



MEMBER OF BASQUE RESEARCH
& TECHNOLOGY ALLIANCE

Greener pathways for Surface treatments and composite materials

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AMI 2030 Workshop on Sustainable materials- 21st Nov2023- San Sebastian-Spain

> Quick flavour

cidetec>

MEMBER OF BASQUE RESEARCH
& TECHNOLOGY ALLIANCE

1. Cidetec overview: institutes and expertise

2. Greener functional surfaces

Nickeffect project

3. Greener printed electronics

Reform project

4. Intrinsically Recyclable, Reprocessable and Repairable (3R) fibre
reinforced thermosets composites

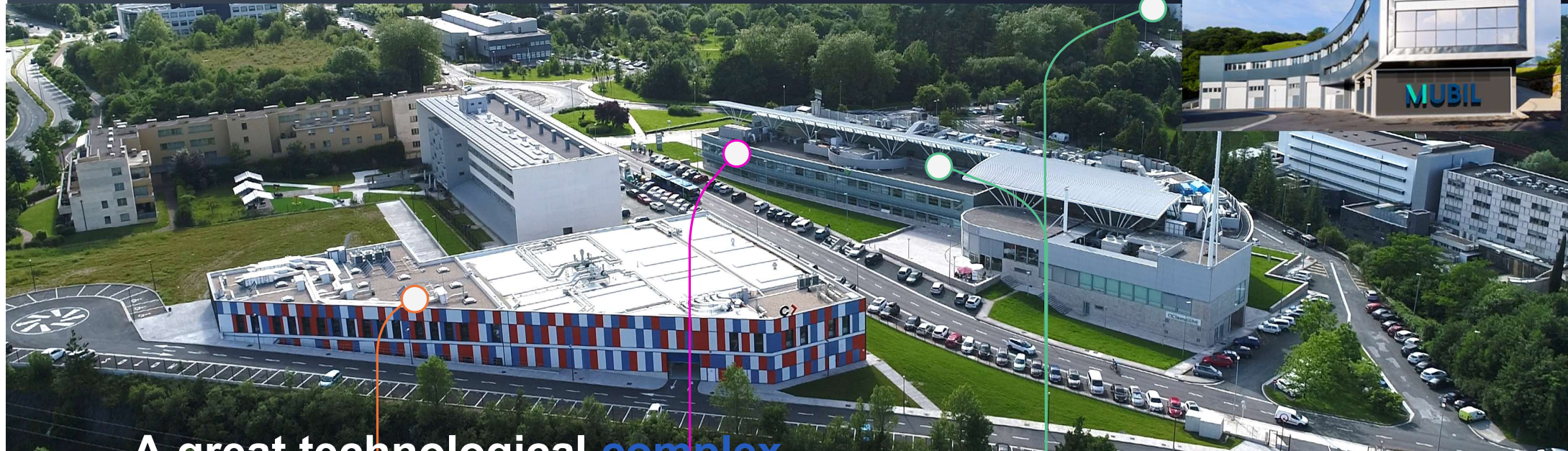
Surpass, MC4, Genex, Carbo4Power, Biouptake

5. Polymeric materials with biodegradability on demand

Unlock project



1. CIDETEC OVERVIEW



A great technological complex

cidetec 
surface engineering

cidetec 
nanomedicine

cidetec 
energy storage



18 M€

Turnover
in 2022

250

People in
the workforce

82

European participated
projects
(22 as coordinator)

214

ISI publications
over the last 5
years

15 M€

Invested
(2018-2022)

54 %

Doctors on
Research staff

27 %

European funding

32

Patent families

**Key
Data**

20

Thesis in
progress

260

clients engaged
in R&D over
the last 5 years

8

Spin-offs



Public-private partnership

Nanomedicine institute



Drug-delivery via inhalation

CIDETEC is a leader in the development of nanotechnology for the administration of drugs via pulmonary route. Our experience in the encapsulation of antibiotics enables solutions for both hydrophilic and hydrophobic compounds



Drug-delivery via skin

Intelligent release systems for increased effectiveness of the active ingredients in cosmetics and dermocosmetics.



Coatings for prostheses

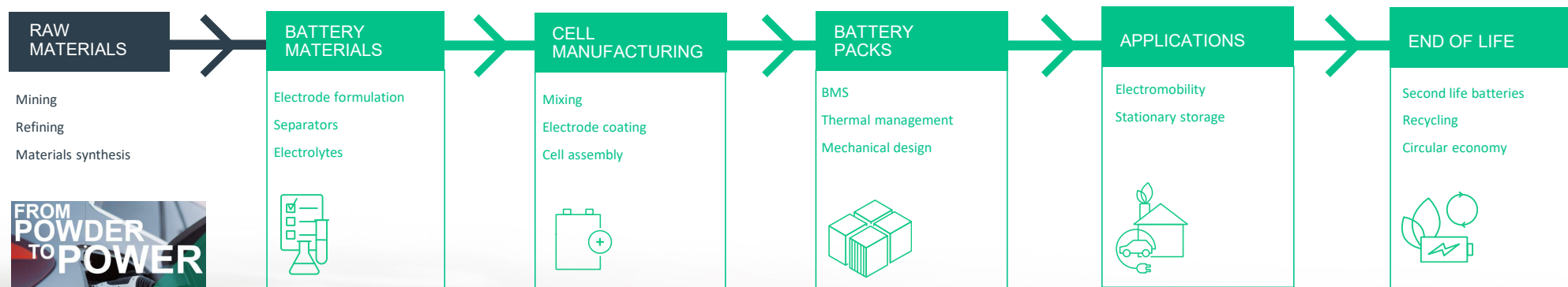
Highly hydrophilic coatings that mimic the surface of the cartilage and reduce the wear of the prosthesis up to 80% and hydrogels of both synthetic and natural origin to generate solutions for the treatment of glaucoma.



