

MARKET RESEARCH STUDY
RECOVERY OF PLATINUM
METALS FROM PEMFCs

August 2022

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1.0 Introduction

In 2022 the Department of Energy released two distinct but related supply chain reports entitled (1) Water Electrolyzers and Fuel Cell Supply Chain and (2) Platinum Group Metal Catalysts – Supply Chain Deep Dive Assessment. Both reports provide significant information regarding the projected 2050 demand for Polymer Electrolyte Membrane Fuel Cells (PEMFC) and Polymer Electrolyte Membrane Electrolyzer Cells (PEMEC).¹ PEMFC have been developed in large part to wean the automotive industry away from fossil fuels, which contribute significantly to CO₂. This report focuses on methods to collect and extract platinum group metals (PGM) from PEMFC, so that these rare and costly materials can be recycled. A brief introduction to fuel-cell electric vehicles sets the stage for this report.

According to the McKinsey study "a cost reduction of roughly 70-80% for fuel cell vehicles would be possible given an annual production volume of 150,000 vehicles".

1.1 Slow Growth of FCEV market

Fuel-cell electric vehicles (FCEVs) share many of the same components as battery electric vehicles (BEVs). The major difference is the energy source. While BEVs use energy stored in a battery, FCEVs use hydrogen.² Unlike a battery, a fuel cell does not run down or need recharging as long as the fuel (hydrogen) is provided. PEMFCs run at low temperatures and can provide greater mileage than BEVs.³

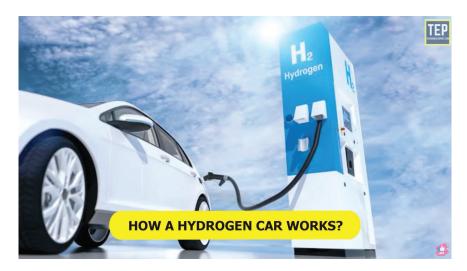


Figure 1: How does a hydrogen car work?

Click to see video

¹ Polymer Electrolyte Membrane Fuel Cells (PEMFC) are also known as Proton-Exchange Membrane Fuel Cells (PEMFC).

² Richard Folkson (Editor), *Alternative Fuels and Advanced Vehicle Technologies for Improve Environmental Performance* (Cambridge, United Kingdom: Woodhead Publishing Limited, 2014)

^{3 &}lt;u>Fuel Cell Basics</u>. Hydrogen and Fuel Cell Technologies Office accessed August 27, 2022.

Honda, Hyundai, and Toyota introduced FCEVs in Europe, Asia, and the U.S. several years ago.⁴ However, some automakers are temporarily changing direction as the hydrogen refueling infrastructure does not yet exist to support the sales of FCEVs. At the end of 2020 there were 540 FCEV refueling stations worldwide – with Japan having the largest number (140 stations).⁵ According to a report prepared by Argonne National Laboratory, in July 2022, only 91 FCEVs were sold in the United States – bringing the total sold in

the U.S. in 2022 to 1,924. Since the first introduction of FCEVs in 2014 into the U.S., a total of 14,207 FCEVs have been sold into the U.S. market.⁶ As a result of low global sales, in 2021 Honda stopped production of the Clarity⁷ and Hyundai froze its third generation FCEV.⁸ By contrast, Toyota announced the introduction in December 2022 of what is described as a high-end, elegant sedan - the Toyota Mirai. The Mirai is positioned as a luxury car with an estimated range of 402 miles and a price tag of approximately \$49,500.⁹ Toyota also gives Mirai owners a hydrogen gas card that has \$15,000 on it. That's about three years of hydrogen gas covered by Toyota that you don't have to pay for!¹⁰

While some automakers are temporarily ceasing production of FCEVs, Toyota has announced its plan to introduce a new luxury sedan in December 2022 – the Toyota Mirai.

Apart from the need to build the hydrogen infrastructure, another challenge is the current cost of FCEVs. In a February 2022 article by Ballard, the author notes that the price of fuel cell vehicles has been dropping for years and predicts a further 70-80% reduction in price as volume scales. According to the McKinsey study "a cost reduction of roughly 70-80% for fuel cell vehicles would be possible given an annual production volume of 150,000 vehicles". Today fuel cells are made in the thousands, compared to hundreds of thousands of batteries and millions of diesel engines. One contribution to reducing the cost of fuel cells is recycling the catalysts, re-using the bi-polar plates and refurbishing fuel cells.¹¹

Ballard Power Systems, a major supplier of PEMFC notes that the typical lifespan of a fuel cell is five to ten years, depending on the application. By this time, the membrane electrode assembly (MEA) wears out. However, the MEA can be replaced, and the fuel cell refurbished and returned to the original user with the same specifications as a new stack at a fraction of the cost of producing a new PEMFC.¹² Ballard offers its customers a refurbishment program for fuel cells that reach the end of life. "The customer returns the

- 4 Pollet, Bruno G. et al. "Current status of automotive fuel cells for sustainable transport". Current Opinion in Electrochemistry, Volume 16, (August 2019): pages 90-95.
- 5 Bermudez, Jose M and Hannula, Ilkka. "<u>Hydrogen More efforts needed. Tracking Report."</u> IEA, November 2021, accessed August 28, 2022.
- 6 "Light Duty Electric Drive Vehicles Monthly Sales Updates." Argonne National Laboratory
- 7 Raynal, Wes. "Honda Discontinues it Clarity Fuel Cell Vehicle." Autoweek, June 17, 2021.
- 8 Taylor, Michael. <u>"Hyundai Freezes Third-Gen Hydrogen Fuel Cell EV Development."</u> Forbes, December 31, 2021.
- 9 "Zero Emissions in Style: The 2022 Toyota Mirai Pricing Announced," Toyota Newsroom. November 18, 2021.
- 10 Bzeih, Reema. "<u>Driving the Toyota Mirai: My Hydrogen Fuel Cell Car Experience."</u> Climate Change, September 17, 2021
- 11 Pocard, Nicholas. Fuel Cell Price to Drop 70-80% as Production Volumes Scale. Ballard, February 11, 2022
- 12 Howe, Terry. Benefits of Fuel Cells: Refurbishing Leads to Zero-Waste. Ballard, April 5, 201

fuel cell stack to Ballard where we replace the MEA while reusing the existing hardware and plates. The used MEA is then sent to a 3rd-party for recovery of the platinum and other precious metals."¹³ Ballard Power Systems has sold over 30,000 PEMFC for buses since it first introduced a Ballard-powered fuel cell bus in Canada in 1993.¹⁴ Today, up to a thousand PEMFCs a year are returned and refurbished by Ballard and the PGMs extracted from the MEA and recycled.

Continued Growth of Catalytic Converter market

The fuel-cell electric vehicle market will co-exist for an extended period with the markets for internal combustion engines and the growing demand for battery electric vehicles. Information is included in this section to demonstrate that the demand for PGMs using conventional removal methods will continue for catalytic converters for an extended period. A market study on the catalytic converter market released in 2019 by MarketsandMarkets shows projections through 2025. During this period stringent emission guidelines and fuel efficiency regulations are expected to increase demand for catalytic converters and new approaches will be used to continue to enhance the performance of

The fuel-cell electric vehicle (FCEV) market will co-exist with the market for internal combustion engines and the growing market for battery electric vehicles (BEV) for an extended period.

catalytic converters and reduce the amount of PGMs required. The use of three- and four-way catalytic converters is also expected to increase, as well as the use of nanoparticles of platinum for catalytic reduction.

Table 1: Global Catalytic Converter Market, by Vehicle Type, 2018 – 2025 ('000s of units)

Vehicle Type	2016	2017	2018-е	2020-р	2022-р	2025-р	CAGR (2018-2025)
Passenger Car	83,438.2	86,289.5	89,797.9	98,297.0	1,06,343.3	1,17,473.4	3.91%
LCV	23,345.4	24,529.4	25,886.6	28,865.8	31,570.5	35,544.0	4.63%
Truck	5,291.1	6,759.9	7,473.2	9,250.8	10,433.9	11,748.2	6.68%
Bus	575.3	601.0	677.3	814.6	908.6	1,011.8	5.90%
Total	1,12,650.0	1,18,179.9	1,23,835.0	1,37,228.2	1,49,256.3	1,65,777.5	4.26%

e-estimated, p - Projected

Reprinted with permission of MarketsandMarkets¹⁵

Although the projected growth rate of the catalytic converter market in Europe, Canada and the U.S. is expected to be between 3.47% and 6.81%; it is predicted to be between 8.83% and 12.35% in other parts of the world. The catalyst supply chain for internal combustion engines will remain robust during the near term.

^{13 &}quot;Technical Note: Recycling PEM fuel Cells: End-of-Life Management." Ballard Power Systems, Inc.

^{14 &}lt;u>"Our History,"</u> Ballard, accessed on August 27, 2022.

^{15 &}quot;Catalytic Converter Market, Global Forecast to 2025". MarketsandMarkets, 2019. [Subscription]

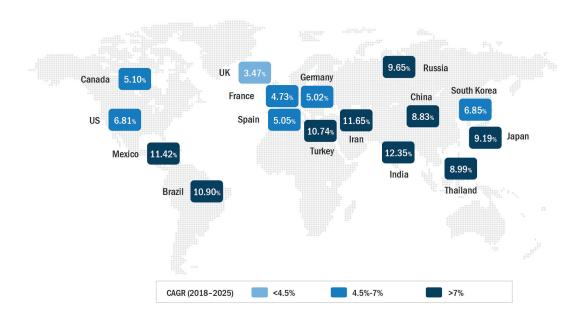


Figure 2: Catalytic Converter Market by Country Reprinted with permission of MarketsandMarkets¹⁶

In looking at the catalytic converter market by material, the largest projected demand will be for palladium. Palladium is less expensive than platinum and is used as an oxidation catalyst. It also tolerates higher temperatures better than platinum. Platinum is used both for reduction and oxidation. In diesel engines, platinum and rhodium are used. Rhodium also performs very well at high temperatures.

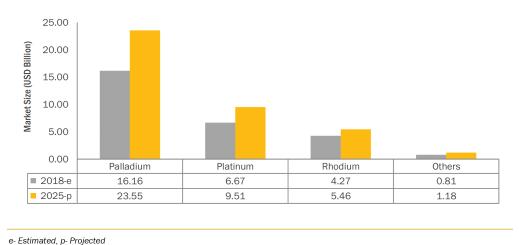


Figure 3: Catalytic Convert Market by Catalyst Reprinted with permission of MarketsandMarkets¹⁷

In this 2019 report by MarketsandMarkets fuel-cell electric vehicles were not even mentioned. However, battery electric vehicles were considered a threat to the catalytic converter market.

 [&]quot;Catalytic Converter Market, Global Forecast to 2025". MarketsandMarkets, 2019. [Subscription]
 "Catalytic Converter Market, Global Forecast to 2025". MarketsandMarkets, 2019. [Subscription]

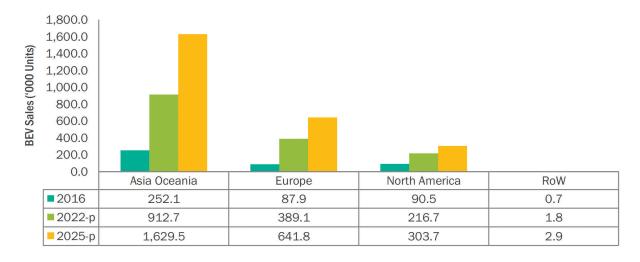


Figure 4: Global Actual and Predicted Battery Electric Vehicle Sales
Reprinted with permission of MarketsandMarkets¹⁸

2.0 Platinum Group Metals in PEMFC and PEMEC

Both PEMFC and PEMEC use a variety of rare platinum group metals (PGM) which can be recovered using existing and novel recovery methods.¹⁹

Table 2: Platinum Group Metals used in PEMFC and PEMEC²⁰

Device	Component	Material	Existing Recovery Method	Novel Recovery Method
	Anode	Pt	HMT, PMT	SED, TD, AP
PEMFC	Cathode	Pt	HMT, PMT	SED, TD ,AP
	Electrolyte	Ionomer	n/a	AD, AP
	Anode	Ir, Ru	HMT, PMT	TD
DEMEC	Cathode	Pt	HMT, PMT	SED, TD, AP
PEMEC	Electrolyte	Ionomer	n/a	AD, AP
	Bipolar Plate	Ti	HMT	n/a

The price of the recovered metals varies depending on the commodity market. The following is the current fair market value as posted on the <u>DOE Business Center for Precious Metals Sales and Recovery</u>.

^{18 &}quot;Catalytic Converter Market, Global Forecast to 2025". MarketsandMarkets, 2019. [Subscription]

AD= alcohol dissolution; AP= acid process; HMT= hydrometallurgical technology; PMT= pyrometallurgical technology; SED= selective electrochemical dissolution; TD= transient dissolution

^{20 &}quot;Water Electrolyzers and Fuel Cell Supply Chain." U.S. Department of Energy Response to Executive Order 14017 "America's Supply Chains", February 24, 2022.

Table 3: Current PGM Fair Market Value, accessed on August 30,2022

Name	Symbol	/larket Gram
Gold	Au	\$ 55.62
Silver	Ag	\$ 0.60
Platinum	Pt	\$ 27.91
Rhodium	Rh	\$ 450.11
Palladium	Pd	\$ 68.64
Iridium	Ir	\$ 136.64
Osmium	Os	\$ 12.86
Ruthenium	Ru	\$ 16.88

Due to the limited supply of these platinum group metals – an effective end-of-life recovery process is necessary for all these materials.

