polynSPIRE
Innovative technologies for plastic recycling

Production of POLYNSPIRE training materials

Deliverable 10.4 (v1)

WP10 Communication and dissemination

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Deliverable 10.4 (v1) Production of POLYNSPIRE training materials	CIRCE	24/02/2023	PU

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VERSION RECORD

Version	Date	Author	Description of changes
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V1	27/01/2023	CIRCE	Document creation: Contents v1
V1a	10/02/2023	EuPC	Content's revision
V1a	14/02/2023	IKMIB	Content's revision
V2	21/02/2023	CIRCE	Review comments included
V3	24/02/2023	CIRCE	General review
V4	24/02/2023	CIRCE	Final version submitted to EC

APPROVALS

Author/s	Reviewers
CIRCE	Reviewer 1: EUPC
	Reviewer 2: IKMIB

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EXECUTIVE SUMMARY

During this task, various training and dissemination materials have been developed to train and inform stakeholders in the plastic recycling value chain.

Three training materials were created to cover the three innovation pillars of the project: (i) training module on chemical recycling (including both recycling technologies, microwave and smart magnetic materials) (ii) training module on upgrading recovered material through additive techniques, and (iii) training module on the valorisation of plastic waste as a carbon source in steel furnaces.

Standard presentations were also created using the data collected for these training materials and were adapted for general audiences for use in dissemination events. Additionally, a guidebook was developed to be published on the project website, as well as a brochure to show the solutions of the project. Furthermore, three specific training workshops focused on the project innovation pillars were held during the final polynSPIRE event in November in Mestre, Italy, to promote among industrial sectors the adoption of innovative technologies for reducing plastic waste and reduce plastic pollution. Additionally, some training activities have been organised for schools during the project.

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OVERVIEW OF THE DELIVERABLE

WP: 10

Task: Training resources for the stakeholders involved in the plastic recycling value chain (Task 10.4)

Title: Production of POLYNSPIRE training materials

Training material for the professionals and staff in charge of the processes design and operation have been developed within this task. In addition, standard materials related to the POLYNSPIRE innovations have been carried out, looking to an easy replication in other locations. All partners have contribute to the engagement of professionals and managers from the chemical, plastic and waste management sectors to be aware of the available training resources. Three training materials about plastic recycling technologies and products have been prepared: (i) Training module on Chemical recycling, oriented to the market uptake of the two new recycling technologies (ii) Training module on Upgrading of recovered material through additivation techniques. (iii) Training module on Valorisation of plastic fibres and the use of plastic waste as carbon source in steel furnaces. Finally, a guidebook gathering a standardized version of these training materials has been developed and published on the website. A specific brochure for policy makers has been developed and distributed in order to make them aware of the opportunities in the plastic recycling industry. Apart from the material for the formative modules, three specialized training workshops (one per each training module) have been performed during the final polynSPIRE exploitation event. These workshops have been industry-oriented so the content of the training material used have been adapted to the specific audience.

LIST OF ABBREVIATIONS AND ACRONYMS

D – Deliverable
MW – Microwave
SMM – Smart magnetic materials
WP – Work Package

1 INTRODUCTION

This deliverable 10.4 gathers the different training materials and training activities that have been developed during the project for the stakeholders involved in the plastic recycling value chain. These material and activities are:

- Three training materials of the innovation pillars of the project: (i) training module on chemical recycling (including both recycling technologies, microwave and smart magnetic materials) (ii) training module on upgrading recovered material through additive techniques, and (iii) training module on the valorisation of plastic waste as a carbon source in steel furnaces.
- Three standard presentations of the innovative technologies.
- A general guidebook of the project to be published on the website.
- A specific brochure for policy makers.
- Three training workshops oriented to the industrial sector.
- Additional training activities for schools

Each point is described in detail in the following sections.

2 CREATION OF TRAINING MATERIALS

In this task three specific training materials have been developed for the professionals and staff in charge of the process design and operation of the innovative project processes. Once the project's demo cases are under operation, it is important to ensure that the people involved are well informed about how the different technologies work and are aware about different health and safety issues. For this purpose, these training materials have been performed to gather all the information available from different innovative technologies.

Three different presentations have been developed (in PowerPoint format) within this task: (i) Training module on Chemical recycling, oriented to the market uptake of the two new recycling technologies, microwave and Smart magnetic materials (ii) Training module on Upgrading of recovered material through additivation techniques, (iii) Training module on valorization of plastic waste as carbon source in steel furnaces.

Figure 1. Training modules of polynSPIRE innovations.

Each formative module is divided into three different chapters:

- Introduction

  This part includes a brief description of the fundamental concepts of each technology, advantages of using these innovative technologies (vs. conventional processes) and kind of materials involved in each technology.

- Installation, starting up, operation and turnoff

  This chapter describe the main tasks that have been carried out during the project to develop each innovative technologies and show a brief description of what kind of equipment are involved and how they work.

- Health and safety issues.

  This part collects the main health and safety issues identified for each technology, to be sure that all partners involved in these technologies are clearly aware of them.

CIRCE has coordinated and gathered the information that has been included in the training resources, but the support of the partners involved in the project technologies development has been essential.

PolynSPIRE partners have provided their feedback in those parts of the module where they have been most involved during the project. The following table shows the contribution of the different partners.

Table 1. Contributions of the partners to the training materials.

Innovative technology	Fundamental concepts	Installation, starting up, operation and turnoff	Healthy and safety issues
MW chemical recycling (WP3)	NIC	CIRCE / F&M	F&M
SMM chemical recycling (WP4)	IONIQA	IONIQA / CPPE	IONIQA / CPPE
Additivation techniques (WP5)	TUE / AITIIP	TUE / AITIIP	TUE / AITIIP
Valorisation in steel sector (WP6)	RINA / IBLU	HTT / RINA	HTT / RINA

The training materials have been created in PowerPoint format, and once finalised they have been sent to all the partners of the project.

These presentations will be included in annexes.

3 CREATION OF STANDARD MATERIALS

Taking advantage of all the information gathered for the training materials developed for each innovative pillar, different standard presentations have been created.

Training materials presentation have been adapted to create three presentations oriented to the plastic sector public. These presentations can be used for all the partners to show the results of the project in workshops or in other events to try to engage the professionals and managers from different industrial sectors (plastic, chemical and waste management sectors), and looking for an easy replication of the technologies in other locations.

The standard materials have been created in PowerPoint format, and they have been shared with all the partners of the project.

These presentations will be included in the annexes.

4 CREATION OF GUIDEBOOK

Apart from the training and standard presentations, a guidebook gathering all the information has been developed.

This guidebook includes all the important information regarding the different innovation technologies developed in polynSPIRE project. The information required for the development of this guidebook has been mainly obtained from the previous information gathered for the training materials and additional information have been extracted from several deliverables submitted by different partners in previous months.

The guidebook includes the following information about the project:

1. Overview of PolynSPIRE project
    1.1. PolynSPIRE background
    1.2. PolynSPIRE innovation
2. Innovation pillar A: Chemical recycling
    2.1. MW recycling
    2.2. SMM recycling
3. Mechanical recycling
    3.1. Vitrimers
    3.2. High energy irradiation (gamma radiation)
    3.3. Additives and fillers
4. Valorisation of plastic waste in steel furnaces

The guidebook will be published on the website to be available to anyone interested in the project solutions looking for an easy replication of the technologies in other locations.

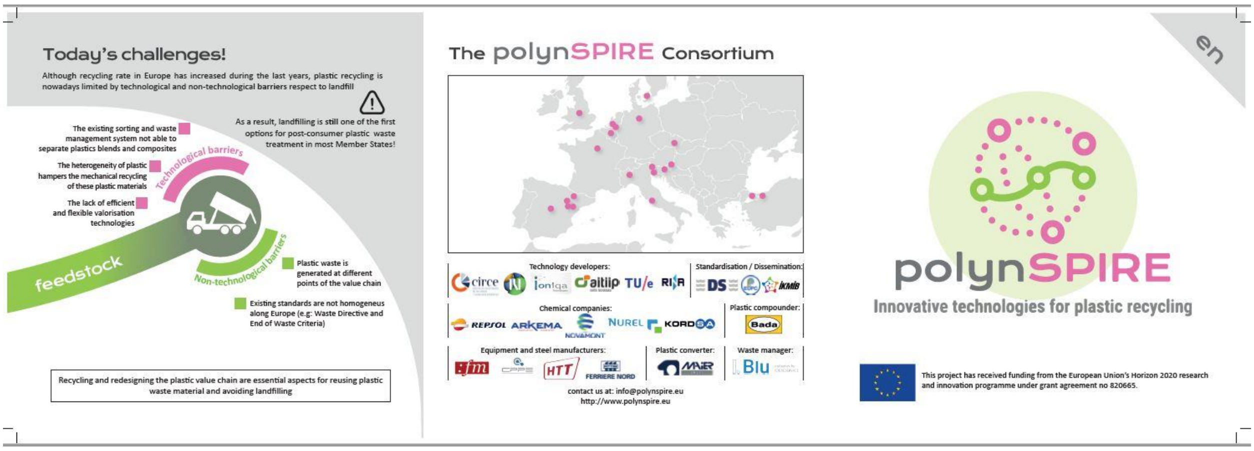
This guidebook will be included in the annex.

5 CREATION OF BROCHURE

A specific brochure for polynSPIRE project has been created. This brochure provides information about the concept and the innovative solutions of the project. It includes a brief overview of the today´s challenges in the plastic recycling sector, a description of the solutions proposed, and shows the partners involved in the project.

This brochure is a key means of communication and it has been distributed in different events, as well as in the offices of project partners. The finality of this brochure is to inform and promote the project to different stakeholders such as researchers, policy makers, industry partners, funding organizations, clients and the public in general. It aims to attract attention and interest, establish credibility, and showcase the project's value and impact.

The brochure's design aligns with the polynSPIRE logo, consistent with the rest of the project's communication materials. The two versions of the brochure can be accessed on the website (www.polynspire.eu). If reading this document on a digital device, the reader may find the English version here and Spanish version here.



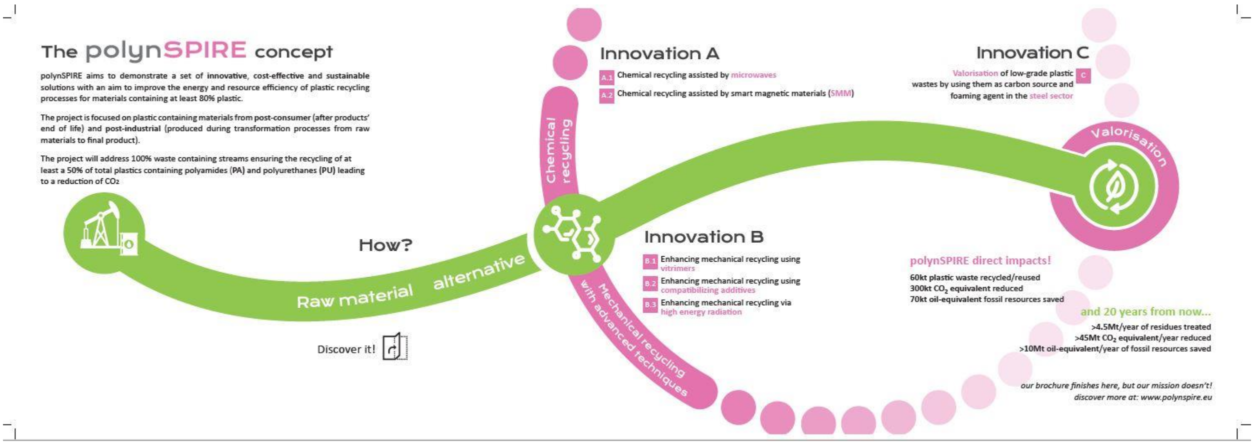
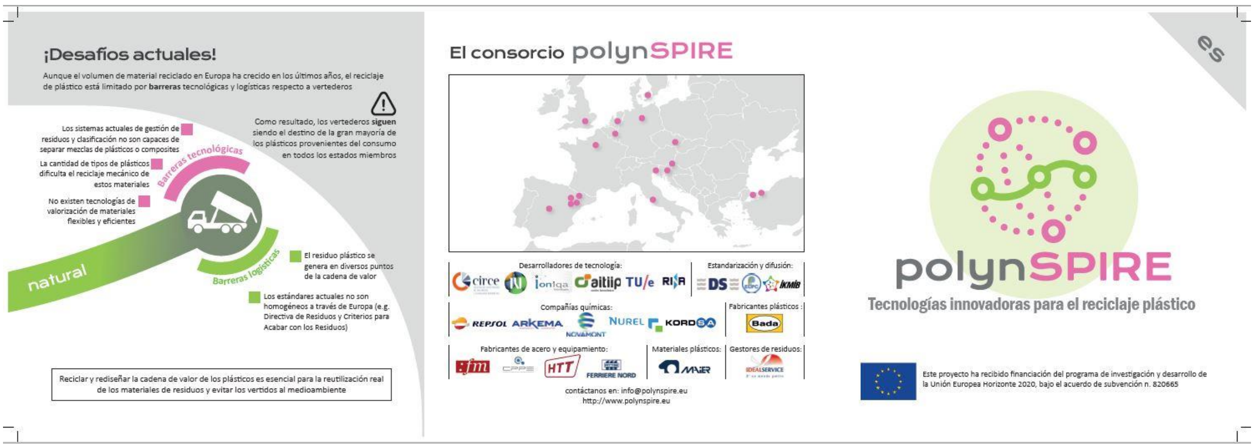


Figure 2. polynSPIRE Brochure (English version)



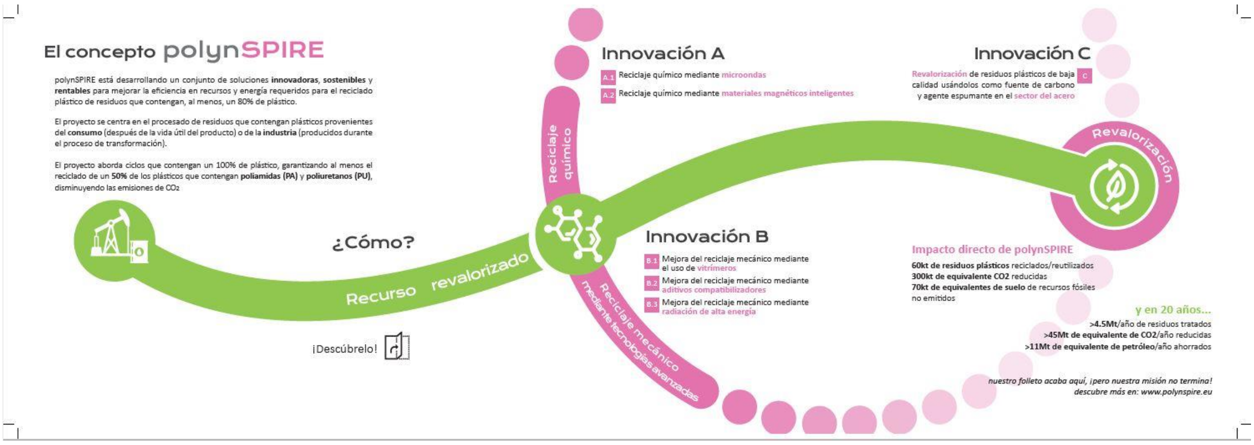


Figure 3. polynSPIRE Brochure (Spanish version)

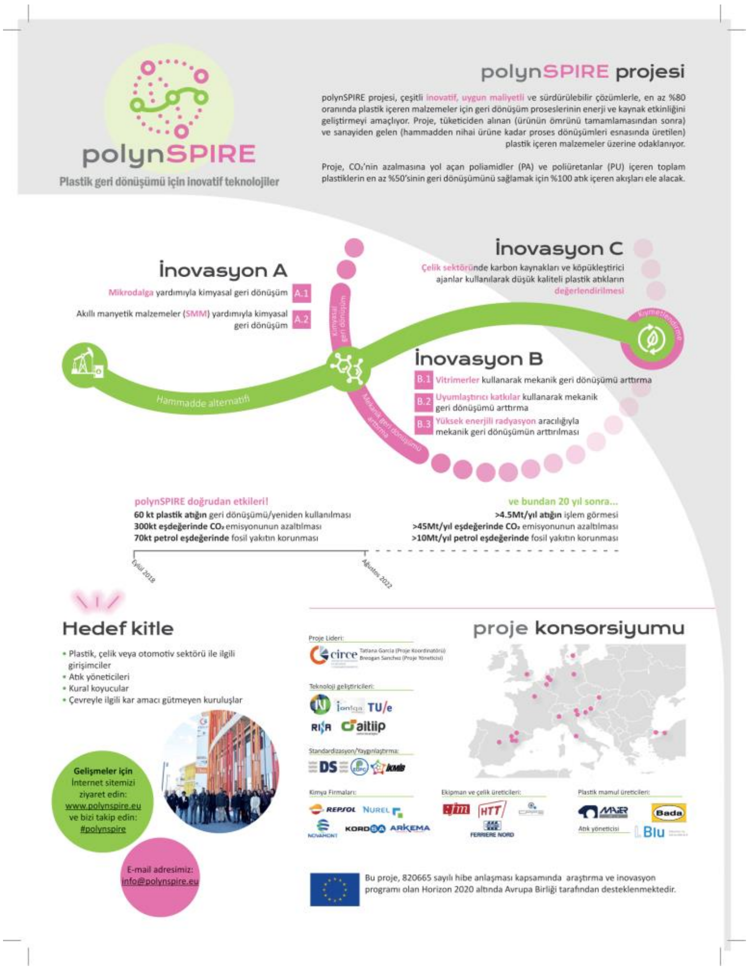


Figure 4 polynSPIRE brochure (Turkish version)

6 TRAINING MODULES & WORKSHOP

As stated in DoA, apart from the material for the formative modules, three specialized training workshops (one per each training module) were to be performed. Due to COVID-19 situation, and the difficulties to organize presential activities with external attendees, the workshops were delayed until the last project period and incorporated as part of polynSPIRE's final event, taking advantage that a presential industry-oriented event was to be organized within the exploitation work package (WP9).

The workshop was divided into the three formative modules developed by polynSPIRE: Chemical recycling, (i) Training module on Chemical recycling, oriented to the market uptake of the two new recycling technologies (ii) Training module on Upgrading of recovered material through additivation techniques. (iii) Training module on Valorisation of plastic fibres and the use of plastic waste as carbon source in steel furnaces.

The trainers were the main partners from polynSPIRE involved in the development of each innovation. In order to dynamize participation of the attendees, the workshops were imparted in form of round tables, were innovation developers participated as presenters, and a Q&A session was enhanced by another relevant partner acting as moderator of the table and the audience.

Figure 4 Training module I (Chemical Recycling)
From left: Seda Araci (KORDSA)- Moderator; Alberto Frisa (CIRCE); Katarina Babic (IONIQA) and David Pahovnik (NIC)

In each roundtable, the following questions were discussed, and noted as the main topics of interest for the industry representatives:

<u>Chemical recycling</u>

- Advantages of the technologies (Microwave and SMM) versus other depolymerization technologies.
- How the microwave technology and SMM technology developed in this project differentiate from other technologies developed for PA and PUR recycling? Such, e.g. technologies developed by Pyrowave (for PS recycling), Gr3n (for PET), ENVAL (for mixed plastics), Microwave Chemicals (for PMMA and other plastics)? (SEDA)

- What about additives that we find in plastics? Mineral fillers, pigments, plasticizers… can they interfere during depolymerization or isolation?
- During the presentations, the results of recovered monomer polyol are shown. What about isocyanate part of the polymer? What is the main problem of isocyanate part recovery?
- How important is quality of the monomer for the process?
- The hydrolysis of polyamides with HCl as catalyst should generate chlorinated amine or amine salts. How difficult it is to free the amine groups?
- What is the biggest technical challenge that you faced during the project?
- What is the specific energy consumption (ex in MJ/kg of monomer or polymer) at the depolymerization stage and at the purification stage? How the energy consumption compares with virgin monomer?
- Looking at the various polyamides tested, which ones are the easiest to depolymerize and purify? Between aminoacids and diacids-diamines is there a major difference? What about polyphthalamides?
- The microwaves are absorbed better by some sensitizing agents like carbon (graphite, carbon fibers…, but also Silicon Carbide…). Is there a need for these additives with these polymers and with the HCl catalyst?
- All product tested were post-industrial wastes or also post-consumer wastes? Did you consider some specific challenges with the post-consumer wastes?
- Magnetic Materials could contaminate also the recycled monomers. Did you investigate that? What is the retention rate? Is there any leaching of the magnetic material?
- Microwaves have also a limitation in penetration depth. What would the size/capacity of a standard plant? How do you address the issue of penetration depth?
- How much is the reduction of the batch time in microwave and smart magnetic particles with respect to conventional technologies?
- What is the maximum scale that you consider can be reached with a microwave reactor?
- Can these technologies be implemented in existing reactors?
- How is the microwave reactor be cooled? How long does it take to cool down? Is it needed another vessel to cool the product?
- Can foam be fed into the microwave in a continuous way, or only at initial before heating?

