glass is dissolved in aqua regia to obtain 2 fractions:
     - Pt in liquid phase and foil containing membrane and carbon.
   - Leaching with HCl (36% conc., 200mL) and HNO_3 (63% conc., 50mL).
   - 1h heating and stirring (nominal power of heating plate: 630W), afterward 1h stirring.
2. **Filtration**. After leaching, a filtration stage removes the membrane (ionomer) and carbon particles from the PGM-containing solution.
   Filtration steps:
   - The liquid is filtrated to obtain a clear Pt solution into a volumetric flask using blue ribbon filter paper.
   - The Pt concentration has been quantified by ICP-OES to determine the concentration of Platinum (ICP nominal power: 1300W, time: 0,5 h; little consumption of Argon gas, Scandium, buffer (NaCl).

    - Pt concentration dissolved out of CCM after leaching process with aqua regia: 90-95%

3. **Precipitation + Filtration.** In this case, the Pt-rich stream coming from the separation process is treated with e.g., ammonium chloride (NH_4Cl) to precipitate platinum as $(NH_4)_2PtCl_6$, which is filtered and recovered in solid form as the final product of the process.

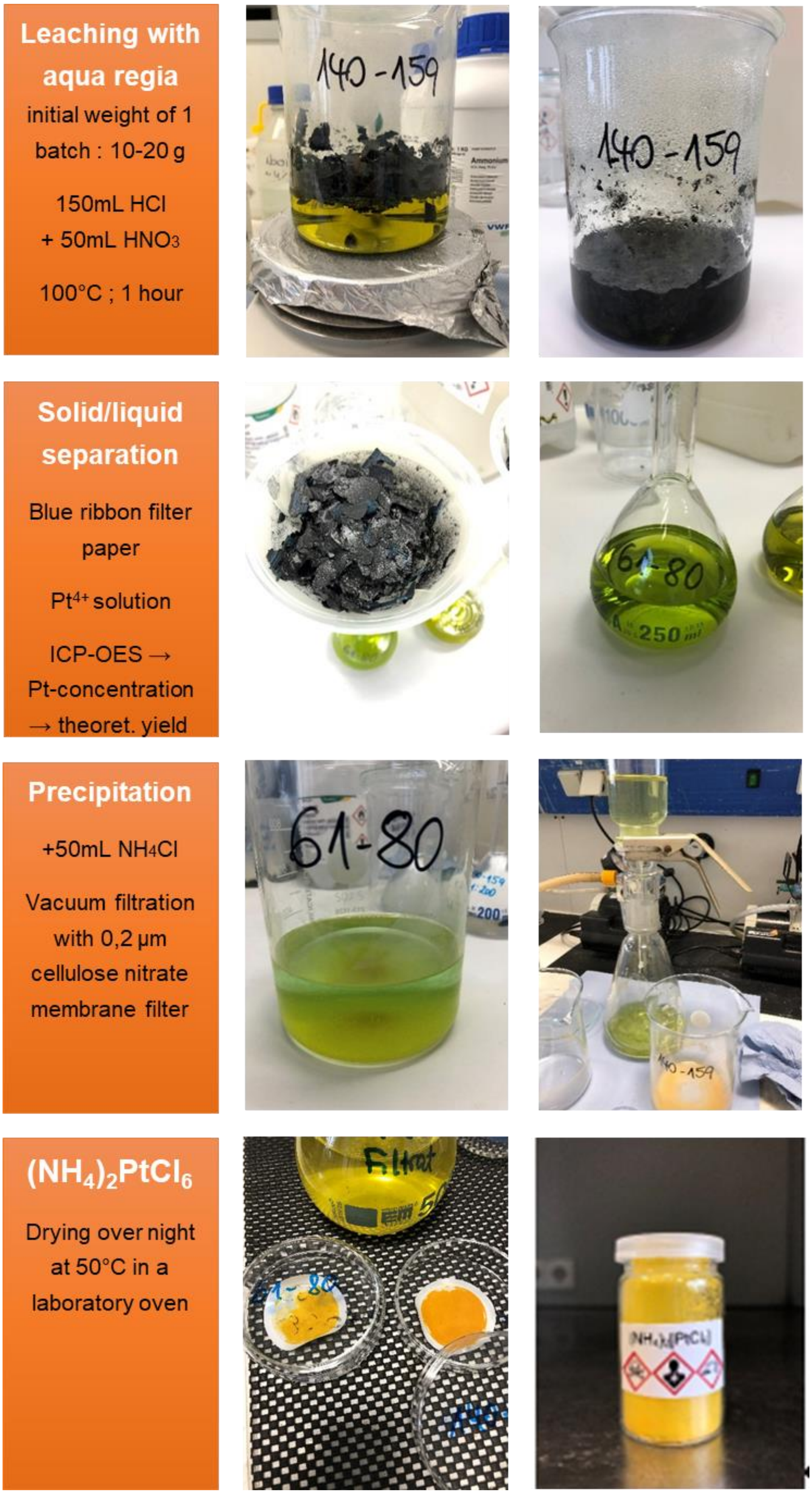
   Precipitation and filtration steps:

    - At last, the concentration is precipitated with ammonia chloride: NH_4Cl (saturated solution 250g/L) 20mL using a piston pipette
    - As a result, Pt salt is obtained from the saturated solution (diammonium hexachloride)

○ vacuum filtration using cellulose filter paper 2µm to obtain 2 fractions in the end: Pt as Pt salt in solid form and the filtrate (waste solution)

    - Pt-salt is dried in the oven before final weighing: drying Pt-salt $(NH_4)_2PtCl_6$ in drying cabinet/furnace (nominal power:1400W, drying time: 24h at 50°C)

Figure 18 reports each step description.