3.0 Comparison of Disassembly Processes (Catalytic Converters and PEMFC)

The location and type of devices that contain the platinum group metals within FCEVs and automobiles with internal combustion engines are totally different. Within today's cars PGMs are found within the catalytic converter that sit underneath the car as part of the exhaust system. The "cat" as it is often called, lasts up to ten years, and has a replacement cost of between \$1,000 and \$2,600 considering parts, labor, and state emissions requirements.²¹ The honeycomb material contains the PGMs.

^{21 &}lt;u>"How Much does a Catalytic Converter Replacement Cost?"</u> AutoZone, accessed on August 27, 2022; <u>How Much Does It Cost to Replace a Catalytic Converter?</u> Insurance Panda, last updated on August 7, 2022.

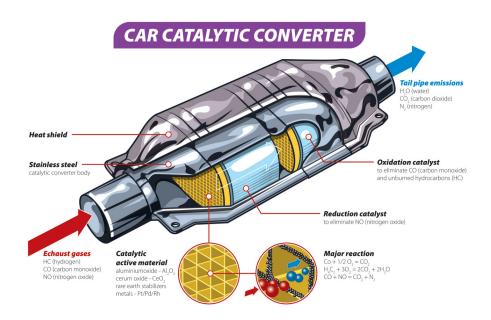


Figure 5: Schematic of Car Catalytic Converter Showing Honeycomb material

By comparison the PEMFC sits inside the frame of the fuel-cell electric vehicle and is a complex structure comprised of stacks of cells that contain bipolar plates, a membrane electrode assembly, seals, cooling plates and end plates. Although replacement data are not currently available for PEMFC, it is known to be one of the most expensive parts of a fuel-cell electric vehicle.

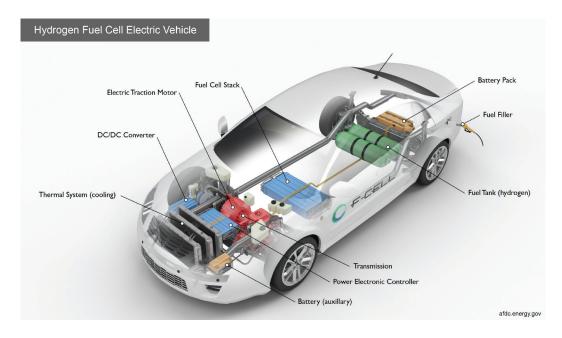


Figure 6: Image of Hydrogen Fuel Cell Electric Vehicle Source: U.S. Department of Energy

Figure 7 demonstrates the assembly of a PEMFC. One can readily see the difference between the MEA which contains the platinum group metals within the fuel-cell and a catalytic converter. Given the difference in the complexity of the components and the function that each system plays, it is questionable whether the collection and separation stage of the supply chain would be the same for PEMFC, as it is for catalytic converters. In fact, the previous discussion on the refurbishment program of Ballard Power Systems indicates that the disassembly of their PEMFCs takes place within Ballard facilities and that they re-use all the components. Only the MEA is sent out to a vendor to separate the PGMs. The result is that a less expensive, high-quality replacement is returned to the customer.

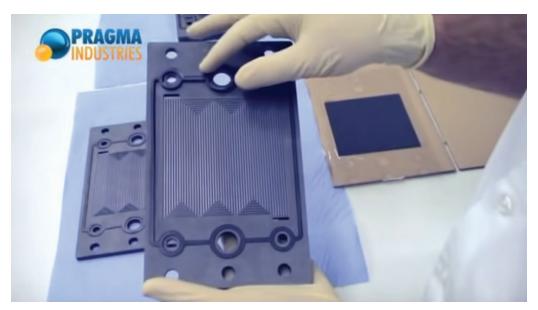


Figure 7: Image of bipolar plates and MEA

Click to see video

3.1 Catalytic Converter Recycling Business Model

Catalytic converters were first introduced into the U.S. in 1973²², followed by widespread use which began in the '80s. Recycling of catalytic converters didn't start until the 90's. Up until that time they were shredded.²³ Today there is an established business model where catalytic converters are collected and sent to a central facility for processing and PGM recovery.

The catalytic converter recycling supply chain can be viewed as having two primary groups: **Producers and Buyers.** Producers are those shops that process catalytic converters from vehicles. These companies are actually consolidators that collect, stockpile, and dismantle the catalytic converters to separate the monolith from the stainless-steel converter shell that surrounds it. The stainless-steel converter shells are recycled offsite. This group of producers includes muffler shops, junk yards and auto shops. The buyers are brokers and smelters.²⁴

²² Morgan, Chris. <u>"40 Years of Cleaner Air: The Evolution of the Autocatalyst."</u> Johnson Matthey Technical Review, 54, 40.

^{23 &}lt;u>"The Price List" and its Evolution – Catalytic Converters,"</u> PMR. Accessed on August 28, 2022.

²⁴ Dove, Nigel. "The Lifecycle of Catalytic Converts – and What it Means to Scrap Metal Dealers." K2Casting, June 29, 2021.

Catalytic converters contain a ceramic honeycomb coated with salts of platinum, palladium, and rhodium. The amount of PGMs found in the catalytic converter depends upon the emission ratings for a given state. The stricter a state's emission rating, the higher the concentration of PGMs. Each catalytic converter has an identifying number which identifies the vehicle make, model and year which can in turn be readily translated into dollar value. As PGM materials wear out over time, this has unfortunately led to catalytic converter theft from newer cars.

The type of auto catalyst used, also varies. There are five different types referred to as OEM Ceramic Converters, OEM Metallic Converters, Diesel Particulate Filter (DPF), After markets and Beads. Most refiners are set up to run 2,000-to-8,000-pound lot sizes. Therefore, to meet this volume, "producers" often mix the catalyst materials from various types of catalytic converters to assure these volume requirements are met. This adversely affects the quality and the value of the lot. One must be particularly careful if the mix includes DPF produced after 2006 which includes a silicon carbide additive. If this DPF is not treated property by the smelter it can ignite and cause an explosion in the arc smelter. For this reason, some smelters will not accept a blend containing DPF or pay less for the blend.

Companies such as <u>Vortex De-Pollution and Recycling Equi</u>pment have designed equipment to expedite the removal of the PGM salts from catalytic converters.



Figure 8: Click to see the video of the Double Cate Guillotine

Catalytic converter recycling consists of two main parts: (1) physical and (2) chemical recycling. In physical (disassembly), the company does not need environmental permits because no chemicals are used in the physical recycling process. According to Ecotrade

²⁵ Hope, Cliff. <u>"Time to rethink what can be accomplished with catalytic converter recycling data".</u> Recycling Product New, December 11, 2018.

Group, technically de-canning can be done at little or no cost. This process is efficient as it ensures that stainless steel or steel housing is reclaimed.²⁶



Figure 9: De-canned catalytic converter with honeycomb bloke Source: Ecotrade.²⁷

The recyclers can recover 100% of all metals in the catalytic converter through the decanning. The convertor is conveyed hydraulically to a shear mill which pulverizes the scrap core. These small pieces land on a shaker table where a heavy magnet is used to separate ferrous and nonferrous metals. The honeycomb or the ceramic block is collected and stored in packages. The packages containing the honeycomb materials are subsequently sold to refiners or brokers for further processing and metals recovery. The figure below provides an overview of the supply chain.²⁸

²⁶ Ecotrade Group. "Maximizing Profitability from Catalytic Converter Recycling." November 28, 2021.

²⁷ Ecotrade Group. "Maximizing Profitability from Catalytic Converter Recycling." November 28, 2021.

²⁸ CJD Cycling. April 18, 2022. https://www.cjdecycling.com/scrapping-recycling-catalytic-converters/

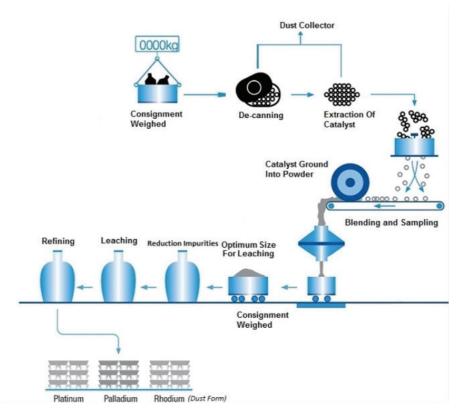


Figure 10: Catalytic converter recycling process Source: Ecotrade Group.²⁹

Smelters are the "real" refiners, melting metals to recover the actual precious metals. The powder from the catalytic converter is melted in a furnace and iron is added to attract molecules. In this process, the metal is filtered out and ground up and the iron is extracted. The remaining powder is reconstituted to recover PGMs and make salts. The salts are sold to manufacturers who use them to coat the ceramic honeycombs for new catalytic converters.³⁰ PGMs are sold to original equipment manufacturers that build new catalytic converters for auto manufacturers.³¹

3.2 PGM Recovery Methods

The technology for PGM recovery is complex and consists of many stages. What was reviewed in the previous section was the first two steps of the Pre-Treatment stage (See Figure 11) The actual process of recovering the platinum group metals requires heavy investment, and, hence, has traditionally been carried out by large organizations that can refine an extensive range of metals such as BASF, Umicore and Johnson Matthey.³²

^{29 &}quot;Refining Scrap Catalysts." Ecotrade Group, accessed August 25, 2022

³⁰ Dove, Nigel. "The Lifecycle of Catalytic Converters – and What It Means for Scrap Metal Dealers." K2Castings. June 29, 2021.

Taylor, Dylan. <u>"There's an Army of Thieves Coming for Your Catalytic Converter."</u> Popular Mechanics. May 23, 2022.

MarketsandMarkets. "Catalytic Converter Market by Type (FWCC, TWCC, SCR, DOC, and LNT), Material (Platinum, Palladium, and Rhodium), Vehicle Type (Passenger Cars, LCV, Bus & Truck, Construction, and Agriculture & Mining), Aftermarket and Region - Global Forecast to 2025." March 2019. https://www.marketsandmarkets.com/Market-Reports/catalytic-converter-systems-market-128255548.html

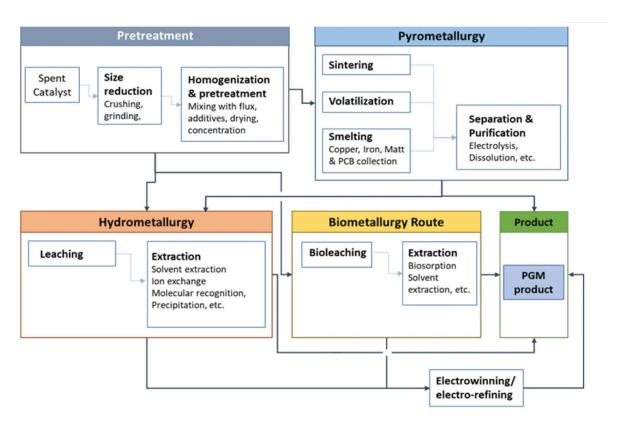


Figure 11: Process Flow for PGM Recovery³³

Pyrometallurgy is the most common industrial solution to recover PGMs (often shared facilities) due to its scalability and proven economic viability. Several PGM refining companies or primary producers such as <u>Johnson Matthey</u>,³⁴ <u>Heraeus</u>, <u>BASF</u>, etc. have continued to build new recycling plants based on the pyrometallurgy technology. For example, in February 2022, BASF and Heraeus formed a joint venture (BASF: 50%; Heraeus: 50%) to recover precious metals from spent automotive catalysts. The new company named BASF HERAEUS Metal Resource Co., Ltd is in Pinghu, China. The plant is based on pyrometallurgy technology.³⁵ In 2019, Ravindra-Heraeus, India's leading precious metals service provider, set up the first pyrometallurgical smelter (Plasma Metal Recovery System) in India.³⁶ Large amounts of energy are required with the consequent large CO₂ footprint. Other techniques like hydrometallurgy or bioleaching are being investigate or implemented.

Hydrometallurgical recycling involves metal leaching with acid/basic solution in the presence of an oxidant, further separation/concentration, and a final recovery either in the metallic or salt form is required. Depending on the material to recycle, a chemical or mechanical pretreatment is often required to (i) reduce refractory Pt oxides formed

^{33 &}lt;u>"Platinum Group Metal Catalysts – Supply Chain Deep Dive Assessment".</u> U.S. Department of Energy, February 24, 2022.

³⁴ Giovana Nichol. "Platinum Group Metals Recovery Using Secondary Raw Materials (PLATIRUS): Project Overview with a Focus on Processing Spent Autocatalyst." 2021.

^{35 &}lt;u>"BASF and Heraeus to form a joint venture offering world-class precious metal recycling solutions in China."</u> BASF. February 11, 2022.

^{36 &}quot;Ravindra-Heraeus sets up first pyrometallurgy smelter in India for precious metal catalyst recycling." Heraeus Group. February 18, 2019.

during its use as a catalyst, (ii) eliminate problematic organic compounds, or (iii) reduce the particle size of the initial sample, creating environmental problems. Companies are also investing in new plants. For example, in January 2022, BASF announced plans to build a plant for lithium-ion battery recycling in Germany. It will have an annual processing capacity of 15,000 tons of electric vehicle batteries and production scrap. It will create around 30 new production jobs, and it's expected to launch in early 2024. The black mass will then go to BASF's yet to be build a commercial hydrometallurgical refinery for metals recovery. The recycled batteries will recover lithium, nickel, cobalt, and manganese – the key metals used to produce cathode active materials.³⁷

4.0 Key Players in PEMFC

The automotive catalytic converters recycling business has a highly efficient logistics chain and economic incentives postulated throughout the literature. However, the business model for collection, dismantling and recycling of fuel cells and MEAs to recover PGMs is not as straight forward. In this section, a brief review of the key players that provide catalyst materials, MEAs and fuel-cells is provided.

