TITLE: End-of-life of Fuel Cell and Hydrogen products: A state of the art

AUTHORS: Ana María Ferriz^{1*}, Alfonso Bernad¹, Mitja Mori², Sabina Fiorot³.

¹Fundación para el Desarrollo de las Nuevas Tecnologías del Hidrógeno en Aragón, PT.Walqa Ctra. N330A km 566 22197 Huesca (Spain)

²University of Ljubljana – Faculty of Mechanical Engineering, Aškerčeva 6, 1000 Ljubljana (Slovenia).

³Environment Park S.P.A. Via Livorno, 60 10144 –Turin (Italy)

**Corresponding Author:*

Telephone: 0034 974 215 258

Fax: 0034 974 215 261

E-mail: aferriz@hidrogenoaragon.org

ABSTRACT:

The Fuel Cells and Hydrogen (FCH) technologies will play an important role in a future where greenhouse gasses emissions need to be reduced. Nevertheless, a huge implementation of these technologies must be addressed taking into account an eco friendly scope not only from the manufacturing perspective but also from the end-of-life scope.

A classification of the materials has been done considering the importance of each of them. To obtain a complete overview of the problem, different criteria have been compared: the cost, the scarcity of the material and the affections these materials can cause not only to the environment but also to the humans. This classification has been used to identify which are the most critical materials.

Moreover, other transversal issues have been studied as the regulations that apply to FCH technologies from a paneuropean perspective and the strategies to face the end-of-life of these equipments. A holistic point of view has been considered in order to see how the process of dismantling and recycling faces different problems and which milestones to achieve in a future with a deep market penetration are.

KEYWORDS: recycling, fuel cell, hydrogen, electrolyzer, materials.

1. Introduction

The EC has established its energy targets at a 40% reduction of greenhouse gas emissions compared to 1990 levels, 27% of renewable energy consumption and 27% of energy savings [1] where FCH technologies have the potential to decarbonize the EU transport and energy sectors and will play a key role by means of substitution of pollutant applications.

In this sense, a penetration of around 9% - 13% of the total fleet in Europe by 2030 [2] is expected at the same time as a European market of hydrogen fuel cell vehicles of more than 135 million units by 2050. Hydrogen as an energy vector will work in synergy with other technologies; storage of renewable energies being chemical energy storage a reliable solution (the underground storage in Europe (EU-28) have a capacity of approximately 1,200 TWh [3]), allowing the storage of surpluses of electricity for long periods of time and in high capacity, balancing then the use of Variable Renewable Energies (VRE) by means of pure hydrogen or power to gas, reducing also the GHG emissions, generating at the same time large amounts of “green” hydrogen; or considering the implementation of more efficient devices with zero emissions (more than 50,000 stationary units until 2017[4] and 200,000 units, also in Europe in the same time horizon. On the other hand, to cover all the hydrogen demand that these

applications will require, an installed power of electrolyzers between 90 and 140 GW is estimated at that time.

However, the development of these technologies must also include their recycling and dismantling or possible future uses, a fundamental aspect for which neither the whole of technology nor society is being prepared in a future context of complete commercialisation. These devices include a number of critical materials for its operation. In particular, Platinum Group Metals (PGM) and Rare Earths Elements (REE) are used in the catalysts, electrodes and electrolytes of fuel cells and electrolyzers [5].

Furthermore, there is a widespread lack of specific end-of-life strategies focused on reusing products, i.e. saving materials and taking advantage of still valuable components and subsystems. It is necessary to apply innovative recycling processes that involve both manufacturers of the hydrogen technology sector (by redesigning products that seek greater recovery of critical materials and greater compatibility with recycling and dismantling) and end users, in order to ensure collaboration and supply of products within a methodological framework of reverse logistics.

Moreover, one of the key aspects for a complete market development of these technologies is a specific legislation regarding the end-of-life of these technologies. In this context, this work analyzes these underdeveloped aspects, identifying and discussing all the needs and barriers that impede the complete deployment of FCH technologies, seeking to anticipate the implementation of the technology and facilitate the development of future real implementation actions and advances in legislation on the recycling and dismantling of hydrogen technologies, including both hydrogen production systems by electrolysis and its use in integrated fuel cells.

