

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



## TRAINING MODULE 2

### Chapter\_4

Description and manual to perform the novel and optimized recovery & recycling technologies (SOFC) (Cathode recovery)

#### Document Details

Prepared by	ENVI
Input from	POLITO

#### Document Details

- x PU - Public
- CO - Confidential, only for members of the consortium (including the EC)



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe Research.



## Abbreviations

FC	Fuel cells
SO	Solide Oxide
Co	Cobalt
La	Lanthanum
Ni	Nickel
YSZ	Yttria-stabilised zirconia
POLITO	Politecnico di Torino
Ni-GDC	nickel-gadolinium doped ceria
LSM	lanthanum manganite
LSCF	strontium-doped lanthanum cobaltite ferrite



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.



## Contents

1	Executive Summary .....	4
2	Introduction .....	5
3	Overview of full SOFC EoL Cell, anode and cathode recovery process .....	6
3.1	LSC cathode removal .....	7
3.2	Lanthanum and Cobalt recovery process .....	8
4	References.....	10



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.



# 1 Executive Summary

Within project BEST4Hy, a training kit is to be developed. This will be subdivided into 4 Modules with this content:

- Module 1: How to dismantle a fuel cell
- Module 2: Recycling technologies
- Module 3: Technical results and economical aspects
- Module 4: Measures towards to take up.

Module 2 is dedicated to the recycling technologies, providing knowledge on the adaptation and designing of existing and novel processes and an overview of the processes developed within the project.

Module 2 should be organized as follow:

- Chapter 1: Introduction to the current existing recovery & recycling technologies
- Chapter 2: Description and manual to perform the existing and optimized recovery & recycling technologies (PEMFC);
- Chapter 3&4: Description and manual to perform the novel recovery & recycling processes developed within the project (PEMFC/SOFC).

Video tutorials on how to perform the recovery and recycling processes above described are also available. For more information, users can consult the associated deliverables describing the activities performed in BEST4Hy:

- D1.2: Technical report on the adaptation of existing technology (hydrometallurgy process) for PEMFC material recovery: results and design
- D1.5: Pilot-scale plant (TRL5) based on 3 recycling technologies for PEMFCs
- D2.3: Report on the evaluation of MEA including recycled materials in a small single cell of PEMFC
- D2.6: Report on the evaluation of MEA including recycled materials in PEMFC stack\_PU
- D3.3: Pilot-scale plant (TRL5) based on two integrated existing recycling technologies for SOFCs\_PU
- D3.5: Technical report on open loop analysis of different scenarios
- D4.3: Technical report on developed recovery technologies for LSC cathode materials\_PU

The present document refers to Chapter 4: Description and manual to perform the novel and optimized recovery & recycling technologies (SOFC) (Cathode recovery).



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.



## 2 Introduction

This chapter focus on the recovery and recycling processes of critical materials and rare earths from the anode component of Solid Oxide Fuel Cells (SOFCs).

Well-established technologies for recovery and recycling of end-of-life SO fuel cells still do not exist. Project BEST4Hy developed a process for the recovery of the anode supported electrolyte by adaptating and combining existing technologies based on hydrothermal (HT) and hydrometallurgical (HMT) methods. An efficient and scalable recovery strategy for End-of-Life solid oxide fuel cells has therefore been defined. The current chapter reports achievements of recovery and recycling process at laboratory scale.

Within project BEST4Hy, research activities have been carried out by Politecnico di Torino with the support of Elcogen as provider of EoL fuel cells materials. More details on the topics explained in this chapter are available in the following public deliverable:

D4.3: Technical report on developed recovery technologies for LSC cathode materials\_PU



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.

