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

Deliverable D1.2

Technical report on adaptation of existing technology (hydrometallurgical process) for PEMFC material recovery: results and design

Document Details

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Document Details

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Clean Hydrogen Partnership

Co-funded by the European Union

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Abbreviations

AB	Advisory Board
AD	Alcohol Dissolution
BPP	Bipolar Plate
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 liquids
LCA	Life Cycle Assessment
LCC	Life Cycle Costing
MAS	Magic Angle Spinning
MEA(s)	Membrane Electrode Assembly/Membranes Electrode Assembly
NMR	Nuclear Magnetic Resonance
NP	Nanoparticle
PFSA	Perfluorosulfonic acid
PEMFC	Polymer Electrolyte Membrane Fuel Cell
PGMs	Platinum Group Metals
Pt	Platinum
TGA	Thermal gravimetric analysis
US bath	Ultrasound Bath
WP	Work Package

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1 Executive Summary

The **second Deliverable** (**D1.2**), due on the 30th of June 2022, lies within the **WP1**, which is under the leadership of **Hensel Recycling Deutschland (HRD)**, and deals with the **technical report on adaptation of existing technology (hydrometallurgical process) for PEMFC material recovery.** Thus, it is meant to report the **results and design** achieved in the first 18 months of work performed.

WP1 is structured in different tasks to shape and implement the recycling processes according to the final cell requirements in terms of purity and quality testing. The outcomes of WP1 are crucial to the whole project as the recovered materials will be characterized and reused both in cells and in a stack in WP2. The successful validation of the employed recovery technologies will be transferred to WP5 for the LCA/LCC.

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.

In the previous Delivery (**D1.1**) we reported about the recovery of 80% of Pt content minimum via **hydrometallurgical process (HTM)**, while an **alcohol dissolution process (AD)** is presently ongoing and should prove successful in recovering 90% of Pt: The recovery of ionomer is supposed to be over the quote of 80% and is currently under investigation. Finally, **electro-leaching and electrodeposition** should salvage 95% of the overall Pt quantity (this task lies under the responsibility of CEA).

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

- Increase the **Platinum recovery** from disassembling MEAs, avoiding loss of material (pure CCM).
- Obtain **Pt** under the form of **Pt salt (**as **diammonia hexachloroplatinate)** as final process product to reuse it in a closed loop recycling

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- 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 harmful 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, finally requiring a sustainable handling. The issue is not only ensuring a correct disposal path within a clear regulatory frame, but also ensuring the full recovery of the critical materials used. This is crucial not only to safeguard a lower environmental impact of new hydrogen technologies and devices compared to their counterparts over the whole life cycle, but also to grant the recirculation of 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 critical raw materials (CRMs) is needed. This is paramount for supporting the new hydrogen economy along with the development of lower environmental impact technologies, and the whole hydrogen stream delivery.

Currently, according to the findings of previous research initiatives, such as the HyTechCycling[1] project, there are very few examples of viable and up-scaled technologies

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[1] http://hytechcycling.eu/

for the recovery and recycling of CRMs from fuel cells (FCs) and electrolysers. Aside the current low volumes of streams – 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 such as harmful emissions and hazardous materials, and finally low level of technology readiness.

The innovative technologies developed within the WP1 concentrates on Pt (high material value and high criticality[2]) and ionomer (PFSA, medium material value, as well as 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 streams (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%). However, to give the correct contribution, an increase of the input rate is necessary. PGMs present a high value of recovery and have an interesting monetary value which makes them particularly attractive for recycling, presenting a high added value both in closed loop, as well as in open loop recycling (significative revenues).[3]

The available 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 to re-manufacture the PEMFC. Novel technologies are also studied and developed to recover ionomer via alcohol dissolution (AD) process and reintegrate it in a cell; the electrochemical

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[2] 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

[3] JRC, "Critical Raw Materials and the Circular Economy, Background Report", 2017

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. PEM 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 "**technical report on adaptation of existing technology (hydrometallurgical process) for PEMFC material recovery**" describes the main steps to adapt the technologies tested and described in the previous homologous document, according to the final cell requirements in terms of purity and quality testing. The results are linked to the work done in the first eighteen months of the BEST4Hy project.

The processes below are applied and shortly described as:

1. Identification and optimisation of an efficient and effective disassembling method of the MEAs: this has been named "**hybrid method**" and it is the fifth and last among the previous methods tried and described in the D1.1. The method consists of both manual and mechanical disassembling techniques.

This method proved ideal within a laboratory scale environment, since it leads to a highly pure Catalyst Coated Membrane (CCM) fraction which is the quality of material fitting best for the successive laboratory treatment.