Figure 5 Training module II (Training module on Upgrading of recovered material through additivation techniques)
From left: Carlos González (CIRCE)- moderator; Hans Heuts (TUe); Julio Vidal (AITIIP) and Víctor Peinado (BADA)

Mechanical recycling

- What is the time spent in material treatment?
- As we would be using radioactive material as a source of radiation for the crosslinking generation, what would be the proper procedure to deal with the material after? Or would we have radioactive activity in the material?
- Is it possible to use this technology with other materials such as thermoset materials or any other thermoplastic material?
- What does produce the color change in the material during radiation?
- What is the most appropriate procedure: radiating the materials before or after extrusion?

Figure 6 Training module II (Valorisation in steel sector)
From left: Elisa Marchensan (IBLU); Alberto Frisa (CIRCE) - Moderator; Filippo Cirilli (CSM) and Jaroslav Brhel (HTT)

Valorisation in steel sector

- What is the influence of the material heterogeneity of plastic during the process? Should been selected specific types of material?
- Different parameters and elements are present in the injected plastic waste material. Presence of chlorine seems to be a challenge in the process. Can it be ensured that the developments carried out overcome this difficulty?
- Can it be confirmed that 1 kg of coal is equal to 1 kg of plastic material substitution?
- Does a complete replacement of coal with polyolefins make sense or is it technically necessary to keep a proportion of coal? Can it be increased in the future the current replacement percentage?
- [to HTT] About the plastic material injector:
    - Has the useful life been studied? The process is more aggressive over the injector than in other type of injector in the same system?
    - What is better, one big injector or to integrate several injectors in different points of the EAF?
- Is there some influence over electrical consumption?
- Have some risk or difficulties the storage of plastic material in silo?
- About achieved results, how much tests have been performed? Are they relevant for a feasible process?

- To talk about advantages in relation to the EU-ETS, measurement of the reduction of the carbon footprint, exchange of emission rights between participating companies, relevance of the proximity between I.BLU and FENO due to lower emissions in material transport.
- What are the main impacts achieved in the EAF process?

7 TRAINING ACTIVITIES FOR SCHOOLS

The partner IBLU has organised some dissemination and training events in three selected schools of the Italian region Friuli Venezia-Giulia. The focus of the interventions was placed on the activities of WP6, and therefore to the use in steelmaking of recycled polymers (R-PMIX-SRA, BLUAIR) deriving from the recycling operation of mixed plastic packaging waste that is otherwise considered non-recyclable. The selected schools are the following:

- Istituto Saleniano Bearzi (Udine), in particular the sections of: Technical and Indutrial Institute and the Professional Training Centre
- Enaip (Enaip FVG - Centro Servizi Formativi di Pasian di Prato (UD)), in particular the Professional Training and Education school section
- IC Rilke – Secondary School Carlo De Marchesetti (I level)

The above listed events were planned during the month of February 2023, to disseminate the activities carried out and the results of the project achieved in Work Package 6, in the context of the broader plastic recycling industry and sustainability practises.

8 CONCLUSION

During the project different training and dissemination materials have been developed for the stakeholders involved in the plastic recycling value chain.

Firstly, three specific training material have been created in the form of PowerPoint presentations; one per innovation pillar of the project: (i) training module on chemical recycling (including both recycling technologies, microwave and smart magnetic materials) (ii) training module on upgrading recovered material through additive techniques, and (iii) training module on the valorisation of plastic waste as a carbon source in steel furnaces.

Subsequently, three standard presentations have been created using all the data collected for the training materials developed for each innovation technology. These presentations have been adapted for the general public in order to be available for all the partners and be used in different dissemination events, showing the benefits of the project.

In addition to these training modules, a guidebook has been developed to be published on the project's website. This guidebook will provide a standardized version of the training materials that can be used in a wider range of contexts and will not be specific to the demonstration sites. Also, specific brochure for policy makers has been created and distributed in order to make them aware of the opportunities in the plastic recycling industry.

Finally, during this task three specific training workshops (one per training module) were held during the final polynSPIRE event which took place on Tuesday 8th November in Mestre (Italy). These workshops have been oriented to the industrial sectors as an important step towards increasing the adoption of innovative technologies for reducing plastic waste and addressing the issue of plastic pollution.

Additionally, some dissemination and training activities have been organised in three Italian schools focused on the use of recycled polymers in steelmaking.

LIST OF FIGURES

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ANNEXES- MATERIALS DEVELOPED

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ANEX A. TRAINING MATERIALS

TRAINING MODULE ON CHEMICAL RECYCLING

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Innovative technologies for plastic recycling

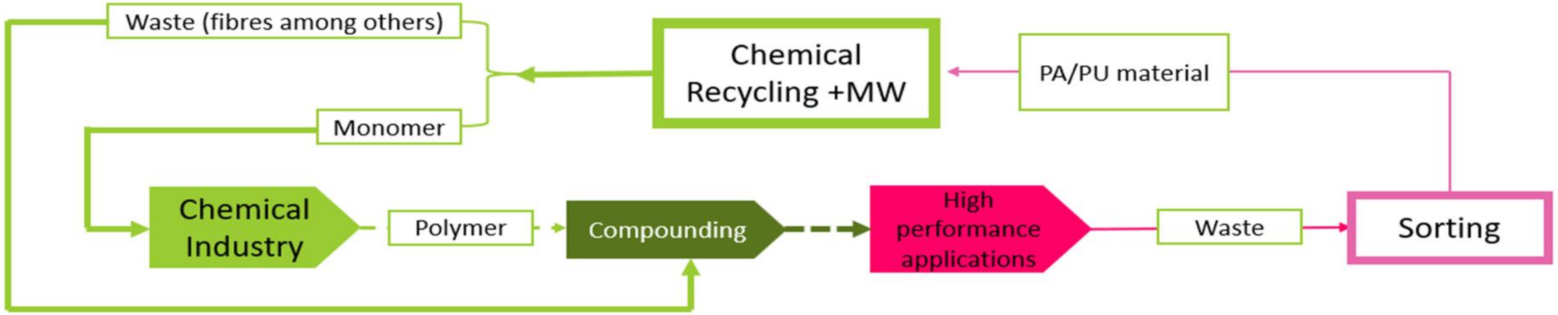
Chemical recycling assisted by microwaves & Chemical recycling assisted by smart magnetic materials

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

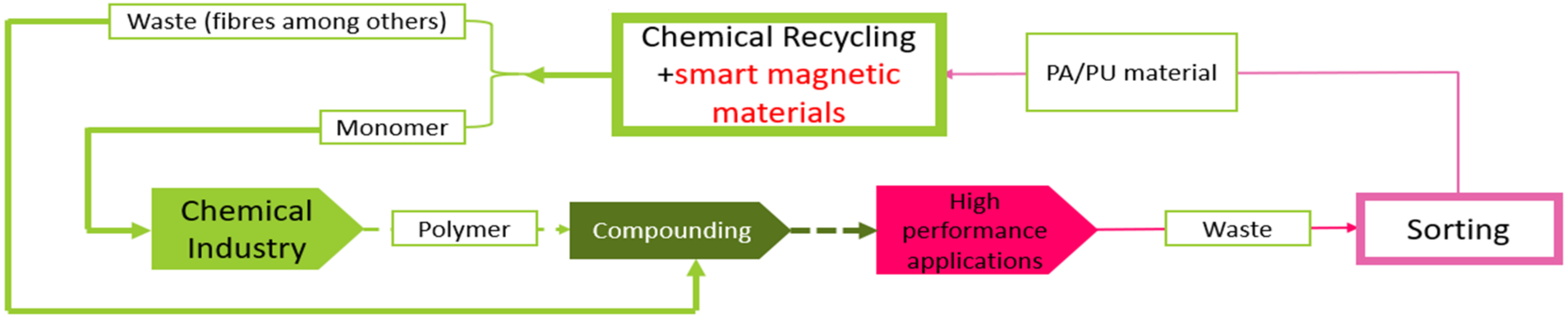
Chemical recycling

- The following technologies are used to depolymerize plastic materials to obtain their monomers (raw materials for polymer production)

Chemical recycling assisted by microwaves



Chemical recycling assisted by smart magnetic materials



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Innovative technologies for plastic recycling

Chemical recycling assisted by microwaves

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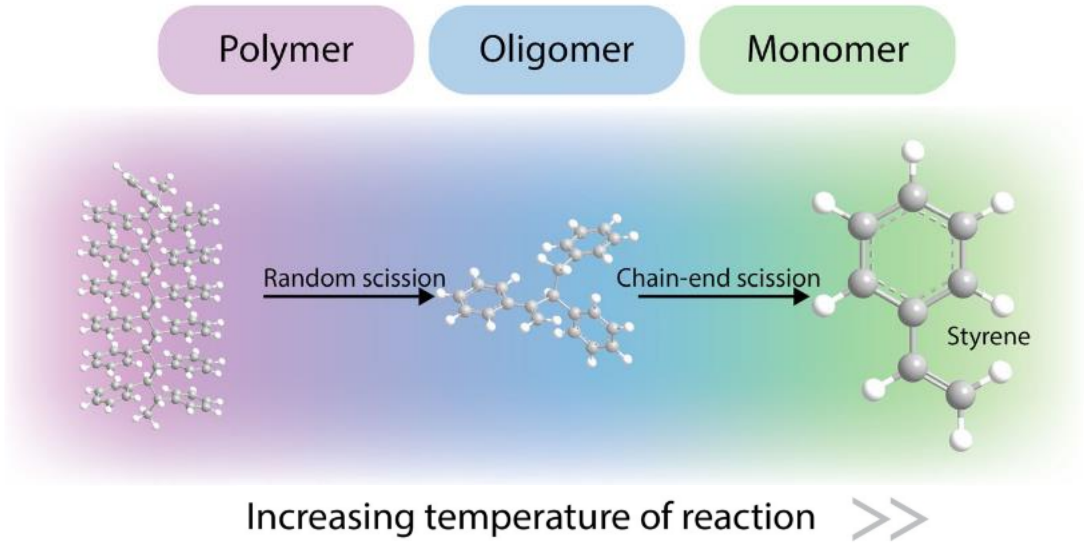
Fundamental concepts

Microwave heating for plastics recycling

- *Microwaves are electromagnetic radiation with wavelength ranging from 1 mm to 1 m in free space with a frequency from 300 GHz to 300 MHz*
- *Microwave heating (MW) allows efficient recycling of different kind of plastics (PA, PU) to recover valuable secondary products (monomers, polyols) for synthesis of new products with comparable or the same characteristic.*



Plastic waste (pellets, foams, …)

Highly MW-absorbing digestion media

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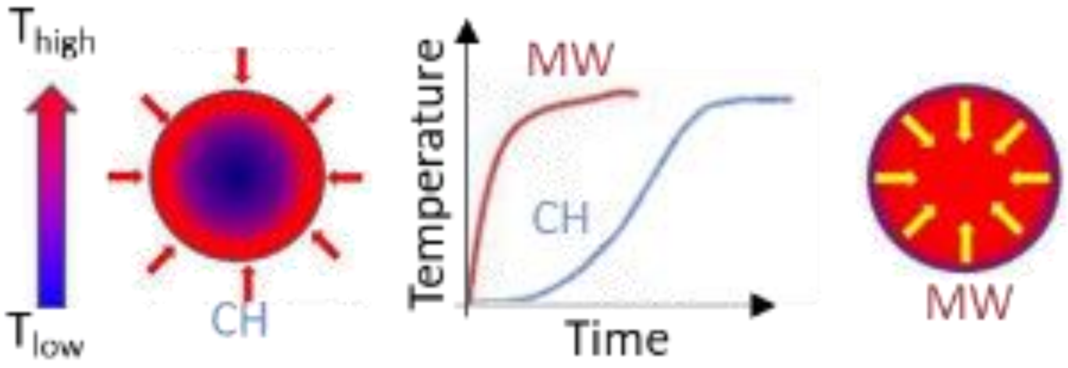
Fundamental concepts

Microwave heating for plastics recycling

- *In MW heating, energy is delivered directly to materials through molecular interactions with electromagnetic field.*
- *Less temperature gradient compared to conventional heating.*
- *Selective and volumetric heating. Improvement in process control.*
- *High energy efficiency.*
- *Rapid operating times.*
- *Electrification: operation driven by renewables. CO_2 footprint reduction.*
- *Microwave heating (MW) allows efficient recycling of different kind of plastics (PA, PU) to recover valuable secondary products (monomers, polyols) for synthesis of new products with comparable or the same characteristic.*



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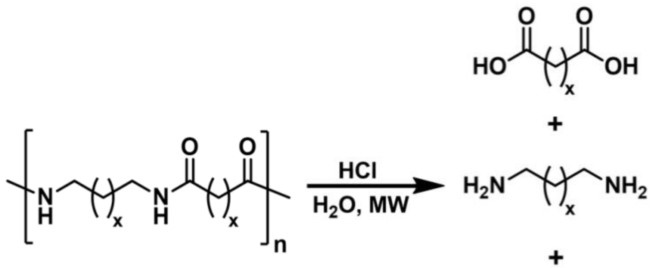
Main results of the project

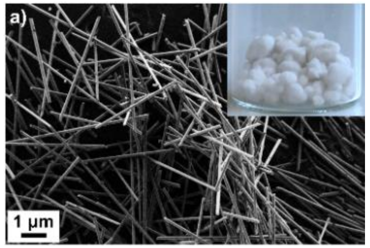
MW-assisted polyamide depolymerization to recover the recycled diacid and diamine monomers (PA66, PA1010) or amino acid monomers (PA11, PA12)



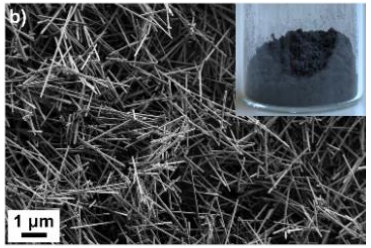
PA66: x = 4
PA1010: x = 8

Reinforcement Additives

Reinforcement additives



Glass fibers



Carbon fibers

Hot filtration if necessary

PA

HCl/H_2O

Microwave heating

Crystallization to recover diacid or aminoacid monomers

Distillation to recover diamine monomers

Monomers

Sample	n(HCl)/n(amide)	MW conditions T (°C)	MW conditions t (min)	Recovered monomer/ Reinforcement additive	Yield (%)	Purity* (%)
PA66	1.25	200	10	AA HMDA	90 86	100.6 ± 0.2 100.1 ± 0.3
$PA66\text{-}GF_{35}$	1.25	200	15	AA HMDA GF	83 81 97	100.7 ± 0.2 100.1 ± 0.4 /
PA11	2.5	200	10	11-AUDAxHCl	93	100.1 ± 0.2
$PA11\text{-}GF_{30}$	2.5	200	15	11-AUDAxHCl GF	71 97	99.8 ± 0.1 /
$PA11\text{-}CF_{30}$	2.5	200	20	11-AUDAxHCl CF	72 99	99.3 ± 0.2 /
PA12	2.5	200	10	12-ADDAxHCl	97	99.9 ± 0.5
$PA12\text{-}GF_{50}$	5	200	15	12-ADDAxHCl GF	77 97	97.1 ± 0.3 /
PA1010	2.5	200	17	SA 1,10-DDA	89 78	100.4 ± 0.8 100.0 ± 0.2

U. Češarek, D. Pahovnik, E. Žagar *ACS Sustain. Chem. & Eng.* **2020**, *8*, 16274

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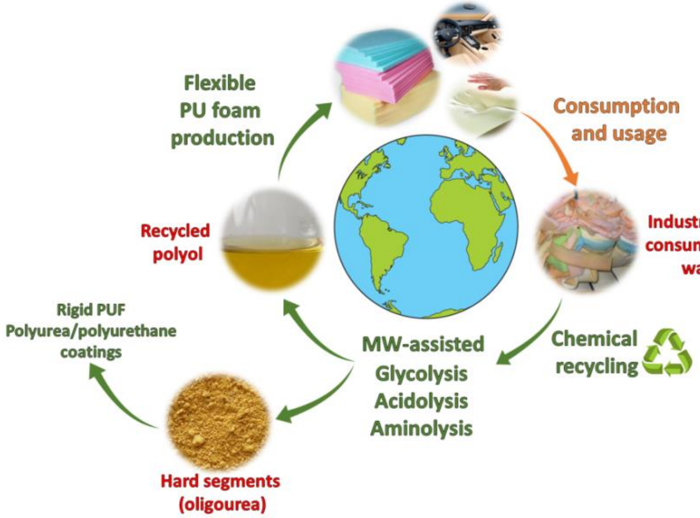
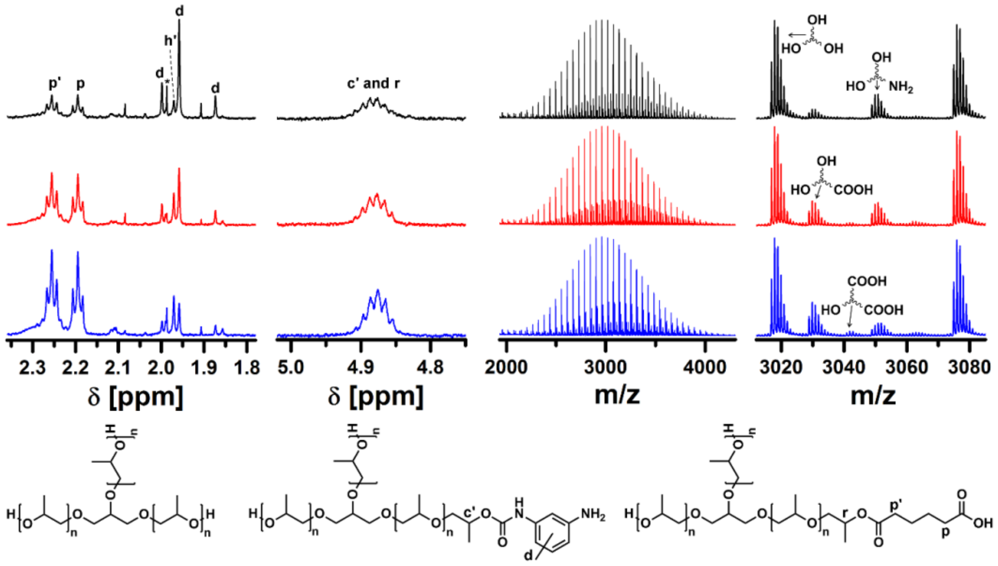
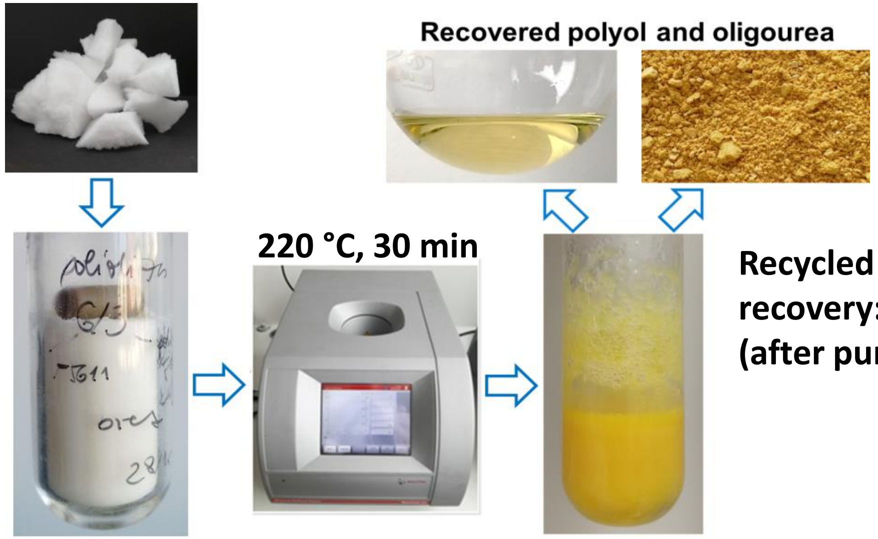
Main results of the project

