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Platinum recovery through electrochemical process

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

Platinum recovery through electrochemical process

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Conference E3MS

Orvieto, September 15th 2022

www.best4hy-project.eu



Co-funded by the
European Union

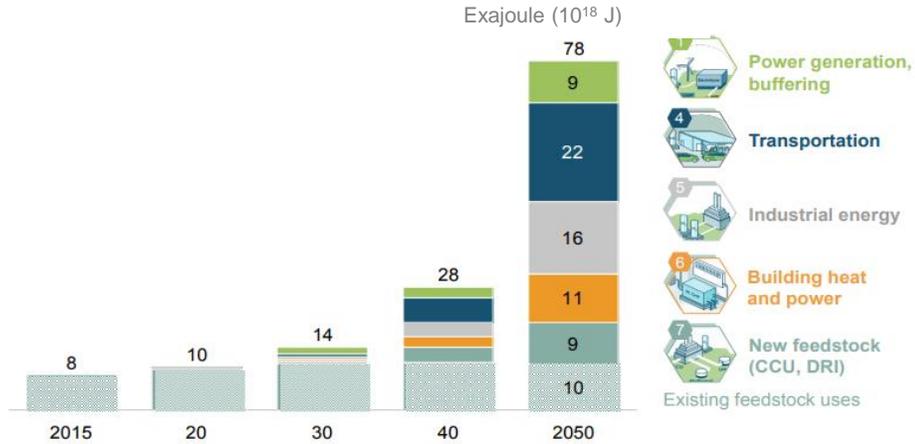
This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking (now Clean Hydrogen Partnership) under Grant Agreement No 101007216. This Joint Undertaking receives support from the European Union's Horizon 2020 Research and Innovation program, Hydrogen Europe and Hydrogen Europe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

Context

Increase in Hydrogen demand and PEMFC production limitations

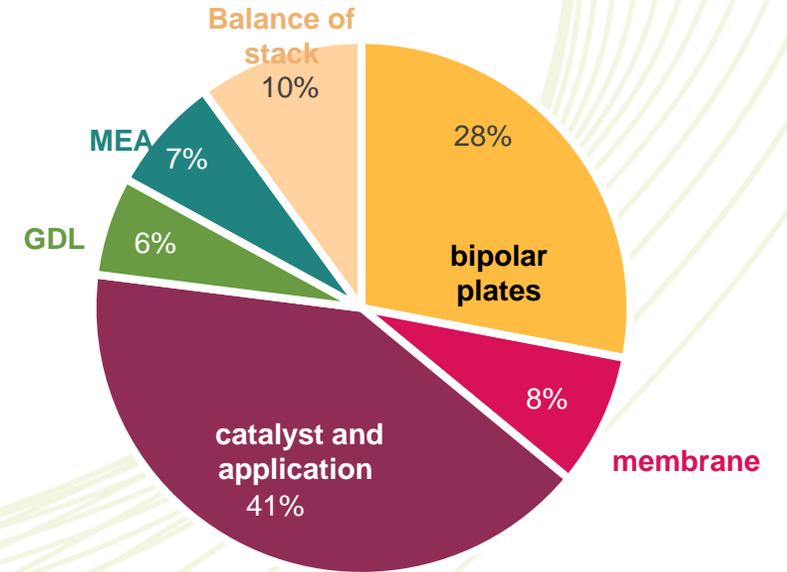
Why do we need to recycle Pt ?

Global energy demand supplied with hydrogen, EJ



SOURCE: Hydrogen Council

PEMFC components costs



Component cost breakdown at a production volume of 500,000 units/year for the fuel cell stack [2]

- ➔ Hydrogen demand increase expectations by 10-fold by 2050
- ➔ PEMFC catalyst (Pt) and electrolyte (membrane) costs limits the development
- ➔ Pt and membrane recovery techniques are required for full development of FCH technologies

[1] Hydrogen council

[2] Thompson, S. T. et al., Direct hydrogen fuel cell electric vehicle cost analysis : system and high-volume manufacturing description, validation, and outlook, *J Power Sources*, 399, 304-313, 2018