4.1 Materials Manufacturers

The manufacturers acquire materials (e.g., catalysts, polymers, etc.) from different suppliers to produce both stack and other components. PGMs catalyst suppliers include:

- BASF SE (Germany)
- <u>Umicore</u> (Germany)
- Johnson Matthey (U.K.)

With its headquarters in Brussels, Umicore develops "materials for a better life" and is positioned as a leading circular materials technology company with extensive experience in material science, chemistry, and metallurgy. The company produces catalysts for fuel cell and stationary applications. With respect to recycling Umicore echoes the nascent stage of the fuel cell industry and of recycling.

"As the fuel cell and electrolyzer industry is still in its early stages, the stream of end-of-life materials is limited and there is no separate stream treatment at the moment. However, we are keeping a close eye on developments in the sector: should the need arise, we can easily separate the fuel cell & electrolyzer waste flows from other waste streams." ³⁸

²⁷ Lewis, Michelle. <u>"BASF is going to build a commercial battery-recycling, black-mass plant."</u> Elektrek. June 20, 2022

^{38 &}quot;Towards circular fuel cells: overcoming 3 challenges," Umicore, accessed August 29, 2022.

The three hurdles that Umicore describes are the need to:

- develop more efficient catalysts in line with international roadmaps for PEM technology;
- 2. **recover PGMs by recycling fuel cells and electrolyzers**. They anticipate that both dismantling and metallurgical treatment to recover PGMs will bring both challenges and opportunities for business growth; and
- cope with fluorine through capturing and managing the fluorine recycling process.

4.2 MEA Manufacturers

With respect to MEAs' most companies manufacturing MEAs specialize in high volume production, such as:

- <u>Ballard Power Systems</u> (Canada)
- Johnson Matthey (U.K.)
- <u>Danish Power Systems ApS</u> (Denmark)
- BASF SE (Germany)
- W.L. Gore & Associates (U.S.A.)
- Plug Power (U.S.A.)

In 2020, Ballard Power Systems announced the company is expanding manufacturing capacity for production of its proprietary membrane electrode assemblies by 6x in 2021. The upgraded capacity for production of six million MEAs annually, equivalent to approximately 1.66 Gigawatts of product, will make Ballard's Vancouver facility the largest fuel cell MEA production operation globally for commercial vehicles.³⁹ However, there are many companies which produce custom or low quantity MEAs, in different shapes, catalysts or membranes to be evaluated as well, which include Giner, Inc.,⁴⁰ FuelCellStore, FuelCellsEtc, HIAT gGmbH, Ion Power and many others.

Johnson Matthey is not only the world's largest secondary refiner of platinum group metals, but also produces MEAs using a highly customized approach to meet the needs of its clients.⁴¹

^{39 &}quot;Ballard Expanding MEA Production Capacity 6x to Meet Expected Growth in Fuel Cell Electric Vehicle Demand." Ballard, September 28, 2020.

^{40 &}quot;Specialty Membrane Electrode Assemblies," GinerLabs, accessed August 25, 2022.

^{41 &}lt;u>"Fuel cells recycling."</u> Johnson Matthey, accessed August 20, 2022.



Figure 12: PGM Refining by Johnson Matthey
Click to see video

4.3 Fuel Cells Manufacturers

The earlier discussion regarding Ballard Power Systems' refurbishment program opened the door to explore what other PEMFC manufacturers may be doing to address the challenge of recycling and refurbishment. The starting assumption was that others might also be using a novel approach for the collection and disassembly of PEMFC components; recognizing that most have not been fielded for an extended period. Another difference at this time, is that unlike the catalytic converter market that is focused on the automotive industry, most of the early fuel cell manufacturers have focused on fleets of buses, trucks,

Most of the early fuel cell manufacturers focused on fleets of buses, trucks, delivery vans and/or material handling equipment with established routes within proximity to hydrogen refueling stations.

delivery vans and/or material handling equipment. These fleets have established routes within proximity to hydrogen refueling stations. As the anticipated life of fuel cells is ten years and given the nascent stage of this market – the volume of PEMFC that need to be disassembled annually is assumed to be low.

The other fuel cell manufacturers reviewed are listed below. Although it is common for most of these companies to make mention of the importance of a circular economy and recycling, only two companies from this list provided details regarding their approach: Ballard Power Systems (discussed earlier) and Plug Power.

- Ballard Power Systems (Canada)
- Plug Power (U.S.A.)
- Hydrogenics recently purchased by Cummins (U.S.A.)
- Nuvera Fuel Cell (U.S.A.)
- Horizon Fuel Cell Technologies (Singapore)
- <u>Nedstock Fuel Cell Technology</u> (The Netherlands)

- <u>ITM Power</u> (U.K.)
- AVL (Austria)
- Intelligent Energy (U.K.)
- W.L. Gore & Associates (U.S.A.)
- <u>Pragma Industries</u> (France)
- <u>Umicore</u> (Belgium)
- Shanghai Shenli Technology Company (China)
- Johnson Matthey (U.K.)

4.3.1 Plug Power, Inc.

The headquarters for Plug Power is in Latham, NY; while its MEA facility is in Rochester, NY. In 2022 the company reported an installed base of 50,000 fuel cells - most of which are used in material handling equipment by companies such as Amazon, Walmart, Wegmans and others. The following video provides a 2020 tour of their fuel cell facilities.



Figure 13: Plug HQ Facility Tour 2020 Click to see video

In Plug Power's ESG Report 2021, four end-of-life treatment options were described:

- 1. Rental program: During high-demand times, Plug Power will rent products from vendors when their life cycle ends, and we will return them to the vendor
- 2. Reclamation of used fuel cell components
- 3. Once a product has been used, we will deconstruct it and reuse any pieces that can be leverage in day-to-day operations
- 4. Refurbish and resell products once deconstructed.

In a May 2022 report, mention was made of using automation to disassemble fuel cells and creating secondary markets involved with standardization for reuse for both fuel cells and electrolyzers.⁴²

4.3.2. European Union, Directive 2000/53/EC

In the European Union, end-of-life vehicle recycling is governed primarily by Directive 2000/53/EC. Under this Directive, automobile producers including those that provide electric vehicles are liable to take-back, treat and recycle end-of-life vehicles (ELV). Several different business entities are involved in the process of collecting and dismantling systems. All are ultimately certified as Authorised Treatment Facilities. However, according to Wittstock et al, it is clear that all ELV sold in Europe are not being disposed of in the prescribed manner. It is believed that many ELV vehicles are exported to other EU countries and could therefore be available for recycling in the receiving countries after a second use phase. This practice has an impact on the amount of PGM material that is available for recycling.⁴³

Wittstock et al also suggest that the disassembly process of the fuel cells needs to be manual. "The recycling of fuel cells requires a manual disassembly procedure. Although automated processing of small fuel cells with a low power range—such as those used as battery replacement—may be conceivable in the future, such processes require high volumes of end-of-life fuel cells and have hence not been developed to date."⁴⁴ In this excellent review of the literature it is mentioned that there are many different types of fuel cells, each with unique disassembly and processing steps. It is therefore proposed that the use of flow charts, photographic guides and manufacturer catalogues be used to guide the disassembly process.⁴⁵

The European Directive 2000/53/EC makes automobile producers, including those that provide electric vehicles, liable to takeback, treat and recycle end-of-life vehicles (ELV).

5.0 Technologies for Recovery of PGMs From MEAs

The core component of the stack of PEMFCs is the MEA, which involves an anodic layer and a cathodic layer separated by a polymeric membrane as the electrolyte. The MEA performs three main functions: reactant barrier, electric insulator, and proton conductor. The electrolyte commonly is a perfluoro sulphonic acid membrane (Nafion type). A PEMFC stack has a determined number of unit cells, associated in series, along with supporting components to produce electricity.⁴⁶ There are many ways of assembling MEAs such

⁴² Swift Lyons, Karen. "Plug Power Perspective on Hydrogen Economy Recycling Needs." May 26, 2022.

⁴³ Wittsotck, Rikka et al. "Challenges in Automotive Fuel Cells Recycling". Recycling, 1, 3 (2016)343-364

⁴⁴ Wittsotck, Rikka et al. "Challenges in Automotive Fuel Cells Recycling". Recycling, 1, 3 (2016)343-364

Schiemann, J.; Kerßenboom, A.; Prause, H.J.; Peil, S. Handbuch Verwertung von Brennstoffzellen und deren Peripherie-Systeme; Institut für Energie- und Umwelttechnik e.V.: Duisburg, Germany, 2007. [Google Scholar]

⁴⁶ Mahbod Moein Jahromi, Hadi Heidary. "Automotive applications of PEM technology." PEM Fuel Cells,

as the catalyst-coated membrane (CCM) and the catalyst-coated backing (CCB). In the CCM concept, a catalyst layer coats both sides of the electrolyte, and carbon-based gas diffusion layers (GDLs) are assembled to each side of the CCM. In the CCB concept, the catalyst layers adhere to the GDLs instead of adhering to the electrolyte.⁴⁷ The table below summarizes recovery technologies.

Table 4: Recovery technologies for relevant fuel cell stack materials

	Recovery technolo	Recovery technologies			
Material	Current	Novel			
Ni; NiO	Hydrothermal treatment; hydrometallurgical treatment	N/A			
Ag	Hydrometallurgical treatment	N/A			
YSZ	Hydrothermal treatment	N/A			
lr	Hydrometallurgical treatment; pyro-hydrometallurgical treatment	Transient dissolution			
Ru	Hydrometallurgical treatment; pyro-hydrometallurgical treatment	Transient dissolution			
lonomer	N/A	Acid process; alcohol dissolution			
PGM	Hydrometallurgical treatment; pyro-hydrometallurgical treatment	Selective electrochemical dissolution; transient dissolution; acid process			
LaMn03; LaCr03	N/A	N/a			

Source: Valente Antonio, Diego Iribarren, and Javier Dufour. (2019).⁴⁸

PGMs are the main electrocatalysts used in PEM stacks. Conventional recovery methods including hydrometallurgical technology (HMT) and pyrometallurgical technology (PMT) are discussed in this section. Recovery methods consist of:⁴⁹

 "Hydrometallurgical treatment: This method can be considered suitable for the recovery of other PGMs such as iridium and ruthenium. The hydrometallurgical pathway involves the dissolution of target elements from solid matrices through caustic or acid attacks. This step is typically followed by separation via precipitation, solvent extraction, distillation, ion exchange, cementation or filtration. The main

⁽²⁰²²⁾ Pages 347-405.

⁴⁷ US20060147791A1, "Platinum recovery from fuel cell stacks,"

Valente Antonio, Diego Iribarren, Javier Dufour. "<u>End of life of fuel cells and hydrogen products: From technologies to strategies."</u> International Journal of Hydrogen Energy, Volume 44, Issue 38 (2019):20965-20977.

⁴⁹ Férriz, A. M., Alfonso Bernad, Mitja Mori and Sabina Fiorot. <u>"End-of-life of fuel cell and hydrogen products: A state of the art."</u> International Journal of Hydrogen Energy (2019).

strengths of hydrometallurgical methods include high selectivity to metals, relatively low energy consumption, and the possibility of recycling reactants. However, hydrometallurgical processes involve the need for mechanical pretreatment to increase the active surface exposed to the reactants, a large volume of solutions, and the generation of wastewater, which may be corrosive and/or toxic. Hydrometallurgical processes to recover PGM catalyst from PEMFCs generally involve pre-treatment, leaching, separation and purification sub-processes. In order to improve the overall techno-economic performance of the process, further sub-processes can be involved, e.g. solvent regeneration. The individual efficiencies of leaching, separation and precipitation lead the overall recovery efficiency.

- **Hydrothermal treatment** involves the processing of waste materials with steam at relatively high temperature and pressure. This process can result relatively lengthy due to its complexity. Water at 200–240 °C is used for the disintegration of the YSZ-containing materials. The powder is collected by filtration and subsequently dried at 50 °C. Afterwards, the dry powder is milled and sieved until achieving a size below 100 µm. The sieved micro-powder undergoes uniaxial pressing followed by cold isostatic pressing. The resulting material is sintered at 1400–1600 °C for 2 h to obtain recycled zirconia.
- Pyro-hydrometallurgical treatment: When applied to PEM systems, pyro-hydrometallurgical treatment involves a calcination process in which GDLs, membranes and electrodes are incinerated. The ashes resulting from the combustion are processed through acid dissolution, and PGMs are subsequently recovered via precipitation. Pyro-hydrometallurgical processes do not require mechanical pre-treatment and they present higher recovery efficiency but higher energy demand. The number and simplicity of the stages involved in pyro-hydrometallurgical treatment facilitate its applicability. However, the severe conditions in terms of operating temperature and pH raise safety and environmental concerns."

In summary, the published literature indicates that the two major methods for PGM recovery

when applied to MEAs are not ideal. Pyrometallurgy is not suitable for treating large volumes of fuel cell MEA recycling, owing to the presence of fluorine compounds. Likewise, hydrometallurgy process has also been used for MEA recycling, but the high concentration of strong acids and oxidants used can react with fluorinated compounds and end with highly toxic vapors. For PEMEC, the recycling process is more complex due to the presence of supporting materials (e.g., membrane, GDL, carbon nanoparticles). Expression of the presence of supporting materials (e.g., membrane, GDL, carbon nanoparticles).