A short description of the a.m. tasks follows hereunder:

- The Bipolar Plates (BPP) will be disassembled manually
- Shortly after, the rubber sealings will be cut off by means of a professional lever cutting machine
- Subsequently the Gas Diffusion Layers (GDLs) will be "stripped off" manually

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- Finally, the remaining CCM will be shredded with a regular office paper shredder.

A video was realised to better illustrate the methodology and can be found hereunder: https://best4hy-project.eu/lab-scale-optimization-results-for-pem-recycling/

The pure CCM fraction obtained via the fifth hybrid method was later sent to IDO-Lab to be recovered as forecasted in the project, which steps are summarized hereafter:

2. Design and pilot construction (M15-M24)
3. The optimized routines and operational steps will feed the design of a pilot plant
4. A pilot-scale equipment will be designed. The design will take in consideration: number of EoL cells to treat, quantity of scraps, volume of alcohol/ water to feed, specifics of filters separation and dimension.

3 Disassembling – 5th hybrid method

3.1 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

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The 5th hybrid method was chosen being ideal to recover pure CCM fractions; other materials resulting from the disassembling operations (manual and mechanical) can be held for recycling purposes as well (BPP, for instance).

Figure 2: BPP detail during manual disassembling

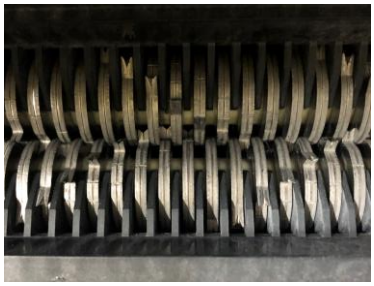

Figure 3: Internal view of paper shredder with CCM fractions traces

Figure 4: Freshly shredded CCM material

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4 Hydrometallurgical process to recover Pt-salt

The activities are based on the optimization at TRL3 of reliable, cost-efficient, and overall efficient operating procedures, varying different parameters to obtain the best recovery of platinum:

i) the %vol oxidants influence the leaching and the separation efficiency

ii) time of the processes and

iii) optimization of Pt separation by ion exchange resin. 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. An existing method to obtain Platinum salt as diammonia hexachloroplatinate is described in L. Duclos et al.[4]

In the scheme hereunder reproduced, the graphic process is replicated:

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[4] "Process development and optimization for platinum recovery from PEM fuel cell catalyst"

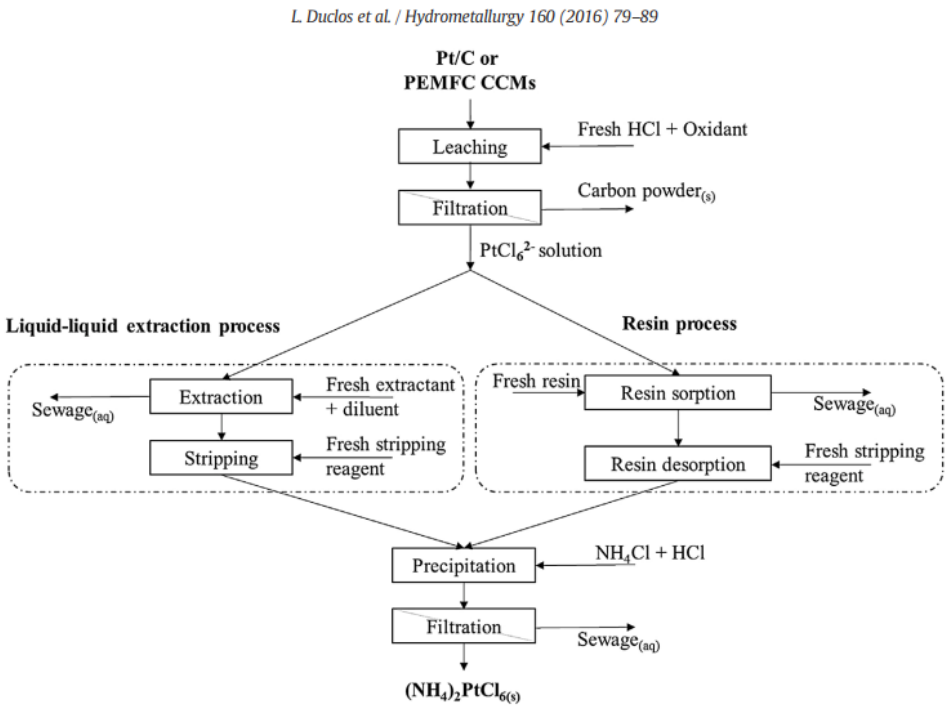

Figure 5: Graphic Description of the L. Duclos et al. Process

HMT process at IDO-Lab:

Hereunder the tailor-made process replicated at IDO-Lab:

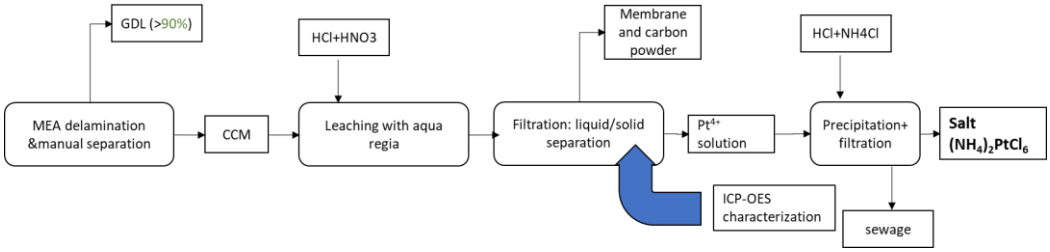

Figure 6: HMT process at IDO-Lab

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. 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.

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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), afterwards 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 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 concentrated solution 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 Pt rich solution
   - 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)

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- 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)

Deviation from the specified literature:

In “L. Duclos et al.” a resin method or a liquid-liquid process has been proposed. Oxidizing agents that require such a process, e.g., hydrogen peroxide, were used. Since nitric acid was applied instead in the beginning of the IDO-Lab process, this step was not considered and skipped. This could be proven in the further course of the laboratory optimization.

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Figure 7: Laboratory scale of Hydrometallurgical process to recover Pt as Pt-salt

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4.1 HMT before laboratory optimization

First attempts were performed with material coming from different mechanical trials, mostly mixed fractions (GDL + rubber sealing). The average yield was **~70%**.

Table 1: Various mechanical trials before 5th hybrid method

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

Laboratory samples can be categorized as follows: virgin cells of type A and used cells of type A and B. According to EKPO, the Pt loadings of cell type A and B differ from each other. While type A has 0.6 mg/cm^2, type B has a Pt load of 0.5 mg/cm^2. The measured Pt concentration at IDO-Lab refers to the initial weight. This analysis provides the theoretical yield after calculation by mass balance. The value of the Pt concentration also gives information about the Pt loading of the CCM when converting the units.

Table 2: Pt salt results before laboratory optimization

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: Pt salt results before laboratory optimization

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%	

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4.2 HMT during laboratory optimization

Some parameters have been changed to improve the process, for instance, batch size, choice of filter, and filtration techniques. Again, for these trials mixed fraction samples were dissolved in aqua regia. Because of the oxidizing effect of the leaching reagents, a rinsing process or an extraction were no longer needed. In addition to that, the precipitation solution was kept in acidic condition by hydrochloric acid. The average yield increased up to **~80%**.

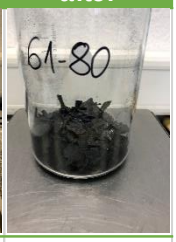
Main issues during laboratory optimization	before	after
Batch size before: max. 5 g in flask after: 50-100 g in beaker glass		
Filtration: choice of filter paper	white ribbon	blue ribbon
Choice of Filtration technique: before: Buchner funnel after: Vacuum Filtration		
Vacuum Filtration: mesh size of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

Figure 8: Main issues during laboratory optimization

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Process	parameters to be optimized
Leaching	Volume of aqua regia depending on batch size
Implementing another resin or extraction step	Extraction or regeneration by ion exchange resin adoption Tests with "Amberjet" inefficient 
Precipitation	pH of Pt-solution temperature of precipitation solution

Figure 9:Parameters to be optimized

The laboratory optimization included fundamental questions and improvements:

- Weighing / Leaching scalable: what is the maximum to reach for lab scale in a range of 50-100 g and leaching with 400 mL
- Temperature / duration: does the reaction depend on the temperature in the range of 100°C – 200°C
- Quality of precipitation: the precipitating reagent of ammonium chloride is made up with deionized water and has ~5.5 pH afterwards. Tests in more acidic zone (pH 1-4) by adding hydrochloride acid will show the effect after precipitating. Temperature ranges from room temperature to 50°C should be considered, too.

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*

Table 4: Pt salt results during laboratory optimization

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4.3 HMT and results after laboratory optimization

After adjusting various parameters, high yields of Pt were achieved. Key changes were the sampling method and the batch size. Pure CCMs (deriving from the 5th hybrid disassembling method) were treated more efficiently with aqua regia than the former heterogeneous mixed material. The larger batch size prevents significant losses during the individual steps.