Leaching with aqua regia

initial weight of 1 batch : 10-20 g

150mL HCl
+ 50mL HNO_3

100°C ; 1 hour

Solid/liquid separation

Blue ribbon filter paper

Pt^{4+} solution

ICP-OES → Pt-concentration → theoret. yield

Precipitation

+50mL NH_4Cl

Vacuum filtration with 0,2 µm cellulose nitrate membrane filter

$(NH_4)_2PtCl_6$

Drying over night at 50°C in a laboratory oven

Figure 18 Laboratory scale of Hydrometallurgical process to recover Pt as Pt-salt

The figure below reports the Pt salts recovered via manual dismantling of the incoming material and the CCM HMT. The salts are sent to CEA for the characterization under WP2: ~1,5 g of fresh MEA and ~ 4,9 g of aged MEA (table below).

Figure 19 Pt recovery of first EKPO delivery after manually disassembling (only CCM)

Table 3 reports for each material treated the HMT process conditions and the final Pt yield %. In particular: Pt salt from NMG-366-240 presents iron contamination and the Pt salt obtained by fresh MEA is contaminated by filter paper (light brown). The Pt from NMG-207-10 presents a final % yield of 100. This recovery % is explainable with the contamination of other components, e.g. nickel (see the light green colour). This batch was rinsed insufficiently after precipitation. Further investigations of the platinum rich solution will show co-components and possible contaminations more precisely (see section 4.2).

Stack	Cell type	operating hours	initial weight	Leaching	Filtration	Pt-conc. in solution	Precip.	Vac. Filtr.	Pt salt	yield	Remarks
NMG-207-10	B	200	10,053 g	130 mL	Blue ribbon	6,36%	50 mL	0,2 µm	1,453 g	100%	any contamination?
NMG-366-240	A	200	11,133 g	130 mL	Blue ribbon	~9,00%	50 mL	0,2 µm	2,014 g	88%	iron contamination
MEA-A 271-200	A	2800	10,634 g	200 mL	Blue ribbon	8,59%	50 mL	0,2 µm	1,456 g	70%	
MEA-A	A	0	10,000 g	200 mL	Blue ribbon	9,23%	50 mL	0,2 µm	1,419 g	68%	

Table 3 Batch 1 Pt salts from different MEAs sent to CEA

Preliminary characterization results (linked to WP2 and reported in D2.1). With regards to batch 1, Co, Fe and Cr have been detected in the salt MEA-A NMG-207-10 (200 hours of operation), whereas the TGA spectrum shows a peak at 380°C, corresponding to the theoretical degradation temperature. But no Fe has been detected into the salt derived from MEA-A NMG-366-240, whereas it seems to be contaminated by Fe from tweezers (with red colour) and the TGA spectrum really differs with a degradation peak higher than 380°C. The Fe quantity must be under the threshold detection of the XRF.

Figure 20 and Table 4 report the second Pt salts' batch. The Pt salt from 2800h aged MEA presents the crystallization of the ammonia chloride after precipitation. Fe and Co have been detected in the unwashed salt, confirming the impure Pt salt.

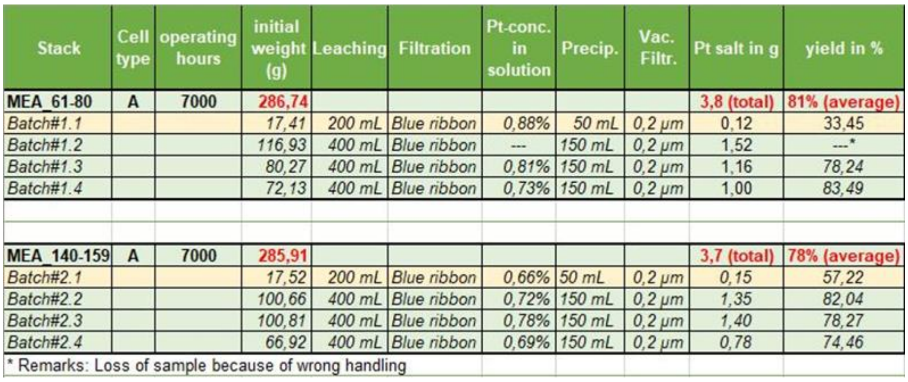
Figure 20 Batch 2 of Pt recovery

Stack	Cell type	operating hours	initial weight	Leaching	Filtration	Pt-conc. in	Precip.	Vac. Filtr.	Pt salt	yield	Remarks
MEA-A 271-200	A	2800	22,274 g	400 mL	Blue ribbon	9,16%	50 mL	0,2 µm	5,317 g	115%	voluminous sample, crystallization
MEA-A (Sample A)	A	0	15,154 g	200 mL	Blue ribbon	9,20%	50 mL	0,2 µm	1,419 g	45%	loss after filtration
MEA-A (Sample B)	A	0	18,335 g	200 mL	Blue ribbon	9,05%	70 mL	0,2 µm	2,574 g	68%	