2. Methodology

Looking for the best way to analyze the end-of-life of the FCH technologies, the work has been split into three different tasks: identification of critical materials, strategies and technologies for the end-of-life and a study of the regulation, needs and challenges of this stage. The technologies considered in this study have been: Proton Exchange Membrane Fuel Cells (PEMFC), Solid Oxide Fuel Cells (SOFC), Proton Exchange Membrane Water Electrolyzers (PEMWE) and Alkaline Water Electrolyzers (AWE).

Identification of critical materials has focused on the analysis of the materials for each of the technologies studied, identifying the component in which the material is located, the name and its classification [6], [7]. Once the identification of all the materials is finished, materials are classified according to three different criteria: hazardousness, scarcity and cost.

A hazard material is the one whose properties when it is considered waste make it dangerous or capable of having a harmful effect on human health or the environment. The Priority List of Hazardous Substances [8], [9], [10] and report from Robert A. Goyer and Thomas W. Clarkson about toxic effects of metals [11] have been took as reference.

Scarcity or criticality refers to their insecurity on the supply and their economic and technological importance. Resources or materials are considered 'scarce' or 'critical' when there is a high demand from industry combined with a risk to their supply. To define scarcity, the EU methodology has been used, considering two assessment components: economic importance and supply risk [12], [13],[14]. The result is a relative ranking of the materials across the two assessment components, with a material defined as critical if it exceeds both the threshold for

economic importance and the supply risk. As a result 27 raw materials are considered as critical by the EU Commission as shown in Table 1.

Cost is related to the price or value of the material. The London Metal Exchange [15] has been taken as reference, thus the materials have been classified into three different groups: low cost materials, those with a price lower than 5 \$/kg, medium cost between 5 and 500 \$/kg and high cost are those whose price is higher than 500 \$/kg.

Subsequently, the technologies and the possible strategies in their implementation during the end-of-life have been studied. The first approach has been understand how the current technologies works, task which has been done in cooperation with recycling centers from batteries and Waste of Electrical and Electronic Equipment (WEEE). After this part, how the process should be in order to recycle the FCH has been designed. Finally, this task has developed a thorough literature review on novel EoL technologies applicable to FCH products. A distinction has been made between EoL technologies for stack components and those for balance-of-plant (BoP) components.

Finally, a study in the regulations, needs and challenges in this end-of-life has been covered. FCH technologies have not any specific regulation in terms of recycling, so each step from the manufacturing to the end-of-life should be study in detail and compared with the regulations that apply to other technologies that can be similar to fuel cells and electrolyzers. The material selection, the different subsystems, and the future end-of-life and disposal techniques fall under different rules and impose barriers that need to be overcome or considerations to take in the manufacturing phase.

Due to it, and looking for a common point among all the European Union (EU), this stage has been based in a review of the main Directives from the EU that apply for electronics, vehicles, EoL, and all the applications of the FCH technologies, looking for regulative barriers in each case that need to be overcome in the future.

Moreover, and out of the legal scope, the role of the actors and problems related with them has been covered, taking special attention to the users and the Extended Responsibility of the Producer (ERP) organisations going through literature reviews and calls with the recycling sector members.

3. Results and Discussion

3.1 Critical materials

The PEMFC have as main elements a conductive electrolyte, the cathode and the anode, forming the so-called set MEA (Membrane Electrode Assembly). The gas is distributed on both sides by the bipolar plates, which in turn perform the functions of current collectors, which is why they must be good conductors of electricity.

Electrochemical reactions occur in the electrodes, generally formed by carbon paper or paper where a catalytic layer of platinum or platinum alloy is applied.

As for the materials used in the membrane, sulfonated polymers are generally used to contain enough water with high proton conductivity. The most common is perfluorosulfonic acid (PFSA). The list of materials for PEM fuel cells is included in Table 2.

SOFC high temperature fuel cells are composed of two porous electrodes separated by a dense oxygen ion conductive electrolyte.

The anode must be stable in the reduction of the fuel, must be an electrical conductor and have sufficient porosity to allow the exit of products from the oxidation reaction. They are generally composed of compound powder mixtures such as YSZ (Yttria-Stabilized Zirconia), GDC (Gadolinium-Doped Ceria) or SDC (Samarium-Doped Ceria) and nickel oxide. On the cathode side, however, compounds are used that are very robust to temperature changes, which allow them to adapt to changes in the electrolyte, such as Lanthanum-doped Calcium Manganite (LCM) or Strontium-doped Lanthanum Manganite with (LSM) [16].