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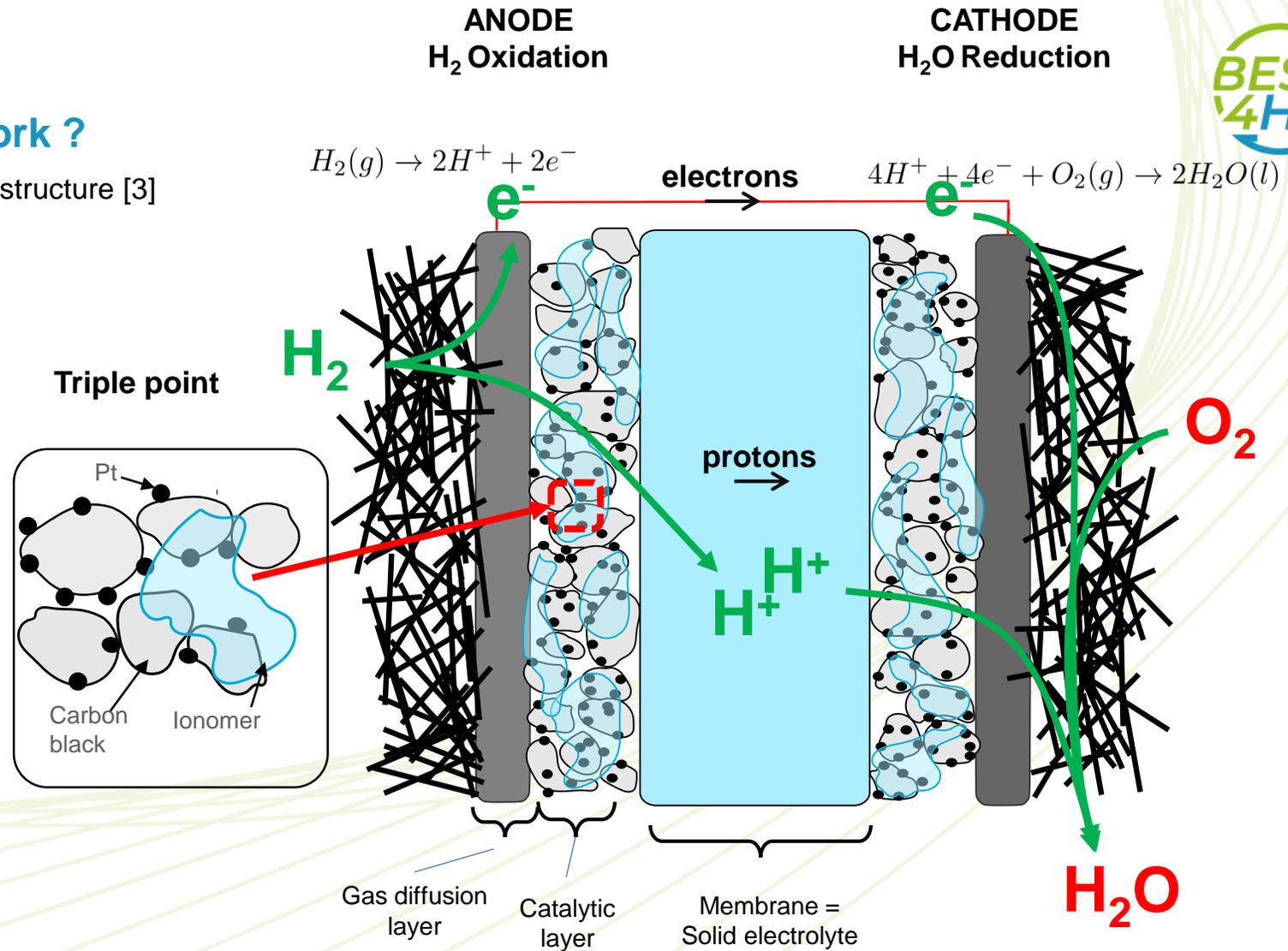
Context

How does a hydrogen fuel cell work ?

Membrane Electrode Assembly components and structure [3]



GDL : Gas diffusion layer
MPL : Microporous layer
CL : Catalytic layer



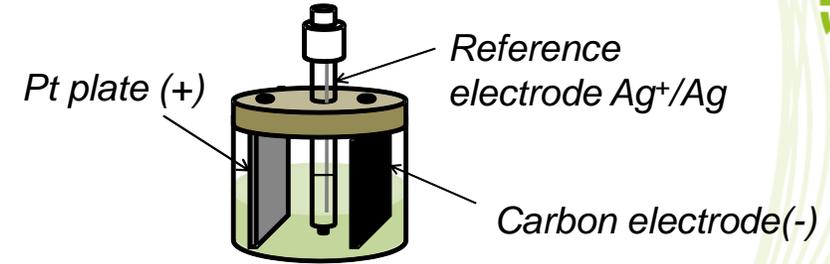
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Pt recovery through electrochemical processes

Scientific approach : environmentally friendly route

Development of an environmentally friendly treatment route for Pt recovery based on EP3263744 A1 2018-01-03 [1]

- No organic solvents
- No acids
- No emission of toxic gases
- One step process



Electroleaching → Electrodeposition

Platinum nanoparticles

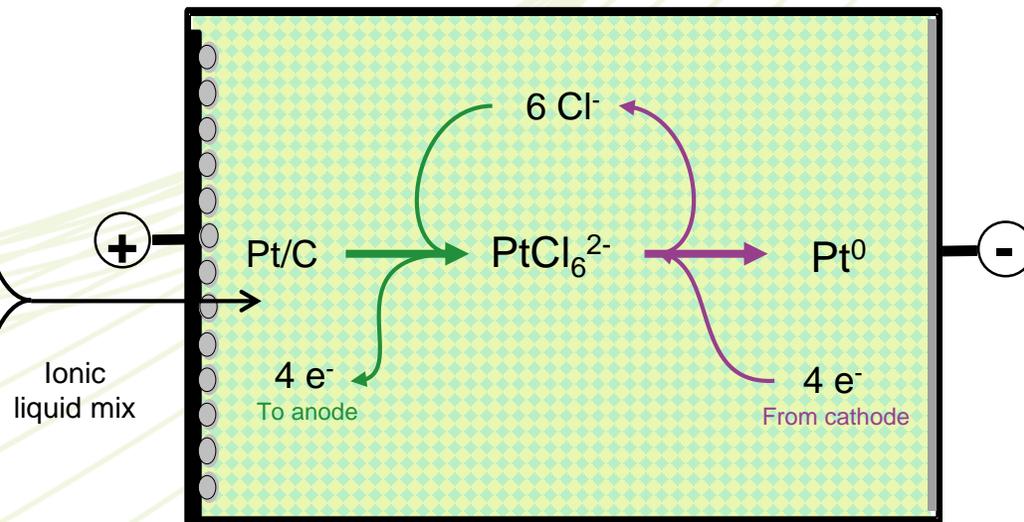
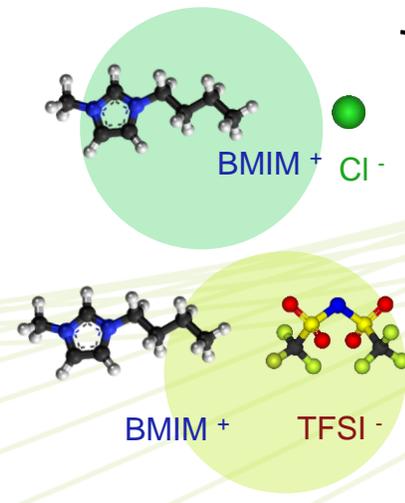
Platinum deposition