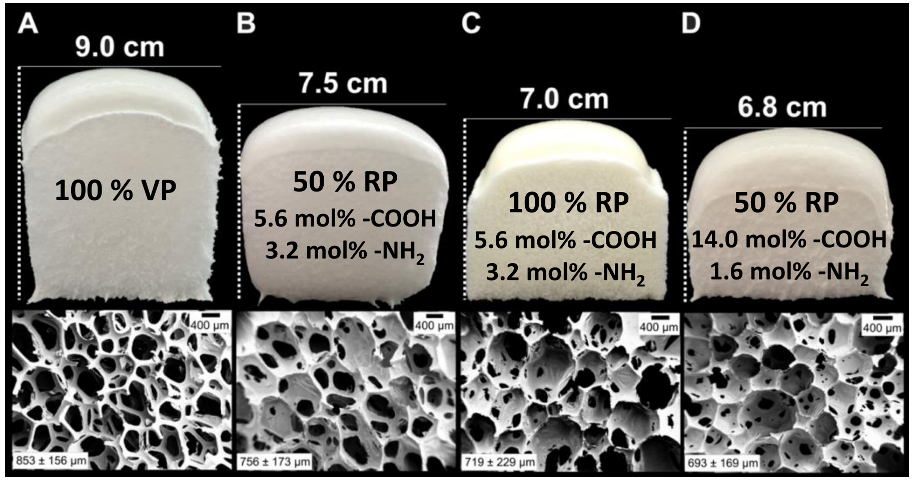
Chemical recycling of flexible polyurethane foams (PUFs) by acidolysis







Recycled polyol recovery: over 85% (after purification)



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ACS Sustain. Chem. & Eng. **2022**, *10*, 1323

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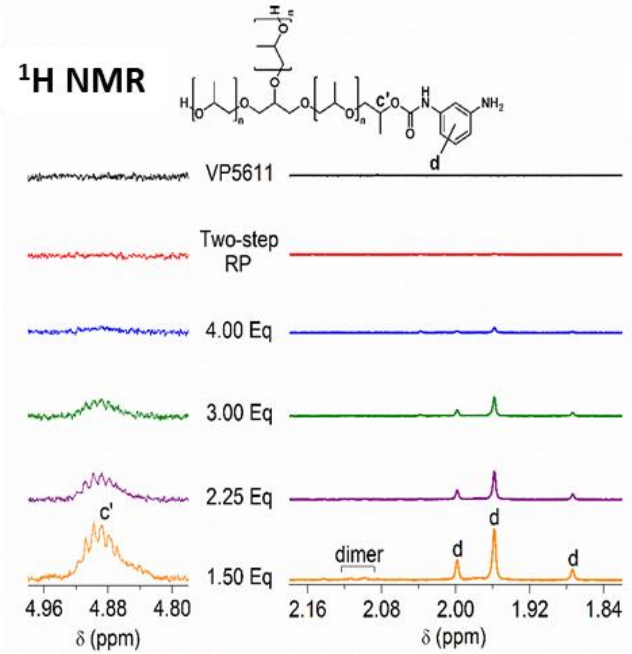
Main results of the project

Chemical recycling of flexible polyurethane foams (PUFs) by aminolysis



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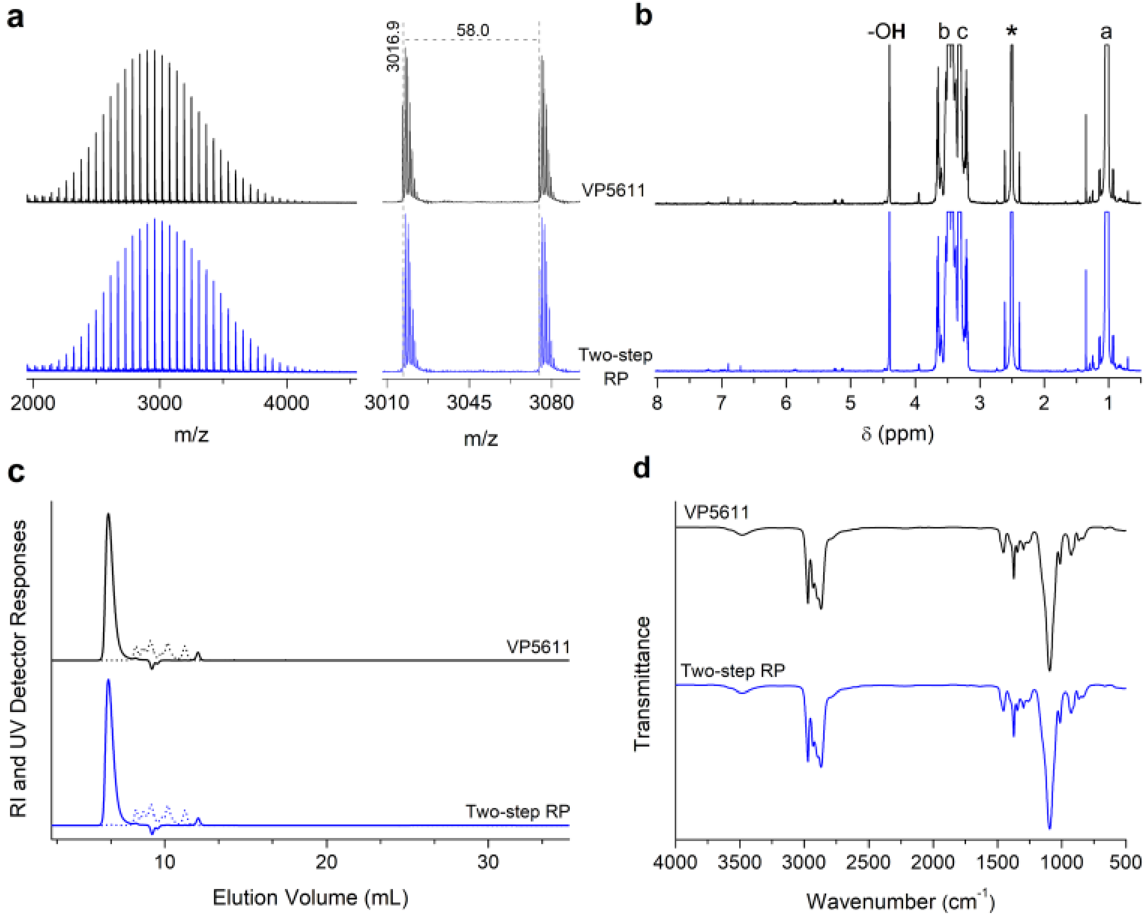

0 20 50 100

Content of recycled polyol in PUF formulation in wt%

Patent application No.: LU501979

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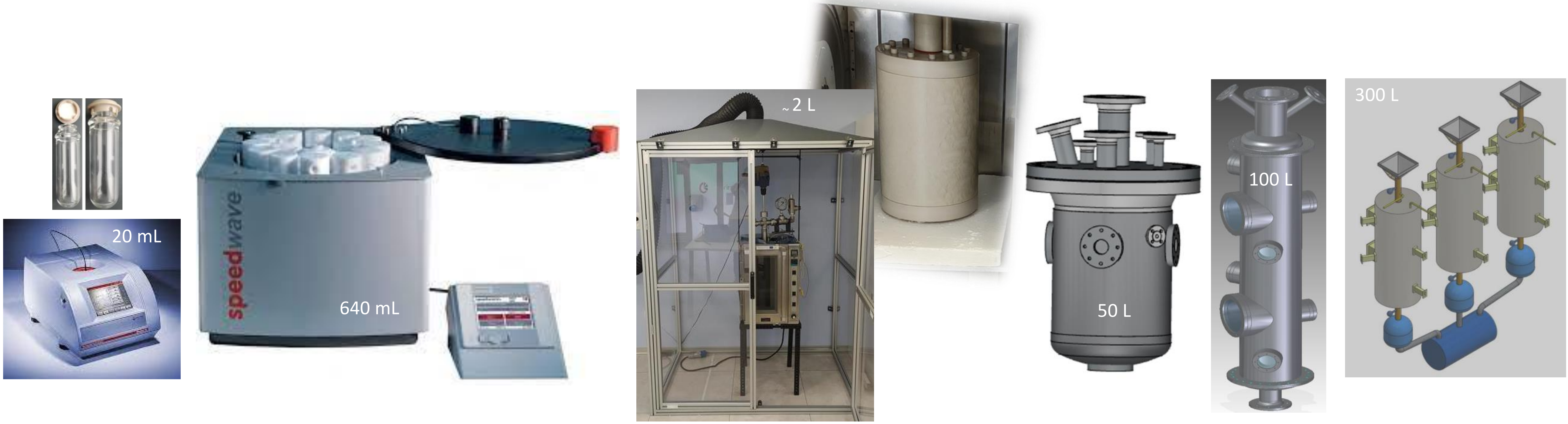
Main results of the project

Upscaling of polyamides (PA) and polyurethanes (PU) MW-assisted depolymerization processes.



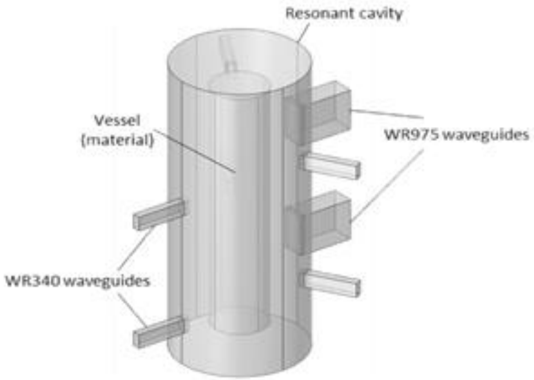
Processing capacity

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

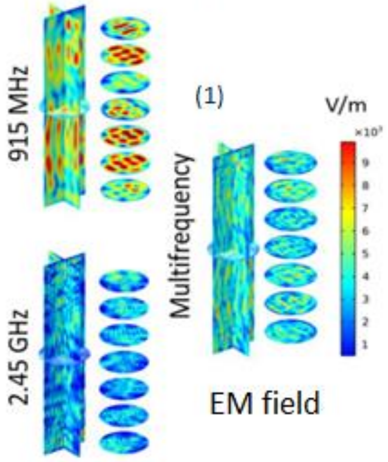
Main results of the project

MW-depolymerisation scale-up pilot: materials and configutations

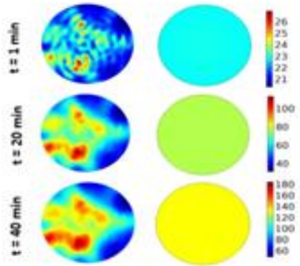
PEEK stirred tank
(T < 300ºC, P < 20 bar)



Initial multifrequency configuration

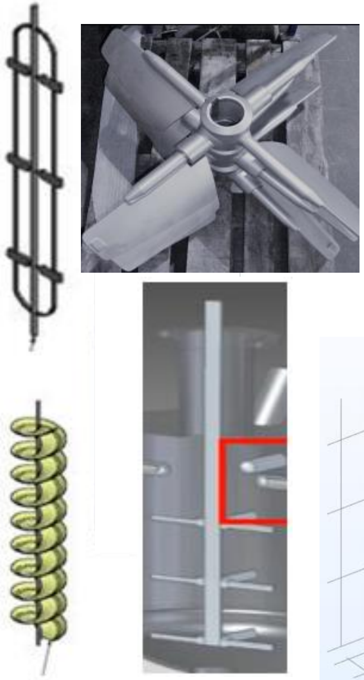


EM field

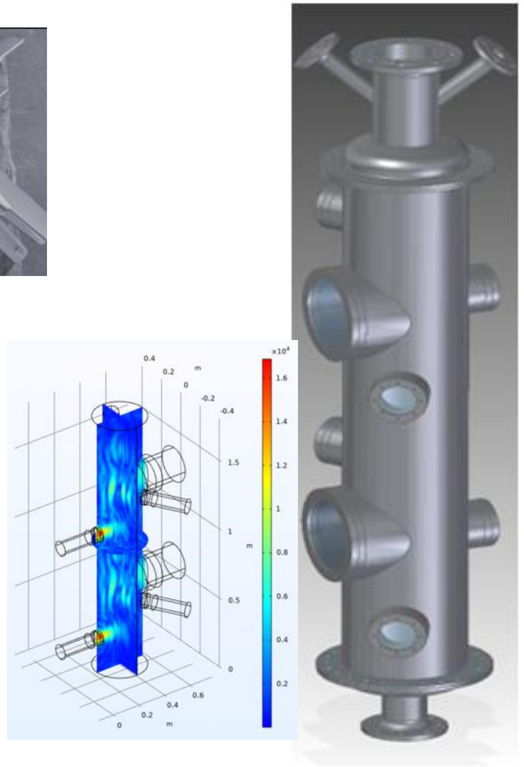


Temperature distribution without/with stirring
(1 m away from reactor bottom)

(1) A. Frisa et al. Cleaner Eng. Technol. 5 (2021) 100297



Mechanical stirrer configurations (axial)



100 L multi-frequency oven (waveguide)

Numbering-up strategies
(basic engineering design)

Teflon-lined SS
(rectangular waveguide)

50 L inconel tank (antenna)

The project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement no 820665

Healthy and safety issues

Microwave reactor:

Microwave radiation. Caution! Danger Zone! Microwave radiation may be present!

Person with pacemaker(s) or any other electronic body implants is not allowed to be near the microwave system

High voltage!!! Caution! Danger zone! Stay away from these areas

Hot surfaces!!! Caution! Do not touch! Surface may be very hot.

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Healthy and safety issues

Chemical materials:

Ethylene glycol

Serious health hazard and danger signals

Hydrochloric acid

Skin corrosion and danger signals

Hexamethylene diamine

Skin corrosion and danger signals

Adipic acid

Health hazard

The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

polynSPIRE

Innovative technologies for plastic recycling

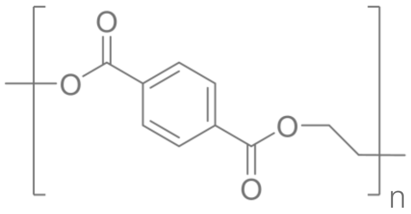
Chemical recycling assisted by Smart Magnetic Materials
WP4

The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

Fundamental concepts

IONIQA **developed and scaled-up** recycling technology for **PET**, both for **packaging** and **fiber** post-consumer feedstock

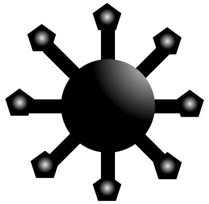
PET



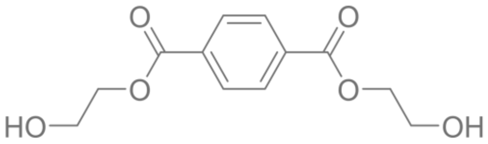
Glycolysis

+ Ethylene Glycol (EG)
and

Smart Magnetic Fluid
(SMM)



BHET
bis (2-hydroxyethyl) terephthalate

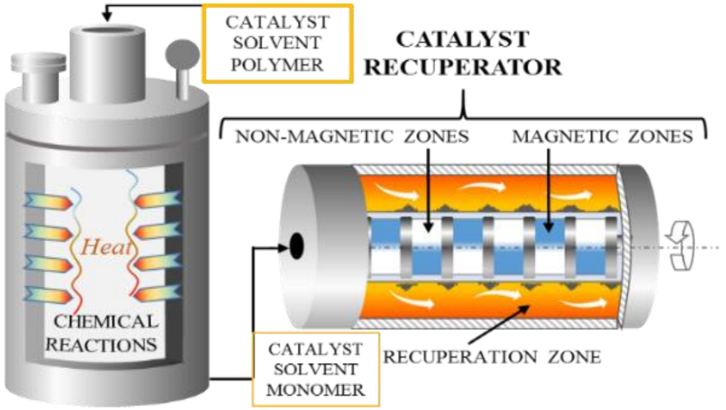


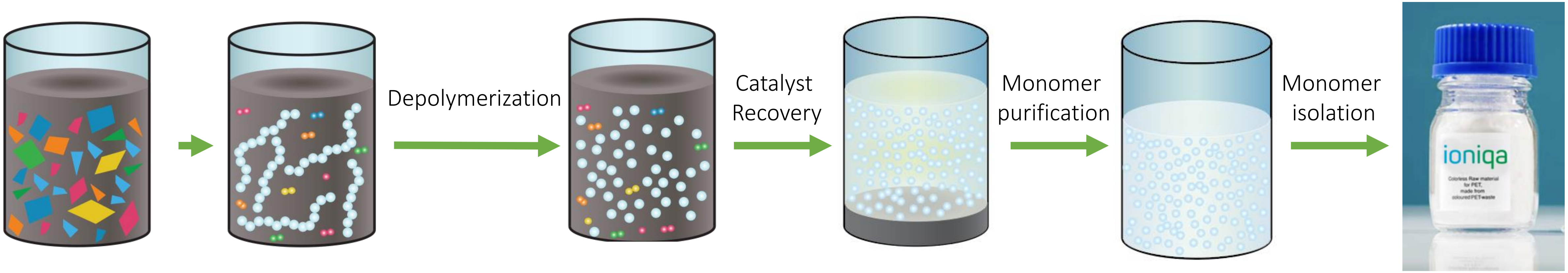
The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

Fundamental concepts

Smart Magnetic Fluid → Functionalized magnetic nanoparticles

- Better yield and selectivity than benchmark catalysts
- Easily recoverable from the reaction mixture and can be reused