Stack	Cell type	operating hours	initial weight (g)	Leaching	Filtration	Pt-conc. in solution	Precip.	Vac. Filtr.	Pt salt in g	yield in %
NM5-366-240	**A**	**200**	**46,94**						**4,7 (total)**	**89% (average)**
Batch#3.1	*only CCM*		*13,18*	*400 mL*	*Blue ribbon*	*9,46%*	*150 mL*	*0,2 µm*	2,475	*87,25*
Batch#3.2	*only CCM*		*33,76*	*400 mL*	*Blue ribbon*	*9,01%*	*150 mL*	*0,2 µm*	6,921	*90,99*

Table 5: Pt salt results after laboratory optimization

The ICP-OES analysis is done on all the Pt salts obtained.

Figure 10: Pt rich solution and 1:100 dissolution ready for ICP-OES

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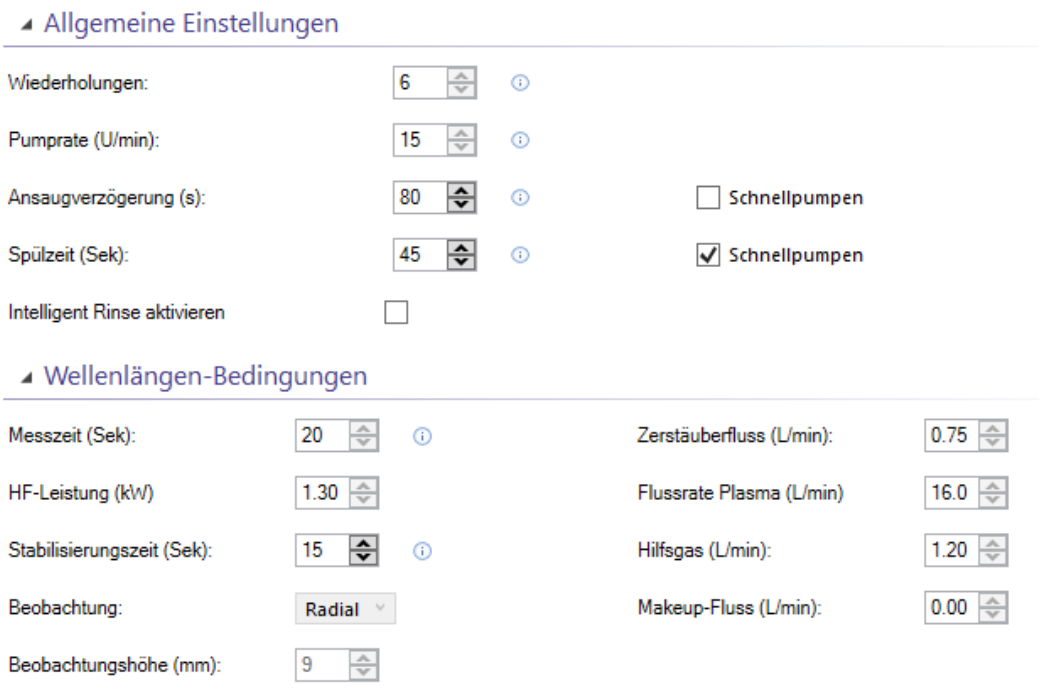
Each element to be analysed was detected by two or more wavelengths. An internal standard (Scandium) was added into the sample solution to ensure drift corrections. The ICP measurement was performed with the following parameters:[5]



[5] **General Settings**: Repetitions [6] Pump Rate (Round per min.) [15] Suction Delay (sec.) [80] Rinse Time (sec.) [45] Speed Pumping (on)
Wavelength Conditions: Measurement Time (sec.) [20] HF Performance (kW) [1.30] Stabilization Time (sec.) [15] Observation [Radial] Observation height (mm) [9] Nebulizer Flow (L/min) [0,75] Plasma flow rate (L/min) [1.20] Auxiliary gas (L/min.) Makeup-Flow (L/min) [0.00]