Table 4 Batch 2 Pt salts from different MEAs sent to CEA

Figure 21 below reports the mechanical trial 2 performed on 7000h aged material. Table 5 reports the third batch of material that will be characterized in WP2.

Figure 21 MEA_61-80 and MEA_140-159 after Mechanical Trial 2 (Erdwich Equipment)



Stack	Cell type	operating hours	initial weight (g)	Leaching	Filtration	Pt-conc. in solution	Precip.	Vac. Filtr.	Pt salt in g	yield in %
MEA_61-80	**A**	**7000**	**286,74**						**3,8 (total)**	**81% (average)**
Batch#1.1			*17,41*	*200 mL*	*Blue ribbon*	*0,88%*	*50 mL*	*0,2 µm*	0,12	33,45
Batch#1.2			*116,93*	*400 mL*	*Blue ribbon*	---	*150 mL*	*0,2 µm*	1,52	---*
Batch#1.3			*80,27*	*400 mL*	*Blue ribbon*	*0,81%*	*150 mL*	*0,2 µm*	1,16	*78,24*
Batch#1.4			*72,13*	*400 mL*	*Blue ribbon*	*0,73%*	*150 mL*	*0,2 µm*	1,00	*83,49*
MEA_140-159	**A**	**7000**	**285,91**						**3,7 (total)**	**78% (average)**
Batch#2.1			*17,52*	*200 mL*	*Blue ribbon*	*0,66%*	*50 mL*	*0,2 µm*	*0,15*	*57,22*
Batch#2.2			*100,66*	*400 mL*	*Blue ribbon*	*0,72%*	*150 mL*	*0,2 µm*	*1,35*	*82,04*
Batch#2.3			*100,81*	*400 mL*	*Blue ribbon*	*0,78%*	*150 mL*	*0,2 µm*	*1,40*	*78,27*
Batch#2.4			*66,92*	*400 mL*	*Blue ribbon*	*0,69%*	*150 mL*	*0,2 µm*	*0,78*	*74,46*

* Remarks: Loss of sample because of wrong handling

Table 5 Batch 3 Pt salts from different MEAs sent to CEA

Both MEA types show similar overall yields of ~80% after the laboratory process standardization to recover Platinum as Pt-salt (details in section 4.1). Batch #1.1 and batch #2.1 are not representative (before lab optimization). The large differences in yield indicate that tests with a weight of minimum 66 g and more give better results. Logically, in the handover of the sample, the steps of precipitation and transferring into the glass are essential factors. A loss of material in a larger sample is less significant than in a small sample (see Table 6 Process optimization).

Latest experiments are very promising, and these good yields need to be confirmed in further trials (Samples coming from Mechanical Trial #5).

4.1 Laboratory optimization

HRD proved capable of producing a sound amount of Pt salt, which optically resembles the characteristics they expected it to have. WP1 goal is to increase the yield in the precipitation “middle step”. In fact, during the precipitation process, the yield may vary, depending on the impregnation of the MEA. This is because many parameters should be optimized – for instance the pH value, the precipitation reagent, the addition of acid, temperature and so on. Activities are continuously ongoing to optimize these parameters and increase the % of recovered material.

Table below reports the main optimization that are linked to batch 3 Pt salts. In particular the batch size, the filtration choice and the mesh size of membrane filter paper during the vacuum filtration.

Table 6 Process optimization

Main issues during laboratory optimization	before	after
Batch size before: max. 5 g in flask after: 50-100 g in beaker glass		
Filtration: choice of filter paper	white ribbon	blue ribbon
Choice of Filtration technique: before: Buchner funnel after: Vacuum Filtration		
Vacuum Filtration: mesh size of membrane filter paper	0,45µm	 0,2µm

Other parameters ongoing to be optimized and more related to the leaching process and precipitation steps: i) volume of aqua regia linked to the batch size during the leaching, ii) implementation of a resin/extraction step, iii) pH and iv) Temperature of the solution during the precipitation.