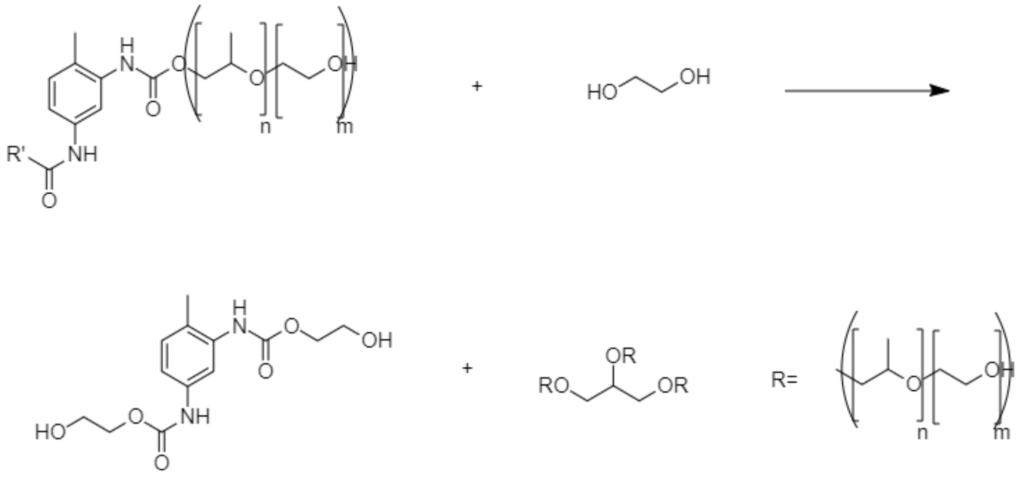


The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

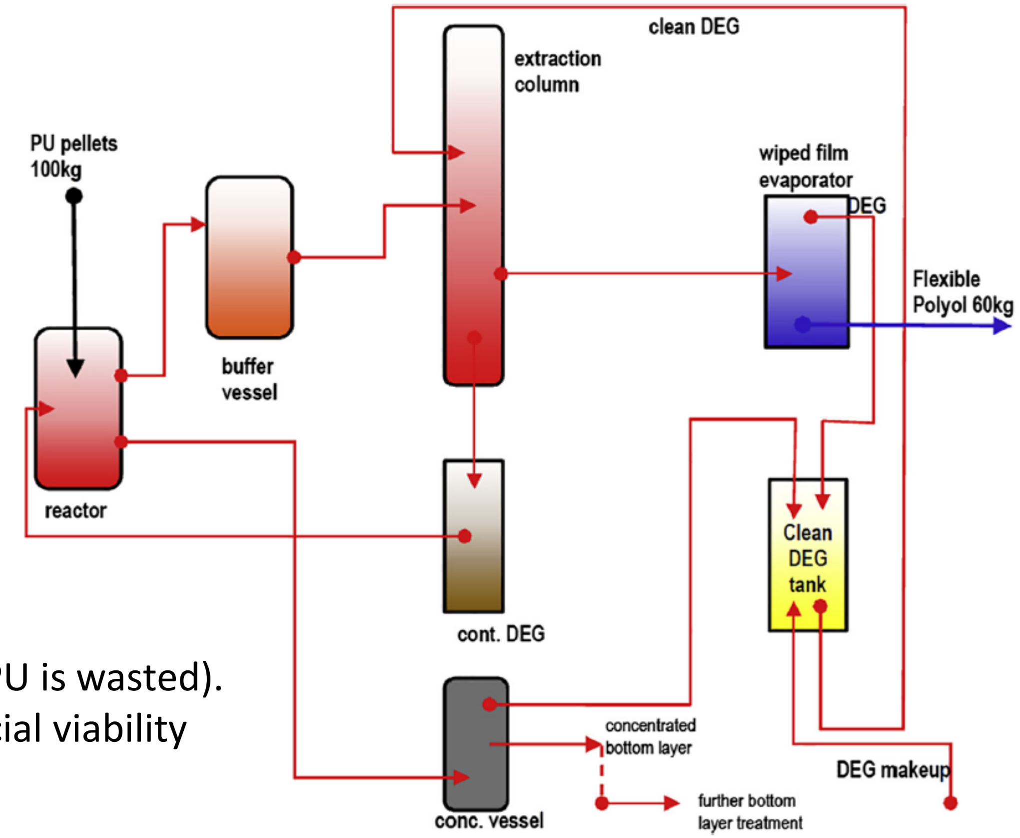
Installation, starting up, operation and turn off

PU recycling:

- Process selection based on literature: split-phase glycolysis → minimal by-products (polyol and carbamate), easier product separation



ICI-Huntsman process (according to literature)



Main obstacles include:

- product purification,
- glycol and catalyst recycling,
- product yield and
- production of large amounts of waste (30-40 wt% of the PU is wasted).

If these obstacles can be overcome, the chances for commercial viability are considered to be positive.

D. Simón, A. M. Borreguero, A. de Lucas, and J. F. Rodríguez, “Recycling of polyurethanes from laboratory to industry, a journey towards the sustainability,” *Waste Manag.*, vol. 76, pp. 147–171, 2018.

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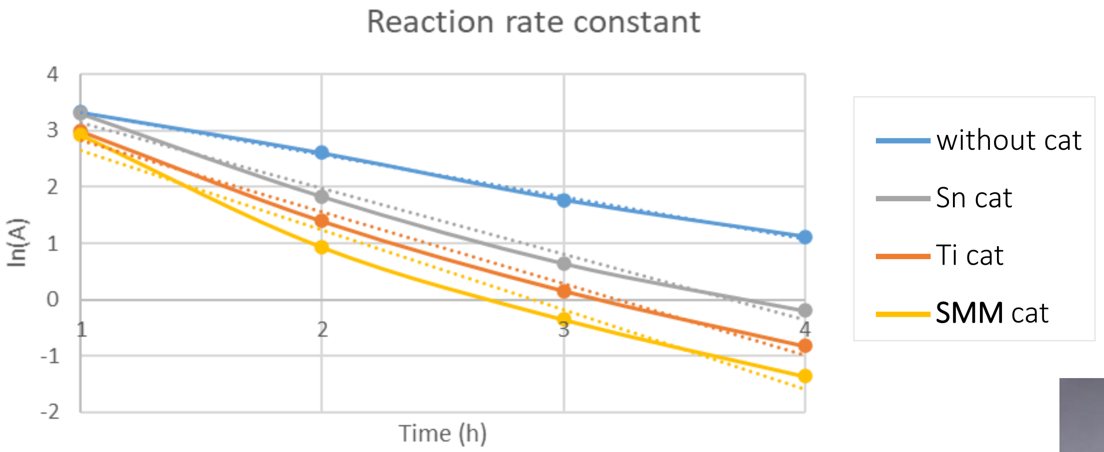
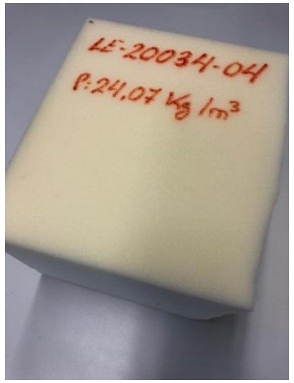
Installation, starting up, operation and turn off

PU recycling by glycolysis:

SMM catalyst performs **better** than state-of-the-art catalysts

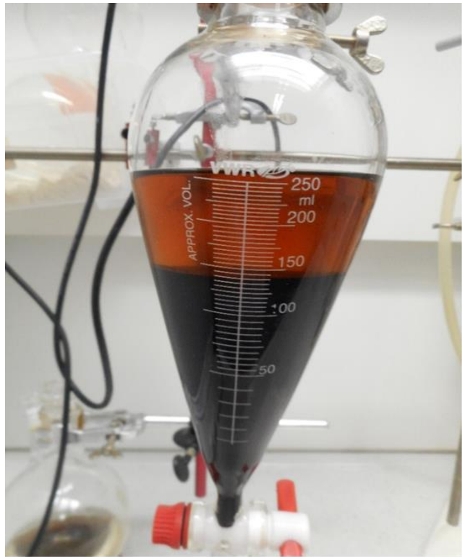


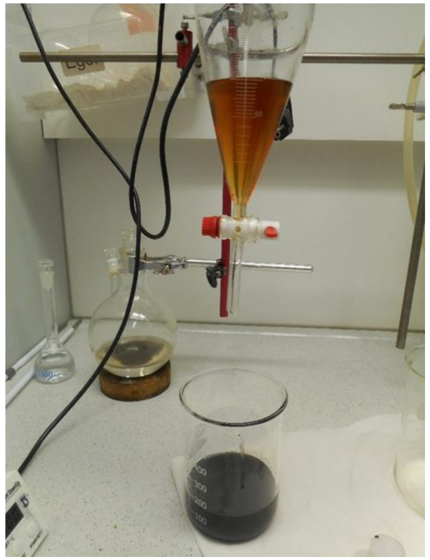
- Higher reaction rate → **faster** reaction.
- Reduced the amount of residual oligomer (and thus, aromatic compounds) in the final polyol product → **better quality** product.
- Heterogeneous → **easier** to **recover** and reuse.



Split-phase glycolysis:

- Glycol solvent and SMM catalyst (bottom layer) is thereby **easily separated** from polyol (top layer)





The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Installation, starting up, operation and turn off

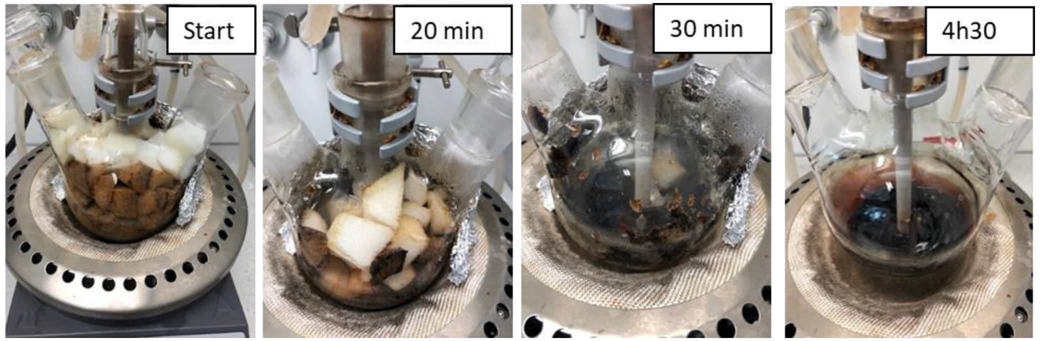
PU recycling by glycolysis:

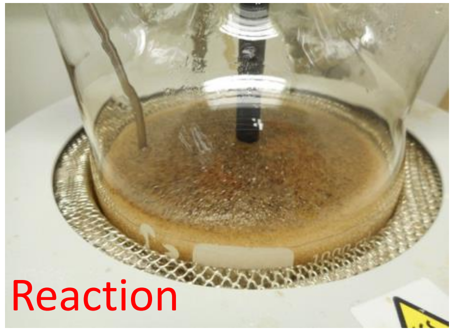
Process with **state-of-the-art catalyst**

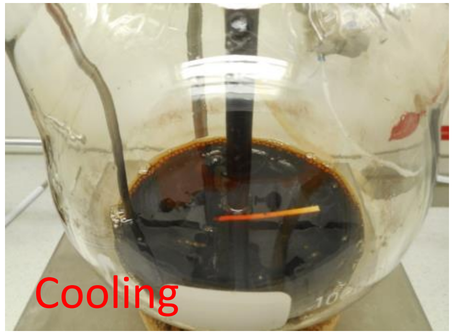
- very poor performance of the initial PU melting
- poor conversion rate
- severe PU degradation resulting in discolouration and side reactions
- catalyst and polyol product cannot be isolated or purified

Process with **SMM catalyst**

Clean reaction mixture, product easy to separate







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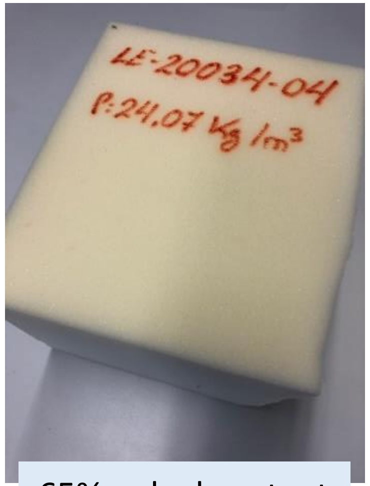
Installation, starting up, operation and turn off

PU recycling by glycolysis:

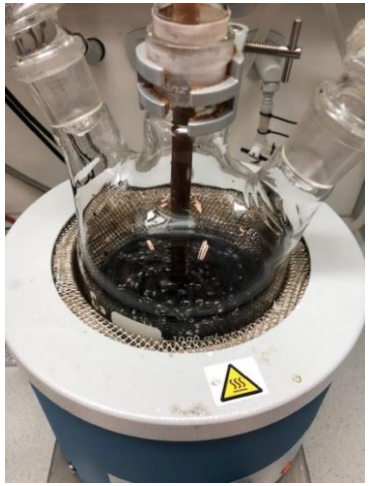
Industrial **PU foam** waste (scrap) from REPSOL



65% polyol content

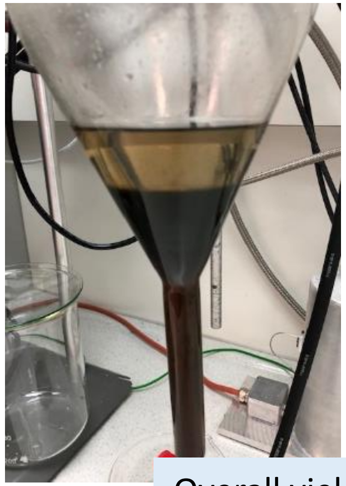
PU glycolysis with SMM

Step-wise addition of PU foam

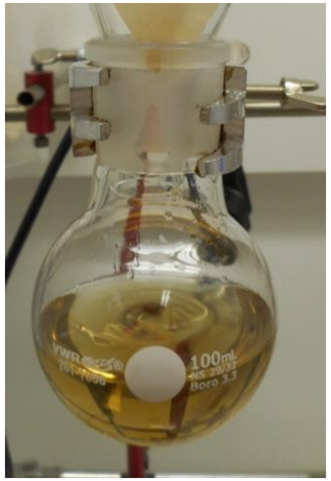


Reaction and cooling

Product **isolation** and **purification** via liquid-liquid extraction



Overall yield: 47.1%
Effective polyol yield: 72.5%



Polyol product after filtration and drying

Next step: **Vacuum distillation** to remove solvent residues

- **Lab-scale** batch: ~15-20 gr of polyol monomer is obtained
- Recycled polyol characterized according to industry ISO/ASTM procedures: recycled monomer close to industry standard – except colour and Si standard
- Si originates from PU foam formulation and needs to be removed to satisfy specs
- Next step: demonstrate technology at **pilot plant scale** (1000L reactor)

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Healthy and safety issues

Magnetic fluid material:

Health Hazards:

H302 Toxic if swallowed
H315 Causes skin irritation
H318 Causes serious eye damage

Risks:
R21/22 Harmful in contact with skin and if swallowed
R36/37/38 Irritating to eyes, respiratory system and skin
R41 Risk of serious damage to eyes

Safety Hazards:
S23 Do not breathe gas
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
S28 After contact with skin, wash immediately with plenty of water
S36 Wear suitable protective clothing
S37 Wear suitable gloves
S39 Wear eye/face protection

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Healthy and safety issues

Chemical materials:

Hydrochloric acid

Skin corrosion and danger signals

Ethylene glycol

Serious health hazard and danger signals

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Partners involved

- WP leader

:fm ioniqa

- Co-leadership (task leaders apart from WPL)

circe RESEARCH CENTRE FOR ENERGY RESOURCES AND CONSUMPTION

CPPE CHEMICAL & PHARMACEUTICAL PROCESS ENGINEERING

- Other partners involved

NUREL KORDSA REPSOL NOVAMONT ARKEMA INNOVATIVE CHEMISTRY

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Thank you for your attention

www.polynspire.eu

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

TRAINING MODULE ON UPGRADING RECOVERED MATERIAL THROUGH ADDITIVE TECHNIQUES

polynSPIRE

Innovative technologies for plastic recycling

Mechanical recycling.
WP5

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

polynSPIRE

Innovative technologies for plastic recycling

Development and optimization of vitrimer formulations for enhancing recycled materials
WP5.1 Training

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

WP 5.1 Fundamentals

polynSPIRE

Innovation of polynSPIRE project:

- Currently post consumer PU waste is either incinerated (pyrolysis) or ends up on landfill sites, both leading to an increase in waste and greenhouse gas emissions. By carrying out high-fidelity laboratory scale experiments by TU/e on post industrial PU foams, an attempt is made to develop and optimize additive formulations, converting these thermosets into dynamic covalent networks.

Specific objectives of the project:

- Lab-scale mixing experiments of polymers with the selected additives will be carried out and the resulting materials will be characterized in terms of structure, (thermo) mechanical and rheological properties.
- Processing conditions of the PU foam and the concentration of catalyst used will be optimized

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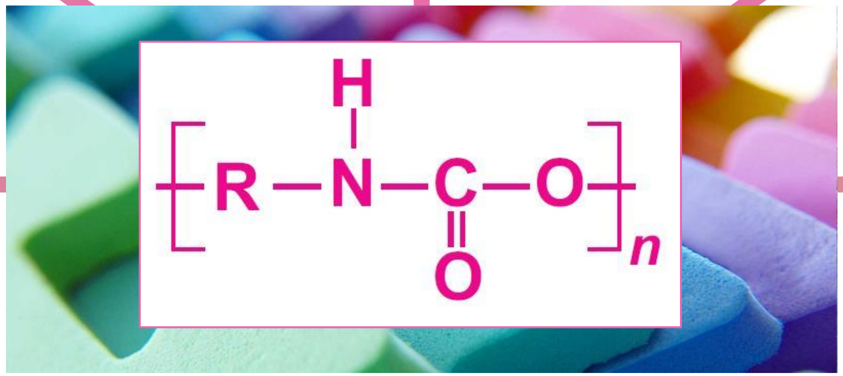
Polyurethanes

Coatings

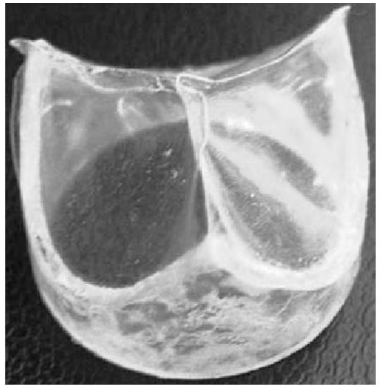
Sealants

Rubbers

Adhesives



Polyurethane Foams



Heart valves

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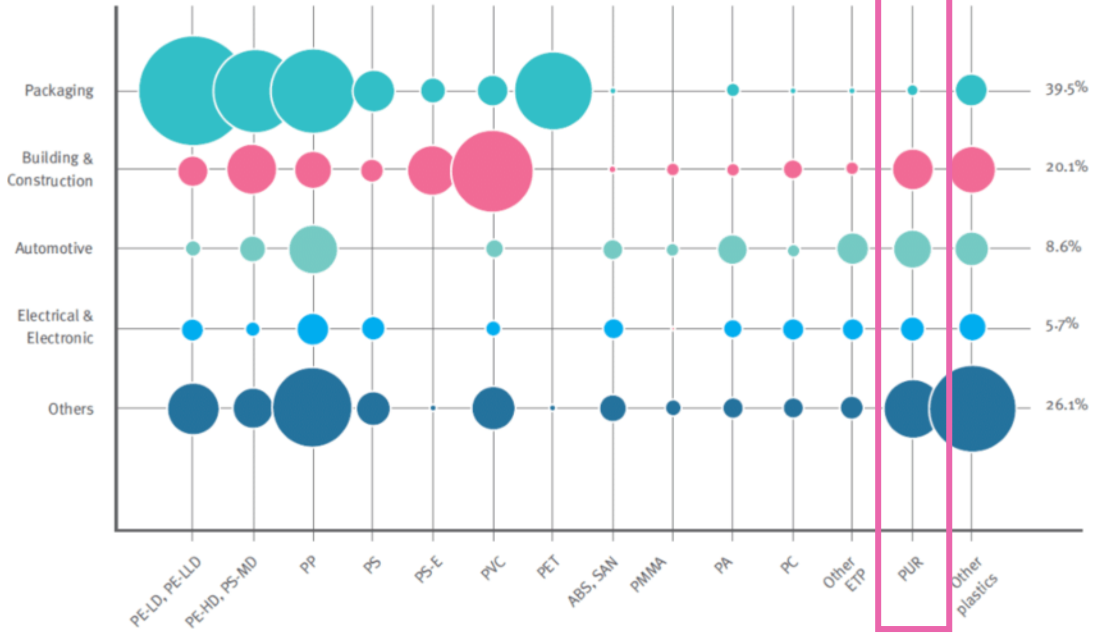
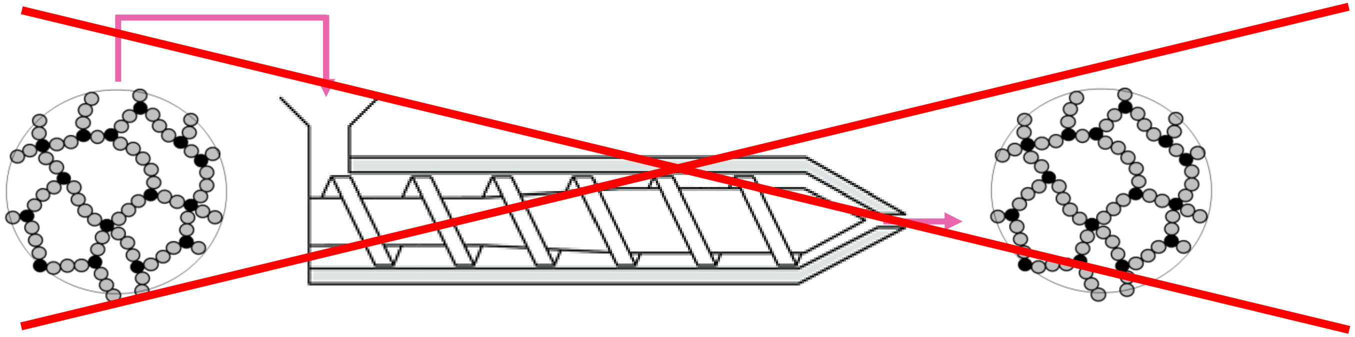
Fundamental concepts

Recycling of polyurethanes



[1]



PU has a 6% global market share [2]

- ≈75 vol% is thermoset PU foam
- Wear and replacement causes a constant stream of waste
- Incineration plants or landfill

Conventional recycling methods

- Melt reprocessing does not work

What does happen?