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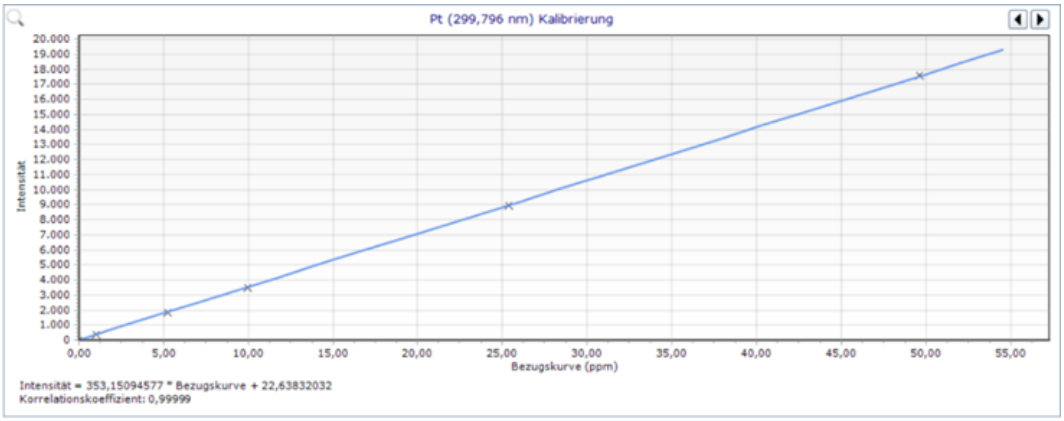

Figure 11: 5-point calibration of Pt 299,796 nm

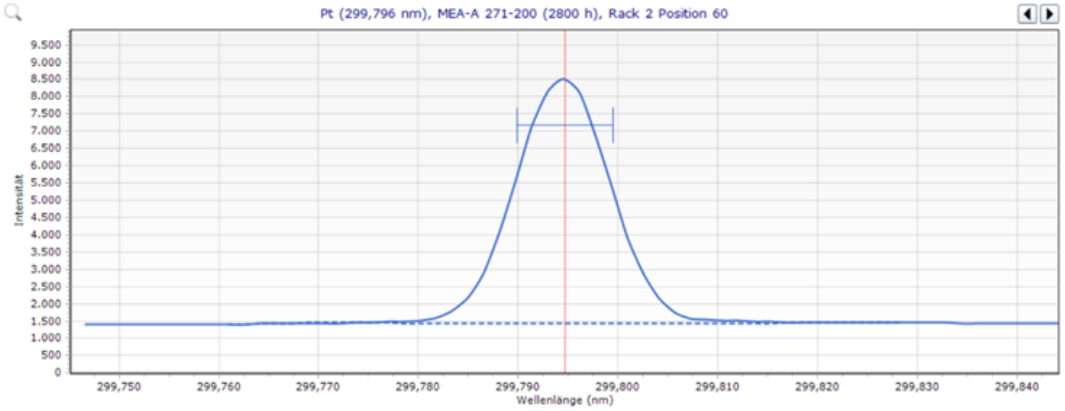

Figure 12: Pt-peak of MEA-A 271-200 (2800h)

Further investigations on other elements in the solution are often carried out as part of WP2 (Task 2.2.1, Quality testing of directly recycled materials, M9-30). Cobalt can be detected qualitatively by ICP-OES, e.g., on wavelength 350 nm, 228 nm and is performed on MEA_61-80 material.

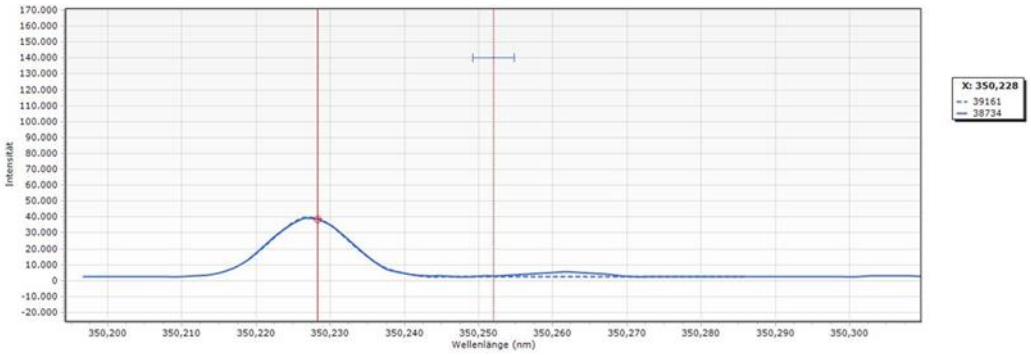

Figure 13: Co in MEA_61-80

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This investigation was further deepened. cobalt was calibrated to quantify the amounts before and after the precipitation process.