The published literature suggests that the two major methods for PGM recovery when applied to MEAs methods are not ideal.

Paiva, Ana Paula et al. <u>"Hydrometallurgical recovery of platinum-group metals from spent auto-catalysts-focus on leaching and solvent extraction."</u> Separation and Purification Technology, Volume 286, April 1, 2022.

⁵¹ Granados-Fernández, R.; Montiel, M.A.; Díaz-Abad, S.; Rodrigo, M.A.; Lobato, J. "Platinum Recovery Techniques for a Circular Economy." Catalysts 11, 8 (July 31, 2021)

L. Sandig-Predzymirska, T. V. Barreiros, A. Thiere, A. Weigelt, D. Vogt, M. Stelter, A. Charitos. <u>"Recycling strategy for the extraction of PGMs from spent PEM electrodes."</u> 2021.

In a research article entitled "Process development and optimization for platinum recovery from PEM fuel cell catalyst,"⁵³ the authors present the development of an efficient hydrometallurgical recovery process of platinum from Pt catalyst of PEMFC electrodes at laboratory scale, whereby leaching, Pt separation, precipitation and filtration steps were combined in order to optimize the recovery process.⁵⁴ In the article, "Gram-size Pt/C catalyst synthesized using Pt compound directly recovered from an end-of-life PEM fuel cell stack" Muralidhar Chourashiya and colleagues recovered platinum with fewer steps and the recycled catalyst as a commercial equivalent. In this investigation, platinum from the end-of-life fuel cell stack is recycled as a fresh Pt/C catalyst. The recycling processes involved the following steps:⁵⁵

Dissolution of Pt from the end-of-life fuel cell electrodes by refluxing in dilute acid (1 M HCl), followed by drying the Pt solution in a rotary evaporator and using the dried-Pt precursor for polyol synthesis of Pt/C. With the aim of their industrial adaptation, a gramscale synthesis of Pt/C using the polyol method is carried out and its electrocatalytic performance is compared with commercial benchmark catalysts. Electrochemical performances of the Pt/C electrocatalysts synthesized using the recycled Pt and the equivalent commercial Pt precursor are found to be comparable.

Antonio Valente, et al present strengths, weaknesses, opportunities, and threats (SWOT analysis) of the PGM recovery technologies from PEMFCs performed under technical, economic, environmental, social, and regulatory aspects. The figure below provides a summary that indicates a "holy grail" solution is not yet feasible.

Duclos, L et al. <u>"Process development and optimization for platinum recovery from PEM fuel cell catalyst"</u>. Hydrometallurgy, Volume 160, March 2016: Pages 79-89.

Duclos, L et al. "Process development and optimization for platinum recovery from PEM fuel cell catalyst". Hydrometallurgy, Volume 160, March 2016: Pages 79-89.

Muralidhar Chourashiya, Raghunandan Sharma, Saso Gyergyek, Shuang Ma Andersen.

<u>"Gram-size Pt/C catalyst synthesized using Pt compound directly recovered from an end-of-life PEM fuel cell stack."</u> Materials Chemistry and Physics, 276.

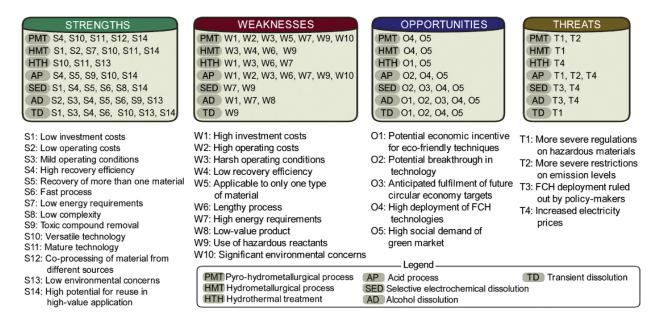


Figure 14: SWOT diagram for EoL technologies applicable to fuel cells and hydrogen stacks.

Source: Valente Antonio, Diego Iribarren, Javier Dufour. (2019).56

Table 5 rates key performance parameters of PGM recovery technologies.

Table 5: Key performance parameters of PGM recovery technologies

Technology	Pyro- hydrometallur- gical	Hydro- metallurgical	Hydrothermal	Acid process	Selective electrochemical dissolution	Transient dissolution
Investment cost	_	+	_	_	+	+
Operating cost	-	+	0	-	0	0
Recovery efficiency	+	_	0	++	+	+
Energy requirements	-	++	_	-	-	++
Hazard/toxicity	_	_	_	_	0	_
Other environmental concerns	_	0	++	-	0	++

Key: (++ very favorable performance; + favorable performance; 0 regular performance; - unfavorable performance)

Source: Valente Antonio, Diego Iribarren, Javier Dufour. (2019).57

Valente Antonio, Diego Iribarren, Javier Dufour. <u>"End of life of fuel cells and hydrogen products: From tech nologies to strategies."</u> International. *International Journal of Hydrogen Energy,* Volume 44, Issue 38 (2019): 20965-20977.

Valente Antonio, Diego Iribarren, Javier Dufour. <u>"End of life of fuel cells and hydrogen products: From tech nologies to strategies."</u> International. *International Journal of Hydrogen Energy,* Volume 44, Issue 38 (2019): 20965-20977.

5.1 Historic DOE Funded PGM Recycling Projects

Within the past two decades the Department of Energy has funded a number of projects aimed at addressing some of these challenges. In 2003 the Department of Energy funded Engelhard Corporation in a cost-shared project to recover precious metals from fuel cells. The project, which had an 80:20 cost share, began is 2003 and ran through 2008. The primary objective of that project was to:

- Develop environmentally friendly processes for recovering and recycling the platinum, palladium, rhodium and ruthenium present in fuel reformers and PEM fuel cell stacks.
- Investigate leaching, industrial microwave, supercritical carbon dioxide, and pyrometallurgical processes, and select the preferred processes for recovering precious metals from the various types of catalysts present in PEM fuel cell systems.
- Develop a commercially-viable process for recovering PGMs from fuel cells without releasing hydrogen fluoride.

In a 2004 progress report, it was noted that simple combustion of MEAs produced a Ptrich ash that was hard to handle. It also gave off significant amounts of toxic and corrosive hydrogen fluoride gas.⁵⁸ In 2009, BASF Catalysts LLC, formerly Engelhard Corporation reported the development of an environmentally-friendly, cost-effective method for recovery of platinum without release of hydrogen fluoride. This was achieved using a combination of milling, dispersion and acid leaching. 99% recovery of Pt was achieved, and this high yield could be scaled up using one vessel for a single leach and rinse.⁵⁹ The final report did not include an estimate of the cost, but stated: "The highlights of the economic analysis are that the process has the capability to recover 98% and the cost of recovering the Pt is a few per cent of the value of the metal, even at a Pt loading of 1% wt./wt."

In 2012, BASF was awarded US Patent 8,124,261, "Process for recycling components of a PEM fuel cell membrane electrode assembly" which currently has an adjusted expiration date⁶⁰ of 6/9/29.⁶¹ This patent disclosed PEM fuel cell can be recycled by contacting the MEA with a lower alkyl alcohol solvent which separates the membrane from the anode and cathode layers of the assembly. The resulting solution containing both the polymer membrane and supported noble metal catalysts can be heated under mild conditions to disperse the polymer membrane as particles and the supported noble metal catalysts and polymer membrane particles separated by known filtration means. A related U.S. patent, 7,635,534 was abandoned by BASF. As described in US 7,635,534, the membrane-electrode assembly can also be cooled to temperatures of the order of -75°C and then pulverized to form a powder. The powder is then treated with a concentrated acidic

- DOE. "Platinum Group Metal Recycling Technology Development (New FY2004 Project)." https://www1.eere.energy.gov/hydrogenandfuelcells/pdfs/iva8_robertson.pdf
- 59 Shore, Lawrence. "Platinum Group Metal Recycling Technology Development Final Report." 2009. https://www.osti.gov/servlets/purl/962699
- The USPTO adjusts—i.e., extends—a patent's nominal expiration date when USPTO delays during prosecution (e.g., waiting too long to issue an Office Action) exceed the applicant's delays (e.g., waiting too long to respond to an Office Action).
- 61 "Process for recycling components of a PEM fuel cell membrane electrode assembly." https://patents.google.com/patent/US8124261B2/en

solution, such as HCI/H_2O_2 , or aqua regia, to dissolve the noble metals. The noble metals are then recovered by electrodeposition and / or any other reduction technique. This patent dealt with pulverizing cryo-embrittled assemblies, combining with a surfactant and then acid leaching to recover precious metals. In addition, BASF stated Hammerhead Engineering of Flemington, NJ was commissioned to design a pilot plant.

During this period, the Department of Energy also funded a number of other companies and universities through the Hydrogen program to explore challenges with PEMFC.⁶³ For those interested in reviewing this rich source of information, please review the 2005 Hydrogen Program Progress Report available from this <u>site.</u>

5.2 Hydrofluoric Acid Issues

A review of the available literature indicates that hydrometallurgical and pyrometallurgical procedures are primarily used for the extraction of PGMs from secondary raw materials. It has been reported that the major drawbacks associated with hydrometallurgy arise from the use of strongly basic or acidic media, which can generate toxic compounds such as NOx gases, ⁶⁴ while, at the same time, high pressure (2800-6200 kPa) and high temperature (up to 270 °C) are required. The temperatures employed in pyrometallurgical processes are even higher (up to 800 °C), rendering them expensive and insufficient from an energy consumption perspective.

"Subsequent mutual separation processes, such as solvent extraction, precipitation separation, or ion exchange, target the PGM ions present in solution. However, since PGMs are precious metals, they are sparingly soluble in protonic acids (e.g., hydrochloric acid (HCl)). Therefore, on an industrial scale, acids containing oxidizing agents such as chlorine gas and aqua regia (a mixture of HCl and nitric acid) are employed. While these oxidizing agents have great potential to oxidize PGMs, they are highly toxic and corrosive. and produce large amounts of highly toxic wastes (Hydrofluoric acid, NOx, among others) that had to be further treated 65 In addition, although pyrometallurgical and hydrometallurgical have good performance and recovery ratios, they require a pre-existent or large-scale industrial facility to be economically feasible." 66

Wittock et al in their review of the recycling literature clarify that "Incineration of the fluoropolymers contained in Nafion®, the hydrophobically treated gas diffusion layer and the catalyst layer's binding agent leads to the formation of hydrogen fluoride (HF), a toxic compound that is both harmful to health and highly corrosive. The application of established pyrometallurgical procedures would thus require the costly and time-consuming refurbishment of existing plants with special protective linings for the furnaces, as well as the installation of elaborate filter and scrubbing systems to eliminate HF from

US 7,635,534 "Simplified process for leaching precious metals from fuel cell membrane electrode assem blies."

⁶³ Department of Energy. "Hydrogen Program: 2005 Annual Progress Report."

Granados-Fernández, R.; Montiel, M.A.; Díaz-Abad, S.; Rodrigo, M.A.; Lobato, J. <u>"Platinum Recovery Tech niques for a Circular Economy."</u> Catalysts 2021, 11, 937.

Kuzuhara S, Ota M, Kasuya R. "Recovery of Platinum Group Metals from Spent Automotive Catalysts
Using Lithium Salts and Hydrochloric Acid." Materials (Basel). Nov 12;14(22) 2021.

Granados-Fernández, R.; Montiel, M.A.; Díaz-Abad, S.; Rodrigo, M.A.; Lobato, J. "<u>Platinum Recovery Tech niques for a Circular Economy."</u> Catalysts 2021, 11, 937

the off-gas.⁶⁷ Despite such investments, any fluorine constituents remaining in the PGM-containing slag could hamper the later separation.⁶⁸ In addition, through incineration the valuable membrane material Nafion® is essentially destroyed in the process and cannot be recycled.⁶⁹ From a recycling viewpoint, the absence of fluorine in the membranes would thus be a major advantage of alternative fuel cell systems, such as the PBI/phosphorous acid fuel cells."⁷⁰

6.0 Summary and Conclusions

The purpose of this report was to explore emerging methods of recycling PGMs from fuel cells. The earliest part of the recycling process begins with collection and removal of the components that contains the spent catalyst. With internal combustion engine vehicles the PGMs are located within the catalytic converter; while with FCEVs the platinum group metals are located in the MEA. Given the difference in the complexity of these components and the function that each plays, the collection and separation activities of the supply chain for catalytic converters and FCEVs are very different. The literature reviewed indicates an interest in reusing and refurbishing as much of the PEMFC as possible. Some articles discuss manual separation, while others allude to potentially using automation to disassemble the PEMFC. However, PEMFC vary greatly by manufacturer. For example, Wittstock mentions that some of the manufacturers mold rather than screw shut the fuel stacks and therefore they cannot be simply disassembled by removing the casing and individual layers. Standardization of some components was suggested to facilitate reuse.

The MEA once removed is sent to a processor to extract the PGMs. As noted in the discussion of catalytic converters, a large volume of materials is desired before the processor applies pyrometallurgical techniques (2,000-8,000 lbs). At this time the volumes of MEAs that need recycling are relatively low, even for those companies which introduced PEMFC earlier such as Ballard and Plug Power. Therefore, it would appear that developing and utilizing techniques to efficiently extract PGMs of interest from smaller quantities of MEAs and gradually scaling as demand increases, remains a challenge. With this in mind, Appendix A includes the results of a patent search intended to surface approaches that companies have developed to address this recycling challenge. No conclusions or recommendations are drawn from the patent search. It is provided as a starting point for those interested in conducting research in PGM extraction from MEAs.