Novel recycling technology [2] :

Pt and membrane recovery by coupling of

- **electrochemical leaching** in chloride-based ionic liquids (IL) mixture
- **Electrodeposition of Pt** in the same compartment as lixiviation

Challenge: application on PEMFC material



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

Balva, M., Process for recycling fuel cell membrane electrode assemblies (MEAs) using ionic liquids, 2019



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TYPE OF MEA AND DISMANTLING PROCESS : APPLICABILITY OF ELECTROLIXIVIATION/DEPOSITION Pt RECOVERY PROCESS



MEA type of assembly

MEA Dismantling

Pt recovery

Results

Catalyst Coated Substrate (GDE)

The diagram shows the assembly of a GDE-based MEA: GDL → GDL+CL → GDE → GDE+PEM+GDE → MEA by CCS. The dismantling process is labeled "Manual dismantlement" with "GDE recovered with CL on GDL support".

Use of the GDL as the anode in the recovery process by electrolixiviation

Results:

- Electrleaching (EL)
- Electrodeposition (ED)
- ED-EL

Catalyst Coated Membrane (CCM)

The diagram shows the assembly of a CCM-based MEA: PEM → CL+PEM+CL → CCM → GDL+CCM+GDL → MEA by CCM. The dismantling process is labeled "Manual dismantlement" with "CCM recovered with CL on membrane support".

Development for new anode material to join with CCM (metal or stainless alloy mesh electrode)

Results:

Mesh electrode stuck with CCM



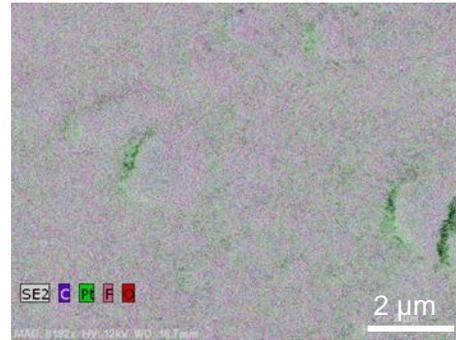
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Pt recovery through electrochemical processes

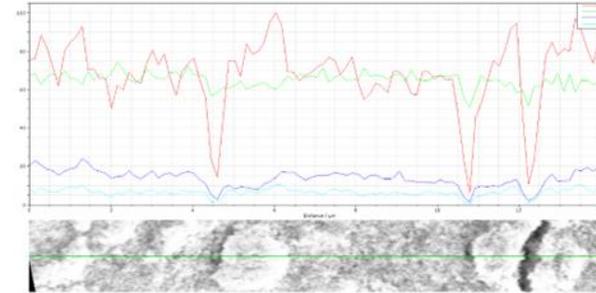
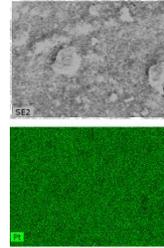
Pt electrolixivation from GDE



Conditions:
 Cylindric cell
 8 g BMIM TFSI ; BMIM Cl **0.3 M** ; 100°C ;
 CA : **2.1 V** ; 22h
 Under Ar flow
 WE : CV + GDE (DEHT);
 CE : vitrous carbon



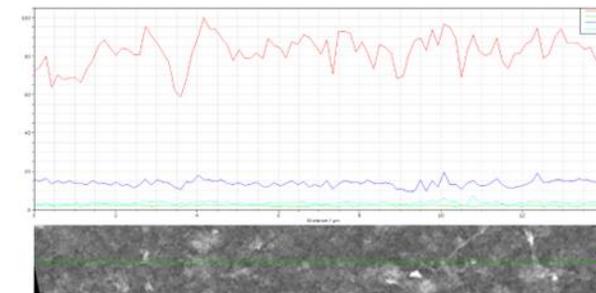
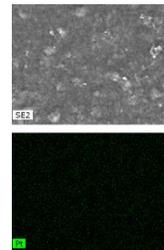
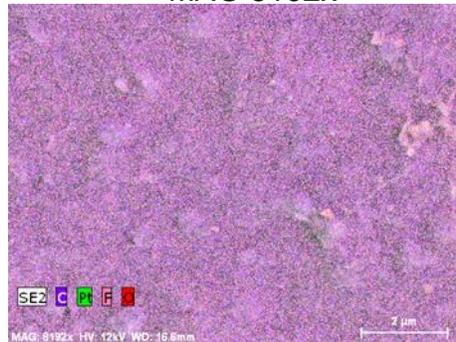
GDE before electroleaching SEM
 MAG 8192x