> Energy storage Institute



R+D Excellence Centre for Advanced Battery Technologies



Customer profile

BATTERY MATERIALS
INDUSTRY



BATTERY CELL
MANUFACTURERS



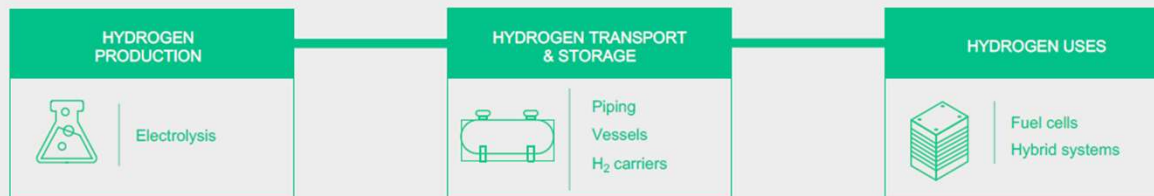
AUTOMOTIVE OEMS



ENERGY COMPANIES



HYDROGEN



Surface Engineering Institute

Focus Areas



Sustainable surfaces & materials

- Sustainable Composites
- Sustainable Thermoplastics
- REACH Compliant Surfaces



Metallisation of thermoplastics & fibre reinforced composites

- Developing polymers and composites.
- Developing sustainable, tailor-made metallisation processes adapted to the nature of the polymeric and composite material and its functional and aesthetic requirements
- Trivalent chromium plating, Cr-free etching for metallization, Electroless metallisation (nickel, copper, etc.), Aero-plating, Brush-plating



Smart Surfaces

- Metallic surfaces with transparency to ADAS systems (radar, cameras, or LIDAR)
- Back-illuminated metallic surfaces for decoration
- Deposition of conductive tracks and layers
- Printed electronics
- Stimuli-responsive surfaces
Design of sensors and non-destructive methods for surface inspection and control



Omniphobic Surfaces

- High repellency towards Water
- High repellency towards Oily substances (oil and organic solvents, greases, etc.)
- High repellency towards Other fluids (moisture in the air, etc.)



Hygienic Surfaces

- Anti-bacterial/ anti-microbial and anti-fungus properties
- Easy-cleaning properties
- Anti-stain properties
- Anti-fouling properties



Post-processing of Additive Manufacturing

- Removal processes
- Finishing processes

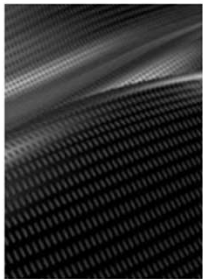


2. Greener functional Surfaces

We offer:

CIDETEC is working on the design of surfaces from a SSbD perspective, searching for alternatives to replace harmful compounds or CRM, enabling companies to comply with regulations (e.g. REACH), while offering safer materials and processes.

The most common treatments affected by the REACH regulation are:



Hard chromium



Decorative chromium



Plastic etching for metallisation



Conversion coatings



Cadmium plating

CIDETEC is working on a range of technologies, including:

- > Electroless nickel
- > Trivalent chromium plating
- > Cr-free etching for metallisation
- > Cr-free conversion coatings
- > Cr-free anodising
- > Sol-gel
- > Anaphoretic e-coating
- > n-Ni plating
- > Cr-free primers
- > Ceramic coatings and cements
- > Metal matrix composite coatings





Motivation: Pt-substitution in materials for energy applications and digital technologies

Use cases: PEM Water Electrolyzers, PEM Fuel cells, MRAMs

Consortium: 12 partners (member of 7 countries)

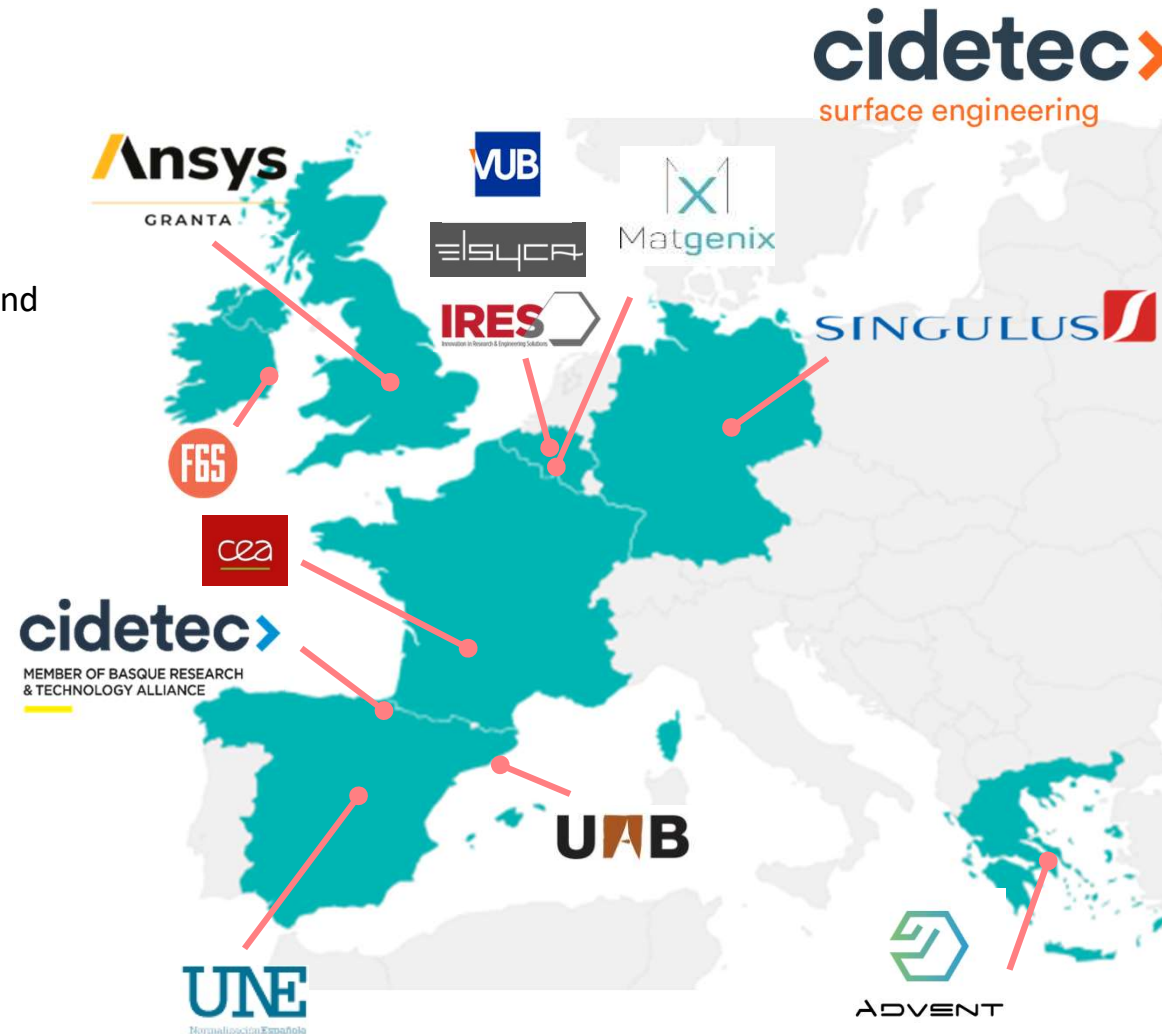
- 5 Small and medium-sized enterprises
- 2 Universities
- 2 Research and Technological Development Centers
- 2 Large Enterprise
- 1 Standardization & Normalization organization