⁶⁷ Stolten, D. Hydrogen and Fuel Cells: Fundamentals, Technologies and Applications; John Wiley & Sons Ltd.: Bognor Regis, UK, 2010. [Google Scholar]

Koehler, J.; Zuber, R.; Binder, M.; Baenisch, V.; Lopez, M. Process for Recycling Fuel Cell Components Containing Precious Metals. WIPO Patent No. WO/2006/024507, 9 March 2006. [Google Scholar]

⁶⁹ Schiemann, J.; Kerßenboom, A.; Prause, H.J.; Peil, S. Handbuch Verwertung von Brennstoffzellen und deren Peripherie-Systeme; Institut für Energie- und Umwelttechnik e.V.: Duisburg, Germany, 2007.

[Google Scholar]

Wittsotck, Rikka et al. "Challenges in Automotive Fuel Cells Recycling". Recycling, 1, 3 (2016) 343-364.

APPENDIX A: Patent Search

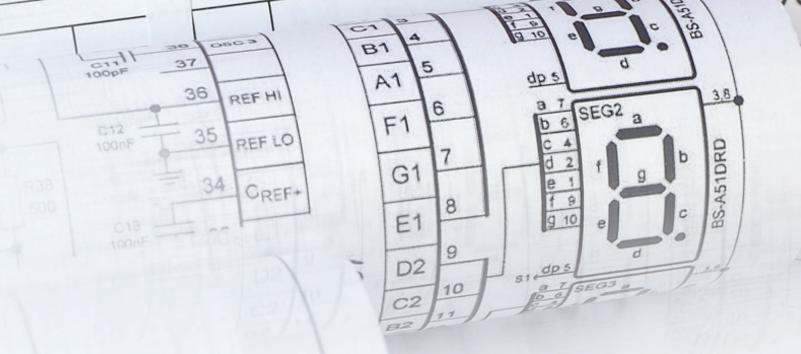
The following section contains the results of a patent search conducted in August 2022, grouped by corporation and/or research institutions. There were multiple objectives for conducting the patent search:

- First, to determine which entities were trying to tackle the problems associated with the efficient recovery/ recycling of PGMs from fuel cells;
- · Second, to categorize the approaches used; and
- Third, to spark continued research by others for improved methods.