The electrolyte must be impermeable to gases to avoid mixing, which motivates the use of structures such as YSZ or ceria doped with rare earths (REE), among others, as shown in Table 3 [17], [18].

Polymer electrolyzers (PEMWE) are very similar to fuel cells of the same type; however, certain elements such as those based on carbon, as for example the catalytic layers in which the catalyst is supported, cannot be used on the anode side of the PEMWE due to the high oxygen concentrations and the high voltages. For this reason, metallic elements based on titanium or alloys are usually used. The detail of all the materials is shown in Table 4.

Finally, as regards to AWE, nickel or nickel alloys are the most commonly used materials in electrodes due to their balance between corrosion resistance, high conductivity and reduced relative cost. The electrolyte is basically formed by polymers such as PFSA or PTFE. Table 5 provides detailed information for AWE.

3.2 Strategies and technologies

Based on the identification of materials that has been carried out, the techniques associated to the recycling of each of the technologies have been differentiated [19].

Regarding PEMFC, the main processes have been defined to recover stack materials. In this type of fuel cells the recycling is focused on critical materials, using two different methods: hydrometallurgical which involves the dissolution of interesting elements from solid matrices through acid or caustic attacks followed by separation via solvent extraction, precipitation, cementation, ion exchange, filtration or distillation. Hydrometallurgical processes to recover PGM from the catalyst layer of PEM stacks (fuel cells or electrolyzers) or from the spent catalyst of SOFC reformers involve leaching, separation and purification sub-processes. Further sub-processes (e.g., regeneration of solvents) may be involved in order to improve the economic and environmental performance of the whole process [20], [21]. The complete processes for PEMFC are shown in Figure 1.

The second one is pyrometallurgical which involves directly a combustion process in which the membrane, GDLs and electrodes are incinerated. The resulting ashes are processed through dissolution and Pt is then precipitated. When compared to hydrometallurgical processes, pyro-hydrometallurgical process present higher recovery efficiency but higher energy demand. This will also include drying and calcination, leaching and purification, and reduction and filtration.

When talking about AWE, two are the main products that could be recovered; the Nickel and the common plastics. For Nickel a hydrometallurgical process is used to treat the spent Raney-Ni catalyst. The process involves leaching with dilute sulphuric acid to produce nickel sulphate and a low percentage of alumina, followed by purification to produce pure NiCO_3 . The treatment of common plastics is the second one; the PTFE scrap is ground into fine powder and then blended with pure PTFE. To remove the inorganic compounds, the scrap is heated before

grinding. Only the extruded forms can be recycled and separated from undesirable impurities. It is then put into a long strand, which is subsequently cut down into small pieces and sent to industries to be used as recycled material for their products.

Finally, about the SOFC the recovery of the anode catalyst in SOFCs is based on the reaction of nickel oxide with sulphuric acid to form nickel sulphate similar to AWEs. For the cathode side in the tubular cells strontium-doped lanthanum manganite should be the one to be recycled, however, no existing recycling process are available. About the planar ones, the best benefits will be obtained on recycling the steel interconnect plates by hydrothermal treatment. Finally regarding ceramic materials the high-temperature environments required during cell fabrication promote the migration of chemical species, and the presence of contaminants –even in trace amounts– leads to performance degradation. Thus, the implementation of ceramic waste recycling processes associated with the SOFC technology is not expected in the near future.

Once the techniques for each material have been established, their combination has been analyzed in search of the best possible strategies for the complete recycling of the fuel cells and electrolyzers studied. In Figure 2 the case for the PEM type fuel cells is shown presenting also the new technologies covered by the literature review, and giving different ways to act or proceed in the recycling phase of the FCH technology.

3.3 Regulation, needs and challenges

Manufacture of the FCH technologies is indeed the stage which allows introducing the biggest amount of changes. In this phase, the REACH Directive [22] and the EcoDesign Directive [23] should be considered. The REACH Directive applies to the materials and, as the case of nickel and nickel alloys, can be a future cause of the prohibition of these materials. The EcoDesign Directive considers not only the design but also the materials selections and the good techniques as standardization and optimization of the manufacturing process. It applies to all energy products, even if FCH technologies are not mentioned.