Coordinator: Fundación CIDETEC, Spain

Start date: 1st of June 2022 (duration 48 months)

Website: <https://nickeffect.eu/>

Main contacts: anicolenco@cidetec.es, mlekka@cidetec.es,
jochoa@cidetec.es, ejubete@cidetec.es, eguinea@cidetec.es,



MOTIVATION



> > > > > > > > > >

PGMs are highly demanded due to their **unique physical properties**. These properties have made them indispensable in different strategic sectors, e.g. **renewable energy, electric mobility and digital technologies**.

Pt is widely used in the following application fields:

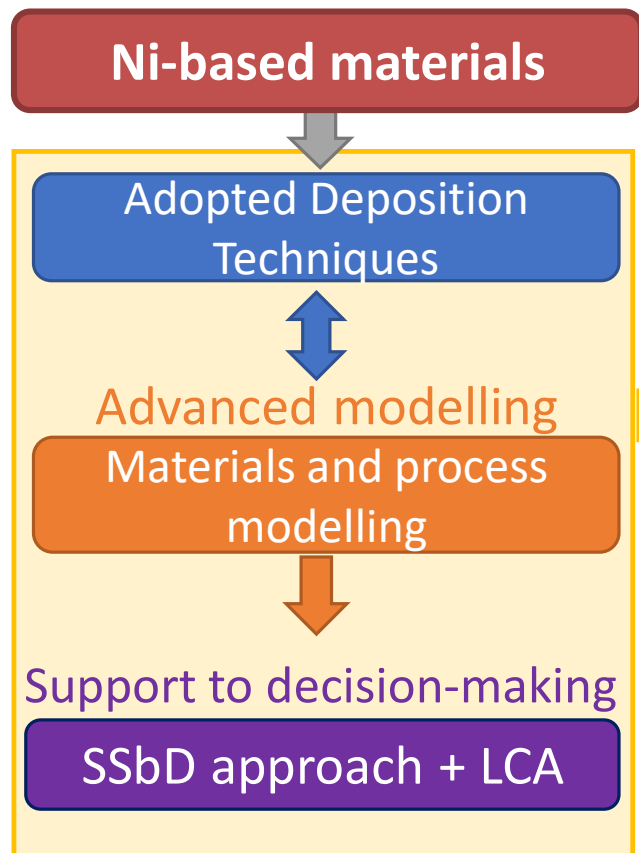
- *Catalytic materials for water electrolysis (WE)*
- *Catalytic materials for Fuel Cells (FC)*
- *Coating materials for low power consumption digital storage devices*



PGMs are **categorized as critical raw materials (CRM) by the EC**.

It is crucial to:

1. Develop methods to recover PGMs for recycling, and/or
2. Find alternative materials to PGMs



Advantages:
↑ S/V ratio
↑↑ efficiency

↑ *efficiency*
ferromagnetic
PGM-free
materials

Advantages:
Voltage-control
↓ E dissipation
Magnetization switch



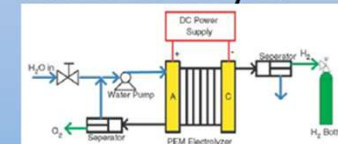
Metal RECOVERY

USE CASES

MEA's coating



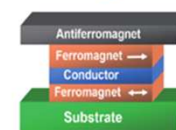
Energy production
water electrolysis



Energy conversion
fuel cell



MRAMs stacks coatings



Digital storage devices
MRAMs



SUPPORT TO DECISION-MAKING USING SSbD APPROACHES



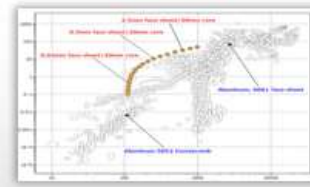
SEARCH & REPORT



LIFE CYCLE ASSESSMENT

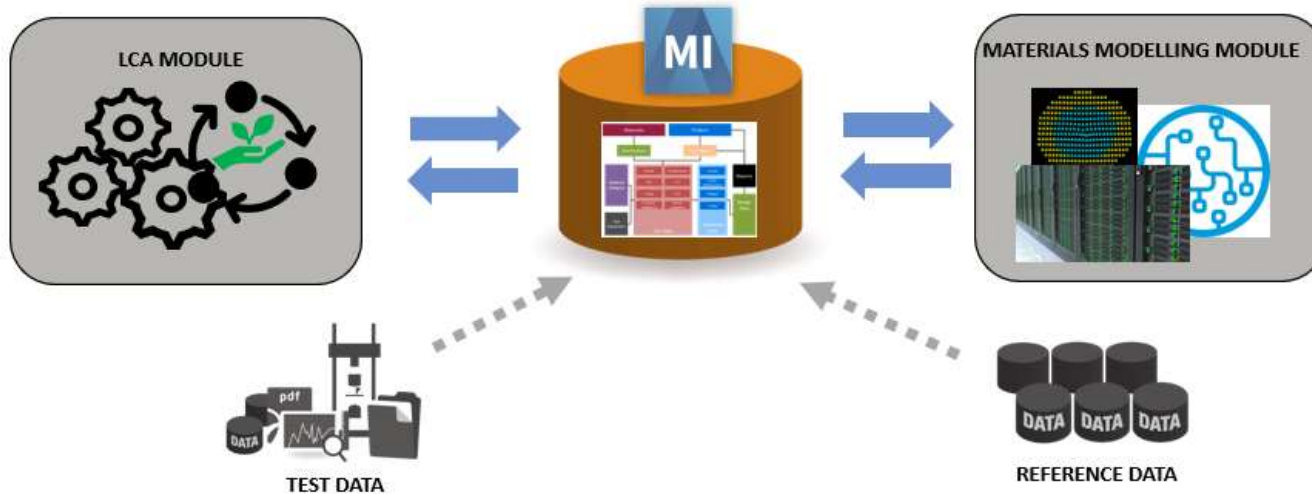


DISCOVERY & PUBLISH



VISUALIZE & PREDICT

Modular decision support tool using input from literature, laboratory testing, environmental testing, reference data, materials modelling, LCA etc.



WP Leader: **IRES**
Innovation in Research & Engineering Solutions

Data base curated by: **Ansys**
GRANTA

3. Greener printed electronics

REFORM Project - pRinted Electronics FOR the circular econoMy

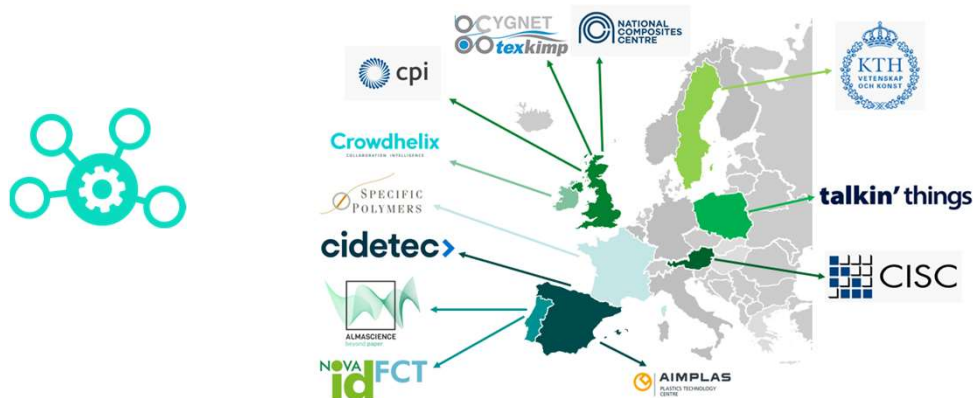
Concept & Consortium



-Address the environmental and sustainability challenges around conventional functional electronics

- Use ecodesign principles to ensure to:

- meet the requirements of multiple high-performance applications
- meet societal and environmental needs for sustainability



cidetec
surface engineering

FUNDING 4,993,610.00 €



European Union
Horizon Europe (HORIZON): 3,588,155.75 €

UK partners: 1,405,454.25 €

Start:
1st
January
2023

End:
30th
June 2026



42
months

8
Countries

6 RTO
5 SME
1 UNI
12
Partners



REFORM project has received funding from the European Union's Horizon Europe research and innovation programme under Grant Agreement N° 101070255. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union. Neither the European Union nor the granting authority can be held responsible for them



REFORM Project - pRinted Electronics FOR the circular econoMy

Main objectives



- Develop environmentally benign electronic 'building blocks' focusing on green, bio-derived

Conductive inks

Flexible substrates

Adhesives



Fully organic conductive inks
Cellulose-based

Cellulose-based substrates
Recyclable thermoset 3R composite

Debondable adhesives

- Integrate into industry-led functional electronics systems, supported by innovations in conformance testing and material recovery methods.



Metal-free on-paper
microsupercapacitors



RFID tags for smart
logistics

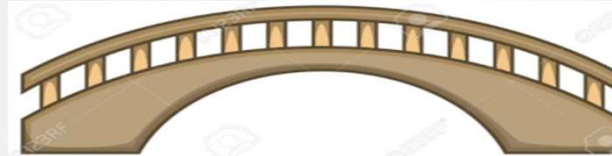
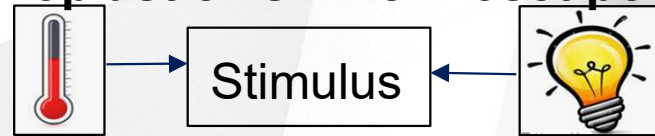


Embedded pressure
vessel sensors



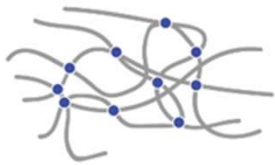
➤ 4. Intrinsically Recyclable, Reprocessable and Repairable fibre reinforced thermosets composites

Thermoplastic vs. Thermoset polymers



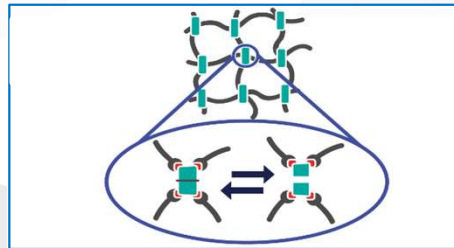
Covalent Adaptable Networks (CANs)

Thermosets



THERMOSET

- Covalently crosslinked
- Remains hard when heated
- Insoluble
- Chemical resistance
- Good mechanical properties
- Non-processable
- Non-recyclable



When $T < T_g$ the network is static → thermoset behaviour
When $T > T_g$ the network becomes dynamic → thermoplastic behaviour

Thermoplastics



THERMOPLASTIC

- Entangled linear polymer chains
- Reprocessable
- Recyclable
- Soluble
- Bad chemical resistance
- Softens when heated