4.2 Characterization via ICP-OES

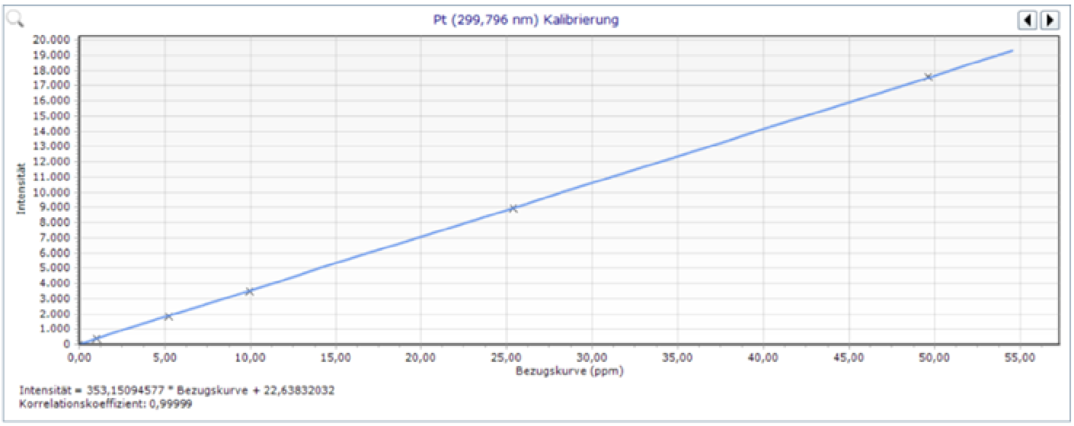
After Leaching process, the Pt-concentration of the solution must be determined to calculate the theoretical yield of the Pt-salt (Diammonium hexachloroplatinate). HRD is able to determine precious metals Pt (Pt, other metals such as Ir, Pd) in solution with Agilent ICP-OES 5100 and PANalytical AxiosMax.

The analysis is done on the CCM's Pt-loading, in order to quantitatively and qualitatively determinate the mg/cm2 of platinum in the CCM from EKPO.

The result on MEA cell type A demonstrated a Pt loading of 0,54mg/cm2. The results are compared with EKPO and the XRF analysis from CEA performed on fresh and aged CCM from EKPO (D2.1).

The ICP-OES analysis is done on all the Pt salts obtained and allows to optimize the processes of separation of the Pt (% of yield reported in Table 3-4-5).

Figure 22: Pt rich solution and 1:100 dissolution ready for ICP-OES



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Korrelationskoeffizient: 0,99999

Figure 23: 5-point calibration of Pt 299,796 nm

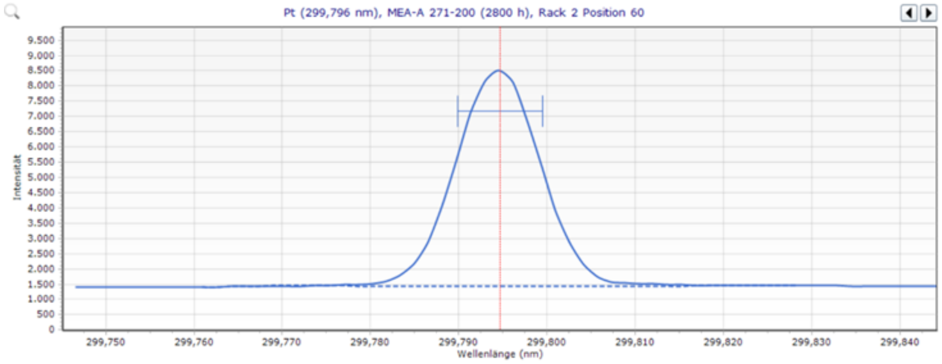

Figure 24: Pt-peak of MEA-A 271-200 (2800h)

Further investigations on other elements in the solution are also useful and ongoing. In particular cobalt can be detected qualitatively by ICP-OES, e.g., on wavelength 350 nm, 228 nm and is performed on MEA_61-80 material.

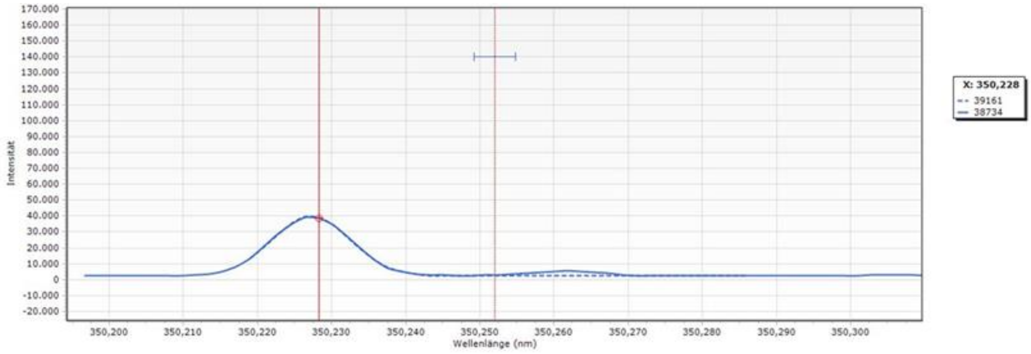

Figure 25: Co in MEA_61-80

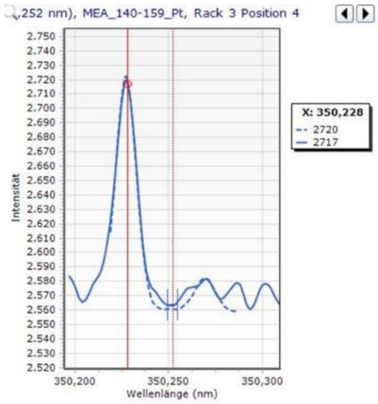

Figure 26: Co in MEA_140-159

Conclusion of this investigation:

- Pt rich solution also contains Cobalt
- Co needs to be quantified
- Other crucial elements? Mn, Fe, Ni, Cr, Mo
- ICP-OES analysis with multi element standard

5 Electroleaching and electrodeposition to recover metallic Pt

5.1 Concept of the process

The aim of this task is to develop an environmentally friendly route to recover platinum from old PEMFC stacks. The process of electro lixiviation/deposition is based on a patented process proposed by CEA institute (EP3263744 A1 2018-01-03) and enables the recovery of platinum without the use of organic solvents nor acids, is free from toxic gas emissions and is convenient as it operates in only one step. The proof of concept is demonstrated in a three-electrode cell as presented below.

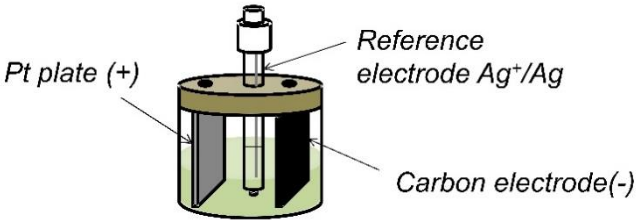

Figure 27 Scheme of the three-electrode cell

In this process, platinum can be recovered from electrochemical leaching in an ionic liquid mixture (IL) containing chloride ions. The ionic liquids selected for the process are 1-Butyl-3-methylimidazolium bis trifluoromethyl sulfonyl imide (BMIM TFSI) and 1-Butyl-3-methylimidazolium chloride (BMIM Cl) which respective molecular structures are presented on Figure 28. The Pt nanoparticles are electrochemically leached at the anode, forming platinum and chloride complexes, and zero-valent platinum is electrochemically deposited at the cathode of the same electrochemical cell.

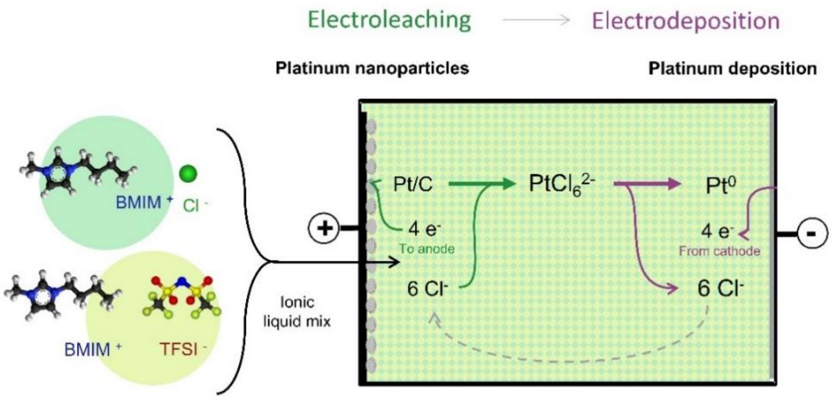

Figure 28 Scheme of simplified electrochemical leaching and deposition process operating mode

5.2 Material and methods

5.2.1 Development of a new electrochemical cell

Several parameters are prevalent for the process operation. The concentration of chloride anions, the concentration of leached platinum in the medium and operating potential are optimized on the three-electrode cell. To improve the process, including the electro lixiviation and deposition of Pt/C coming from MEA material, an electrochemical reactor was developed, with higher electrode surface area, smaller inter-electrode distance, and temperature controlled. (Figure 29). This new vessel should enable the electro lixiviation and deposition of Pt from MEAs, and the next step of our work concerns the optimization of the parameters such as chloride and platinum ions concentration and cathode applied potential.

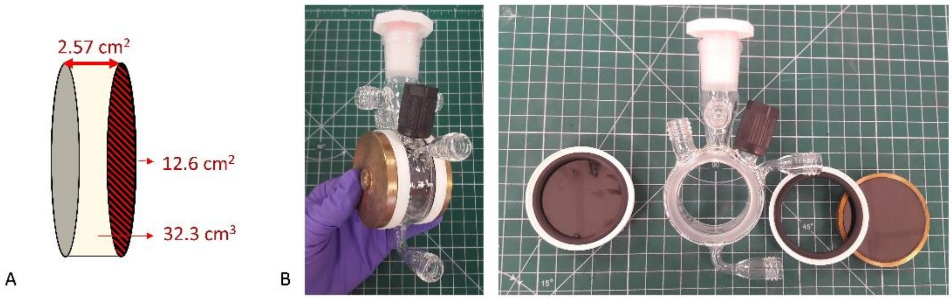

Figure 29 Dimensions of the improved electrochemical vessel (A) and pictures of the vessel (B)

5.2.2 Pt quantification

In order to assess the process efficiency, all forms of platinum need to be quantified. The remaining Pt quantity at the anode side will be assessed by gravimetric analyses, ICP-OES and by SEM-EDX imagery. The same analyses can be applied to assess Pt deposition at the cathode. The concentration of Pt ions in the ionic liquids medium will be measured by atomic absorbance spectroscopy (AAS) and UV-vis spectrophotometry.