Table 6: ICP analysis of Pt rich solution and filtrate (before and after precipitation)

	Pt solution						Filtrate					
MEA_61-80	**Co**	**Cr**	**Fe**	**Mn**	**Mo**	**Ni**	**Co**	**Cr**	**Fe**	**Mn**	**Mo**	**Ni**
Batch#1.3	*653 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*576 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*
Batch#1.4	*675 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*524 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*
MEA_140-159	**Co**	**Cr**	**Fe**	**Mn**	**Mo**	**Ni**	**Co**	**Cr**	**Fe**	**Mn**	**Mo**	**Ni**
Batch#2.2	*621 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*505 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*
Batch#2.3	*668 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*552 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*
Batch#2.4	*598 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*490 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*	*<10 ppm*

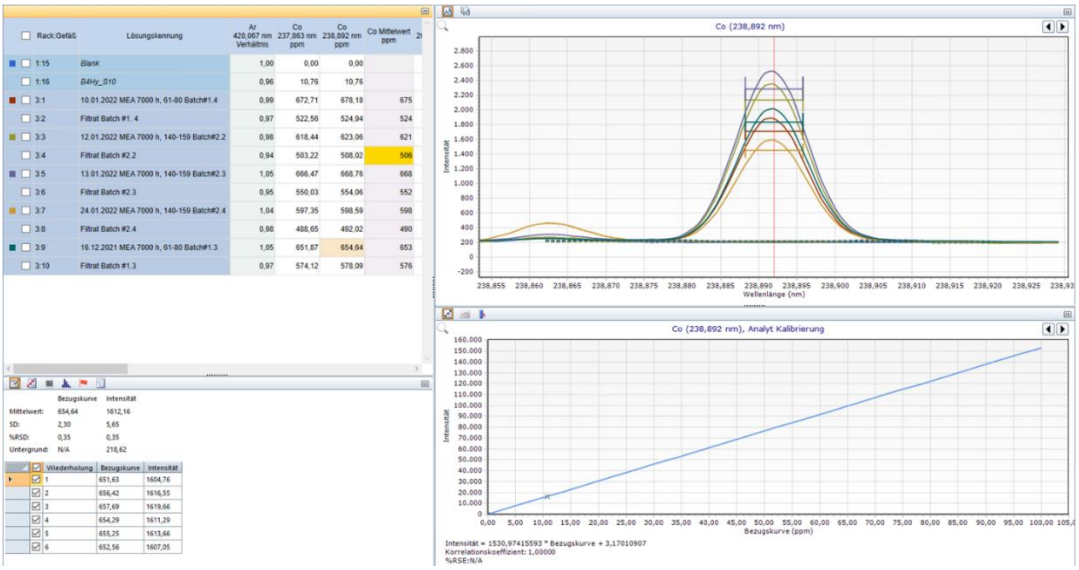

Figure 14: Spectrum of Cobalt measurement (λ Co 238nm)

Cobalt was found in both the platinum rich solution, as well as in the filtrate. This indicates that cobalt **was not coprecipitated**. Since cobalt has been classified by the EU Commission as strategic material and it plays an important role in another part of the BEST4Hy project as well (SOFC recycling), we think that some thought should be given with respect to this element as well.

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4.4 Evaluation of the HMT process: repeatability of high yields

The next step focused on the evaluation of the HMT process. The fact that a laboratory optimization was executed, should confirm repeatability of high yields of Pt. The initial weight was divided into three batches that represent the final lab scale. High yields of Pt were increased once more, in average reaching 93%, as the table hereunder shows.

Stack	Cell type	initial weight (g)	Leaching	Filtration	Pt-conc. in solution	Precip.	Vac. Filtr.	Pt salt in g	yield in %
NM5-366-240	**A (200 h)**	**103,13**						**20,2 (total)**	**93% (average)**
Batch#5.1	*only CCM*	*28,87*	*400 mL*	*Blue ribbon*	*9,24%*	*150 mL*	*0,2 µm*	5,291	*87,2*
Batch#5.2	*only CCM*	*34,31*	*400 mL*	*Blue ribbon*	*8,46%*	*150 mL*	*0,2 µm*	6,374	*96,5*
Batch#5.3	*only CCM*	*39,95*	*400 mL*	*Blue ribbon*	*9,19%*	*150 mL*	*0,2 µm*	8,026	*96,1*

Table 7: Evaluated Pt salt results after laboratory optimization

4.5 Pilot design and construction

According to the goal of the WP1, IDO-Lab projects to set up a pilot plant to scale up the evaluated HMT process. For this purpose, a reactor with a volume of 2 litres will be installed and controlled by an *ad-hoc* software. This application will allow to track various parameters like temperature, stirring or pH level. A picture of the proposed equipment can be found hereunder. The supplier, who was selected after a searching phase, is Heidolph Instruments[6] near Nürnberg and is a very well-known manufacturer of laboratory