### 3 Overview of full SOFC EoL Cell, anode and cathode recovery process

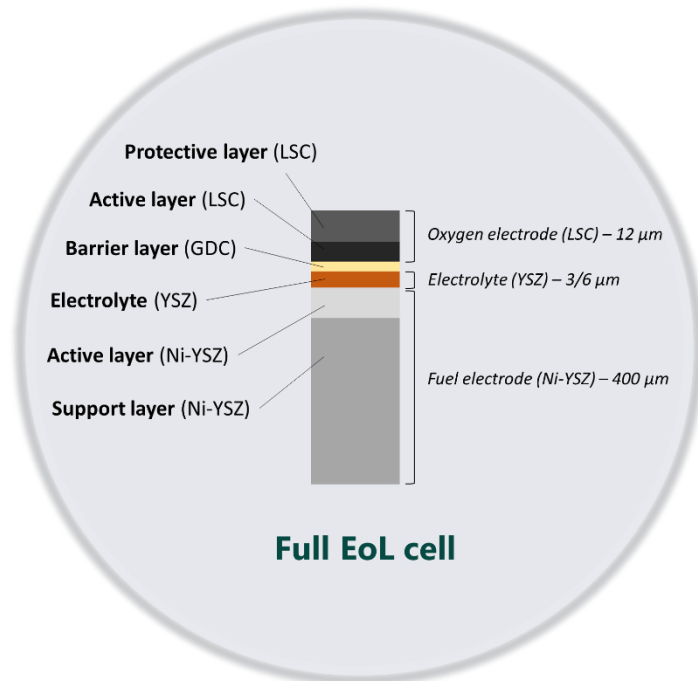


Figure 1 Full SOFC EoL Cell

Protective layer (LSC)   **Electrode (LSC)**   Protective layer (GDC)   Electrolyte (YSZ)   Active layer (Ni-YSZ)  
Support layer (Ni-YSZ)

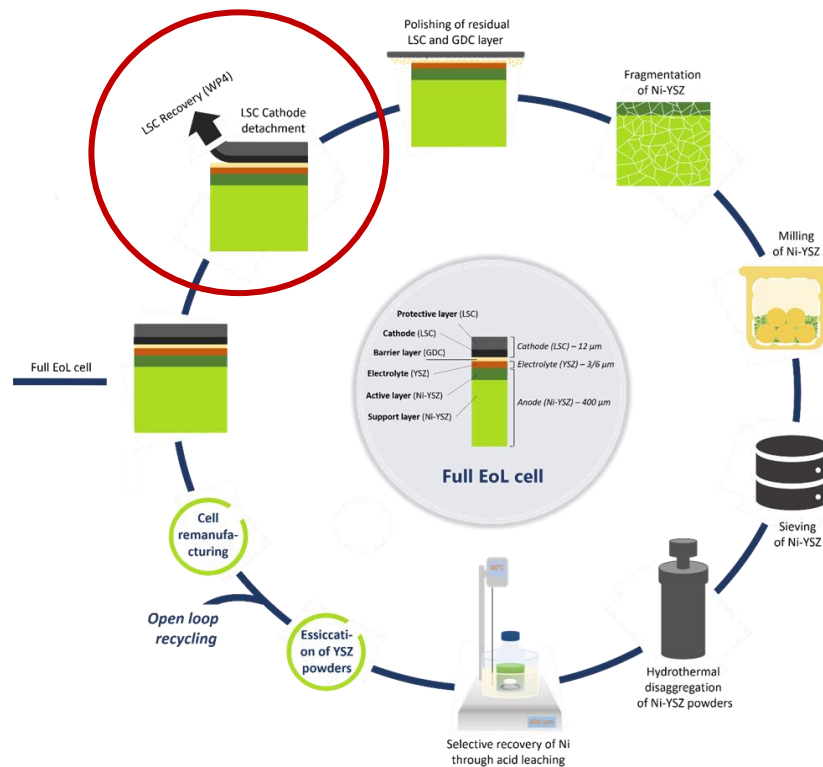


Figure 2 Schematic outline of the pathway followed for the recovery of YSZ and Ni from

Figure 2 - from Module 2 Chapter 3 - shows the multi-step recovery procedure for the EoL cells, including the cathode layer removal, the fragmentation of the half EoL cells and other steps for the recovery of Ni-YSZ from anode component. The figure gives an overview of the full recovery process material for EoL solid oxide fuel cell.

### 3.1 LSC cathode removal

The removal of the LSC electrode and of the GDC protective layer is the first step required to isolate the bottom YSZ electrolyte and Ni-YSZ composite electrode for the subsequent recovery operations. Due to the fragile nature of the cells, the process of removal of the materials needs to be undertaken with the upmost attention.

For a complete and efficient LSC electrode and GDC protective layer removal, polishing was identified as best method, specifically: 600 and 800 grit polishing papers used on a rotating polisher provided with a water stream.