The process efficiency will be enabled by comparing the quantities of Pt electrochemically leached and deposited. The values will be compared with initial material specifications provided by EKPO.

5.3 Work perspectives for the development of the electro lixiviation/deposition process on PEMFC material

There are mainly two types of membrane electrode assembly (MEA): the catalyst coated membranes (CCM) where the catalyst layer (CL) is coated on the membrane, and catalyst coated substrates (CCS), for which the CL is coated on the gas diffusion layer (GDL), forming a gas diffusion electrode (GDE). Depending on the type of MEA, the application of the electrochemical lixiviation/deposition process will require several development steps.

Indeed, the proof of concept was developed on GDE as the carbon paper conducts electrons and could directly be used as the anode. However, an anode should be developed to enable the process operation. For this aspect, stainless materials could be used to enable the lixiviation of platinum. To ensure a large coverage of the CCM surface, a mesh material will probably be used.

The previous dismantling step also affects the operating mode of the electrochemical Pt recovery process. Indeed, according to the dismantling process, the Pt nanoparticles (NP) are recovered in very distinct ways: if liquid-phase dismantling is applied, the Pt NP are in suspension in ethanol or organic solvent solution without any solid support. However, if the vapour-phase dismantling process is applied, Pt/C NP are recovered on the membrane and the CCM is almost intact. In that case, a special anode will be developed to enable the electrochemical leaching of Pt NP.

As a conclusion, the next steps of the electrochemical Pt recovery process are first the parameters optimisation on the improved electrochemical vessel on Pt material, and on GDE material. Then, an innovative anode system that enables the lixiviation of Pt from CCM will be developed. The system parameters will be optimized related to the specific CCM-adapted anode. CCM represent the major MEA assembly type, relatively to catalyst-coated substrates like GDE. Therefore, the system developed to treat CCM will be implemented at a pilot scale to handle higher amounts of MEA material simultaneously.

6 Ionomer recovery by alcohol dissolution process

In this phase of the project a very important milestone is the development of the ionomer recovery technique by alcohol dissolution (AD); the extreme heterogeneity of the ionomer, with many different producers and a variety of products makes it more difficult to identify the best process methodology.

Figure 30 reports the long side chain of Nafion and the short side chain of Aquivion (Solvay).

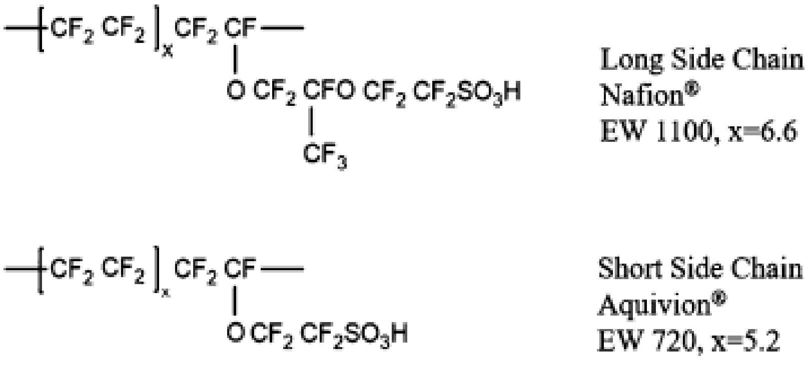

Figure 30 Nafion and Aquivion perfluorinated sulfonic acid ionomer structures[10]

Furthermore, it is also necessary to take in consideration the complex degradation issue of the polymer during PEMFC stack operation, effects linked to the ambient conditions that generate mechanical degradation (due to relative humidity cycling, damaging during MEA manufacturing) and chemical degradation (due to low relative humidity, high temperature and high cell voltage). Membrane can be contaminated also by Ionic Species that come from metal bipolar plates, humidifiers, tubing materials, air, dissolution and repositioning of Pt catalyst (Pt band inside of ionomer membrane). The sulphonate sites have a stronger affinity to metal ions, except Li+, than for protons, leading to exchange of the protons by the metal ion when present and this effect can result in partial drying out of membrane, thus, also reducing the proton conductivity. Bruijn et al. studied that during fuel cell operation, the Nafion crystallinity changes, especially at relatively dry conditions. It is

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[10] Performance Comparison of Proton Exchange Membrane Fuel Cells with Nafion and Aquivion Perfluorosulfonic Acids with Different Equivalent Weights as the Electrode Binders, Ting Li, Jiabin Shen, Guangying Chen, Shaoyun Guo, and Guangyou Xie; ACS Omega 2020, 5, 28, 17628-17636.

believed that a high crystallinity corresponds to open ion-channels, and dehydrated, collapsed channels correspond to a decrease in crystallinity[11].