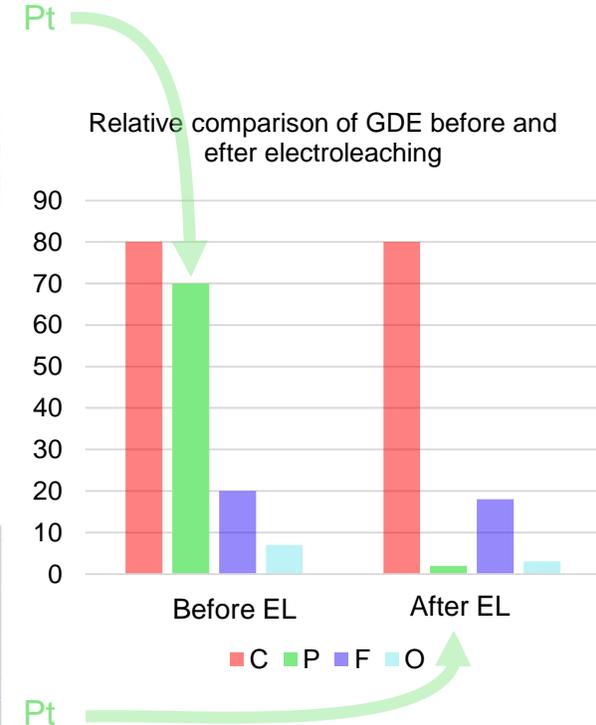
semi quantitative profile SEM-EDX
 before electrolixivation

Analysis:
 Scanning electron
 microscopy
SEM
 with energy
 dispersive X-ray
 spectroscopy
XRD

GDE after electroleaching SEM
 MAG 8192x



semi quantitative profile SEM-EDX
 after electrolixivation



Conclusion: Electrolixivation of Pt can be processes **from GDE in contact with vitrous carbon**



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Pt recovery through electrochemical processes

Pt electrodeposition from GDE

SEM-EDX image of GDL after electrodeposition



Conditions:

Cylindric cell

3 g BMIM TFSI ; BMIM Cl **0.3 M** ; 100°C ;

With 18 mM [Pt]

Ref: in ionic liquid BMIM TFSI and

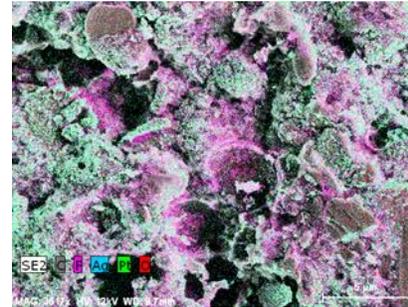
BMIM Cl 0.3M [Cl⁻]

CA : - 0.7V

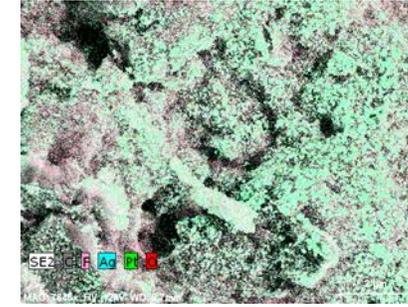
Under Ar flow

WE : vitreous carbon+ gdl;

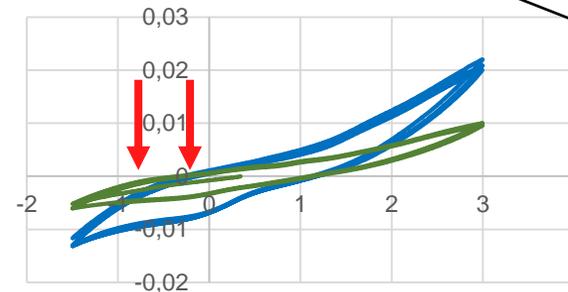
CE : vitreous carbon+ gdl



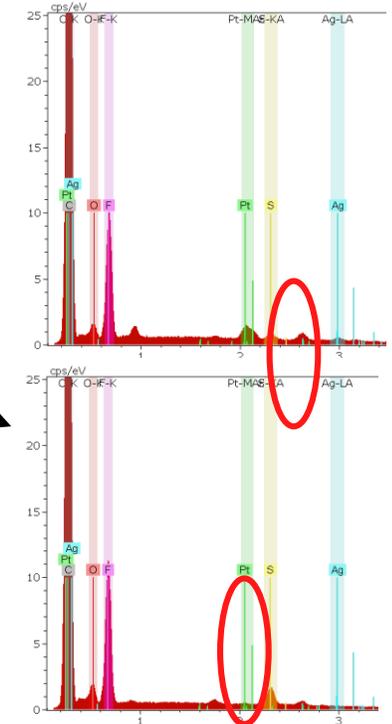
Electrodeposition at -0,2 V (vs Ag⁺/Ag)



Electrodeposition at -0,7 V (vs Ag⁺/Ag)



Cyclic voltammetry in BMIM TFSI + BMIM Cl (0.025 M)
on vitreous carbon electrode +GDL at 100°C Vb = 20
mV.s⁻¹



Control without electrodeposition
deposition

Conclusion: Electroleaching in ionic liquids is possible from GDE. and Electrodeposition of Pt are possible from Pt to recycled GDL

Additional results: Simultaneous electrodeposition to GDL and electrolixivation from Pt plate was enabled

Perspective : Identify conditions to ED-EL Pt to GDL from GDE



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TYPE OF MEA AND DISMANTLING PROCESS : APPLICABILITY OF ELECTROLIXIVIATION/DEPOSITION Pt RECOVERY PROCESS