➤ Epoxy vitrimers based on aromatic disulfide exchange

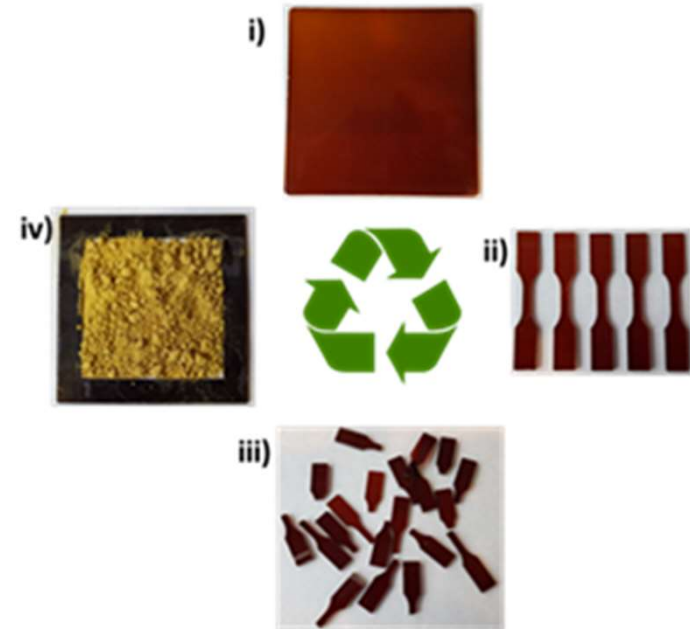
The dynamic character of the epoxy network offers new challenges:

✓ Reparable thermoset resins



✓ a small scratch performed on a specimen was repaired just by applying heat a pressure with a household iron

✓ Recyclable thermoset resins



<https://www.cidetec.es/es/top-achievements-3/3r-leading-technology-2>

A. Ruiz de Luzuriaga *et al.*, *Material Horizon*, **2016**, 3, 241.

➤ *Dynamic fibre reinforced composites based on aromatic disulfide*

3R Composites

- A new generation of **Reprocessable, Repairable and Recyclable** high-performance fibre-reinforced thermoset composites.
- They can be manufactured following traditional methods but the resulting material can be reprocessed, repaired and recycled.



<https://www.cidetec.es/es/top-achievements-3/3r-leading-technology-2>



Patented technology:

EP 3 149 065 B1 – “Thermomechanically reprocessable epoxy composites and processes for their manufacturing”.

Sustainable composites



DYNAMIC EPOXY RESIN



Benefits:

The new generation of advanced thermoset polymers and composites based on dynamic covalent chemistries is one of the most attractive technical solutions providing environmentally friendly alternatives to traditional materials.

The introduction of dynamic covalent chemistry enables a series of "smart" properties, creating a new generation of thermoset polymers and composites that contribute to:



Recyclability

We offer:

- Tailor-made 3R epoxy resin formulations for specific applications and manufacturing processes.
- Reprocessing of cured 3R epoxy composites into complex shapes.
- Fast, cost-effective repairing of delamination damage of 3R composites.
- Debondable on-command 3R adhesives.
- Chemical and mechanical recycling paths.



CONTRIBUTING TO:



High performance



Competitiveness
(reduction in production
and maintenance costs)



Re-processability



Reparability



Recyclability

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> *Dynamic fibre reinforced composites based on aromatic disulfide*

> 3R Composites:

Reprocessing,

Repairing,

Recycling

- ✓ 3R composite laminates can be heated above the T_g and re-shaped in a few minutes applying pressure, which allows the thermoforming of cured 3R laminates to obtain 3D geometries, in a similar way to thermoplastic composites.

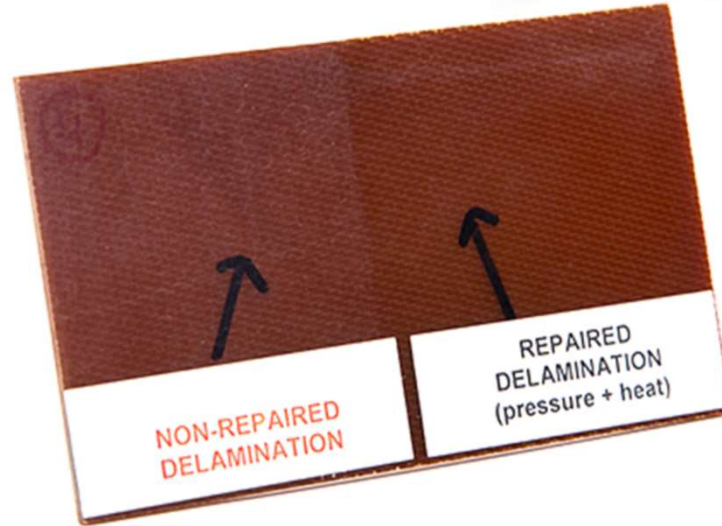


This technology enables the development of high production rate manufacturing processes for thermoset composites **reducing the manufacturing costs of thermoset CC parts by over 35% vs autoclave manufacturing.**

> *Dynamic fibre reinforced composites based on aromatic disulfide*

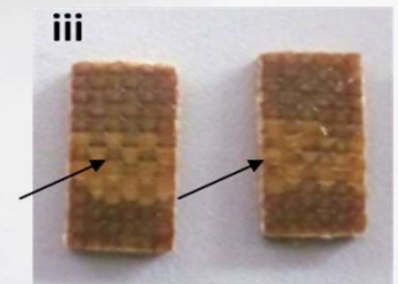
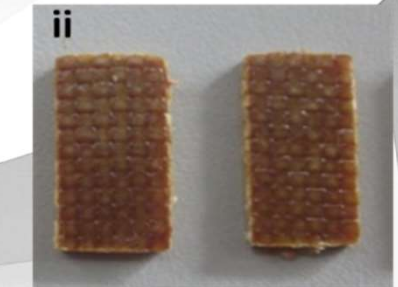
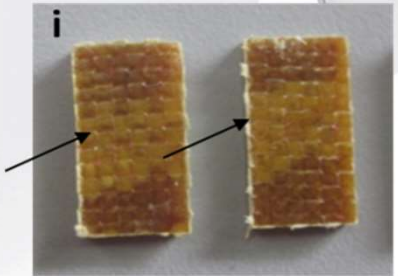
> 3R Composites:

Reprocessing,
Repairing,
Recycling



Repair of damages based on resin/fibre delaminations and resin micro-cracks by applying heat and pressure to the damaged part.