- Mechanical grinding → use as a filler material
- Chemical recycling → Glycolysis

Need specialised equipment

[1] Ragaert, K.; Delva, L.; van Geem, K. Mechanical and Chemical Recycling of Solid Plastic Waste. Waste Management. Elsevier Ltd November 1, 2017, pp 24–58. https://doi.org/10.1016/j.wasman.2017.07.044.
[2] Kemona, A.; Piotrowska, M. Polyurethane Recycling and Disposal: Methods and Prospects. *Polymers (Basel).* **2020**, *12* (8). https://doi.org/10.3390/POLYM12081752.

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

polynSPIRE

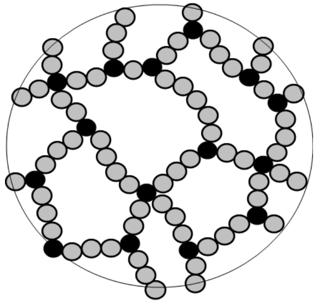
Dynamic Covalent Network (DCN)

Bridging the gap between mechanical and chemical recycling

Mechanical recycling

Chemical recycling

Immobile network

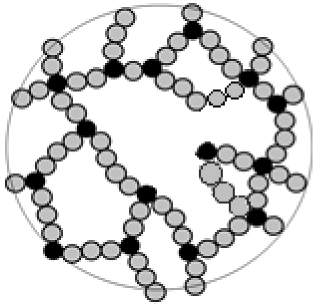


Dynamic Covalent Network

low temperature

high temperature

Mobile network



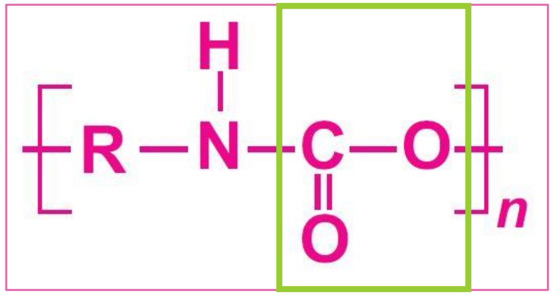
Reactive Extrusion

Using industrial equipment that is already there

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

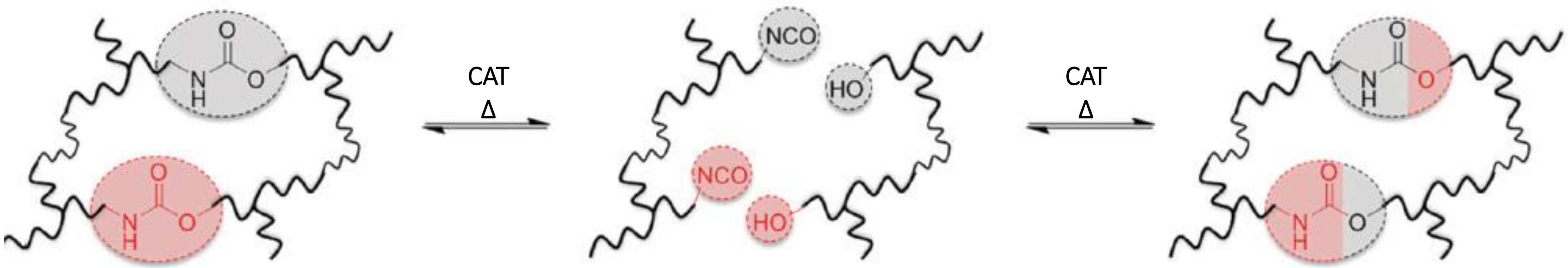
DCN chemistry



Carbamate group

Exchange of the backbone

Transcarbamoylation



By using the reactivity that is already there, by employing the urethane group, effective exchange of the backbone can occur. This allows for mobility of the network

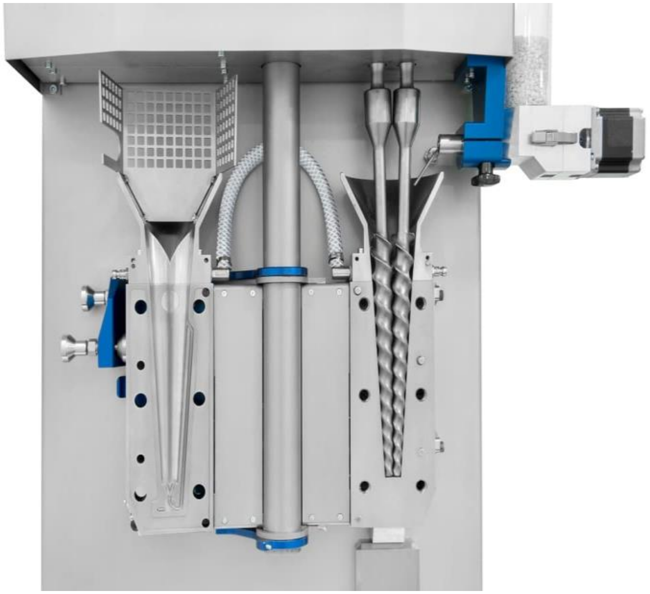
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

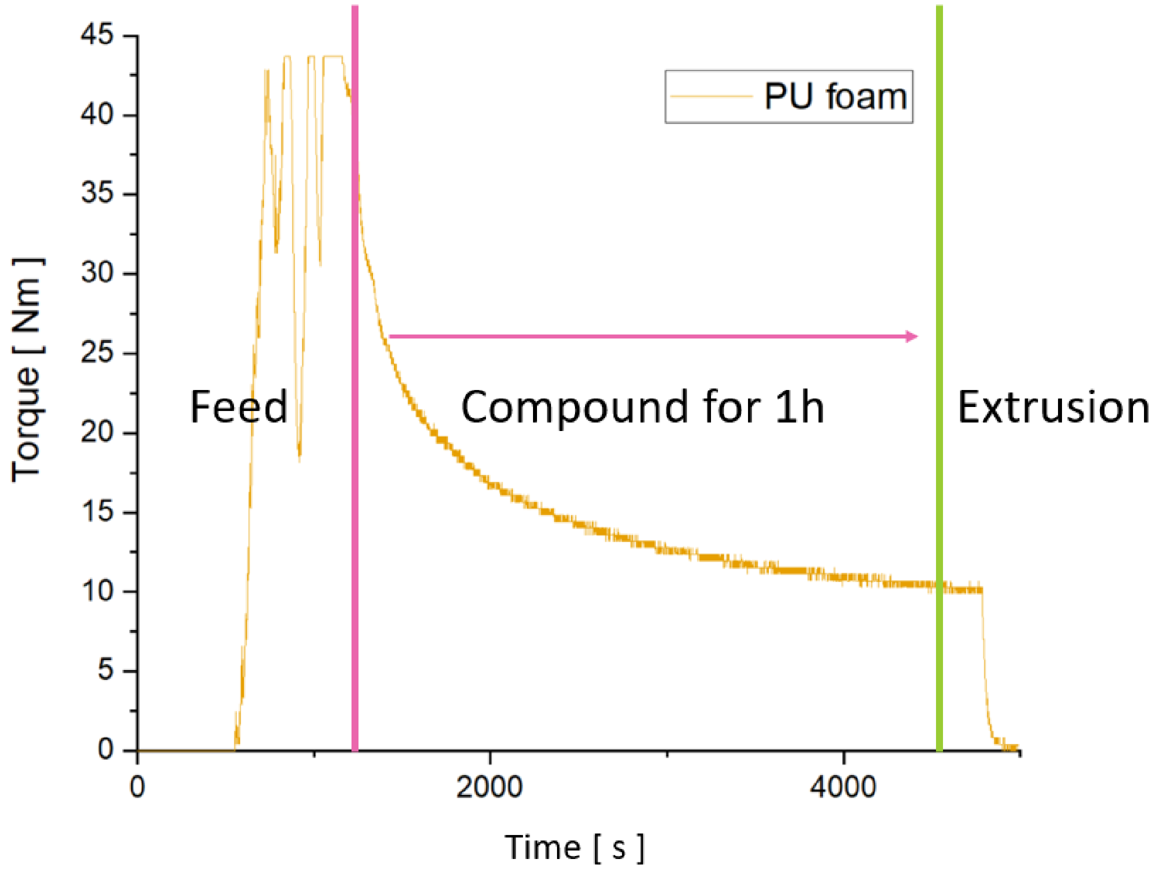
Reactive Extrusion

Extruder as reactor vessel

- Micro Compounder was used for tests
- Gathering torque data during reaction
- 15 mL volume



Example of torque data



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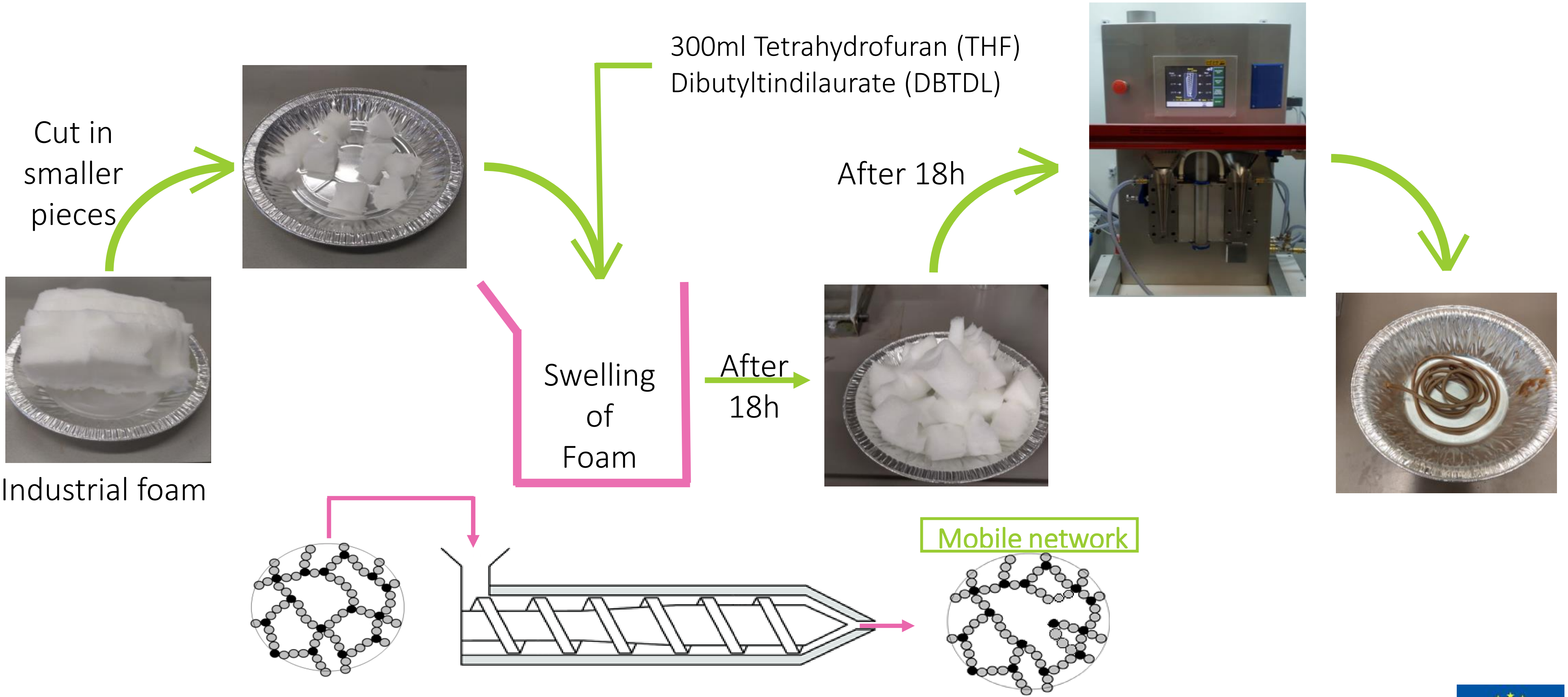
Main results of the project

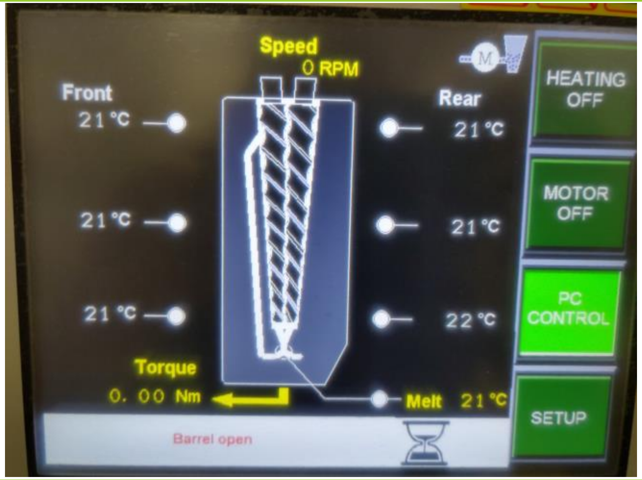
Pre treatment of polyurethane foam (for 15g)



The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

MC-15-HT overview*



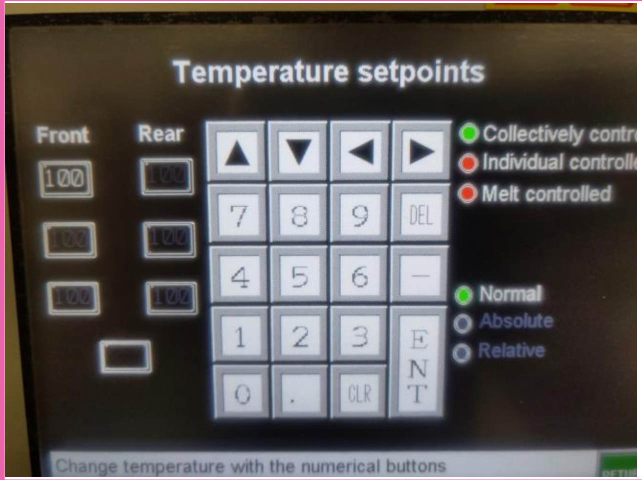
Overview screen

From here you can access any of the required settings. By pressing any of the displayed temperatures in white you access the temperature settings of the extruder. By pressing the yellowed text or numbers (Speed, Torque, Melt) you access the settings for the screws.

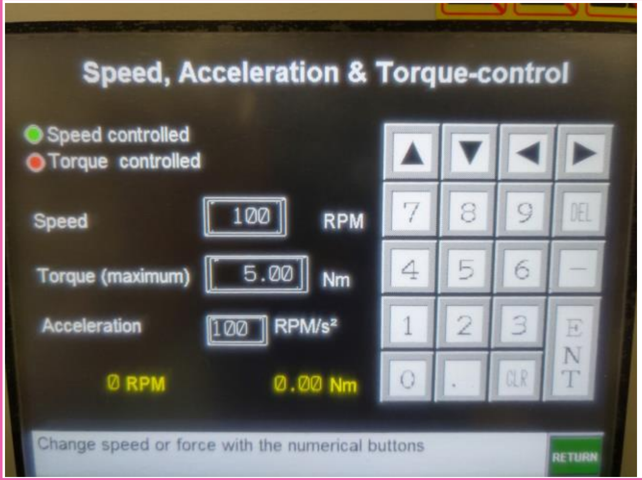
Green button functions

Heating Off – On	Switches the heating on and off
Motor Off – On	Turns the screws on or off. (Only works when barrel is closed)
Pc control	When enabled, settings can be changed via PC software
Setup	Allows access to calibration and software updates

Settings used during experiments



Temperature settings:
During experiments it is important that the temperature remains constant as this is the main influence for the rate of reaction. Therefore, the temperature was set to collectively controlled.

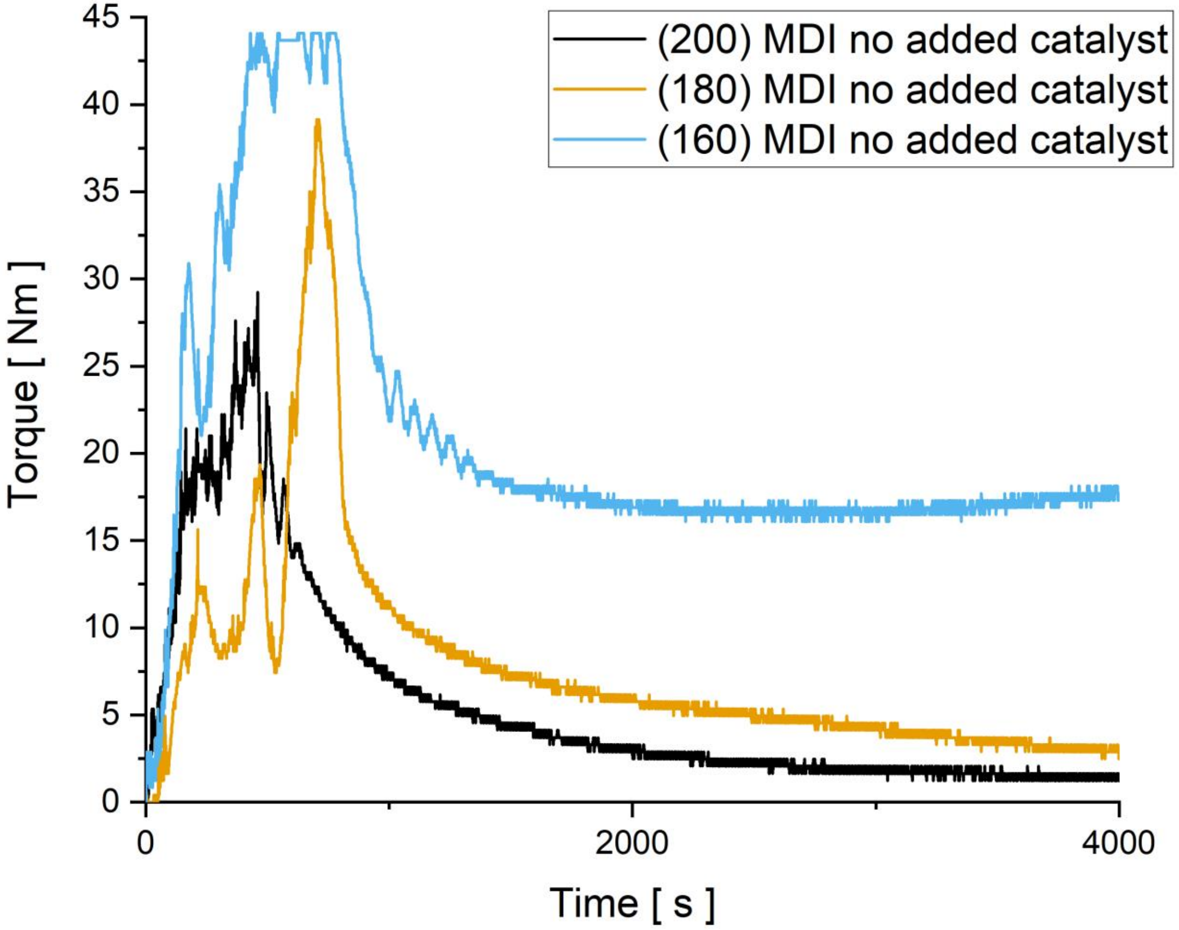


Speed & Torque settings:
As crosslinked polymers are added to the microcompounder, the force required to process them are high. Hence, the speed was set to only 50 RPM with a torque maximum of 40 Nm (machine limit)

*Always refer to the manual for complete instructions and safety procedures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Interpretation of results



Without added Catalyst

- Lowering T leads to an increase in Torque
- Decreasing the amount of thermal degradation

With 0.33 mol% DBTDL

- Still high effect Temperature on Torque
- Catalyst stabilizes torque response

With 6.68 mol% DBTDL

- Torque only becomes measurable at 160 °C
- Low torque indicates loss of network integrity

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Comparison and overview of result interpretation

[CAT]

	No additional catalyst	0.33 mol% DBTDL	6.68 mol% DBTDL
200°C	 Torque at extrusion 1.2 Nm Gel content 87%	 Torque at extrusion 0 Nm Gel content 87%	 Torque at extrusion 0 Nm Gel content 54%
180°C	 Torque at extrusion 2.9 Nm Gel content 87%	 Torque at extrusion 0.4 Nm Gel content 75%	 Torque at extrusion 0 Nm Gel content 75%
160°C	 Torque at extrusion 18.1 Nm Gel content 88%	 Torque at extrusion 9.9 Nm Gel content 87%	 Torque at extrusion 0.8 Nm Gel content 90%

Temperature

Gel Content

Gel Content

Gel content indicates a network is still present. But a loss of network integrity can be observed at high catalyst concentration and/or high temperatures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Health & Safety*

As working with an extruder or compounder can have risks to the worker or close environment, health and safety standards must be applied by employee and employer. As an employer it is important to always follow national regulations (e.g. OSHA, British Factory Act, and others) to avoid injuries.