The ionomer recycled, delivered in solution, will be evaluated by CEA by incorporating it in a catalytic ink using reference commercial catalyst to manufacture a MEA. The comparison of the in-situ performance on a single cell between this MEA and a reference MEA manufactured with commercial ionomer will permit to characterise the impact of the recycled material.

The viscosity of the ionomer solution could be measured with a rheometer. After that the ionomer will be tested incorporating commercial catalyst at MEA level.

The involvement of Solvay (as internal Advisory Board member) will help the consortium to determine the re-usability of the recovered material and add some meaningful information (degradation, contamination, lower quality). In particular, Solvay will contribute the degradation analysis: the degradation of the ionomer chain via backbone rupture can be detected via gel permeation chromatography (GPC) and the C-HSO3 cleavage via Solid-state NMR spectroscopy and MAS and/or titration.

Preliminary tests were performed with a mixture of methanol and water (90/10) by reflux dissolution at ~50°C. The mixture was added and heated smoothly to avoid evaporating. Finally, Pt/C ink and ionomer were solved in solution. A separating process via filtration was performed but with no significant separation between the ink and the ionomer.

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[11] Review: Durability and Degradation Issues of PEM Fuel Cell Components; F. A. de Bruijn, V. A. T. Dam and G. J. M. Janssen, G.J.M. Fuel Cells 2008, 3-22.

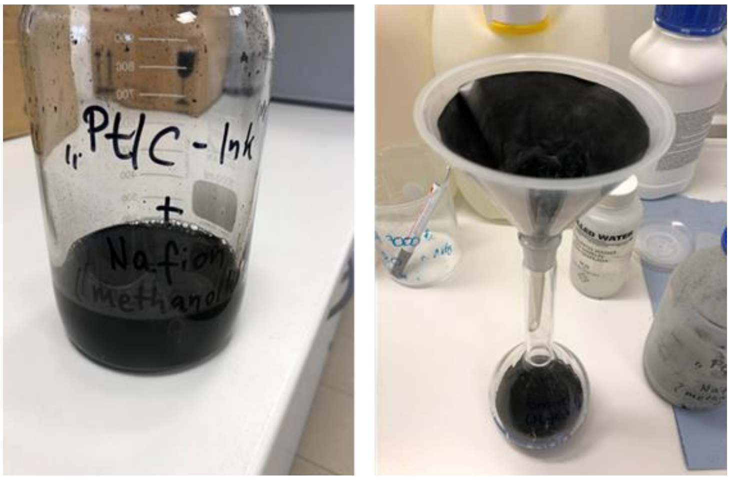


Figure 31 Preliminary results from AD: dispersion and filtration

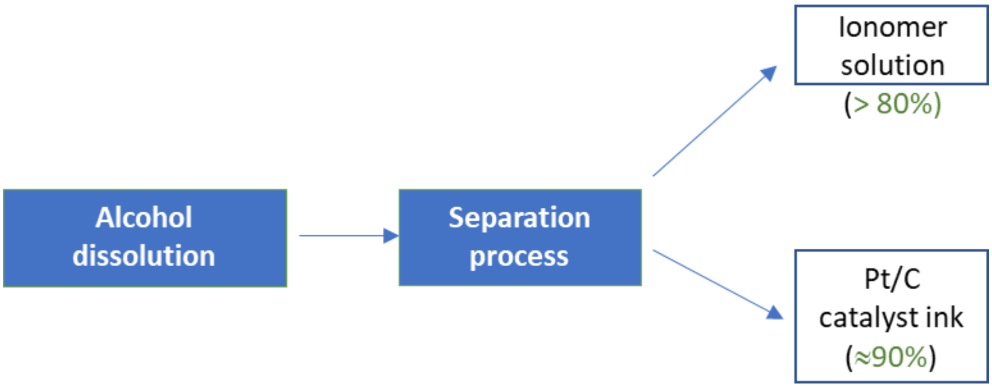


Table 7 Scheme of IDO-Lab process

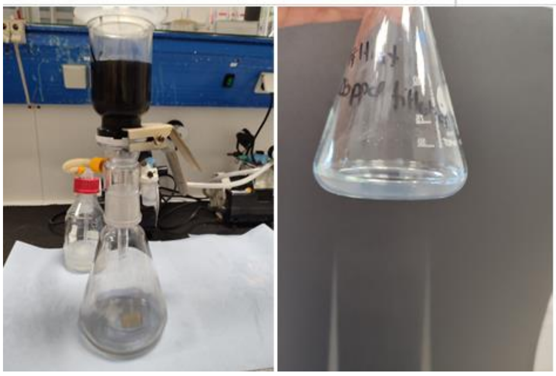
dissolution of material from: EKPO (NMG-366-240, 200h) SOLVAY (as reference)	Ethanol (more ecofriendly) Isopropyl alcohol Separation by Vacuum filtration with cellulose nitrate filter paper; mesh size 0,45µm and 0,2µm	✓
	initial weight: 5g +135 mL Ethanol +15 mL water 80°C, stirring for 30 mins vacuum filtration result: blurry solution	

Table 8 2nd test on NMG-366-240 incoming material with ethanol at IDO-Lab

The second test on NMG-366-240 material is performed dissolved with ethanol/water (90/10) at 80°C. 5 g of this sample was treated with 150 mL mixture. The material was stirred for 30 minutes, and the separation was conducted by vacuum filtration with cellulose nitrate filter paper (0,45 µm). The filter quickly became clogged, so the filtration process was aborted. The result was a 15 mL blurry solution due to the colloidal material that apparently doesn’t affect the quality of the solution negatively. Using ethylene glycol this effect should easily go away.