ILSS: $37,2 \pm 2,81$ MPa



ILSS: $38,0 \pm 2,4$ MPa

50%

reducing the
current costs
of MRO.



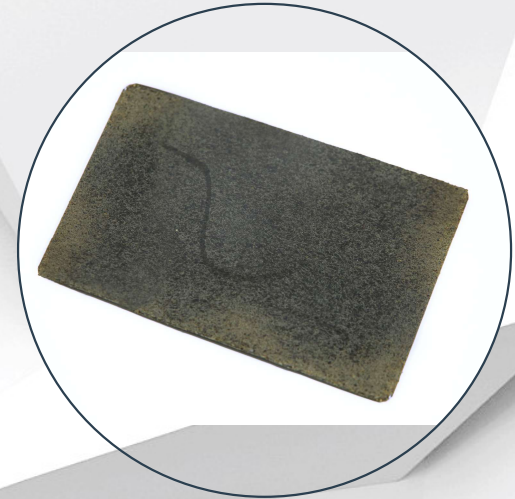
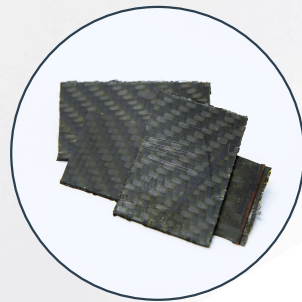
This technology enables to **reduce the current costs of MRO associated to the reparation or replacement of thermoset CC parts by 50%** (nowadays damaged parts are often rejected due to the high costs and repair times of the traditional patch techniques).

> *Dynamic fibre reinforced composites based on aromatic disulfide*

> 3R Composites:

Reprocessing,
Repairing,
Recycling

- ✓ **Mechanical recycling:** the cured 3R composite can be ground into flakes or pellets which can then be reprocessed by heating 80°C above the T_g and pressing, obtaining a new short fibre reinforced 3R composite.



This technology enables the valorisation of the scraps generated during the manufacturing of thermoset CC offering 3 different environmental and industrial advantages:

**REDUCTION
OF LANDFILL
WASTE.**

**REDUCTION
OF THE COSTS
OF WASTE
MANAGEMENT
OF SCRAPS.**

**MANUFACTURING OF COST
COMPETITIVE AND
SUSTAINABLE NEW
THERMOSET CC PARTS BASED
ON VALORISED SCRAPS.**

> 4. Dynamic fibre reinforced composites based on aromatic disulfide

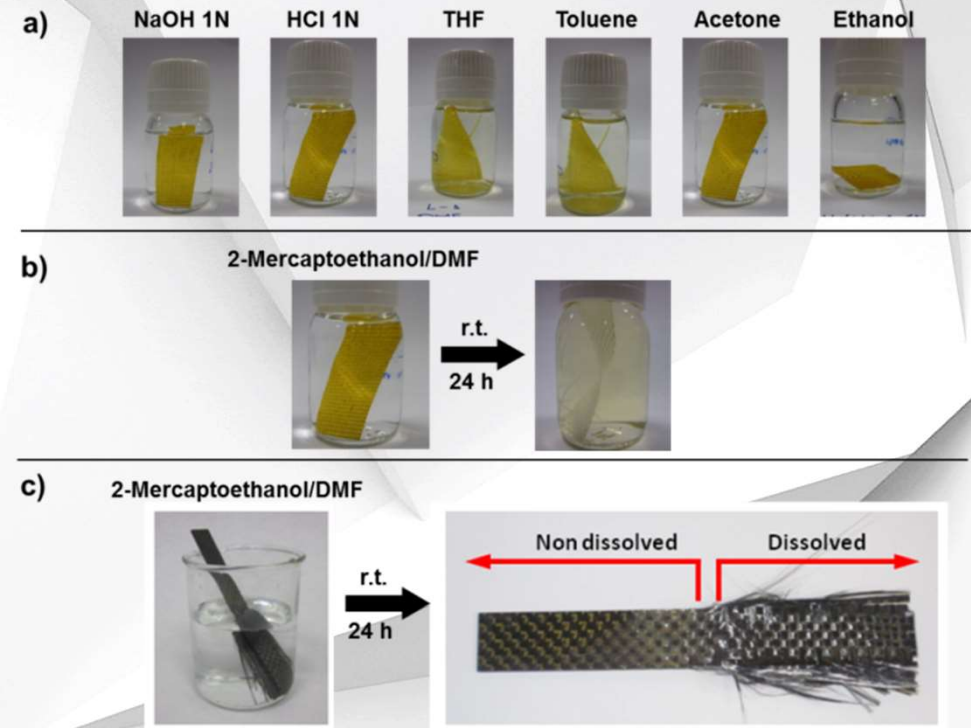
> 3R Composites:

Reprocessing,

Repairing,

Recycling

- ✓ **Chemical recycling:** the 3R matrix can be completely disrupted by the addition of a specific chemical agent without affecting the reinforcement.



This technology enables the recycling and reuse of the carbon fibre reinforcement offering environmental and industrial advantages:

**REDUCTION OF
LANDFILL
WASTE.**

**MANUFACTURING OF COST
COMPETITIVE AND SUSTAINABLE NEW
THERMOSET CC PARTS BASED ON
VALORISED SCRAPS.**



Examples of european projects related to 3R composites





COORDINATED

MOST RELEVANT PROJECTS IN THE PREVIOUS H2020 PROGRAMME

cidetec
surface engineering



Bio-based recyclable, reshapable and repairable (3R) fibre-reinforced EPOXY composites for automotive and construction sectors



BBI2016.R7:

“Biopolymers with advanced functionalities for high performance applications”

GA n°744311

Total budget: 4,85 M€

Duration:
01/06/2017 – 30/11/2020

www.ecoxy.eu



ThermoformAble, repairable and bondable smart ePOXY based composites for aero structures

MG-1.3-2017:

“Maintaining industrial leadership in aeronautics”

GA n°769274

Total Budget: 6,5 M€

Duration:
01/09/2018 – 28/02/2022

www.airpoxy.eu



RECYSITE

Production of fully recyclable and reusable green composites based on bioresins and natural fibres



LIFE Programme:

“The LIFE programme is the EU’s funding instrument for the environment and climate action”

LIFE15 ENV/BE/000204

Total Budget: 2,08 M€

Duration:
01/07/ 2016 – 31/12/2018

www.recysite.eu



Hierarchical multifunctional composites with thermoelectrically powered autonomous structural health monitoring for the aviation industry

MG-1.4-2017:

“Breakthrough innovation”

GA n°769140

Total Budget: 4 M€

Duration:
01/09/2018 – 31/08/2021

www.harvest-project.eu



ONGOING EUROPEAN PROJECTS FOCUSED ON 3R TECHNOLOGY



New generation of offshore turbine blades with intelligent architectures of hybrid, nano-enabled multi-materials via advanced manufacturing

H2020-EU.2.1.3. – INDUSTRIAL LEADERSHIP:
“Leadership in enabling and industrial technologies – Advanced materials Programme”

GA n° 953192

Total Budget: 7,8 M€

Duration:
01/11/ 2020 – 31/10/2024

www.carbo4power.net



Multi-level Circular
Process Chain for Carbon
and Glass Fibre Composites

Multi-level Circular
Process Chain for
Carbon and Glass Fibre
Composites

HORIZON-CL4-2021-
RESILIENCE-01-01 :
“Ensuring circularity of
composite materials”

GA n° 101057394

Total Budget: 7,0 M€

Duration:
01/04/2022 – 31/03/2025

www.mc4-project.eu



Safe-, sUstainable- and
Recyclable-by design
Polymeric systems
A guidance towardS next
generation of plasticS

HORIZON-CL4-2021-
RESILIENCE-01-11:
“Safe- and sustainable-by-
design polymeric materials
”

GA n° 101057901

Total Budget: 5,0 M€

Duration:
01/06/2022 – 31/11/2025

www.surpass-Project.eu



New end-to-end digital
framework for optimized
manufacturing and
maintenance of next
generation aircraft
composite structures

HORIZON-CL5-2021-D5-01-06:
“Next generation digital aircraft
transformation in design,
manufacturing, integration and
maintenance”

GA n°769140

Total Budget: 5,7 M€

Duration:
01/09/2022 – 28/02/2026

www.genex-project.eu



BIOcomposites in smart
plastic transformation
processes to pave the way
for the large-scale
UPTAKE of sustainable
bio-based products

HORIZON-CL4-2021-TWIN-
TRANSITION-01-05:
“Manufacturing technologies
for bio-based materials”

GA n° 101057049

Total Budget: 6,0 M€

Duration:
01/12/2022 – 31/05/2026

Context

- **Plastic waste** outlives us on this planet as it **takes centuries to break down**
- The **hazard of leached substances** not only **pollutes** land, air, water and **increases greenhouse** gas emissions, but also has **adverse effects on health**
- Still, **70% of plastic waste** collected in Europe is **landfilled or incinerated**

The consortium

- 13 partners from 6 different European countries



General Objective

- The general objective is to equip European SMEs with a **digital guiding tool that will impart knowledge and provide Safe, Sustainable, Recyclable by Design (SSRbD) Assessment and guidance** to support them in their development of **new polymers** that contribute to the transition towards a **green economy**

Approach

1. Develop SSRbD alternatives with no potentially hazardous additives through industrially relevant case-studies, targeting the 3 sectors representing 70% of the European plastic demand:

Building

Bio-sourced polyurethane foams (PU) with enhanced vitrimer properties to replace polyvinyl chloride (PVC) for windows frames – with improved insulating properties, and reduced carbon footprint

Transport

Intrinsically recyclable and repairable fiber reinforced epoxy-vitrimer based composite integrating no-releasable flame retardant moieties, as lightweight alternative to metal for the train structure reducing its energy consumption

Packaging

MultiNanoLayered (MNL) reprocessible films involving no compatibilizers to replace currently non-recyclable films composed of multiple high-performance layers, associated with a decontamination process allowing the up-cycling in the food packaging value chain

2. Optimize reprocessing technologies adapted to the new SSRbD systems to support achievement of ambitious recyclability targets
3. Develop a scoring-based assessment that will guide material designers, formulators and recyclers to design SSRbD polymeric materials
4. Merge all data and relevant methodologies in a digital infrastructure



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European Union



SURPASS

June 2022 –November 2025

Safe-, sUstainable- and Recyclable-by design Polymeric systems

A guidance towardS next generation of plasticS

cidetec
surface engineering

<https://www.surpass-Project.eu/>

PROBLEM to be solved

currently		<ul style="list-style-type: none">Most structural parts of the train are made of metalComposites used for secondary structures are lighter but ruled by Fire, Smoke and Toxicity (FST) requirements (EN45545)<ul style="list-style-type: none">Glass/CF have good fire propertiesThe resin has to be improved with flame retardants
recently		<ul style="list-style-type: none">Composites developed (e.g. car-body in MAT4Rail) which meet FST requirements (EN45545)Intrinsically non-recyclable composites: once cured, cannot be reused and end up in landfill or incinerated.
Currently	<div>Conventional epoxy based composites</div> <div>Non-sustainable recycling processes available</div>	<ul style="list-style-type: none">At the end of life NOT SUSTAINABLE (no intrinsically recyclable)<ul style="list-style-type: none">Pyrolysis: high energy consumption processSolvolyis: toxic, hazardous and dangerous chemicals requiredLandfillIncineration plant <div></div>

CIDETEC's role & Approach



CIDETEC has demonstrated on sample parts (TRL 3) that vitrimers are RECYCLABLE

The project will proceed from up to TRL 5 (more relevant parts)



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European Union



Multi-level Circular
Process Chain for Carbon
and Glass Fibre Composites

April 2022 –March 2025

<https://www.mc4-project.eu/>

cidetec
surface engineering

Multi-level Circular Process Chain for Carbon and Glass Fibre Composites

MC4 is entirely funded by the European Union, under the topic HORIZON-CL4-2021-RESILIENCE-01-01 of the Horizon Europe Framework Programme (HORIZON). Grant agreement N° 101057394

Environmental impact of current carbon and glass fiber composites value chains

- 98% of CF and GF EoL composites is landfilled
- 40% of CF is wasted during the production process
- 6-8000 EoL aircrafts by 2030, with OEMs aiming to recycle at least 90%
- +10.000 wind turbines blades made of GF composites cannot be recycled today
- Regulations are evolving: Since 2015, EU regulations have required recycling of at least 85% of EoL materials in the automotive industry

Consortium

MC4 gathers 15 partners from 7 different European countries covering the whole value chains.