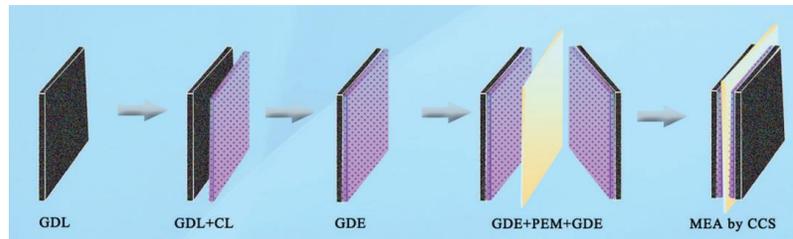


MEA type of assembly

MEA Dismantling

Pt recovery

Results



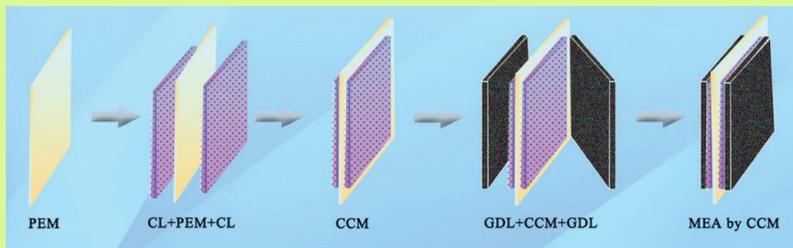
Catalyst Coated Substrate (GDE)



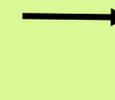
Manual dismantlement
GDE recovered with CL on
GDL support



Use of the GDL as the anode in the
recovery process by
electrolixiviation



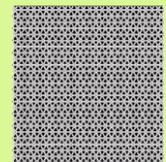
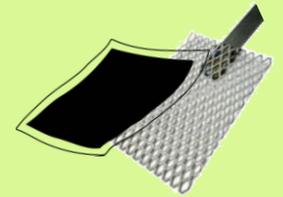
Catalyst Coated Membrane (CCM)



Manual dismantlement
CCM recovered with CL on
membrane support



Development for new anode
material to join with CCM (metal
or stainless alloy mesh electrode)



Mesh electrode
stuck with CCM



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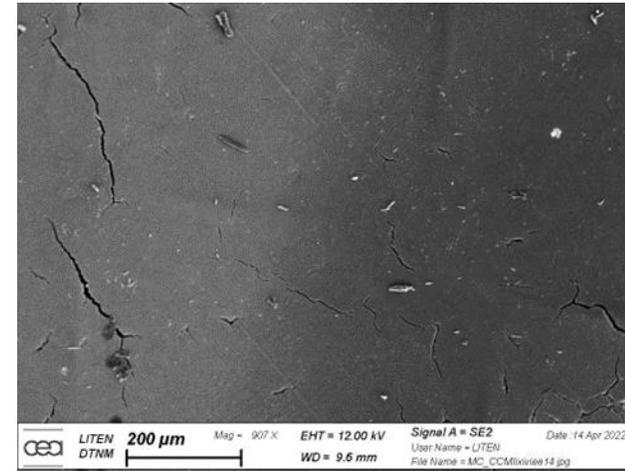
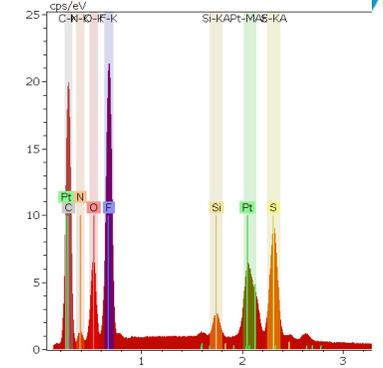
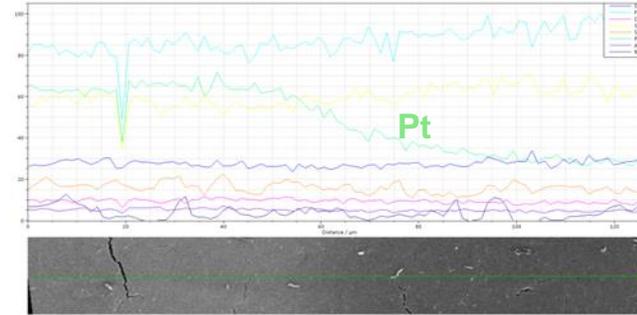
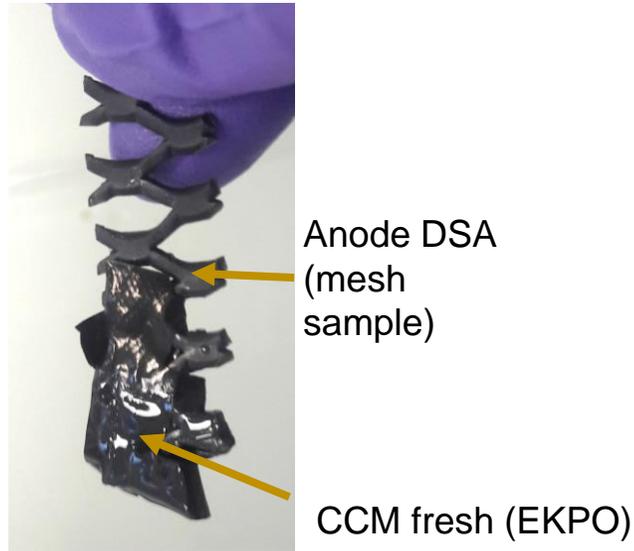
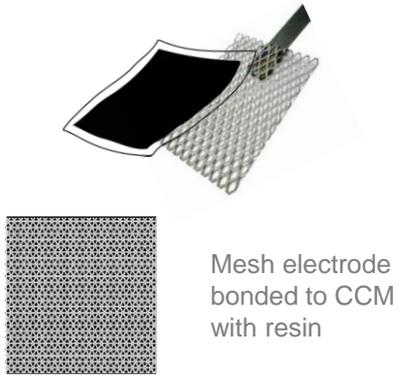
Pt recovery through electrochemical processes