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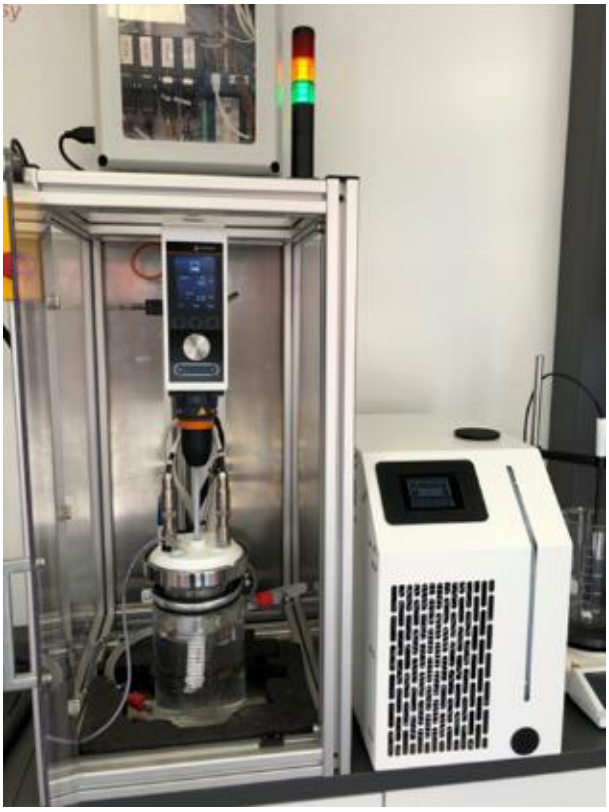
[6] https://heidolph-instruments.com/en/start

equipment. We are currently waiting for an offer from the sales department, which should reach us within the next weeks.



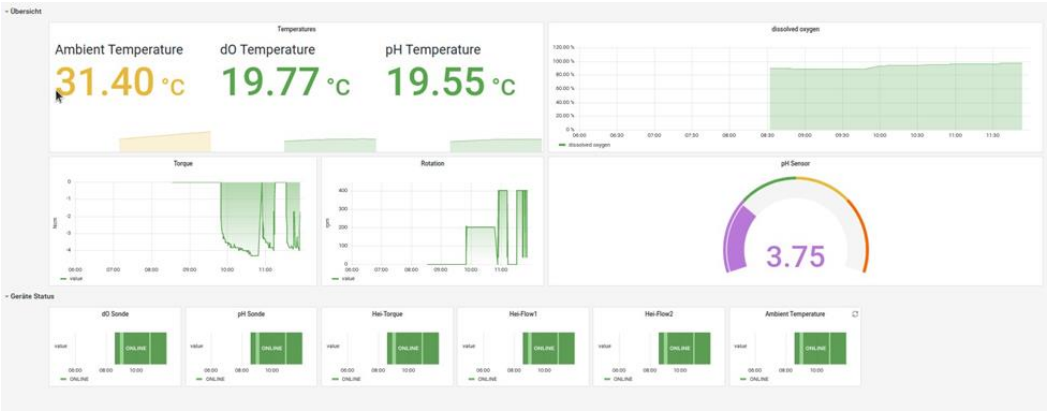


Figure 15: Example of different parameters to be controlled by the Heidolph equipment

"Hei-Control PRO" is a software that allows connecting different devices with each other in one interface. You can monitor and control the entire network of devices from one dashboard, whether at home or in the office. A reactor can be equipped with useful tools and applications that are necessary during a whole process. Some interesting parameters to be mentioned are:

- Temperature control: not only the ambient temperature can be observed, but the temperature of the recirculating chiller can also be useful to monitor the reaction.
- pH sensors

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- Stirrer: the stirrer can be programmed, e.g., if you want to stop stirring at a certain time of the current reaction.
- Pump: installation of a peristaltic pump if you want to feed your reactor continuously.