For specific subsystems as inverters and DC/DC components, specific regulations apply. RoHS Directive [24] drives the manufacturers to avoid the use of some components as lead, mercury. Moreover, the WEEE Directive [25] is related to electric and electronic parts in a fuel cell system, and also to the whole fuel cell system. This Directive imposes a recycling target and due to it, this will cause future problems if the new technologies are neither ready nor available. It also introduces the Extended Responsibility of the Producers and classifies all the equipment into different groups. It should be noted that the whole system may fall under this directive, and in some cases, a fuel cell could be considered a battery under the definition of the Batteries Directive [26] difference that need to be clarified if future problems as double targets wants to be avoided.

Moreover, these specific recycling targets will vary if the fuel cell is part of a FCEV because it will be considered a part falling under the End-of-life Vehicles (ELV) Directive [27]. Due to it, the manufacturers will need to consider these targets and design equipments that are able to be recycled covering that targets.

Finally, in the end-of-life of the FCH technologies, multiples Directives apply and should be properly considered. Hazardous waste Directive [24], Landfill Directive [28] and the Batteries Directive [26] may apply to all the components or just to some subsystems, which implies that

mainly hazardous materials should be avoided and minimized. The Landfill Directive also affects to the recycling center, which should pre-treat the waste prior to the disposal. Nevertheless, the manufacturer is still the member of the supply chain that has more capacity to avoid the problems via the material selection at the same time that looks for an intermediate point between profitability, efficiency, ecodesign and circular economy.

In addition to all the barriers or legal considerations previously presented, the end users and the recycling philosophy play an important role. The end users, which will be the first actor in the recycling route, must be awarded and conscious of the concerns that an improper recycling strategy can cause. Nevertheless as Keramisoglou [29] explained, the recycling behavior is influenced by factors like financial incentives, information and infrastructure.

Following that lines, the challenge is to create the proper recycling infrastructure. A solution will be an Extended Responsibility of the Producer (ERP) system where a Producer Responsibility Organization (PRO), evolving from individual agreements between the recycling centers and the manufacturers, which will be the responsible for the transport of the FCH waste, to a detailed structure which will work with the municipalities, with a detailed logistic infrastructure, and make the complete process easy for the end user.

Taxes and specific aids to manufacturers, recycling centres and logistic companies will be also needed in the future, just in the cases when the recycled process is not profitable, in order to accomplish the regulations and the principles of circular economy. Nevertheless, these taxes should be studied and defined in detail for each specific case.

4. Conclusions

Further efforts are highly required in order to avoid that the end-of-life of FCH technologies hampers their complete deployment and commercialization due to the current underdevelopment observed in terms of end-of-life techniques, strategies, and regulation. This work may serve as a starting point in this direction. Some specific conclusions are:

- There is a possible ratio to improve the design of the FCH technologies from the EoL perspective, going through a reduction of high cost, hazard and scarce materials. Nevertheless, this improvement needs to be aligned with maintenance of the efficiency of the equipments.
- Even if the recycling process is feasible nowadays, improvements on the efficiency are possible. Nevertheless, this work only presents the new process in the lab scale, being necessary study how to scale this new processes and then, comparing them with the current technologies, in order to see how the global process vary.
- Legal framework needs improvements in order to cover properly the FCH technologies and their applications. Recycling targets and how this regulation affects to the FCH technologies, with proper clarification of the status, ERP systems and waste position needs to evolve in the future if the FCH market pretends to increase.

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2017 CRMs (27)			
Antimony	Fluorspar	LREEs	Phosphorus
Baryte	Gallium	Magnesium	Scandium
Beryllium	Germanium	Natural graphite	Silicon metal
Bismuth	Hafnium	Natural rubber	Tantalum
Borate	Helium	Niobium	Tungsten
Cobalt	HREEs	PGMs	Vanadium
Coking coal	Indium	Phosphate rock	

Table 1. 2017 critical raw materials list.