Conditions 3 g BMIM TFSI ; BMIM Cl **0.025 M** ;
 100°C ; WE : DSA
 CE : recycled GDL
 Three electrodes cell



Electrolixivation of Pt from CCM (EKPO)

Use of DSA mesh anode



MEB image of fresh CCM after ELED process

EDX analysis of MEB image



Gradient of Pt concentration observed with MEB EDX: electrolixivation of Pt in immersed part of CCM ON BOTH SIDES OF CCM



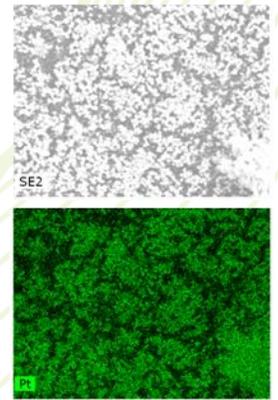
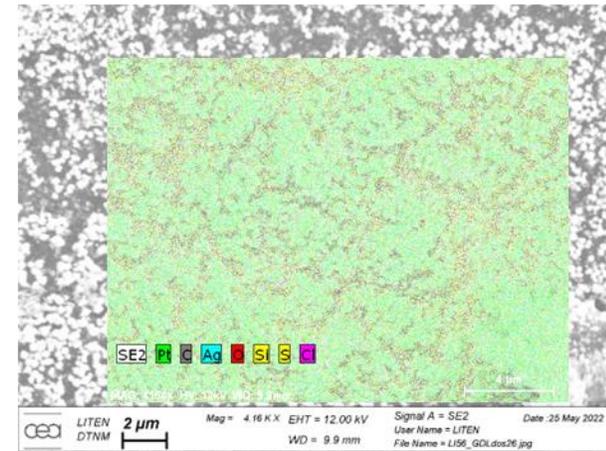
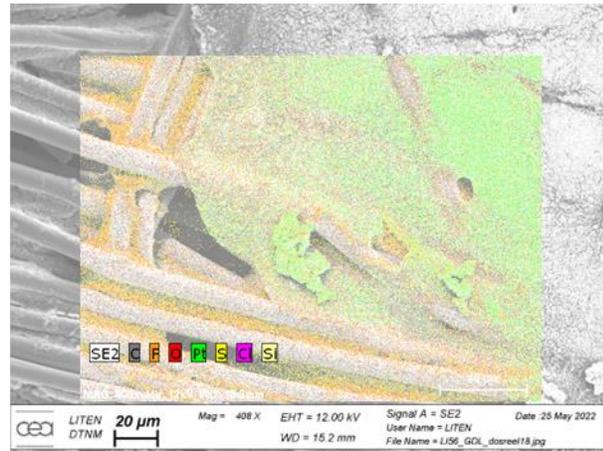
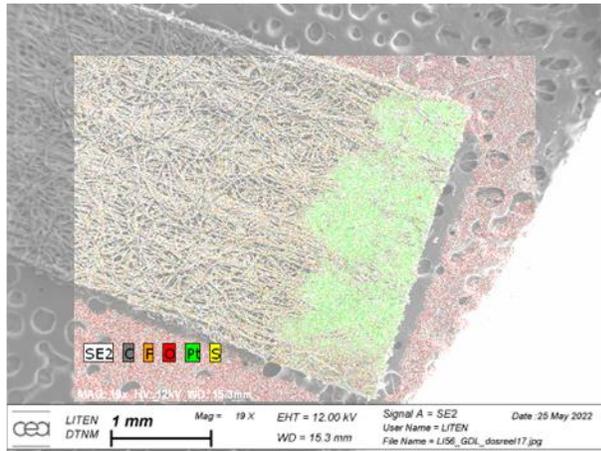
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Pt recovery through electrochemical processes

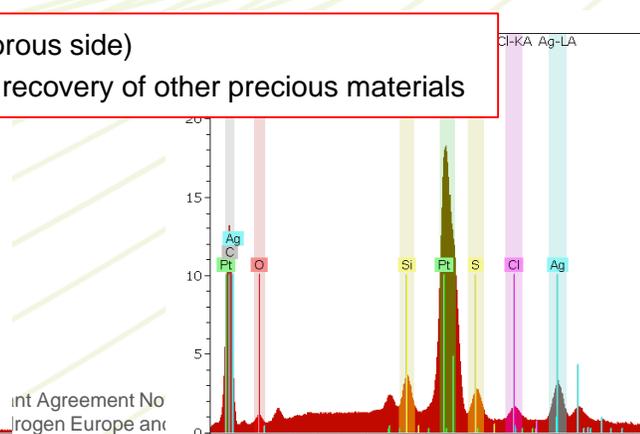
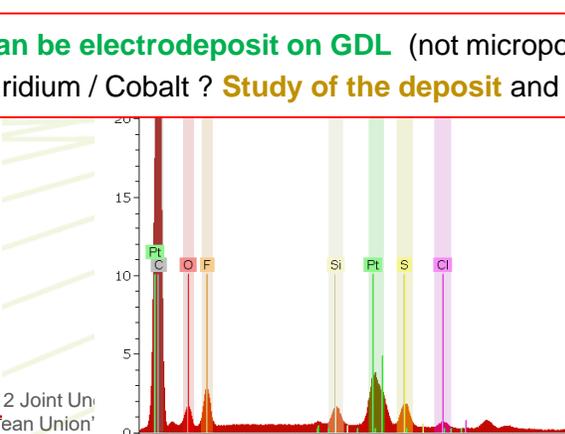
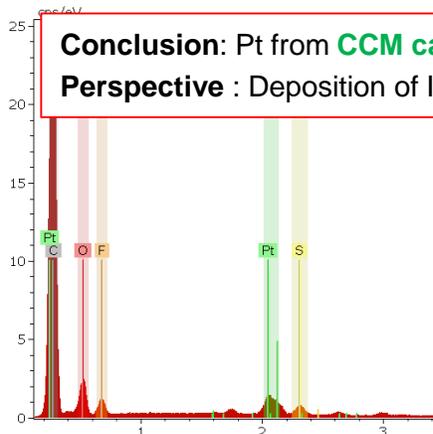
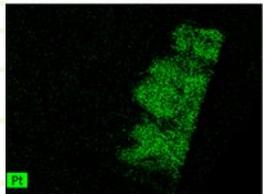
Pt electrodeposition from EL-ED process performed on CCM and recycled GDL