Objectives

- To establish a **multi-level circular process** for carbon and glass fibre composites
- To develop **performant and economically realistic processes**
- To enhance the **EU independency** for raw materials and recycling processes

Approach

	Carbon fiber Composites	Glass fiber Composites
Short term MC4 processes: waste reduction	Re-use prepreg scraps directly in the production line	Re-use of mechanically recycled scraps/EoL parts
Long term MC4 processes: EoL recycling	Chemically recycle CF from EoL parts to make new yarns, fabrics and nonwovens.	Re-shape EoL with the use of a dynamic resin
Additional MC4 processes	<ul style="list-style-type: none"> • Infrared sorting of EoL composite parts • Enhanced quality control to assess the suitability of the recycled materials • Set the path for industrial-sized installations 	

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Multi-level Circular
Process Chain for Carbon
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April 2022 –March 2025

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surface engineering

Multi-level Circular Process Chain for Carbon and Glass Fibre Composites

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PROBLEM to be solved with Glass fiber Composites

Any kind of separation of matrix and fibre will :

...Be economically
infeasible

...will inevitably
damaged the sizing

MC4 Objective: EoL recycling

To avoid the need for matrix/fiber separation by
using a vitrimer as matrix that enables the re-
shaping and the re-use of cured EoL parts.

CIDETEC Background and Role

Vitrimers that allow easy re-work and re-shaping of parts
have been demonstrated on sample parts (TRL 3) by
CIDETEC,
but not at industrial scale.

The re-shaping of large parts and the resulting performance
and quality have not yet been investigated.

The project will close this gap and proceed towards more
relevant parts (**TRL 3 to 5**)



**MANAGING
COMPOSITES**

3B the
fibreglass
company

LAB23
supreme urban living

cidetec
surface engineering

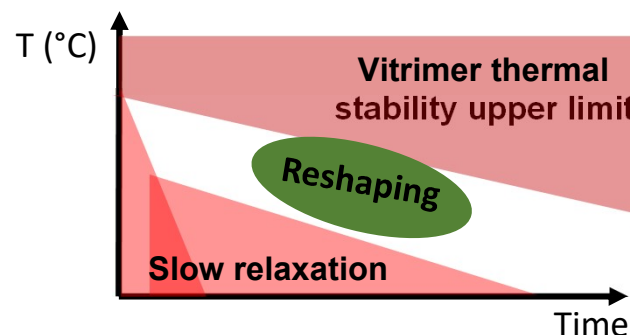
Development and benchmarking of 3R epoxi for kayak manufacturing

GF composite infusion and reshaping trials



Demostration

REQUIREMENTS		Araldite LY1564/ Aradur 3486	MC4 vitrimer
SUITABLE FOR INFUSION?	Viscosity (mPa.s) at 60 °C	23	54
	Infusion temperature (°C)	35	60
	Curing cycle	1.5h 80°C + 5h100°C	1h130°C + 1h150°C
SUITABLE FOR KAYAK APLICACION?	Tg (°C) by DSC / DMA	86 / 91	81 / 94
	Tensile strength (MPa); elongation at break (%)	70.3 MPa	80.4 MPa
		6.4%	6.15%
	Flexural strength (MPa); strain at break (%)	114 MPa	138 MPa
		6.4	6.1%
RESHAPEABLE?	Relaxation time at 180 °C	No	13''



Reshaping conditions:

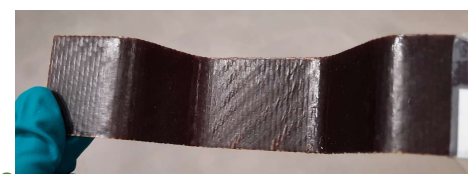
- ✓ Profile: omega, half omega, 90°
- ✓ Mold closing rate: 10-0.5 mm/min
- ✓ Temperature: 180-140 °C
- ✓ Residence time: 10-30 min
- ✓ Pressure/force: 18, 36 KN



Benchmark



Reshaping conditions must
match resin profile to avoid
damage (otherwise behaves
like a thermoset)



- Testing of new geometries and lay-ups and GF sizes.
- Mechanical evaluation of reshaped laminates:
 - NDT: Ultrasound characterization
 - Microscopy
 - Compression tests
- Demonstrations at DEMO level: demonstration of kayak reshaping into new parts.
- LCA analysis
- TRL 3 to 5

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5. Polymeric materials with biodegradability on demand

The use of treated feathers in Plastics...



...increases Circular Bioeconomy

- Use of a **waste** as raw material.
- **Substitute** fossil based resources.

3,6 million tonnes of feather waste generated per year.

CIDETEC has developed a new technology for the valorisation of feather keratin into added value materials (WO 2021/123194 A1)



...adds new functionalities

- Allow **customized biodegradation** of biopolymers.
- **Accelerate disintegration** process.
- May **change the EOL scheme** of biodegradable polymers.
- Input of **Nitrogen** to soil.

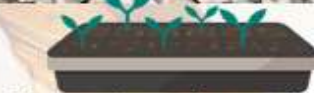


Applications

- **Beneficial** for agricultural applications.
- Other potential applications: **packaging**.



Mulch films



Forest and seed trays



Hydroponic foams



MEMBER OF BASQUE RESEARCH
& TECHNOLOGY ALLIANCE

Thank you very much for your attention

Contact: ejubete@cidetec.es

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