## 3.2 Lanthanum and Cobalt recovery process

Within BEST4Hy, the hydrometallurgical route through acid leaching process has been identified as the best process for the recovery of Lanthanum and Cobalt.

After several investigations and tests, the Nitric acid process has been selected and optimized for the Lanthanum and Cobalt recovery. Experiments showed good yield of recovery and high purity of the recovered material after characterisation.

The leaching process on EoL LSC powders (size < 20  $\mu\text{m}$ ) is carried out based on operative conditions reported by literature [1].

### Leaching step

Input: LSC powders (1,5 g)

Leaching process parameters

- 2 M  $\text{HNO}_3$  (10 g/L concentration);
- 80° C Temperature;
- 3h time;
- $\text{H}_2\text{O}_2$  1,7%V

Output: residual solid phase

### 1<sup>st</sup> recovery step (pH=0,7):

After collection of the residual solid phase, Oxalic Acid is added to recover the La in form of oxalate hydrate

- Oxalic acid (acid/La = 2.3)
- 10 M NaOH until pH 0,7
- 1h time

Output: 1,3 g Lanthanum oxalate hydrate

### 2<sup>nd</sup> recovery step (pH=1):

More oxalic acid is added to increase the pH and recover the Cobalt:

- Oxalic acid (acid/Co = 2)
- 10 M NaOH until pH 1
- 3h time

Output: 1,1 g Cobalt oxalate hydrate

### 3<sup>rd</sup> recovery step (pH=4):

More oxalic acid is added to increase the pH and recover the Strontium:



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.





- Oxalic acid (acid/Sr = 2)
- 10 M NaOH until pH 4
- 2h time

Output: 0,48 g Sodium strontium oxalate hydrate

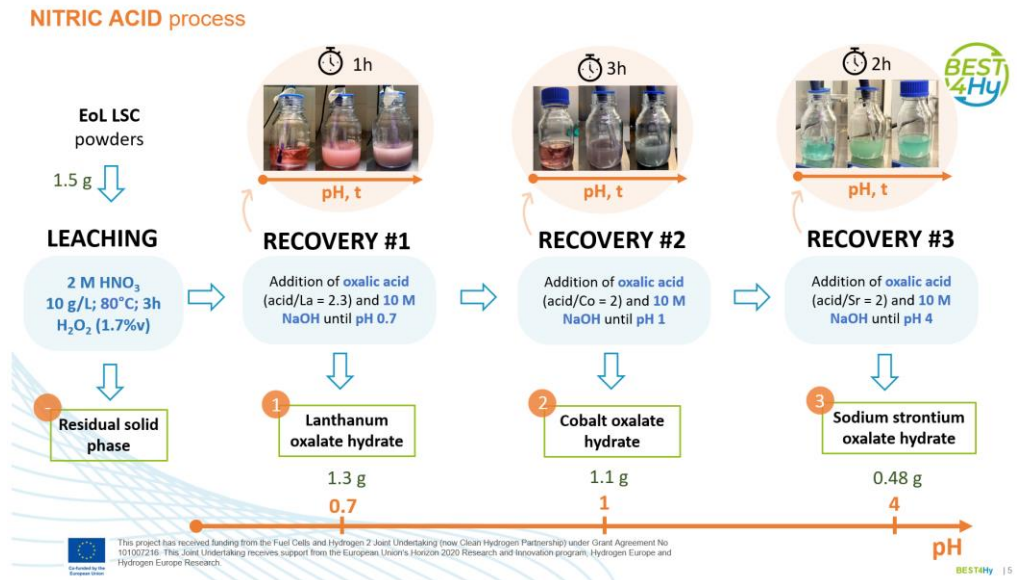


Figure 3 Nitric acid process: leaching and recovery of La and Co



Video on the recycling processes research for SO fuel cells available in BEST4Hy You Tube channel:

[Recycling End-of-Life SOFC](#)



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.



## 4 References

- [1] Lee, C. K., & Rhee, K.-I. (2003). Reductive leaching of cathodic active materials from lithium ion battery wastes. *Hydrometallurgy*, 68, 5–10. [www.elsevier.com/locate/hydromet](http://www.elsevier.com/locate/hydromet)



This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216.