Successful ELED on CCM with DSA as anode (WE) and recycled GDL as cathode (CE)

Pt electrodeposit on GDL, not microporous side

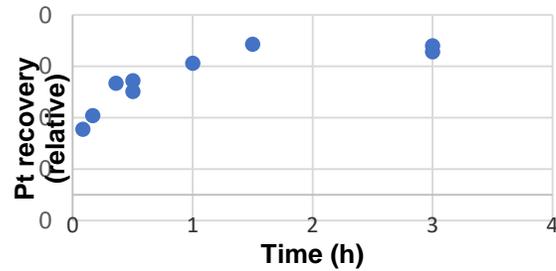


Conclusion: Pt from CCM can be electrodeposit on GDL (not microporous side)
Perspective : Deposition of Iridium / Cobalt ? Study of the deposit and recovery of other precious materials



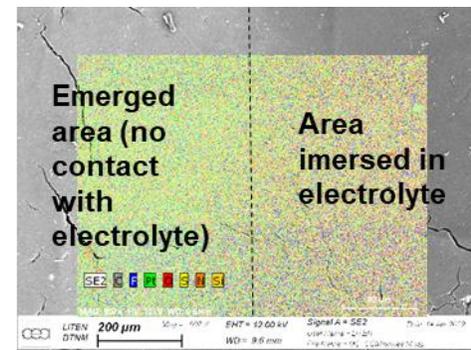
Pt recovery through electrochemical processes

Pt electrodeposition from EL-ED process performed on CCM and recycled GDL

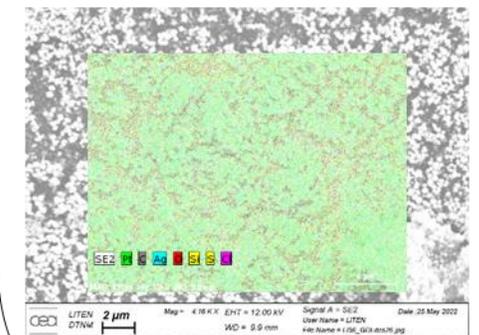
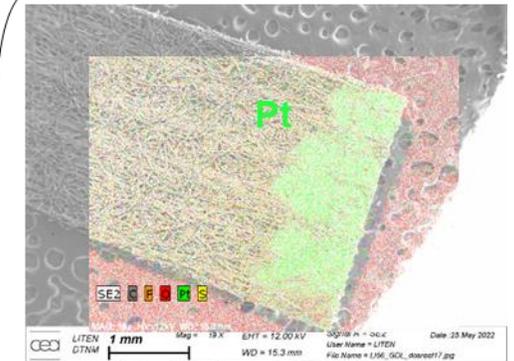
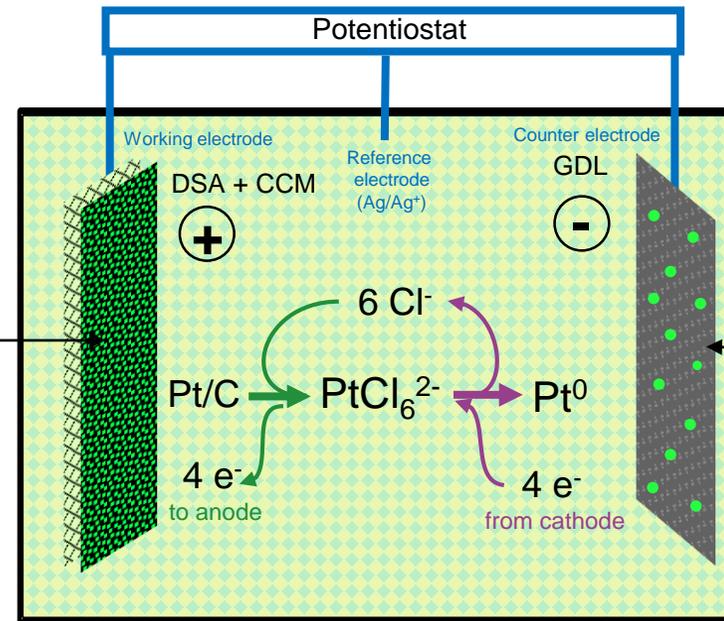


Pt
Electroleaching
from CCM from used fuel cell

Pt
Electrodeposition
On recycled GDL from used fuel cell



MEB image and EDX profile of fresh CCM after ELED process



SEM-EDX analysis :
Pt electrodeposit on GDL

Pt recovery from electrochemical process



Experimental set up

Optimisation of EL-ED of Pt from CCM

Exp name	Objective	E (V vs Ag/Ag+)	Duration (h)	[Pt] mM	[Cl ⁻] M	EL	ED
ELED-01	EL-ED	2,3 V	22,2	14,5	0,3		
ELED-02	EL-ED	2,3 V	22,2	9,1	0,23		
ELED-03			...				