Two patent searches were conducted. The first focused on the recovery and recycling of PGMs from fuel-cells, while the second focused on issues related to the formation of HF. There was considerable overlap between the two searches. This search included patents registered under the US Patent & Trademark Office, as well as foreign patents and was conducted by a patent agent using the following search strategies:

```
Search 1 – (recover platinum pefmc) (c22b11)
Search 2 – ((fuel cell)) ((c22b11)) ((recovery) OR (recycle))
Search 3 – (recover platinum mea) (c22b11)
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The corporate and the research sections each begin with a summary table with patents organized by the assignee. Included in this summary table are the types of recovery processes used accompanied by more details in the adjacent column. After the table more detailed information is provided on a subset of these patents – that covered challenges of particular interest. This section includes information on the process of reclaiming one of more of the PGMs. The last Table in the Appendix is a list of patents that address the challenge of hydrofluoric acid.



PEMFC Recycling

Relevant Corporate Patents and Patent Applications

Table 6: List of Corporate Patents Resulting from the Patent Search Companies highlighted in blue are discussed in detail following this table

Company	Title	Method	PEMFC/MEA processed whole?
Company	ritie	Wethou	PEWIFC/WEA processed whole?
Audi Aktiengesellschaft	Method for operating a motor vehicle with a fuel cell device and a motor vehicle	chemical	Process PEMFC/fuel cell stack as whole
Basf Catalysts Llc	Simplified process for leaching precious metals from fuel cell membrane electrode assemblies	mechanical/chemical	MEA pulverized to form powder
Basf Catalysts Llc	Efficient process for previous metal recovery from fuel cell membrane electrode assemblies	mechanical/chemical	MEA ground to powder
Basf Catalysts Llc	Process for recycling components of a PEM fuel cell membrane electrode assembly	chemical/heat	MEA as whole, chemical and heat processing
Basf Catalysts Llc	Method for Recovering Catalytic Elements from Fuel Cell Membrane Electrode Assemblies	mechanical/chemical	MEA converted to particulate, by grinding or granulating
Chengdu Guangming Paite Precious Metals Co., Ltd., Chengdu Guangming Optoelectronics Co., Ltd.	Method for recovering noble metal platinum from proton exchange membrane of hydrogen fuel cell	chemical	MEA dissolved
Daimler Ag	Method for recycling membrane electrode unit of fuel cell for use as alternative energy source for vehicle, involves performing filtration of laden with solvent to obtain filter material, and recovering metals from filter material	ultrasound/mechanical/chemical	MEA crushed into chips, flocs or flakes
Datasi Nantong Information Technology Co	Method for recovering metal platinum from platinum-carbon catalyst/microporous polymer composite membrane	drying/chemical	Disassembling PEMFC, drying MEA, chemical processing of MEA as whole
Datasi Nantong Information Technology Co., Ltd. OR Datax Nantong Information Technology Co., Ltd.	Method for recovering metal platinum from platinum-carbon catalyst	drying/mechanical/chemical	disassembling PEMFC, grinding MEA, chemical processing
Dongguan Zuoyou Electronic Technology Co., Ltd.	A kind of method for reclaiming platinum in waste and old fuel cell	ultrasound/mechanical/chemical	MEA separated from PEMFC, processed in organic solvent, pyrolized, ultrasonically cleaned, soaked in iodized salt solution, leached
Ekpro Gmbh, MAGNUM PIREX AG, DE	Method for recycling electrochemical proton exchange membrane (PEM) fuel cell, involves discharging platinum group metal (PGM) components contained in carrier of fuel cell during electrochemical reaction	electrochemical/chemical	Processing PEMFC/fuel cell stack as whole, with a solvent, electrochemically processing by charging fuel cell electrodes to detach platinum constituents from the MEA
GM Global Technology Operations LLC	METHOD FOR RECYCLING A COATED BIPOLAR PLATE OF STAINLESS STEEL	chemical	not platinum recovery - recovering other metals from the bipolar plates between MEA in a fuel cell stack
Guangdong Bangpu Recycling Technology Co., Ltd.	Method for recycling hydrogen fuel cell of new energy automobile	electrochemical/heat	disassembling PEMFC, heating MEA to ash, chemical processing/leaching
Heesung Metal Co., Ltd.	Method for collecting High-Purity Platinum from MEAmembrane electrode assembly used Ultrasonic	ultrasound/mechanical/chemical	MEA processed in organic solvent, followed by ultrasonic separation of PT catalyst
Heesung Metal Co., Ltd.	Method and apparatus for recovery of platinum in MEA	chemical	MEA processed in organic solvent
Heesung Metal Co., Ltd.	Recovery method of platinum group metals	chemical/heat	MEA pulverized, processed in organic solvent
Heesung Metal Co., Ltd.	Method for retrieving platinum of mea	chemical	MEA processed in organic solvent

Table 6: List of Corporate Patents Resulting from the Patent Search Companies highlighted in blue are discussed in detail following this table

Heesung Metal Co., Ltd.	Method for recovering platinum from spent membrane-electrode assembly	chemical/heat	MEA pyrolyzed, chemical processing
Heesung Metal Co., Ltd.	Method for collecting alloy-metal from MEA		MEA processed in organic solvent, followed by ultrasonic separation of PT catalyst
Hubei Huadelai Energy Saving and Emission Reduction Technology Co., Ltd.	Method for preparing nano catalyst by utilizing platinum group metal in exhaust gas treatment catalyst	mechanical/chemical	waste gas treatment catalyst - no mention of how the waste catalyst is created
Ion Power, Inc.	Recycling of used perfluorosulfonic acid membranes	chemical/heat	Recycling of used catalyst-coated perfluorosulfonic acid membranes by dissolving the used membranes in water and solvent, heating the dissolved membranes under pressure and separating the components.
Jiangsu Yaoyang New Energy Technology Co., Ltd., Jiangsu Hydrogen Pure Energy Technology Co., Ltd.	Recovery processing method of fuel cell stack material	chemical/heat	disassembling PEMFC, chemically/heat processing the MEA to obtain a Nafion slurry
Neo DM Co., Ltd.	Method for recovering platinum from spent catalyst and manufacturing method of platinum chloride using the same	chemical	chemical processing of "spent catalyst" - no mention of how catalyst is received
Robert Bosch GMBH	Method and apparatus for recovering gold, silver and platinum metals	chemical	particulate starting materials processed in electrolyte solution processing
Robert Bosch GMBH	Method and apparatus for recovering gold, silver and platinum metals	chemical	particulate starting materials processed in electrolyte solution processing
Robert Bosch GMBH	Process for obtaining platinum and/or ruthenium	chemical/ultrasound	MEA can be treated as a whole without prior crushing in the process since crushing takes place by the ultrasonic treatment.
Robert Bosch GMBH	Method for recovering material that constitutes fuel cell stack	chemical/heat	Process PEMFC/fuel cell stack as whole
Robert Bosch GMBH	Process for recovering gold, silver and platinum metals from components of a fuel cell stack or an electrolyzer	chemical	Process PEMFC constituents as a whole
Robert Bosch GMBH	Method for obtaining platinum and/or ruthenium	mechanical/chemical	MEA crushed to powder then chemical treatment
Toyota Motor Corp	Method and apparatus for recovering catalyst for fuel cell	chemical/magnetic	MEA pulverized
Umicore Ag & Co. Kg	Process for recycling fuel cell components containing precious metals	chemical	MEA pulverized
Umicore Ag & Co. Kg	Process for the concentration of noble metals from fluorine-containing fuel cell components	heat/chemical	MEA melted
Yeda Research and Development Co. Ltd.	Method for platinum group metals recovery from spent catalysts		crushed material
Yeda Research and Development Co. Ltd.	Method for platinum group metals recovery from spent catalysts		crushed material
Yushi Energy (Nantong) Co., Ltd.	Method for recovering key material from fuel cell membrane electrode	chemical	MEA soaked then crushed

Corporations

Audi Ktiengellschaft

Title: Method for operating a motor vehicle with a fuel cell device and a motor vehicle				
Patent or Patent Application Number: DE102020106563A1	Assignee or Applicant: Audi AG Inventors: Markus RUF, Kai Muller, Rene Stemeile	Publication Date: September 16, 2021		

Abstract: The invention relates to a method for operating a motor vehicle with a fuel cell device (1) having at least one fuel cell (2), comprising the steps of acquiring and evaluating the data of at least one sensor monitoring an aging condition of the fuel cell (2), and in the event of a determination reaching the end of the service life of the fuel cell (2) based on the data from the sensor, changing from a normal mode for operating the fuel cell (2) to a recycling mode in which the fuel cell (2) is supplied with a fuel in a sub-stoichiometric ratio. The invention also relates to a motor vehicle with a fuel cell device (1).

BASF Catalysts, LLC

TITLE: Simplified process for leaching precious metals from fuel cell membrane electrode assemblies				
Patent or Patent Application Number: 7,635,534 B2	Assignee or Applicant: BASF Catalysts LLC Inventors: Lawrence Shore, Ramail Matlin	Publication Date: December 22, 2009		

Abstract: The assemblies are cryogenically embrittled and pulverized to form a powder. The pulverized assemblies are then mixed with a surfactant to form a paste which is contacted with an acid solution to leach precious metals from the pulverized membranes.

TITLE: Efficient process for previous metal recovery from cell membrane electrode assemblies				
Patent or Patent Application Number: 7,709,135 B2	Assignee or Applicant: BASF Corporation Inventors: Lawrence Shore, Ramail Matlin, Robert Heinz	Publication Date: May 4, 2010		

Abstract: The method includes grinding the membrane electrode assembly into a powder, extracting the catalytic element by forming a slurry comprising the powder and an acid leachate adapted to dissolve the catalytic element into a soluble salt, and separating the slurry into a depleted powder and a supernatant containing the catalytic element salt. The depleted powder is washed to remove any catalytic element salt retained within pores in the depleted powder and the catalytic element is purified from the salt.

TITLE: Process for recycling components of a pem fuel cell membrane electrode assembly

Patent or Patent
Application Number:

8,124,261 B2

Assignee or Applicant:
BASF Corporation
Name: Lawrence Shore

Publication Date:
February 28, 2012

Abstract: The membrane electrode assembly (MEA) of a PEM fuel cell can be recycled by contacting the MEA with a lower alkyl alcohol solvent which separates the membrane from the anode and cathode layers of the assembly. The resulting solution containing both the polymer membrane and supported noble metal catalysts can be heated under mild conditions to disperse the polymer membrane as particles and the supported noble metal catalysts and polymer membrane particles separated by known filtration means.

TITLE: Method for recovering catalytic elements from fuel cell membrane electrode assemblies				
Patent or Patent Application Number: 8,206,682 B2	Assignee or Applicant: Name: Lawrence Shore, Ramail Matlin, Robert Heinz	Publication Date: June 26, 2012		

Abstract: A proton exchange membrane of the hydrogen fuel cell is placed into a first dissolving container, and hydrochloric acid and hydrogen peroxide are adopted to dissolve the proton exchange membrane under specific process conditions, so that the volatilization of tiny precious metal platinum particles attached to the surface of the proton exchange membrane of the hydrogen fuel cell can be effectively prevented, and the recovery rate of the platinum can be improved; the dissolved residue dissolved for the first time is dissolved again after being sintered, so that the noble metal platinum in the proton exchange membrane of the hydrogen fuel cell can be effectively recovered;

Relevance:

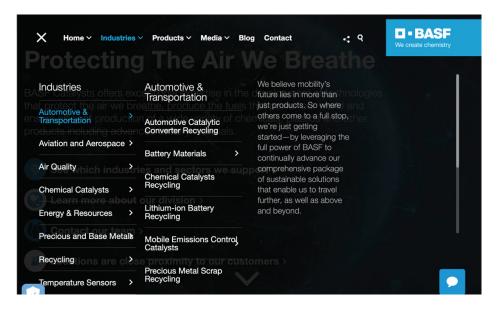
The inventor, Lawrence Shore from BASF Catalysts, LLC. was the technology lead DOE Project DE-FC36-03G01314 between 2003 and 2009. The Project title was <u>V.F.1: Platinum Group Metal Recycling Technology Development</u>. The objective of the study was to develop a new process for previous metal recovery from polymer electrolyte membrane, membrane electrode assemblies (MEAs), while eliminating hydrogen fluoride emission, a disadvantage of the current combustion-based recycling process. The full Technical Report for this project is available <u>here.</u>

Related publications by the inventor can be found at these references.

OSTI.gov. Precious Metal Recovery from Fuel Cell MEAs, 2004. The conference presentation is available from this <u>site</u>.

Shore, Lawrence. "An Environmentally-Friendly Process for Fuel Cell Electrode Reclamation". OSTI

Corporation: BASF Catalysts



BASF's Catalysts division, headquartered in Iselin, New Jersey, USA, is a global division of BASF SE, Ludwigshafen, Germany, and is the world's leading supplier of environmental and process catalysts. The division employs more than 5,000, with over 30 manufacturing sites worldwide. BASF's Catalysts division is the global market leader in catalysis.

BASF is highly specialized in this industry, as we both <u>produce</u> and recycle catalytic converters. As the inventor of the first catalytic convert in the 1970s, we are well-versed in every stage of a catalyst's lifecycle and are dedicated to providing our customers with that expertise. As precious metal contents vary by material, BASF has developed precise analytical and statistical ISO accredited methods, to provide our customers with a high degree of accuracy across material types.

BASF makes PEM fuel cells. See <u>Celtec-P membrane electrode assemblies for high-temperature PEM fuel cells</u>

Daimler AG

To see an English translation of this World patent, which is printed in German, please select the hyperlink under Patent number from the table below and scroll down to read the English translation.

Title: Method of recycling membrane/electrode units of a fuel cell			
Patent or Patent Application Number: W02015010793A2	Assignee or Applicant: Daimler Ag Inventor Name: Markus Paepke	Publication Date: World patent (German) January 29, 2015	

Abstract: The invention relates to a method for recycling membrane/electrode units (MEA) of a fuel cell, comprising proton-exchange membrane material (PEM) and gas-diffusion layers (GDL), said PEM containing a perfluorsulfonic acid polymer (PFSA) and precious metal, under the effect of ultrasound and in the presence of a solvent for separating the precious metal from the fluorine-containing membrane material by means of a filtration process, having the following steps: - comminuting the precious metal-loaded MEA into chips, flocs, or flakes; - transporting the comminuted precious metal-loaded MEA into an ultrasonic bath filled with a solvent (LM) made of aqueous ethanol; - subjecting the MEA membrane material to an ultrasonic treatment in the solvent (LM), said precious metal passing into the solvent (LM); - separating the fluorine-containing membrane material (PFSA) from the precious metal-loaded solvent (LM); - feeding the fluorine-containing membrane material (PFSA) to a thermal or material recovery process; - filtering the precious metal-loaded solvent (LM) and obtaining a precious metal-loaded filter material; and - recovering the precious metal from the loaded filter material using a conventional thermal method.

Corporation:

71

The former **Daimler AG** is now the **Mercedes-Benz Group AG**. Although the company introduced a passenger FCV in 2017, in 2020 they announced that they would cease production of Hydrogen GLC F-Cell FCV and instead will join forces with the Volvo Group in developing a hydrogen fuel cell heavy-duty vehicle designed for long-haul operation.⁷¹

Datasi Nantong Information Technology Co.

Title: Method for recovering metal platinum from platinum-carbon catalyst/microporous polymer composite membrane			
Patent or Patent Application Number: CN112342396A	Assignee or Applicant: Datasi Nantong Information Technology Co Itd Inventors:	Publication Date: February 9, 2021	

Abstract: The invention provides a method for recovering metal platinum from a platinum-carbon catalyst/microporous polymer composite membrane, which is particularly suitable for small-scale recovery of waste MEA in a fuel cell laboratory or a factory.

Mercedes-Benz Ceases Production of Hydrogen GLC F-Cell FCV.

Ekpro Gmbh, Magnum Pirex AG, DE

Title: Method for recycling electrochemical proton exchange membrane (PEM) fuel cell, involves discharging platinum group metal (PGM) components contained in carrier of fuel cell during electrochemical reaction

Patent or Patent Application Number: DE102012109063A1	Assignee or Applicant: MAGNUM PIREX AG, DE Inventors: Sven Jakubith, Stefan Nettesheim	Publication Date: March 28, 2013
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Abstract: The method involves supplying solvents over the external plates (9,10) of fuel cell (7). The PGM components contained in the carrier of fuel cell are discharged during electrochemical reaction. The discharged PGM components are delivered from fuel cell through electrode compartments (2,3) such that particles of discharged metal components are separated from the porous solvents such as alcohol or liquid.

GM Global Technology Operations, LLC

Title: Fast recycling process for ruthenium, gold, and titanium coatings from hydrophilic PEM fuel cell bipolar plates			
Patent or Patent Application Number: US8323415B2	Assignee or Applicant: GM Global Technology Operations Inventors: Mahmoud H Abd Elhamid; Youssef M. Mikhail; Richard H. Blunk	Publication Date: December 4, 2012 (Adjusted expiration)	

Abstract: A method for recovering ruthenium oxide or gold and titanium or titanium oxide from a bipolar plate at the end of the life of a fuel cell stack so as to use these materials in other fuel cell stacks thereafter. The bipolar plate is immersed in a solution including a suitable acid that dissolves the titanium or titanium oxide. The ruthenium oxide or gold will be released from the plate and will float on the solution from which it can be removed. The solution is then heated to evaporate the acid solution leaving a powder of the titanium oxide. The stainless steel of the bipolar plate is thus cleaned of the titanium or titanium oxide, and can be reused.

Related Inventors research:

See Google Scholar for Mahmoud H. Abd Elhamid

Heesung Metal Company, Ltd.

To see an English translation of this World patent, which is printed in Korean, please select the hyperlink under Patent number in the table below and scroll down to read the English translation. The inventors names are not listed.