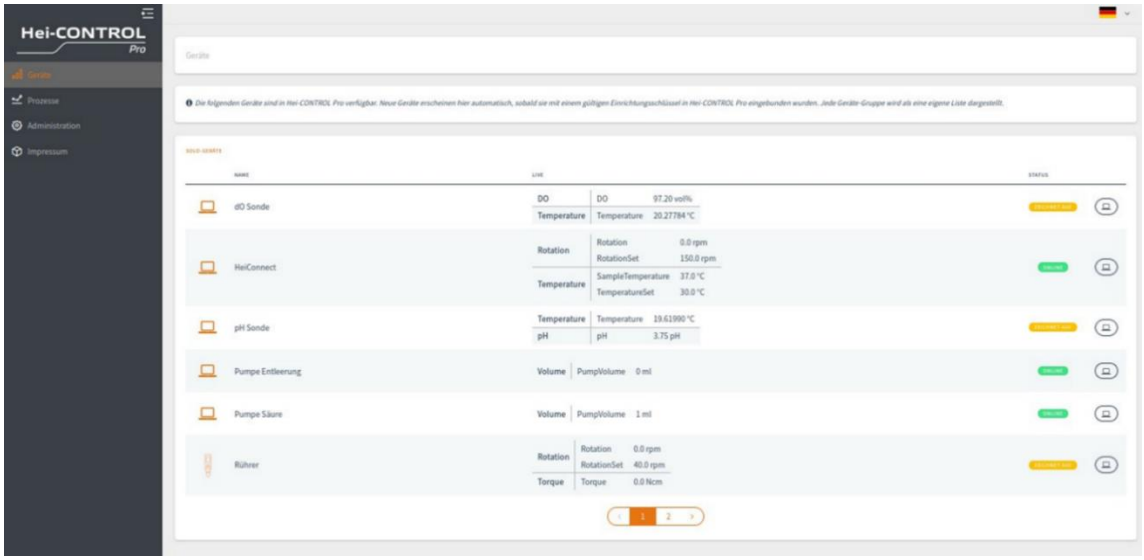

Figure 16: Hei-Control Software example

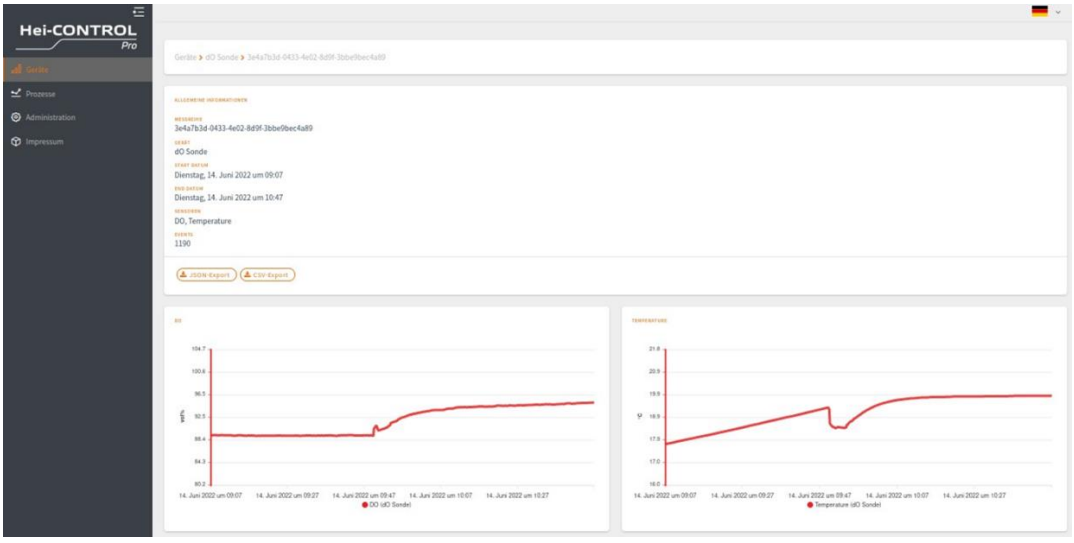

Figure 17: Pictures of Hei-Control software ("Heidolph Instruments")

5 Conclusions

Several highly significant elements and parameters in the recovery process were successfully optimized and implemented in this second phase of the project. It proved paramount to the whole Pt recovery process, to have determined an ideal disassembling

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solution, i.e. the fifth hybrid method. The fact that this process is a mixture between manual and mechanical disassembling is acceptable, due to the laboratory scale of the BEST4Hy project. However, from an industrial viewpoint, in terms of scalability and efficiency, and, last but not least, profitability, a deep reflection must be done in the future in this respect, since a standardization in the disassembling technique will be needed when big streams of EOL materials will be available. HRD is committed to further searching and investigating into this direction.

As far as the TRL3 framework is concerned, and within the duration forecasted for this task by the project, excellent Pt yields were recovered (>90%), even above the goal (~ 80%) which was agreed upon during the writing of the GRANT Agreement. It was also assessed that Pt recovery is not influenced by the age or operative hours of the cells treated, proving another important fact in favour of the recycling of this kind of waste.

Now the challenge in front of us is the realization of the TRL5 level with the construction of the pilot plant which is under study right now and which we are to achieve by the end of the year.

Clean Hydrogen Partnership

Co-funded by the European Union

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