General Health & Safety for when working with the MC-15-HT*

- The instrument should only be operated with original parts provided by, or in consideration with, the manufacturer of the equipment
- Management must make sure that maintenance, assembly activities and inspections are performed by qualified and authorized personnel, who are well informed as to the instrument/installation after acquiring a certain level of technical knowledge by schooling/training.
- It is forbidden to place the instrument in a room where a danger of explosion is present.
- Do not wear loose clothing (for example neckties, open jackets, scarfs, etc.) and tie up long hair
- Do not smoke, eat, or drink in proximity to the instrument.

*Always refer to the manual for complete instructions and safety procedures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

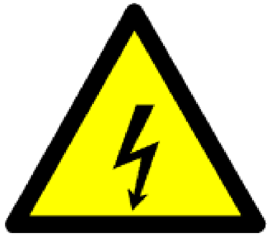
Health & Safety*

- **Labelling and posting.** Indicators and visual signals used to alert individuals should be clearly labelled as to the conditions that prompt actuation of the indicator.
- **Emergency stop.** If dangerous situations occur, switch off the instrument by means of the emergency stop installations. The emergency stop installation does not disconnect the operating voltage.
- **Intrinsic safety.** Moving parts of the instrument should be placed in such a manner that they are unlikely to be touched. A protective guard on the hopper can avoid entrapment by the mixing screws.
- **Overload**. The mixing screws exert a torque when mixing viscous materials in the barrel. The drive system of the instrument should be protected against overload.
- **Notices and symbols**. The following slide gives an overview of the safety installations and warning labels that should be present on or at the instrument.

*Always refer to the manual for complete instructions and safety procedures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

& Safety*

Symbol	Description	Position on the instrument
	Warning: Danger at or near parts under tension. Access only by technically competent personnel	On the outside of the hatch providing access to the electrical compartment and near any other parts that are under tension
	Warning: Danger. Do not open. Risk of electric shock. Disconnect all sources of supply prior to servicing.	On the outside of the hatch providing access to the electrical compartment.
	Warning: Danger of burns if hot parts are touched.	On the barrel and in the display, if present, when the heating is switched on. On other parts of the machine that have the risk of being hot.
	Warning: Danger of bodily damage when touching rotating parts	On any part where the rotating barrel can be reached.

*Always refer to the manual for complete instructions and safety procedures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

polynSPIRE

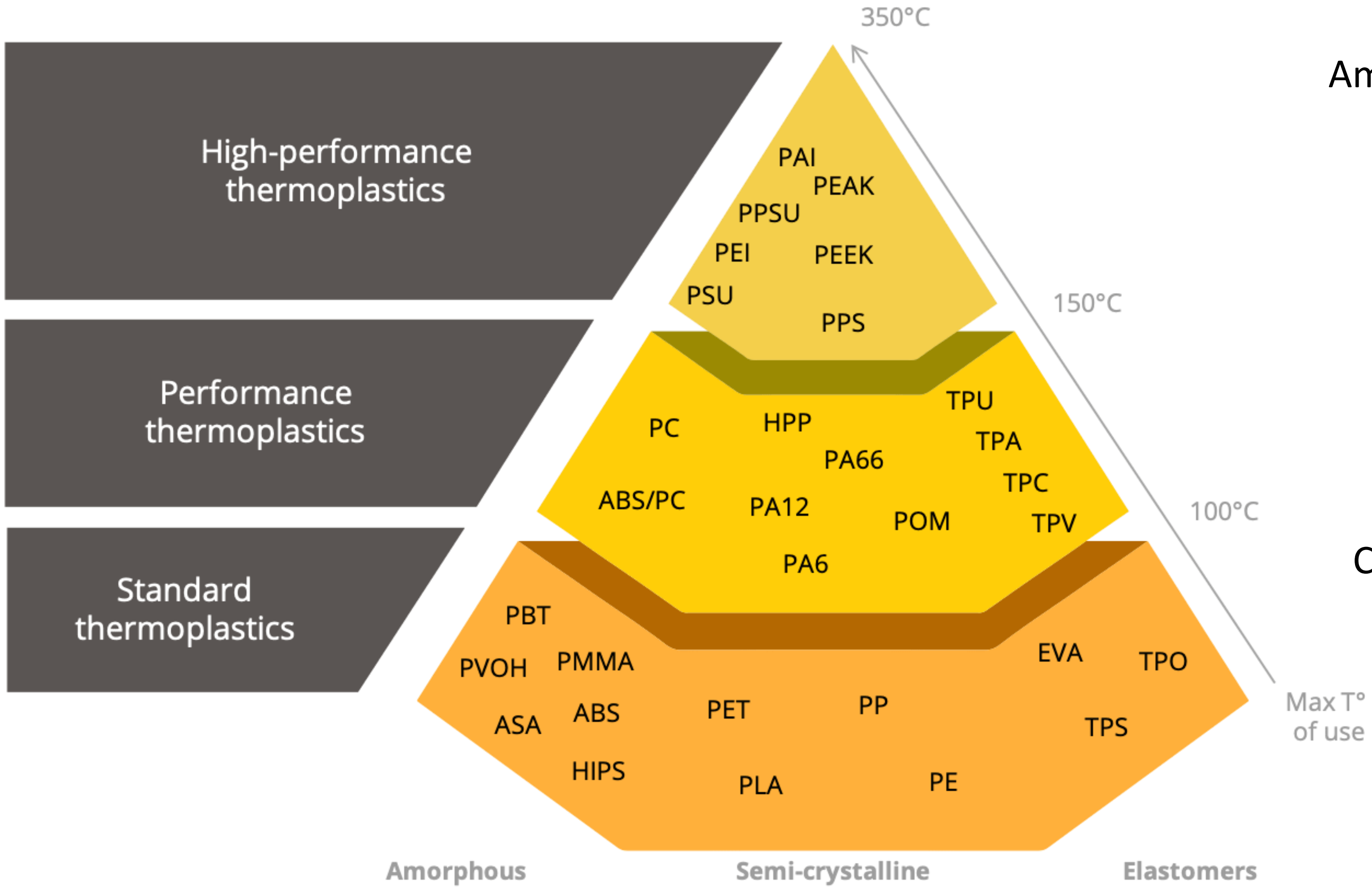
Innovative technologies for plastic recycling

Development and optimization of high irradiation energy as a way for recycling
WP5.2 Training

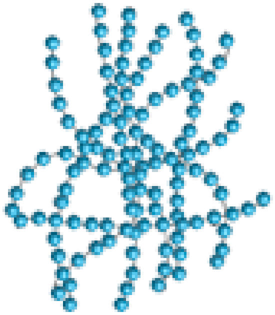
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

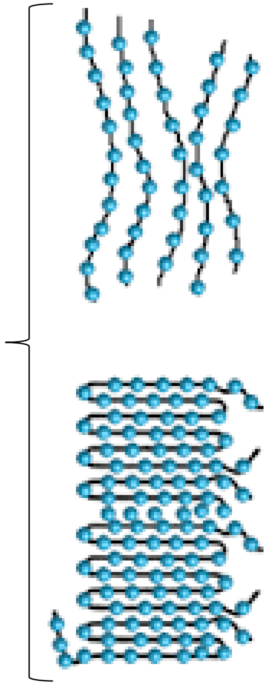
Thermoplastic materials



Amorphous phase



Cristaline phase



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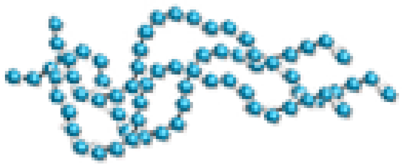
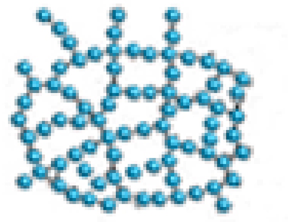
Fundamental concepts

Plastic materials

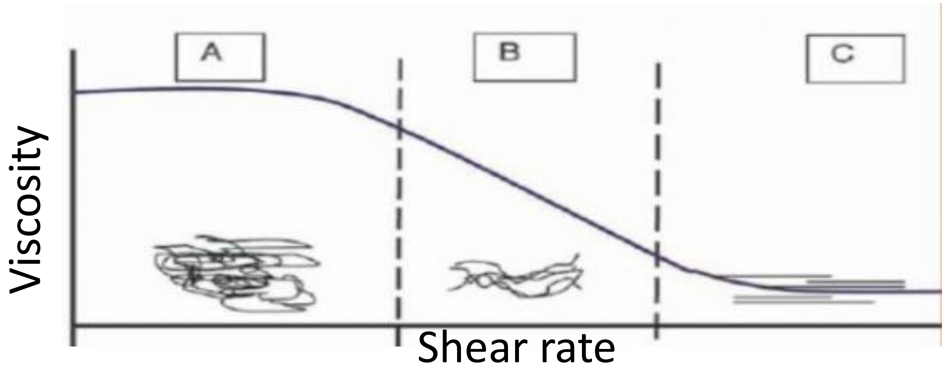




Thermoplastic materials:
- Weak conections among the different polymeric chains
- Can be melted without degrading

Thermoset materials:
- High crosslinking between the diffent polymeric chains
- It can not be melted without degrading

Thermal treatments breaks the bonds between the different polymeric chains or even within the chain itself, modifying the properties of the material itself. As the bonds and interactions among polymeric chains are reduced, it is easier for the material to flow.



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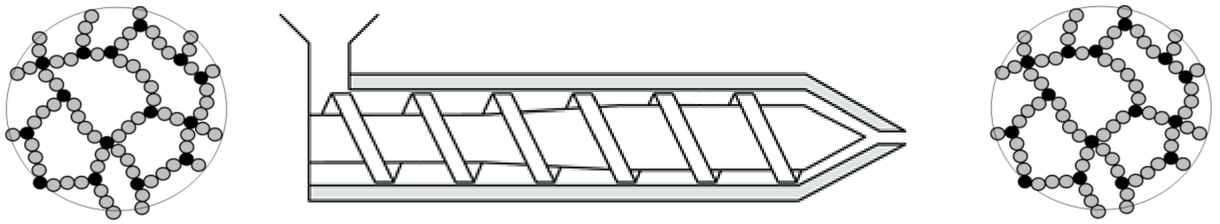
Fundamental concepts

Plastic materials

Although in comparison with other palstic materials (Still above 100 tons per year), polyamide is not so much produced. The high value of this thermosplastic material due to its properties makes it one of the most interesting materials. Besides, it is a thermoplastic material and therefore it is possible to melt it and provide a new shape, a new life



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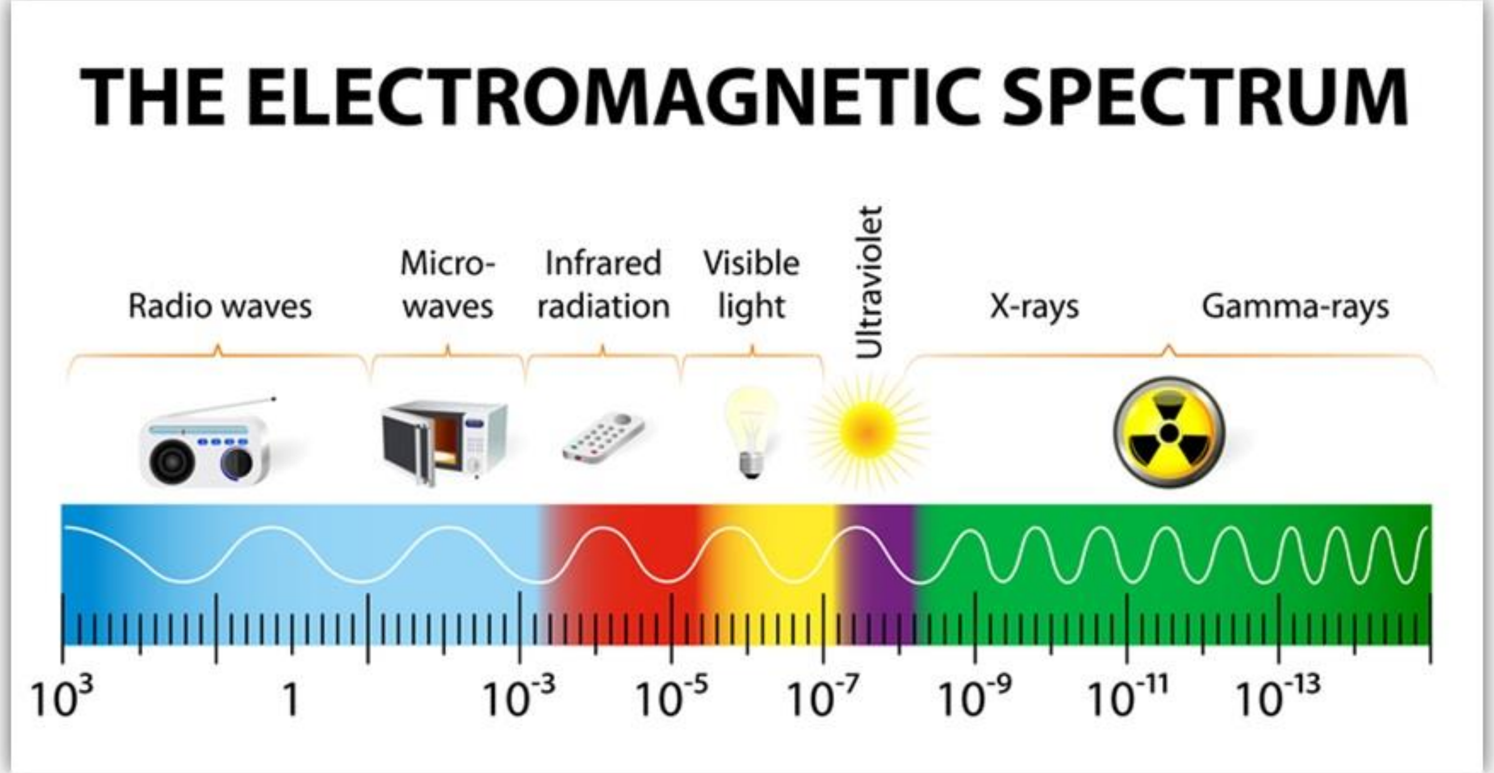
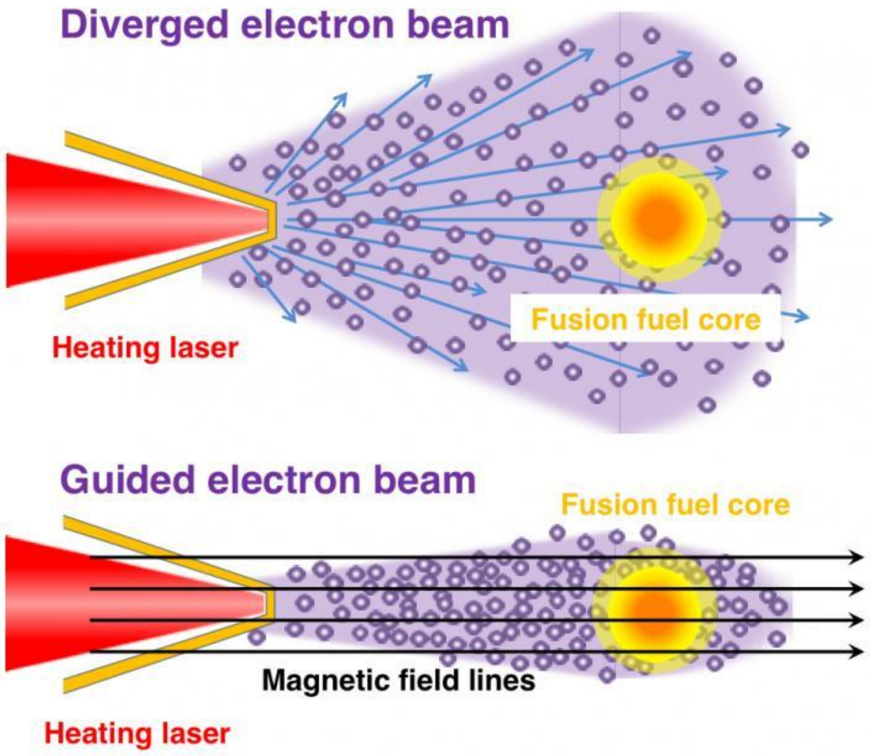
Fundamental concepts

High energy irradiation





Radiations of high energy, those of short wavelenght have the capacity to modify electronically a molecule

Irradiation is measured in Gray (Gy)

$$Gy = \frac{J}{Kg}$$

Guided electron beam has the capacity to charged electronically the molecule, providing the capacity to generate new bonds

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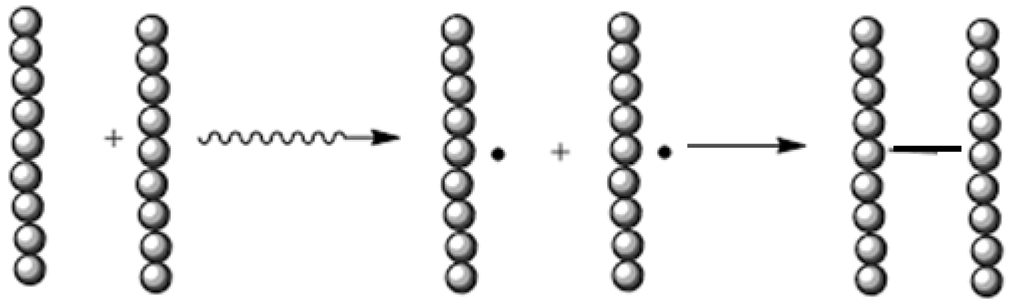
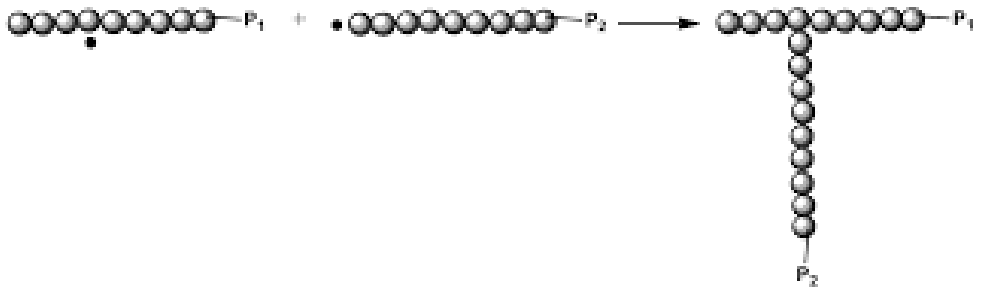
Fundamental concepts

High energy irradiation



The gamma radiation generates free radicals in the polymeric chain.