Polymer Electrolyte Membrane Water Electrolyzer (PEMWE)	Component	Material	Material classification	Material value	Material Criticality
	Electrolyte	Perfluorosulphonic acid (PFSA)	Non-hazardous	Medium	Medium
		Sulfonated polyether ether ketone (s-PEEK)	Non-hazardous	Medium	Low
	Catalyst layer - Cathode	Pt or Pt-alloys	Non-hazardous	High	High
	Catalyst layer- Anode	Iridium and Ir-alloys	Hazardous (irritant, harmful)	High	High
		Ruthenium and Ru-alloys	Hazardous (toxic, carcinogen)	Medium	High
	Anode and Cathode - GDL	Thermally sintered Ti	Non-hazardous	Low	Medium
		Ti or stainless steel mesh	Non-hazardous	Low	Medium
		Graphite or graphite composites (only possible on cathode side)	Non-hazardous	Low	High
	Interconnect	Coated titanium or Ti-alloys	Non-hazardous	Low	Medium
	Sealant	Thermoplastic	Non-hazardous	Low	Low
		Elastomer	Non-hazardous	Low	Low

Table 2. List of common materials in PEMFC.

Solid Oxide Fuel Cells (SOFC)	Component	Material	Material hazardousness	Material value	Material Criticality
	Electrolyte	Yttria-stabilised zirconia	Non-hazardous	Medium	High
	Anode	Nickel-based oxide doped with YSZ	Hazardous (Cat. 1 carcinogen)	Medium	High
		Nickel	Hazardous (Cat. 1 carcinogen)	Medium	High
	Cathode	Strontium-doped lanthanum manganite	Hazardous (Irritant)	Medium	High
	Interconnect	Doped lanthanum chromate	Hazardous (Irritant, harmful)	Medium	Medium-High
		Inert metals/alloys	Non-hazardous	High	Medium-High
	Sealant	Glass/Glass-ceramic	Non-hazardous	Low	Low
		Mineral	Non-hazardous	Low	Low
		Precious metals	Non-hazardous	High	High
	Substrate	Ceramic	Non-hazardous	Low	Low

Table 3. List of common materials in SOFC.

Polymer Electrolyte Membrane Water Electrolyzer (PEMWE)	Component	Material	Material classification	Material value	Material Criticality
	Electrolyte	Perfluorosulphonic acid (PFSA)	Non-hazardous	Medium	Medium
		Sulfonated polyether ether ketone (s-PEEK)	Non-hazardous	Medium	Low
	Catalyst layer - Cathode	Pt or Pt-alloys	Non-hazardous	High	High
	Catalyst layer- Anode	Iridium and Ir-alloys	Hazardous (irritant, harmful)	High	High
		Ruthenium and Ru-alloys	Hazardous (toxic, carcinogen)	Medium	High
	Anode and Cathode - GDL	Thermally sintered Ti	Non-hazardous	Low	Medium
		Ti or stainless steel mesh	Non-hazardous	Low	Medium
		Graphite or graphite composites (only possible on cathode side)	Non-hazardous	Low	High
	Interconnect	Coated titanium or Ti-alloys	Non-hazardous	Low	Medium
	Sealant	Thermoplastic	Non-hazardous	Low	Low
		Elastomer	Non-hazardous	Low	Low

Table 4. List of main materials in PEMWE.

Alkaline Water Electrolyzer (AWE)	Component	Material	Material classification	Material value	Material Criticality
	Electrolyte	Potassium Hydroxide	Hazardous (corrosive)	Medium	Low
	Anode	Precious metals	Non-hazardous	High	High
		Plastic	Non-hazardous	Low	Low
	Cathode	Raney-Nickel	Hazardous (carcinogen)	Medium	High
		Plastic	Non-hazardous	Low	Low
	Interconnect	Plastic	Non-hazardous	Low	Low
	Sealant	Thermoplastic	Non-hazardous	Low	Low
		Elastomer	Non-hazardous	Low	Low
	Diaphragm (membrane)	Asbestos	Hazardous (carcinogen)	Low	Low
		Polymers	Non-hazardous	Medium	Low

Table 5. List of main materials in AWE.

Figure 1. Hydrometallurgical Pt-recovery process (based on [20]).

Figure 2. EoL scheme of PEM systems: hydrometallurgical route.