Title: Method for collecting high purity platinum from mea membrane electrode assembly using ultrasonic Patent or Patent Application Number: KR101167669B1 Assignee or Applicant: Heesung Metal Company Ltd. Publication Date: July 20, 2012

Abstract: The present invention relates to a method for recovering platinum (Pt), in particular, by separating the platinum catalyst electrode and Nafion from the waste electrode assembly (MEA) of the fuel cell using an organic solvent, and then dispersing the platinum catalyst using ultrasonic waves. The present invention provides a method for recovering the total amount of platinum used in a fuel cell by increasing reactivity and selectively dissolving and reducing platinum in the platinum catalyst.

Title: Method and apparatus for recovery of platinum in MEA		
Patent or Patent Application Number: KR101371078B1	Assignee or Applicant: Heesung Metal Company Ltd.	Publication Date: March 10, 2014

Abstract: The present invention relates to a method and a device for recovering platinum (Pt), and in particular, to facilitate separation of the MEA and the platinum catalyst from the spent MEA of the fuel cell through a solvent and agitation, the organic solvent used An apparatus and a method for recovering are provided.

Title: Recovery of metal group metals			
Patent or Patent Application Number: KR101527655B1	Assignee or Applicant: Heesung Metal Company Ltd.	Publication Date: June 9, 2015	

Abstract: The present invention relates to a method for separating and recovering a platinum group metal. The method includes the steps of: S1) pretreating a waste including one or more types of a platinum group metal to obtain a mixture where one or more types of the platinum group metal are mixed; and S2) plasma-treating the mixture to recover one or more types of a platinum group metal powder.

Title: Method for retrieving platinum of MEA		
Patent or Patent Application Number: KR20130013449A	Assignee or Applicant: Heesung Metal Company Ltd.	Publication Date: February 6, 2013

Abstract: A method for collecting the platinum of an MEA (Membrane Electrode Assembly) is provided to improve the separation efficiency of a platinum electrode, to be capable of massively separating in a short time, to minimize a loss by implementing an efficient separation, thereby shortening a processing time. CONSTITUTION: A method for collecting the platinum of an MEA separates a platinum catalyst from a fuel cell MEA scrap using an organic solvent and pure water. The method for collecting the platinum of an MEA includes: a step of supplying a first solution(methanol, ethanol, propanol, iso-propanol, buthanol, pentanol, hexanol, hepthanol, acetone, tetrahydrofurane, and so on) for separating the platinum catalyst and nafion; a step of supplying a reacting time for the first solution for 0.1 to 10 minutes; a step of supplying a second solution(pure water, distilled water) after reacting the MEA with the first solution; and a step of using jigs formed at regular intervals and a robot. The method for collecting the platinum of an MEA further includes: a step of separating the platinum catalyst from the second solution using an air bubbling and stirring at more than 1000 rpm (revolutions per minute); and a step of manufacturing a third solution by implementing an electrode-dispersion on the second solution at 50 to 100 MHz(megahertz). The method for collecting the platinum of an MEA further includes a shower device for separating the platinum catalyst after manufacturing the third solution. [Reference numerals] (101) Organic solvent vessel; (102) Pure water vessel 1; (103) Pure water vessel 2; (301) MEA fixing jig; (302) MEA charging position

Title: Method for recovering platinum from spent membrane-electrode assembly		
Patent or Patent Application Number: KR20160136633A	Assignee or Applicant: Heesung Metal Company Ltd.	Publication Date: November 30, 2016

Abstract: The present invention relates to a method for recovering platinum from a spent membrane-electrode assembly (MEA), wherein the method comprises the steps of: (a) obtaining platinum group metal-including residues by pyrolyzing a spent membrane-electrode assembly including a platinum group metal; (b) separating a platinum group metal hydrochloric acid solution by dissolving the platinum group metal-including residues in aqua regia; (c) forming platinum group metal salts by neutralizing the platinum group metal hydrochloric acid solution with a neutralizing agent; and (d) sintering the platinum group salts. Thereby, the method for recovering platinum from a spent membrane-electrode assembly (MEA) according to the present invention is safe and environment-friendly, and displays a high recovery rate.

Patent or Patent Application Number: KR101312086B1 Assignee or Applicant: Heesung Metal Company Ltd. Publication Date: September 27, 2013

Abstract: The present invention relates to a method for recovering platinum (Pt), cobalt (Co) and palladium (Pd), in particular an electrode containing a platinum / cobalt alloy and a platinum / palladium alloy from waste MEA (Membrane Electrode Assembly) of a fuel cell. After separating through a solvent and an ultrasonic wave, and selectively separating platinum, cobalt and palladium using an acid treatment and a reducing agent in the electrode containing platinum, thereby providing a method for recovering the entire valuable metal used in the fuel cell.

Corporation:

Product Special Features and Merits or



Heesung Metal Co., Ltd. is engaged in manufacturing precious metal, thin film and functional Nano material used in semiconductor, automobile, and display industries. The company is based in Incheon, South Korea with branches in China, Hong Kong, and Taiwan. Heesung Metal Co.,Ltd. was incorporated in 1993 formerly known as Heesung Metal Ltd. It appears that Heesung Metal Co established a branch in China which uses the name **LT Metal**. Their platinum recovery process is described **here**.

Ion Power

Title: Recycling of used perfluorosulfonic acid membranes		
Patent or Patent Application Number: U.S. 7,255,798 B2 US7255798B2	Assignee or Applicant: Ion Power Inventors: Stephen Grot, Walter Grot	Publication Date: August 14, 2007

Abstract: A method for recovering and recycling catalyst coated fuel cell membranes includes dissolving the used membranes in water and solvent, heating the dissolved membranes under pressure and separating the components. Active membranes are produced from the recycled materials.

Related Inventor's Research:

Stephen A. Grot, Ph.D. See ResearchGate list of publications

Corporation

Ion Power is a small business founded in 1999 and located in New Castle, DE. Ion Power Inc. develops, manufactures, and distributes membrane electrode assemblies to the fuel cell and water electrolyzer markets. An interesting note: "Nafion is a brand name for a sulfonated tetrafluoroethylene-based fluoropolymer-copolymer invented in the late 1960s by his father, Walther Grot of DuPont."⁷²

Robert Bosch GMBH

Title: Method for the recovery of platinum and/or ruthenium		
Patent or Patent Application Number: EP3957759A1	Assignee or Applicant: Robert Bosch GMBH Inventors: Ingo Kessel, Christian Diessner, Claudio Baldissone, Fabian Haemmerle	Publication Date: Pending

Abstract: The present invention relates to a method for obtaining platinum and/or ruthenium. This comprises introducing (11) at least one starting material (20) which contains platinum and/or ruthenium and at least one polyfluorinated polymer or copolymer in water or an aqueous first solution (30), comminuting (12) the starting material by means of ultrasound (40) and introducing (14) ozone (61) into a suspension containing the comminuted starting material (20) in an aqueous second solution (50) in which hydrogen chloride (51) is dissolved.

Frank, Walter. "Up and running: Ion Power Inc., bringing new jobs, innovation to Tyrone" Altoona Mirror. December 29, 2019.

Title: Method for recovering gold, silver, and platinum metals from components of a fuel cell stack or of an electrolyzer Patent or Patent Application Number: US20210310098A1 Assignee or Applicant: Robert Bosch GMBH Inventors: Torsten Trossman, Claudio Baldissone

Abstract: A method for recovering gold, silver, and/or platinum from components of a fuel cell stack of a fuel cell or electrolyzer includes treating the components with an aqueous electrolyte solution and with at least one gaseous oxidant in the fuel cell or the electrolyzer in an oxidation step. In a reduction step, the components are treated with a flow of an aqueous electrolyte solution and with at least one gaseous reductant in the fuel cell or the electrolyzer. A device by which the method can be carried out has a reservoir for the electrolyte solution, a line connected to an outlet opening of the reservoir, the line having a pump, an anode inlet connection connected to an anode inlet, and a cathode inlet connection connected to a cathode inlet. An oxidant-introducer introduces a gaseous oxidant into the line. A reductant-introducer introduces a gaseous reductant and/or inert gas into the line.

Title: Method for obtaining platinum and/or ruthenium		
Patent or Patent Application Number: W02021089233A1	Assignee or Applicant: Robert Bosch GMBH Inventors: Alexander Eifert Claudio Baldissone	Publication Date: Pending

Abstract: The present invention relates to a method for obtaining platinum and/or ruthenium. Said method comprises the provision of a solution of ethanol in water (11), the introduction of at least one starting material (20) into the solution (12), the introduction of HCl into the solution (14) and a multiple introduction of ozone into the solution (16).

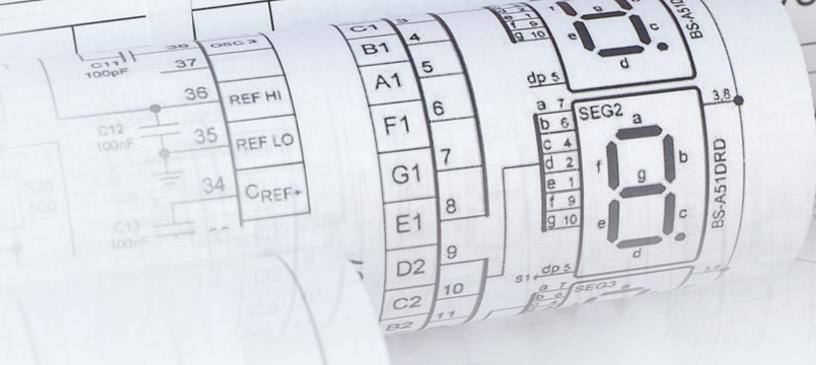
Title: Method of recovering materials forming fuel cell stack			
Patent or Patent Application Number: US20210296658A1	Assignee or Applicant: Robert Bosch GMBH Inventors: Shogo Takamuku, Claudio Baldissone, Katsuhiko Oshikawa	Publication Date: September 23, 2021	

Abstract: To recover materials forming a fuel cell stack by an easy method. Provided is a method of recovering, from a fuel cell stack having a stack structure including a plurality of fuel cells stacked, materials forming the fuel cell stack, the fuel cells each including a membrane electrode assembly and two separators holding the membrane electrode assembly therebetween, the separators each being provided with a gas flow channel configured to supply a raw material gas to the membrane electrode assembly, the method including: a first step of supplying a solvent or a solvent and a reagent to the fuel cell stack through the gas flow channel, collecting the solvent which contains a material, and recovering the material from the collected solvent; and a second step of subjecting the fuel cell stack after the first step to a heat treatment to obtain a molten liquid or gas and recovering a material from the molten liquid or gas, the materials recovered including materials forming the membrane electrode assembly and the separators.

Toyota Motor Company

Title: Method and system for recovering catalyst for fuel cell		
Patent or Patent Application Number: US7553793B2	Assignee or Applicant: Toyota Inventors: Kazuhiro,Tasniwaki	Publication Date: June 30, 2009

Abstract: A method for recovering a catalyst for a fuel cell includes a collection step in which a catalyst is collected by attracting, using a magnetic force, a magnetic material contained in at least one of the catalyst and a carrier on which the catalyst is supported.



PEMFC Recycling

Relevant Patents and Patent Applications from Research Institutions

Table 7: List of Patents Resulting from the Patent Search for Research Institutions Research institutions highlighted in blue are discussed in detail following this table

Assignee	Title	Method	PEMFC/MEA processed whole?
Commissariat à l'Energie Atomique et aux Energies Alternatives	electrode assembly	mechanical/chemical	membrane-electrode assembly in a water/alcohol
Commissariat à l'Energie Atomique et aux Energies Alternatives	fuel cell	chemical	MEA processed in acid/oxidant agent
Commissariat à l'Energie Atomique et aux Energies Alternatives	AN ELECTRICALLY INSULATING SUPPORT	chemical	MEA immersed in ionic liquid
Commissariat à l'Energie Atomique et aux Energies Alternatives	containing same, with an extraction medium consisting of a	chemical	pressurized dense fluid containing an organic ligand
Dalian Jiaotong University	waste fuel batter with proton exchange film	ultrasound/mechanic	catalyst containing platinum
Korea Institute of Geoscience and Mineral Resources	Method for recycling phosphoric acid type fuel cell waste electrode including dry process phosphoric acid recovery method	chemical	dry chemical method - MEA appears to be processed intact
Korea Institute of Geoscience and Mineral Resources	Method for recycling phosphoric acid type fuel cell waste electrode including wet process phosphoric acid recovery method	chemical	chemical leaching - figures imply the MEA is chopped before processing
Korea Institute Of Geoscience And Mineral Resources	Method of recovering acid and platinum group metal from leaching solution of spent catalyst	chemical	unclear whether 'spent catalyst' is process whole
Korea Research Institute of Chemical Technology	Method for recycling platinum from platinum based catalysts	chemical	chemical processing of "platinum based catalyst" - no mention of how catalyst is received
Korea Research Institute of Chemical Technology	Method for seperating platinum group metals using solvent extraction	chemical	MEA processed in aqua regia
Wuhan University of Technology	Method for recovering catalyst palladium in proton exchange membrane fuel cell	chemical	MEA processed whole-immersed in organic solvent
Wuhan University of Technology	High-efficiency and environment-friendly recovery method of noble metal palladium in retired solid oxide fuel cell	mechanical/chemical	disassembling and crushing the retired solid oxide fuel cell
Wuhan University of Technology	Method for recovering platinum catalyst of waste proton exchange membrane fuel cell	electrochemical	possibly processing MEA whole or in particulate form

Commissariat à L'energie Atomique et Aux Energies Alternatives

To see an English translation of this patent, which is printed in French, please select the hyperlink under Patent number in the table below and scroll down to read the English translation.

Title: Method for recovering the platinum found in a membrane-electrode assembly		
Patent or Patent Application Number: EP3000902B1	Assignee or Applicant: Commissariat a lEnergie Atomique et aux Energies Alternatives CEA Inventors: Denis Vincent, Emmanuel BILLY, Richard Laucournet	Publication Date: March 30, 2016

Abstract: Separating the polymer electrolyte membrane from the first electrode and from the second electrode so as to make the catalyst layers accessible - performing leaching on at least the catalyst layers so as to dissolve at least a part of the platinum, the leaching being performed with an acid solution having an acid concentration lower than or equal to 1mol/L, the leaching solution being a mixture of water and HNO3/HCl, the leaching being performed at a temperature comprised between 70°C and 90°C.

Title: Process for the recovery of platinum and cobalt from fuel cells		
Patent or Patent Application Number: EP3574122B1	Assignee or Applicant: Centre National de la Recherche Scientifique CNRS, Institut Polytechnique de Grenoble Universite Grenoble, Alpes, Commissariat a lEnergie Atomique et aux Energies Alternatives CEA Inventors: Rémi VINCENT, Raphaël CHATTOT, Laëtitia DUBAU, Lucien DUCLOS, Lenka SVECOVA, Pierre- Xavier THIVEL	Publication Date: December 4, 2019

Abstract: Processing of the EMA by lixiviation producing a lixiviate containing platinum ions and cobalt ions b) extraction from the lixiviate of platinum ions and cobalt ions c) separation of the cobalt ions from the platinum ions by the formation and precipitation of cobalt oxide.

Title: Method for recovering platinoid particles contained in an electrically insulating support		
Patent or Patent Application Number: EP3815762A1	Assignee or Applicant: Centre National de la Recherche Scientifique CNRS, Universite Claude Bernard Lyon 1 UCBL, CPE- Lyon-FCR, Commissariat a IEnergie Atomique et aux Energies Alternatives CEA Inventors: Emmanuel BILLY, Mathias COUDRAY, Véronique DUFAUD, Paul- Henri Haumesser, Hakima Mendil, Catherine Santini	Publication Date: May 5, 2021

Abstract: A method of recovering platinoid particles (122) contained in an electrically insulating carrier, the method comprising an extraction step in which the electrically insulating carrier containing the platinoid particles is immersed in an ionic liquid solution comprising a first ionic liquid, so as to extract the particles (122) of platinoid from the electrically insulating support.

Title: Method of selective extraction of platinoids, from a support containing same, with an extraction medium consisting of a supercritical fluid and an organic ligand

Inventors: Frédéric Goettmann, Audrey Hertz, Sandrine Mongin
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Abstract: The present invention relates to a method of selective extraction of a platinoid, from a ceramic support containing a metal, comprising the following successive steps: a) the support is brought into contact, with an extraction medium consisting of a pressurized dense fluid containing an organic ligand that is selective for the metal and that is capable of forming a complex with the metal in the 0 state; whereby a ceramic support depleted in the metal, or even free of the metal, and, a medium consisting of the fluid containing the complex of the organic ligand with the metal in the 0 state are obtained; b) the fluid is brought back to atmospheric pressure and to ambient temperature, whereby the complex separates from the fluid; c) the ceramic support depleted in the metal, or even free of the metal, and the complex, are recovered.

Korea Institute of Geoscience and Mineral Resources

To see an English translation of this patent, which is printed in Korean, please select the hyperlink under Patent number in the table below and scroll down to read the English translation. The inventors' names are not listed.

Title: Method for recycling phosphoric acid type fuel cell waste electrode including dry process phosphoric acid recovery method		
Patent or Patent Application Number: KR102284346B1	Assignee or Applicant: Korea Institute of Geoscience and Mineral Resources	Publication Date: August 4, 2021

Abstract: The present invention is a recycling method that does not use aqua regia. More specifically, the present invention provides a recycling method of a waste electrode of a phosphoric acid-type fuel cell including a dry-type phosphoric acid recovery method, in which phosphoric acid is recovered by dry heat treatment, and is oxidized with a high content of oxygen in a state in which a carbon-platinum (Pt-C) electrode and silicon carbide, a separator component, are mixed, and recovers most of platinum group elements and silicon carbide and produces no waste.