These free radicals can give differnt scenarios
1- Oxidation of the polymeric chain due to the interaction with the oxygen in the air
2- Generation of a bond between differnt polymeric chains



Due to the generation of new bonds between different polymeric chains, the crosslinking. The properties lost due to the thermal degradation of materials are recovered

In PolynSpire it has been observed that the most sensitive property is the viscosity. Which in the case of the PA6 is reduced by more than 10% after several extrusion compounding processes (7 Extrusions)

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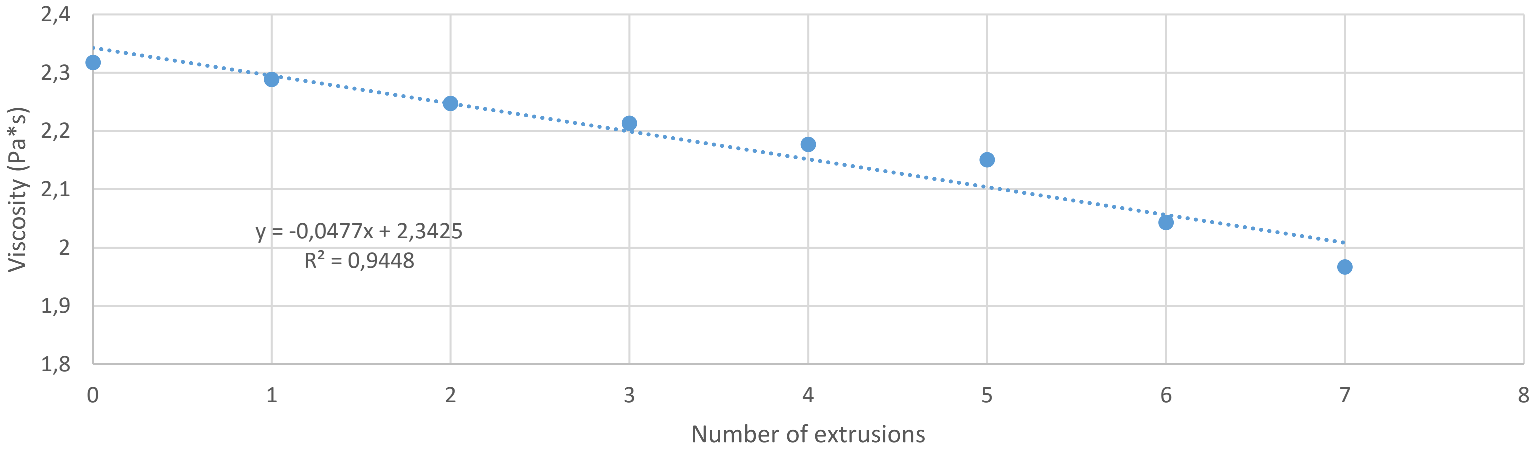
Main results of the project

High energy irradiation

Residue simulated after 7 extrusions



Raw material after irradiation
100 KGy-250 KGy- 500 KGy

Thermally degraded material after irradiation
100 KGy-250 KGy- 500 KGy

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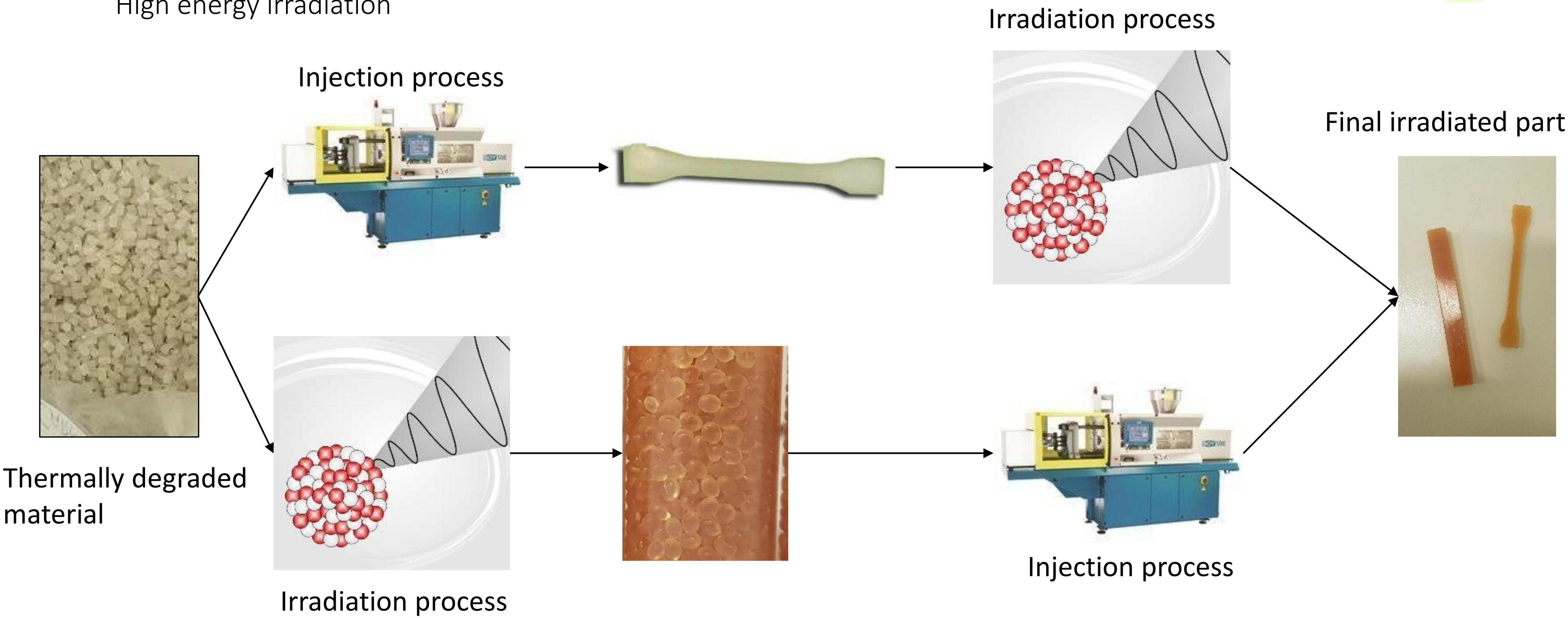
Main results of the project

High energy irradiation



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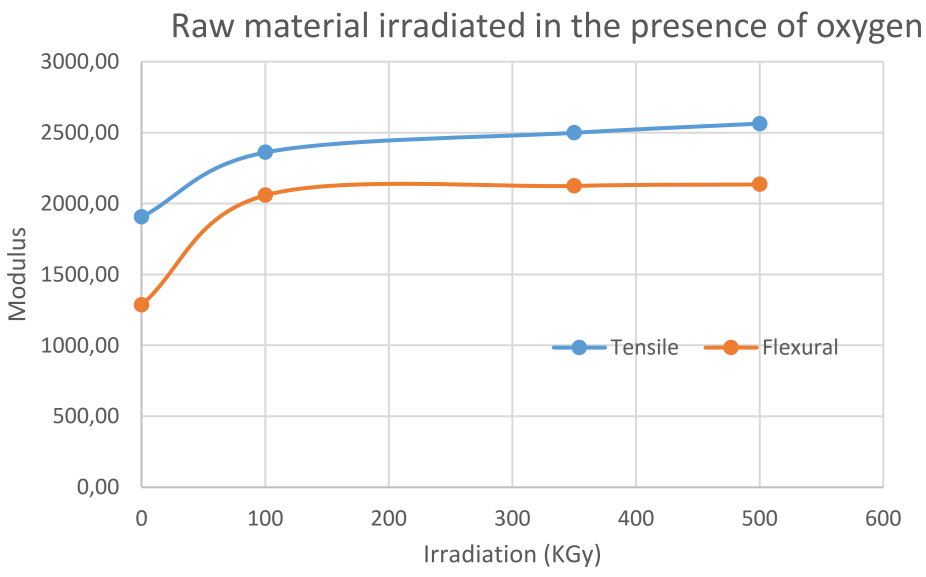
Main results of the project

High energy irradiation



The irradiation process generates crosslinking between the polymeric chains.

The crosslinking affects directly to the mechanical properties. Nevertheless, after reaching a certain point the effect of the irradiation is reduced.

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Health & Safety*

As working with an extruder, an injection moulding machine or any thermoplastic processing equipment can have risks to the worker or close environment, health and safety standards must be applied by employee and employer. As an employer it is important to always follow national regulations (e.g. OSHA, British Factory Act, and others) to avoid injuries.

General Health & Safety:

- The instrument should only be operated with original parts provided by, or in consideration with, the manufacturer of the equipment
- Management must make sure that maintenance, assembly activities and inspections are performed by qualified and authorized personnel, who are well informed as to the instrument/installation after acquiring a certain level of technical knowledge by schooling/training.
- It is forbidden to place the instrument in a room where a danger of explosion is present.
- Do not wear loose clothing (for example neckties, open jackets, scarfs, etc.) and tie up long hair
- Do not smoke, eat, or drink in proximity to the instrument.
- Heat protective globes are mandatory
- Proper air ventilation must be assured by the employer
- Courses and training should be given to the worker in order to understand the process and its risks

*Always refer to the manual for complete instructions and safety procedures

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Health & Safety*

- **Labelling and posting.** Indicators and visual signals used to alert individuals should be clearly labelled as to the conditions that prompt actuation of the indicator.
- **Emergency stop.** If dangerous situations occur, switch off the instrument by means of the emergency stop installations. The emergency stop installation does not disconnect the operating voltage.
- **Intrinsic safety.** Moving parts of the instrument should be placed in such a manner that they are unlikely to be touched. A protective guard on the hopper can avoid entrapment by the mixing screws.
- **Overload**. The mixing screws exert a torque when mixing viscous materials in the barrel. The drive system of the instrument should be protected against overload.

- **Notices and symbols**. The following slide gives an overview of the safety installations and warning labels that should be present on or at the instrument.

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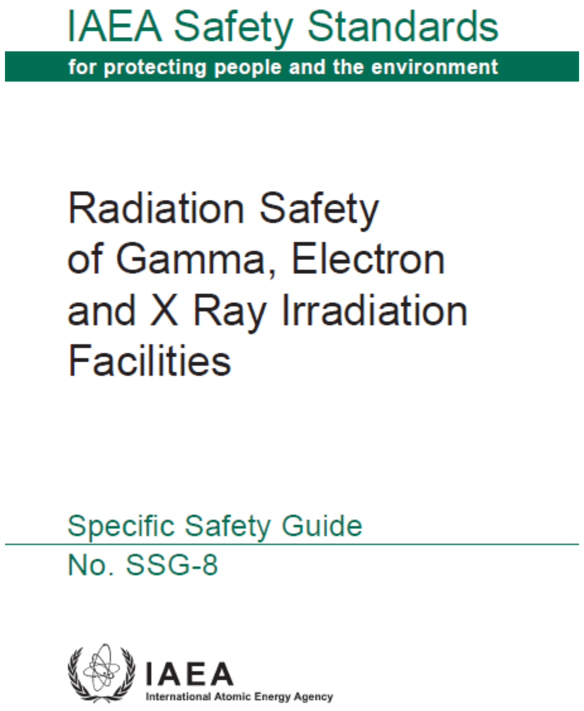
Health & Safety*

Besides, the safety measures necessray to trat the thermoplastic materials. It is necessary to keep in mind that the treatments given to the material to regenerate the chemica bonds, imply gamma rays and therefore radiactive compounds.

These activities must be performed by specialiced entities with special protective equipment.


IAEA Safety Standards
for protecting people and the environment

Radiation Safety of Gamma, Electron and X Ray Irradiation Facilities

Specific Safety Guide
No. SSG-8

IAEA
International Atomic Energy Agency

The safety standards described in the “Radiation safety of gamm, electron and X Ray irradiation facilities” of IAEA must be followed

After the material has been exposed to the gamma rays, these material can be treated as normal plastic material. Due to the fact that it is not emitting any type of radiation

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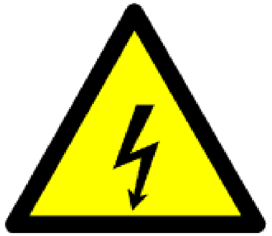
Health & Safety*

Symbol	Description	Position on the instrument
	Warning: Danger at or near parts under tension. Access only by technically competent personnel	On the outside of the hatch providing access to the electrical compartment and near any other parts that are under tension
	Warning: Danger. Do not open. Risk of electric shock. Disconnect all sources of supply prior to servicing.	On the outside of the hatch providing access to the electrical compartment.
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Partners involved

WP leader

- Co-leadership (task leaders apart from WPL)

- Other partners involved

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Thank you for your attention

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research and innovation programme under grant agreement no 820665

TRAINING MODULE ON THE VALORISATION OF PLASTIC WASTE AS A CARBON SOURCE IN STEEL FURNACES

polynSPIRE

Innovative technologies for plastic recycling

Valorisation of plastic waste in the steel sector

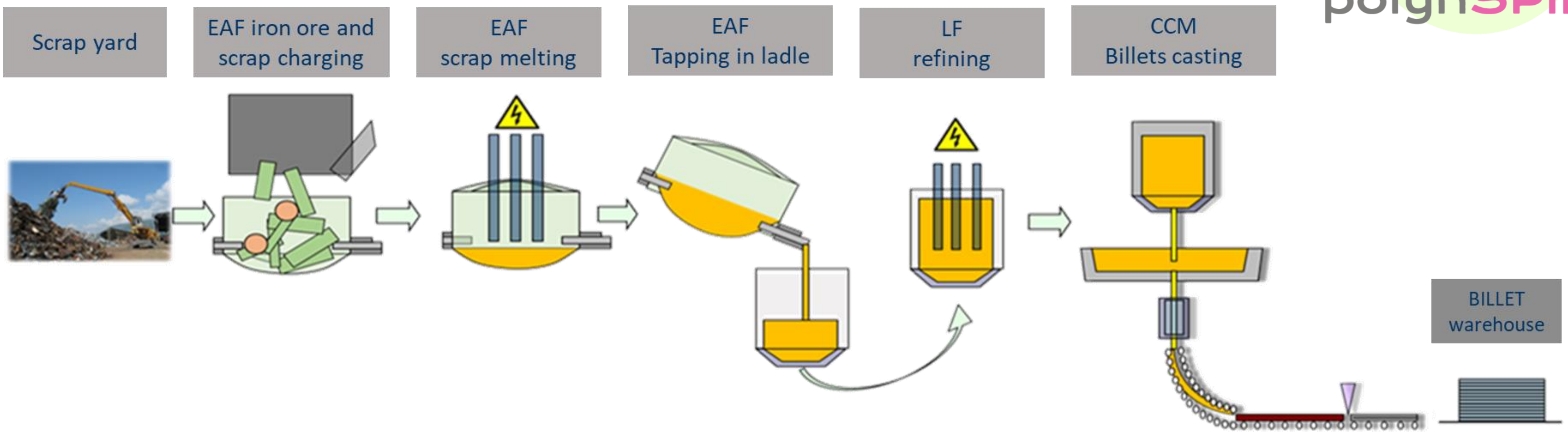
WP 10 Training

The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

Plastic recycling in the Steel sector – fundamental concepts

polynSPIRE

STEEL PRODUCTION



- Steel is produced from **iron ore** and **metallic scrap**, by melting it in an **Electric Arc Furnace (EAF)**. Despite the name, EAF requires a relevant amount of natural gas and coal (solid carbon) to increase process efficiency.
- An average EAF produces **500-1000 kt of Steel per year**.
- Assuming a typical amount of 5-10 kg of coal per ton of produced Steel, about 16-32 kg of CO2 per ton of Steel are released.

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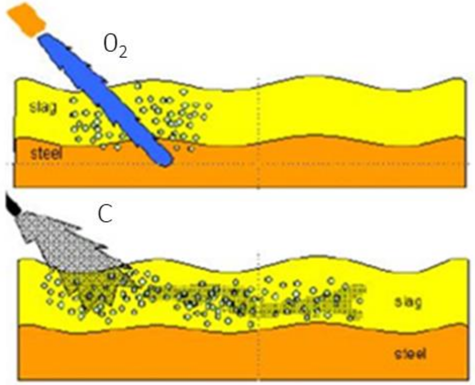
Plastic recycling in the Steel sector – fundamental concepts

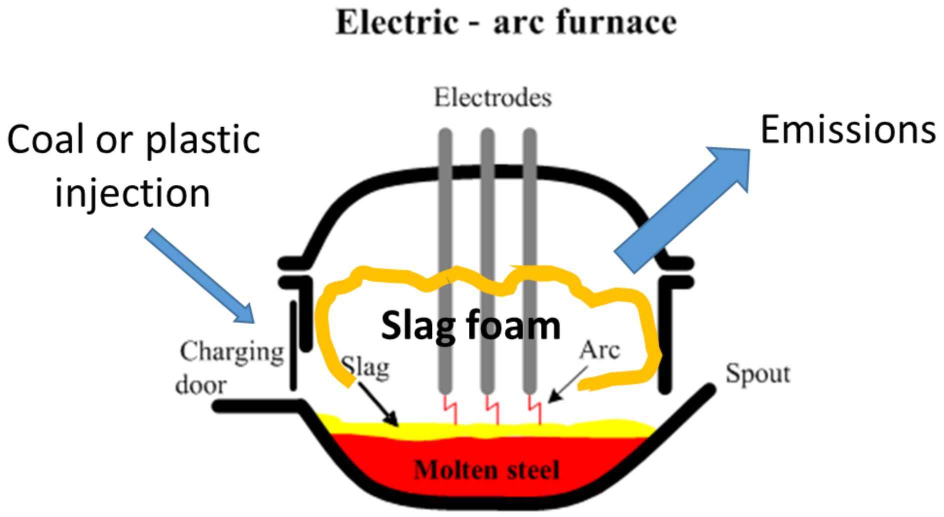
Why is coal used in the EAF process?

- A layer of protective slag is present in the furnace (slag is formed by CaO, FeO, Al2O3, MgO, SiO2, MnO). The foaming of the slag by CO/CO2 gas bubbles occurs naturally in the process by the oxidation of the carbon in the molten steel by oxides in the slag.
- Foaming process is intensified by the injection of carbon, usually fossil coal (a carburizing/foaming agent and reducing agent). C reacts directly with oxygen and iron oxide forming gaseous CO. The gaseous emission forms a foam with slag, reducing the heat losses, NOx and noise emission from furnace.
- The process of carburization and reduction is fundamental to ensure energy efficiency and productivity.

Reactions:

1) $C + \frac{1}{2} O_2 \rightarrow CO$
2) $FeO + C \rightarrow Fe + CO$
3) $FeO + CO \rightarrow Fe + CO_2$
4) $C + CO_2 \rightarrow 2\ CO$





Basic features of slag foaming

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Plastic recycling in the Steel sector – fundamental concepts

INNOVATION OF POLYNSPIRE PROJECT

- This project is focused on the partial substitution of fossil coal with recycled polymers complaints with UNI 10667-17-2021 deriving from post-consumer waste.
- Currently, coal particles are injected when all scrap is molten. Due to the different characteristics of coal and plastics, the core of the Project is to develop an injection system for the utilization of recycled heterogonous plastic materials into Electric Arc Furnace (EAF), in order to reduce the consumption of coal (as carbon supply and reducing/foaming agent) by the use of a significant quantity of plastic waste.