CONCLUSION

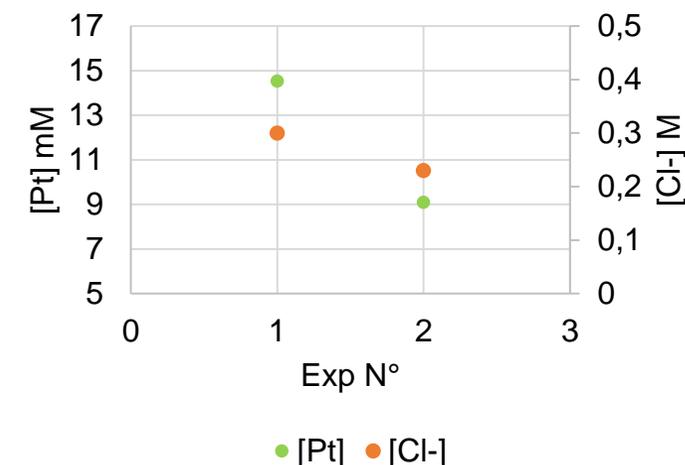
Efficient parameters found to perform ELED on fresh CCM

PERSPECTIVES

Parameter **optimization** : reduce use of Pt, Cl and duration / electrolyte; anode substrate

Understand the nature of Pt deposit (what about Co and Ir) ?

Scale up



Parameters conditions on efficient ELED experiments



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Pt recovery from electrochemical process

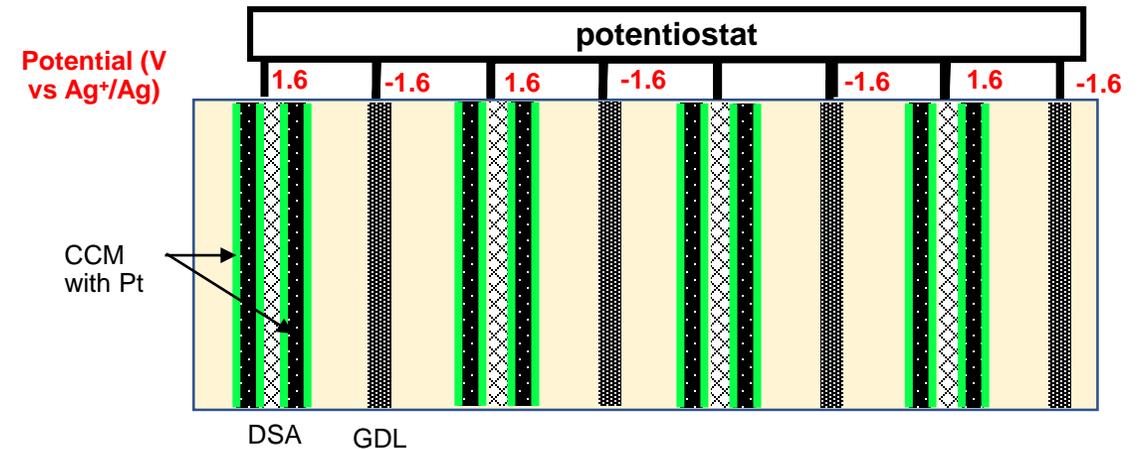
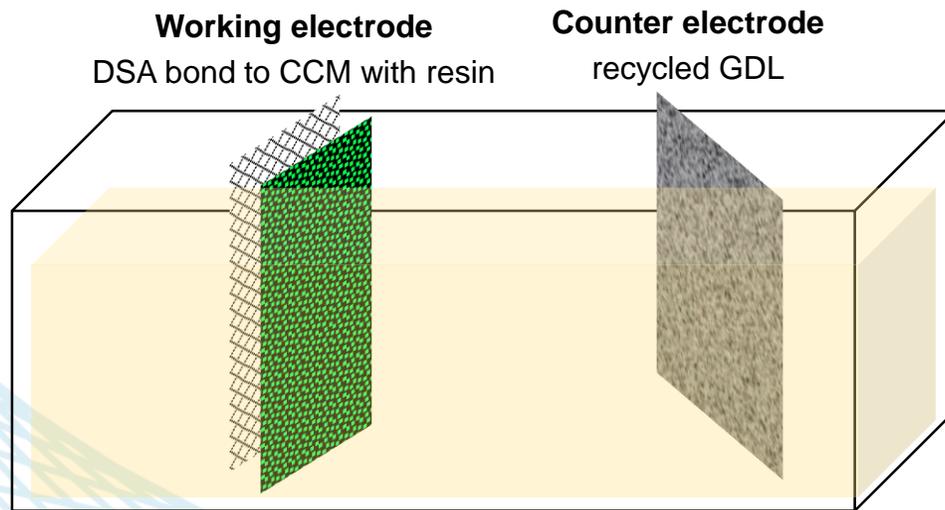


Perspectives

Pilot design for the one-step electrochemical process

Cost of electrolyte : test other electrolyte bath

Recycling of Ionic Liquid bath? What about the degradation of the electrolyte?



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Thank you for your attention

I am happy to answer your questions



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