Conclusions and next steps.

A. Ethanol is considered ideal to perform the alcohol dissolution (AD)
B. The ionomer should be handled with some energy to obtain some results
C. The best results are reached applying pressure (under investigation) and high temperature (>120 Celsius)
D. These conditions (A and B) are best obtained via autoclave and after with a passing in an oven or a drying cabinet (lab optimization).
E. It is not advisable to perform these operations in a microwave apparatus since the temperature is not easily controlled inside the microwave.
F. A part of the ionomer will not dissolve, since it contains Polytetrafluoroethylene (PTFE- Teflon); its function is to strengthen the structure of the film
G. Separation process: the vacuum filtration is a good method, and it is advised to substitute the cellulose filtration paper with a glass frit. A satisfying option could be to centrifugate the product

7 Conclusions

The mechanical disassembling procedures of the PEMFC stacks, especially seen in a scalable context, proved more challenging, given the necessity of a clear separation among the different MEA components.

It is paramount to the process that the metal fractions of the BPP be not mixed with the rest of the MEA, since the metal fractions are the most complex to be treated, leaving spurs of metallic elements in the mix to be analysed and representing both a source of pollution, as well as a potential cause of Pt loss.

All methods used so far proved pros and cons, but none among them was fully satisfactory; in fact, during the tests spurs of Pt were revealed both in the sealings as well as in the BPP shredded fractions in quantities that made give up the first mechanical disassembling method (Granulator).

Using the paper shredder function, the fractions recovered are instead ideally shaped to be further dissolved in the alcoholic solutions, but the fact that the separation of the GDL from the CCM proved not completely successful placed in front of a new challenge.

The fifth, hybrid method to disassemble MEAs should prove successful and bring the expected results both quantitative as qualitative, as far as the transferring to TRL3 level to TRL5 level is concerned.

In parallel, even if not directly applicable to the project, HRD will investigate the use of components such as BPP. Though these showed Au traces, the experience leads to think that a possible re-utilization of the same would be more advisable than recycling of these products. In fact, the energy employed to shred and treat those elements would not be counterbalanced by the commercial value of the Au recovered. Thus, the possibility of second hand BPP should be further investigated possibly with producers of the same. This seemed to be particularly true for graphite BPP, however BPP with a metal base could lead to different conclusions, which relate more to the LCC and LCA part of the project, and of course the eco-design issue.

Gaseous phase dismantling on MEA demonstrated good results and re-use of the solvent guarantee the eco-friendly process. In particular with liquid phase demonstrate that liquid is very efficient dismantling whatever the aging of the MEA's; the high-pressure process makes dismantling easier for MEA's with sealing and ethanol seems to be the suitable solvent for efficient dismantling. US Bath based post-process seems more efficient to remove catalyst from the membrane. Approaching the vapour phase demonstrate a very easy GDL and CCM disassembling and good aspect for GDL and CCM. The membrane exposition to ethanol seems more homogeneous and smoother with vapor compared to liquid exposition. The catalyst ink remains stuck on the CCM and it is easier recovery.

So far, the recovery of Pt using available technologies proved successful, offering results in line with the goals and expectations of the project. The choice of filtration technique led to better separation of the filtrate from the Pt salt. Different aged materials are treated, and the process optimization reached the overall yields of ~80%. Future tests and replicates after ideal sampling (only CCM) should prove the optimal conditions that were reached at laboratory scale. Furthermore, the sample of CCM coming from gas phase dismantling can be processed in parallel with HMT and electro leaching and electrodeposition, comparing the 2 processes results.

The ionomer recovery approach is certainly difficult, given the need to deepen some aspects as the extreme heterogeneity of the ionomer, with many different producers and a variety of products, these technical aspects make the identification of the best process methodology and procedures a challenge.
Preliminary tests demonstrate interesting results, such as the use of ethanol. Some parts of the process will be improved such as the use of autoclave and the final product centrifugation thanks also to the support of Solvay (as AB member) and EKPO as ionomer user in the PEMFC stack.

The latest innovative process based on Pt recovery by coupling of electrochemical leaching in chloride-based ionic liquids (IL) mixture and an electrodeposition of Pt in the same compartment as lixiviation is studied. It must be considered the electrolixiviation process is no easy application because of absence of conductive support. There will be the development for new anode material to join with CCM (metal or stainless alloy mesh electrode). Next steps. Development on real material from GDE with GDL as anode and development from fresh CCM with metal or alloy mesh electrode as anode.