Title: Method for recycling phosphoric acid type fuel cell waste electrode including wet process phosphoric acid recovery method		
Patent or Patent Application Number: KR102284348B1	Assignee or Applicant: Korea Institute of Geoscience and Mineral Resources	Publication Date: August 4, 2021

Abstract: The present invention is a recycling method that does not use aqua regia. More specifically, the present invention provides a method for recycling a phosphoric acid-type fuel cell waste electrode including a wet-type phosphoric acid recovery method, which is a recycling method that recovers most of the platinum group elements and silicon carbide powder and produces no waste, in which phosphoric acid is recovered by wet leaching and oxidation process is performed with high oxygen content in a state where a carbon-platinum (Pt-C) electrode and silicon carbide, a component of the separator, are mixed.

Title: Method of recovering acid and platinum group metal from leaching solution of spent catalyst			
Patent or Patent Application Number: US10190192B2	Assignee or Applicant: Korea Institute of Geoscience and Mineral Resources KIGAM Inventors: Jin-Young Lee, Kyeong-Woo CHUNG	Publication Date: April 14, 2016	

Abstract: The present invention relates to a method of recovering acid and a platinum group metal from a leaching solution of a spent catalyst, more particularly, to a method of recovering acid and a platinum group metal from a leaching solution of a spent catalyst, the method including: filtering a leaching solution of a spent catalyst, providing the filtered leaching solution into a concentration chamber, and heating the filtered leaching solution to recover acid included in the leaching solution; providing a concentrated solution of the leaching solution into a substitution chamber after recovering the acid, and adding a metal for a substitution reaction; and cleaning a solid, which is separated by solid-liquid separation after the substitution reaction, with acid and recovering the platinum group metal.

Korea Research Institute of Chemical Technology

To see an English translation of this patent, which is printed in Korean, please select the hyperlink under Patent number in the table below and scroll down to read the English translation. The inventors' names are not listed.

Title: Method for recycling platinum from platinum-based catalysts		
Patent or Patent Application Number: KR101226946B1	Assignee or Applicant: Korea Research Institute of Chemical Technology	Publication Date: March 14, 2012

Abstract: The present invention relates to a method for recovering platinum from a platinum-based catalyst, and more particularly, to prepare a platinum chloride solution by adding aqua regia to a platinum-based spent catalyst, and then adding acetone together with triphenylphosphine to form tetrakis. It relates to a method for recovering platinum by precipitating (triphenylphosphine) platinum (Tetrakis (triphenylphosphine) platinum). Platinum recovery method of the present invention has no chlorine gas discharge as compared to the conventional method for recovering platinum by firing ammonium chloroplatinic acid, it is possible to recover the platinum at a calcination temperature of $380 \sim 500 \,^{\circ}\text{C}$ mild conditions. In addition, the recovered platinum can be applied again to the production of platinum-based catalysts, etc., and thus it is possible to cope with the increasing demand for platinum.

Title: Method for separating platinum group metals using solvent extraction		
Patent or Patent Application Number:	Assignee or Applicant: Korea Research Institute of	Publication Date: November 13, 2013
KR20130123921A	Chemical Technology	

Abstract: The present invention relates to a method for separating platinum group metals by using a solvent extraction method which comprises the steps of: (1) separating an electrolyte membrane of a membrane electrode bond from a gas dispersion layer; (2) separating platinum and ruthenium by immersing the gas dispersion layer into aqua regia; and (3) increasing the purity at least one between the platinum and the ruthenium by using an extract and a diluent after adding the aqua regia to at least one between the extracted platinum and ruthenium. The method for separating platinum and ruthenium from the membrane electrolyte bond according to the present invention is used to efficiently and economically separate the platinum and the ruthenium.

Syddansk Universitet

Title: Method for recovering platinum group metals from catalytic structures		
Patent or Patent Application Number: US9580826B2	Assignee or Applicant: Syddansk Universitet Inventors: Eivind Skou, Casper Noergaard, Serban Nicolae Stamatin	Publication Date: February 4, 2016

Abstract: A method for recovering platinum group metals from a catalytic structure, such as a fuel cell membrane electrode assembly, involving dissolution of the platinum group metal by treating the catalytic structure in an electrolytic cell with a suitable electrolyte containing a complexing agent and introducing an electric current into the electrolytic cell; and subsequently re-precipitating the platinum group metal by increasing the pH of the electrolyte system and adding a reducing agent.

Wuhan University of Technology

To see an English translation of these patents which are printed in Chinese, please select the hyperlink under Patent number in the table below and scroll down to read the English translation. The inventors' names are not listed.

Title: Method for recovering catalyst palladium in proton exchange membrane fuel cell		
Patent or Patent Application Number: CN110172580B	Assignee or Applicant: Wuhan University of Technology WUT	Publication Date: August 27, 2019

Abstract: The invention discloses a method for recovering catalyst palladium in a proton exchange membrane fuel cell, which comprises the following steps: disassembling the proton exchange membrane fuel cell subjected to the brine discharge treatment to obtain a membrane electrode, and immersing the membrane electrode in an organic solvent; separating solid on the upper layer of the suspension by ultrasonic oscillation, filtering the separated suspension by a screen, filtering the filtered suspension by filter paper to obtain filter residue, soaking the filter residue in an acid solution to obtain the suspension, and filtering to obtain the filter residue; washing the obtained filter residue with deionized water, drying, soaking in nitric acid solution to obtain suspension, and filtering; dropwise adding an alkali solution into the obtained filtrate to completely precipitate palladium, and dropwise adding a hydrogen peroxide solution to reduce palladium hydroxide into palladium; and centrifuging the obtained solid-liquid mixture, and filtering to obtain the metal palladium. The method for recovering the catalyst palladium in the proton exchange membrane fuel cell provided by the invention has the advantages of simple process, less pollution and higher recovery rate.

Title: High-efficiency and environment-friendly recovery method of noble metal palladium in retired solid oxide fuel cell		
Patent or Patent Application Number: CN112522516B	Assignee or Applicant: Wuhan University of Technology WUT	Publication Date: March 19, 2021
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Abstract: The invention discloses a high-efficiency and environment-friendly recovery method of noble metal palladium in a retired solid oxide fuel cell, which comprises the steps of disassembling and crushing the retired solid oxide fuel cell, placing the disassembled and crushed retired solid oxide fuel cell in octylphenol polyoxyethylene ether to remove organic substances, washing and drying; slightly burning the powder obtained in the last step, soaking in a eutectic solvent for a period of time, filtering, and washing filter residues with distilled water for multiple times; placing the filter residue in prepared [Hbet] [Tf2N] ionic liquid for Pd leaching; after leaching is finished, a three-electrode system is constructed by taking leaching solution as electrolyte to carry out Pd electrodeposition, after the deposition is finished, the deposition solution is placed in a centrifugal device to be separated to obtain Pd, and the Pd is washed and dried to obtain the simple substance Pd. The method avoids using a large amount of acid and alkali, and can recover and obtain the noble metal Pd with higher purity on the basis of simple process and environmental protection.

Title: Method for recovering platinum catalyst of waste proton exchange membrane fuel cell			
Patent or Patent Application Number: CN112593084B	Assignee or Applicant: Wuhan University of Technology WUT	Publication Date: April 2, 2021	

Abstract: The invention relates to a method for recovering a waste proton exchange membrane fuel cell platinum catalyst. The method comprises the following steps: 1) platinum in the catalyst layer was electrolyzed using cyclic voltammetry: the catalyst layer is used as a working electrode, a carbon rod is used as a counter electrode, an Ag/AgCl electrode is used as a reference electrode, and a eutectic solvent is used as electrolyte for electrolysis; wherein the potential window is set to be 1-2V; 2) and (3) electrodeposition reaction: and after electrolysis, replacing the catalyst layer with an electrode made of a conductive material, setting a potential window to be-3 to-2V, and performing electrodeposition reaction for multiple times under an ultrasonic condition to obtain the metal platinum. The platinum metal particles recovered by the method of the invention have uniform size, no other metal impurities and high purity.

 Table 8: List of Patents that Address Hydroflouric Acid Gas

Link	Assignee	Comments re: HF emissions/treatment
US7255798B2	Ion Power	In certain embodiments, the disclosure includes processes that allow for the re-manufacture of new catalyst coated membranes (CCMs) from used CCMs extracted from failed fuel cell stacks. This may be accomplished by removing the CCM from the stack, decontaminating the CCM to remove impurities, and then <u>dissolving</u> the <u>ionomer component of the CCM to form a slurry of dissolved PFSA[™] together with the Pt/C catalyst particles</u> . The dissolution may, in certain embodiments, be done at increased pressure in an autoclave, for example. Preferred embodiments include a pressure of from 500 to 2000 psi. These two valuable ingredients are then separated, allowing the PFSA [™] solution to be reprocessed into a new fuel cell membrane. Ideally the recovered catalyst (Pt/C) is redeposited on the re-manufactured membrane so that a completely re-manufactured CCM is the final result. The same process would be used for an end-of-life Chlor-alkali membrane where the separation of the fiber reinforcement and other solids are separated by similar methods.
US8101304B2	Umicore	The hydrogen fluoride formed during the heat treatment of fluorine-containing components is bound by an inorganic additive so that no harmful hydrogen fluoride emissions occur.
US7713502B2	Umicore	a process for recycling fuel cell components containing fluorine-containing and precious metal-containing constituents: in this process, the fluorine-containing constituents are separated off from the precious metal-containing constituents by treatment with a medium present in the supercritical state. Preference is given to using water as supercritical medium. After the fluorine-containing constituents have been separated off, the precious metal-containing residues can be recovered in a recycling process without harmful fluorine or hydrogen fluoride emissions. The fluorine-containing constituents can likewise be recovered.
US7635534B2	BASF Catalysts	The bulk of the membrane electrode assembly is carbon-based; therefore, a standard method to recycle precious metals, including platinum, involves a combustion step to remove carbon material. However, membrane electrode assemblies have high fluorine content due to polytetrafluoroethylene (PTFE) impregnated on the carbon fibers and from common polymer electrolyte membrane materials, such as Nafion® (DuPont Co., Wilmington, Del.), which results in a large, undesirable discharge of HF upon combustion. Removal of HF gas involves scrubbing and dedicated equipment that can withstand the corrosive nature of HF gas. Isolating the combustion from existing infrastructure is recommended to localize maintenance needs caused by the effects of HF gas.
<u>US7709135B2</u>	BASF Catalysts	Regardless which vessel or apparatus is used to filter the solids from the leachate solution, the corrosive nature of the slurry requires materials of construction that can withstand corrosion. FIG. 5 illustrates the corrosion of various materials, in percent weight loss, when exposed to chlorine. A Hastelloy C276 metal coupon was tested and corroded badly. Glass lined vessels, such as those fabricated by De Ditrich and Pfaudler, have been shown to fare better when exposed to acid, but such vessels are typically limited to pressures of less than about 10 bar (150 PSIG). For example, according to vessel manufacturer data, a 20% HCl mixture at 160° C. corrodes away about 20 mils per year from a glass liner. However, trace HF from the degraded perfluoropolymer membrane could attack the glass liner. The addition of dispersed silica acts as a fluorine getter to protect the glass liner, and testing has shown that 100 ppm of SiO ₂ added to the mixture reduces the glass liner corrosion to about 2 mils per year at 160° C. Boric acid can also be used as a fluorine getter.
US8206682B2	BASF Corp	to optimize the recovery of catalytic elements from a fuel cell MEA, the efficiency of the leaching process can be improved based on parameters including, but not limited to, the leach medium, the concentration and quantity of leach medium per weight of catalytic element sought to be recovered, and the temperature, pressure, and cycle time of the leach step or steps. In the experiments discussed herein, leaches have been performed in several reactor vessels, including open glass beakers and sealed fluorinated polymer vessels.

KR102284348B1	Korea Institute of Geoscience and Mineral Resources	As a recycling method of a waste electrode of a phosphoric acid type fuel cell that recovers phosphoric acid by wet method, recovers platinum group elements and silicon carbide (SiC), and does not emit waste, leaching the waste electrode of the phosphoric acid type fuel cell to wet-recover the phosphoric acid; an oxidation step of removing carbon by oxidizing the waste electrode of the phosphoric acid type fuel cell obtained by wet recovery of the phosphoric acid; and Leaching the oxidized phosphoric acid type fuel cell waste electrode with a mixture of an acid compound and a halogen gas to separate the silicon carbide (SiC) and the platinum group element, and to recover the silicon carbide (SiC) and the platinum group element
KR102284346B1	Korea Institute of Geoscience and Mineral Resources	As a recycling method of a waste electrode of a phosphoric acid type fuel cell that recovers phosphoric acid by dry method, recovers platinum group elements and silicon carbide (SiC), and does not emit waste, dry-recovering the phosphoric acid by heat-treating the waste electrode of the phosphoric acid type fuel cell at a low temperature; an oxidation step of removing carbon by oxidizing the waste electrode of the phosphoric acid type fuel cell obtained by dry recovery of the phosphoric acid; and Leaching the oxidized phosphoric acid type fuel cell waste electrode with a mixture of an acid compound and a halogen gas to separate the silicon carbide (SiC) and the platinum group element, and to recover the silicon carbide (SiC) and the platinum group element
CN112421067B	JIANGSU YAOYANG NEW ENERGY TECHNOLOGY CO LTD	Since the fluorine proton exchange membrane releases hydrofluoric acid harmful to human body during combustion, calcium oxide must be used for adsorption to remove fluorine, thereby increasing the complexity of the process.
JP2005289001A	Toyota Motor Co	To efficiently recover a precious metal and a fluorine-containing polymer without using a solvent or the like from a membrane-electrode bonded element (MEA) of a used solid polymer electrolyte fuel cell. SOLUTION: The membrane-electrode bonded element (MEA) is constituted of an electrolyte membrane comprising the fluorine-containing polymer having a sulfonic acid group, a conductive carrier which is bonded to the electrolyte membrane and carries a catalyst metal, and a gas diffusing electrode (b) of which the major constituent material is a catalyst layer comprising a proton conductive polymer. The precious metal and/or the fluorine-containing polymer having the sulfonic acid group are recovered from the membrane-electrode assembly (MEA) by this recycling method. The recycling method has two processes, (1) a process in which the membrane-electrode assembly (MEA) is solidified, and the electrolyte is expanded to a degree wherein the plastic deformation of the catalyst layer after the solidification becomes easier, (2) a process in which the membrane-electrode assembly (MEA) after the electrolyte has been expanded is solidified while imparting a stress. By these processes, the electrolyte membrane comprising the fluorine-containing polymer having the sulfonic acid group, and the catalyst layer comprising the conductive carrier carrying the catalyst metal and the proton conductive polymer are made easily separable.
US20140056786A1	Heraeus Precious Metals GmbH	Ashing plant for enriching noble metals from fluorine-containing materials, comprising a thermal treatment chamber (1) having a refractory insulating lining on the inside of the thermal treatment chamber (1), and an exhaust gas cleaning system, whereby the insulating lining is resistant to hydrofluoric acid and the exhaust gas cleaning system comprises at least one or more acid scrubber(s) (3, 4) and at least one alkaline scrubber (5).
US20210047708A1	Battelle Energy Alliance LLC - NOTE, funded by DOE	a method of recovering palladium from a palladium-containing material includes exposing the palladium-containing material to a <u>leaching solution</u> including an acid, an oxidizer, and iron ions (e.g., ferric ions (Fe ³⁺), ferrous ions (Fe ²⁺)) dissolved therein. The acid may include a source of halide ions. For example, the acid may include one or more of hydrochloric acid, hydrofluoric acid, hydrobromic acid, or hydroiodic acid.
US10964967B2	Johnson Matthey Hydrogen Technologies	A process for the <u>recovery of a perfluorosulphonic acid ionomer</u> from a component comprising a perfluorosulphonic acid ionomer is disclosed, the process comprising immersing the component comprising the perfluorosulphonic acid ionomer in a solvent comprising an aliphatic diol and heating. Also disclosed is the use of the recovered perfluorosulphonic acid ionomer, for example in to prepared a proton conducting membrane or a catalyst ink.