Specific objetives of the project:

- To obtain a complete characterization of the injected coke and of the recycled polymers available to be utilized in EAF.
- To design and manufacture an injection system suitable for recycled plastic polymers (SRA, Secondary Reducing Agents).
- To demonstrate at industrial scale the feasibility of the systems, aiming to reduce carbon consumption in steel industry.

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Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Characteristics of plastic products

- Plastic grains are produced from MPW (mixed plastic waste).
- This mix is recovered at the end of the collection and recycling chain, and it is generally considered difficult to recycle due to its high variability.
- The transformation of MPW into a product compliant with the technical standard UNI 10667-17 (addressed as Secondary Reducing Agent or SRA) and its valorisation in the steel sector allows to close the loop of plastic recycling.

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Plastic recycling in the Steel sector – installation, starting up, operation and turn off

SRA production

- The selection process carried out by I.BLU allows for a maximisation of the plastic content in the flow and the minimization of non-relevant materials. The selection process is essential to ensure that, when reaching the recycling plant, the flow is suitable for SRA production, in such way that the final product complies with relevant technical standards and presents adequate physical and chemical characteristics in view of its final use.
- With this in mind, the sorting and selection phases have been improved. Moreover, the same purposes have been further pursued through several targeted technical interventions applicable to the production process itself. The latter can include, according to different final utilizations and in-flow material characteristics, the following production phases:
  - Shredding
  - Floatation phases
  - Densification
  - Milling/Grinding
  - Sieving
  - Further selection

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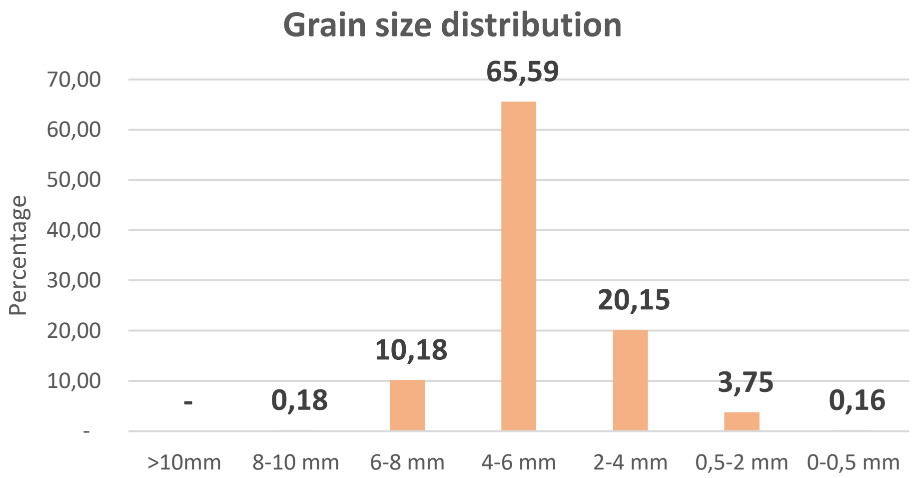
Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Chemical parameters outlined in the technical standard UNI 10667-17:2021.

Parameter	Unit	Value D6.3
Density	g/cm^3	0,3686
Moisture	%	0,3700
HHV	MJ/Kg SS	39,8700
HHV	MJ/Kg	39,2200
LHV	MJ/Kg SS	36,8900
LHV	MJ/Kg	36,7100
Hydrogen	% SS	12,1700
Chlorine	% SS	0,9300
Lead	mg/Kg SS	10,0000
Cadmium	mg/Kg SS	0,7000
Mercury	mg/Kg SS	<0,3
Cadmium + Mercury	mg/Kg SS	0,85
Ash	%	4,30
Total Carbon	%	75,50
Fixed Carbon	%	1,50 - 2,00
Oxygen	%	8,00
Thermogravimetric analysis	%	90,00

Parameter	Requirement
Heterogeneous Plastics Content	≥ 80% in weight in the dry matter
Low Heating Value (LHV)	≥30 MJ/Kg
Chlorine (Cl)	≤ 2%
Cadmium (Cd)	≤ 8 mg/Kg
Lead (Pb)	≤ 100 mg/Kg
Mercury (Hg)	≤ 0,6 mg/Kg
Moisture	Max 10% in weight



All parameters included in the Italian technical **standard UNI 10667-17** are largely compliant with their relative legal requirements.

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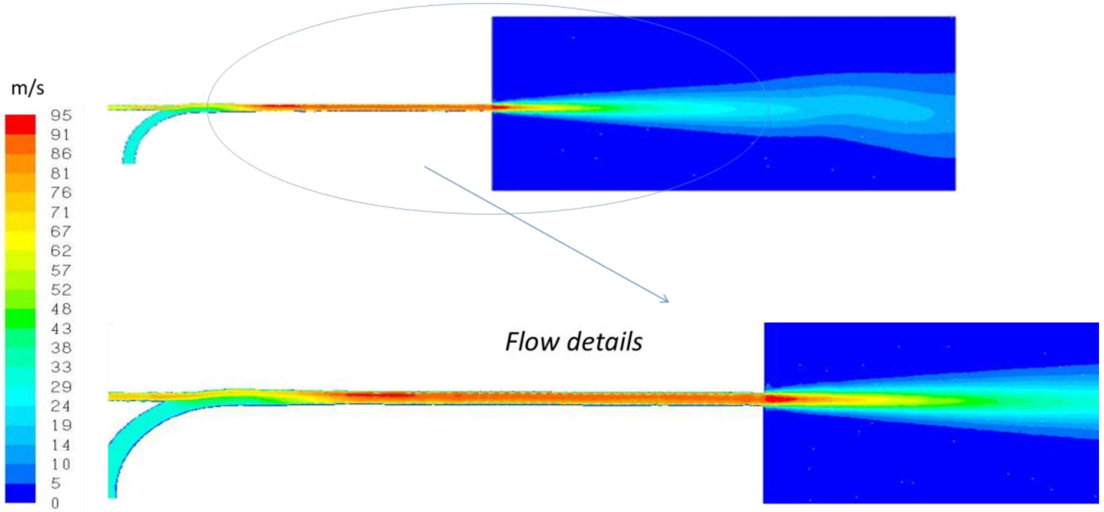
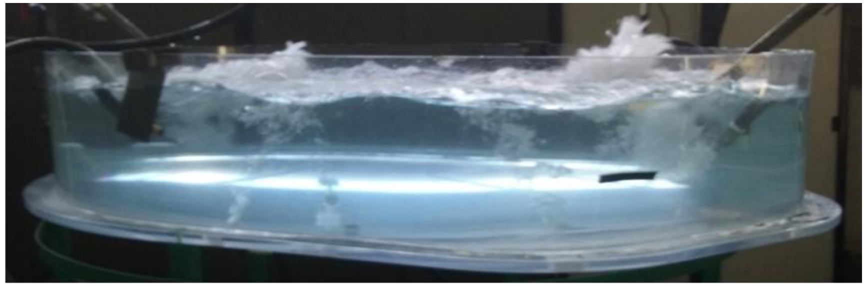
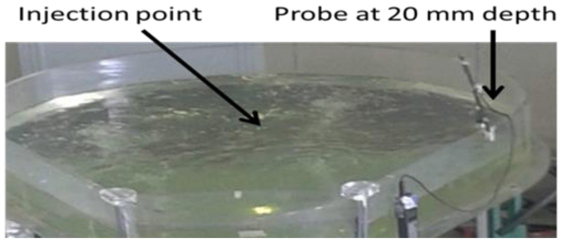
Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Design of the injection system

- HTT worked on the design of the injector and the whole pneumatic system (silos, hopper, air compressor system, piping hopper..) in cooperation with FENO. A pilot facility of pneumatic system has been realized by HTT to check material behavior before industrial trials.
- CFD simulation and tests with physical model are carried out by CSM with HTT cooperation in order to support the engineering of injection system.







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Plastic recycling in the Steel sector – installation, starting up, operation and turn off

NEW INJECTION AND PNEUMATIC SYSTEM

The injection and pneumatic system designed and installed in polynSPIRE project is a prototype fully representative of industrial system. It composed by following sections:

- Plastic grains charging station
- Silos
- Weighting system
- Dispenser
- Injector; this equipment has been purposely designed within this project and it is not a commercial component.

Even if not originally foreseen, the system is equipped with ATEX components (protection from explosion), in order to guarantee the highest safety working conditions.

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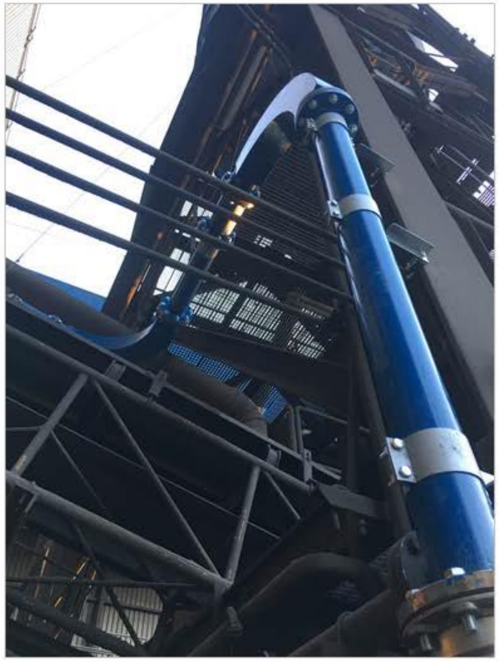
Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Plastic grains charging system

- Plant has been designed in order to charge the silo directly from the truck.
- Truck is connected to a loading pipe with a pneumatic pinch valve that opens and closes by air pressure.
- The filling sequence is remotely controlled on the safety PLC (programmable logic controller).



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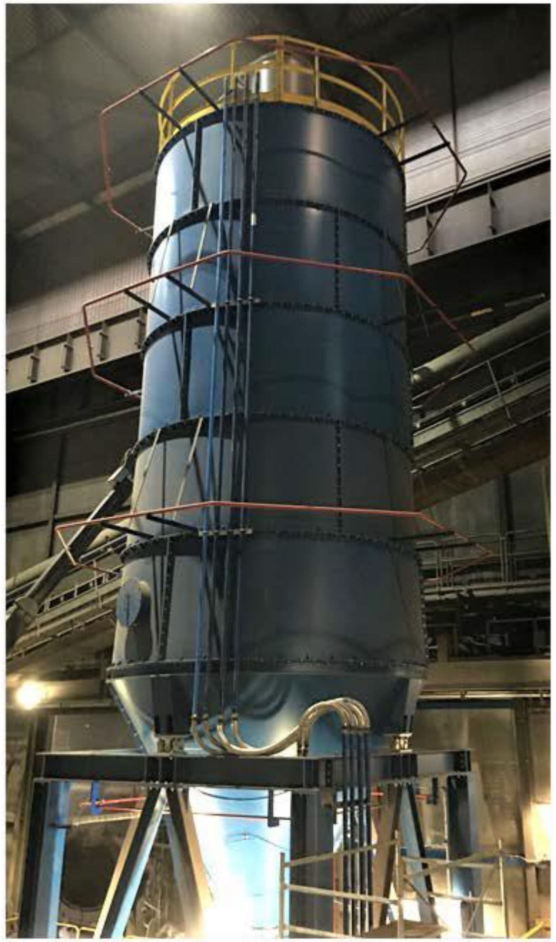
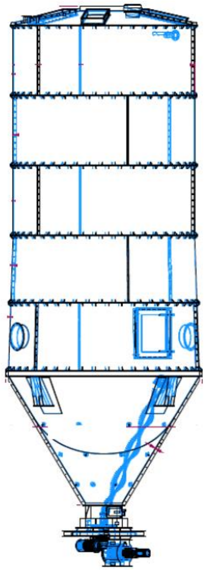
Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Silo equipment



- The silo has a capacity of 100 m^3.
- It is equipped with vibro-fluidification and screw extractor to avoid grains packing.
- The silo is weighted using load cells and using radar transmitter to check the content level within it.
- The internal temperatures on the silo are monitored using two PT100 probes.



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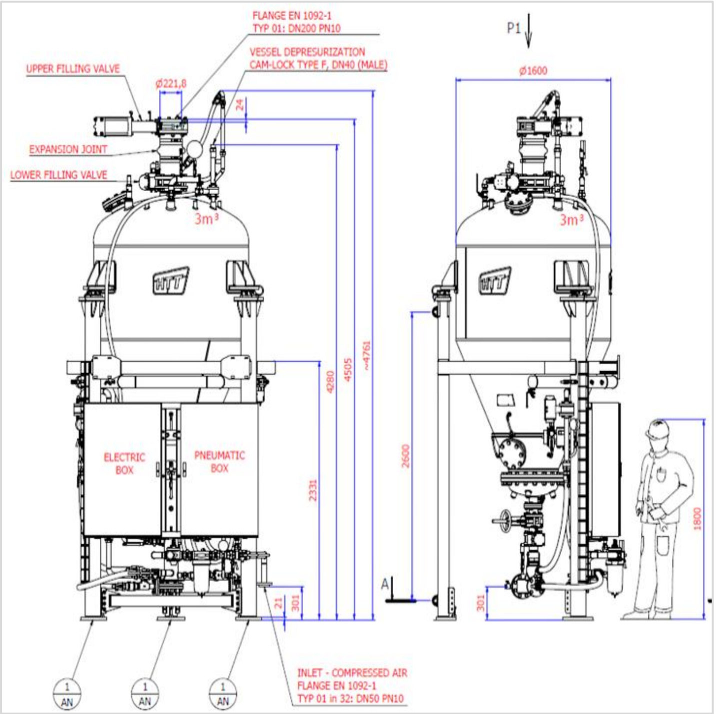
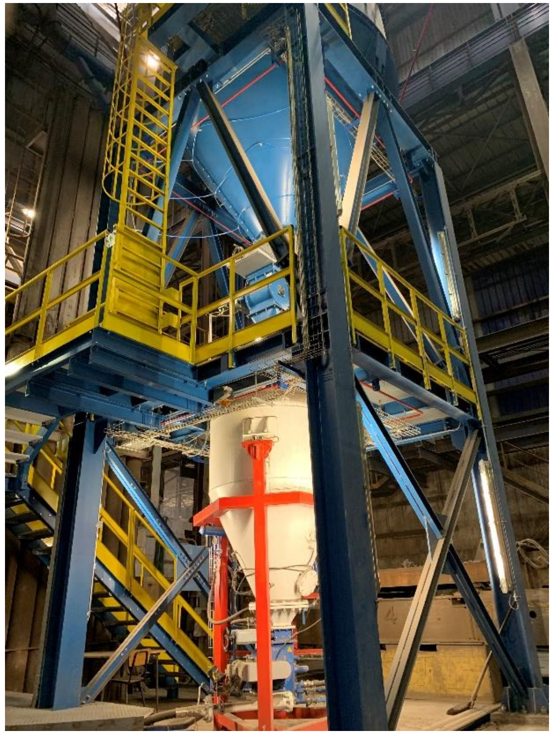
Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Industrial pneumatic and injection dispenser



Injection dispenser layout



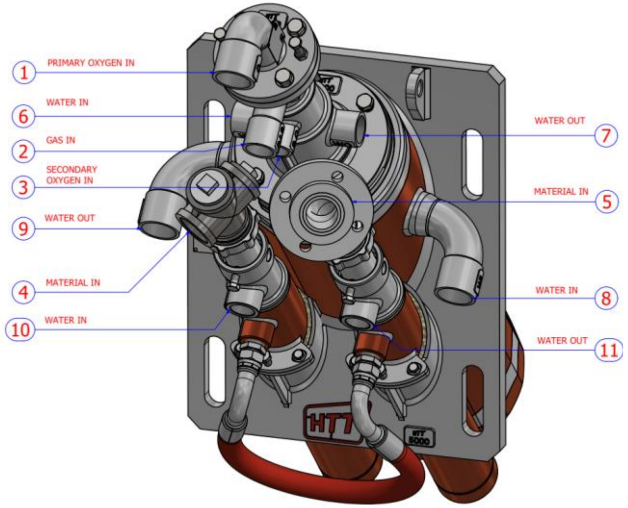
- The silo and pneumatic system has been purposely designed for plastic grain, with possibility to use inert gas.
- Compliance with ATEX Directive.
- The injection system is fully automatized and controlled from the Furnace control cabinet.
- It works with pressurized air.

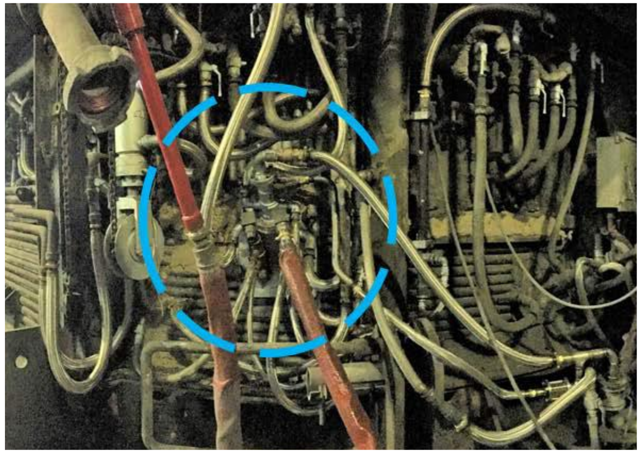
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Plastic recycling in the Steel sector – installation, starting up, operation and turn off

Injector and its installation at the EAF

- During standard operation, there are 3 anthracite lances used with a flow of 10 – 25 kg/min.
- One of them has to be switched off and substituted by the new polymer injector (approximately 30% of the injected anthracite is replaced by polymers).
- The injector is a water-cooled lance already equipped for simultaneous utilization of: Plastic grains, oxygen (to promote foaming as explained in paragraph above), or other grains (as anthracite, or slagging agents).





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Plastic recycling in the Steel sector – installation, starting up, operation and turn off

INDUSTRIAL TRIALS

- Plastic injection has been tested in industrial trials. The substitution percentage of coal was about 30 % (355 Kg of anthracite replaced by 345 Kg of polymers). The substitution ratio is more or less 1:1
- More than 40 process variables were considered as performance indicator and the outcomes of the tests have been considered positive: no production anomalies detected (average power consumption in agreement with usual values).
- The pneumatic and injection system worked properly, without significant problems.
- A measurement campaign at the chimney has been performed both with polymers and without, on the same process configuration. The results highlighted no differences.

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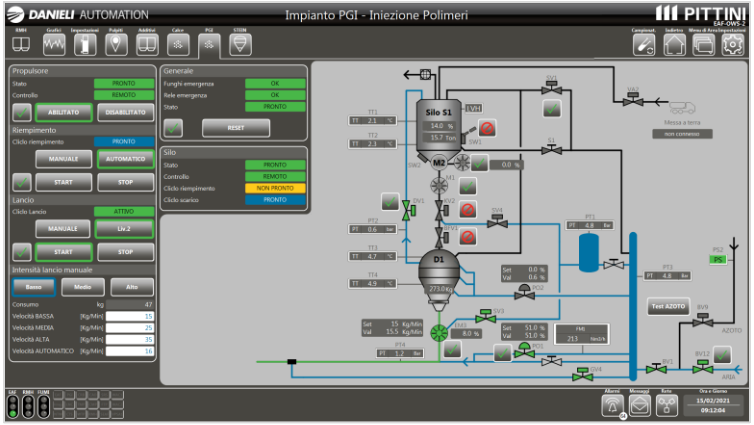
Plastic recycling in the Steel sector – healthy and safety issues

- ✓ The system is fully compliance with fire protection and is equipped with ATEX Zone 22 components (protection from explosion), in order to guarantee the highest safety working conditions.

ATEX zones		Level of danger
Gas	Dust	
Zone 0	Zone 20	Very high level of danger where explosive atmospheres exist continuously, or for long periods or frequently
Zone 1	Zone 21	High level of danger where explosive atmospheres can exist occasionally during normal operations
Zone 2	Zone 22	Normal level of danger where explosive atmospheres are not likely to be present during normal operations but, if they do occur, they will persist for a short period only

- ✓ A control system has been installed and couples with plant PLC and HMI for process control and real-time data acquisition.

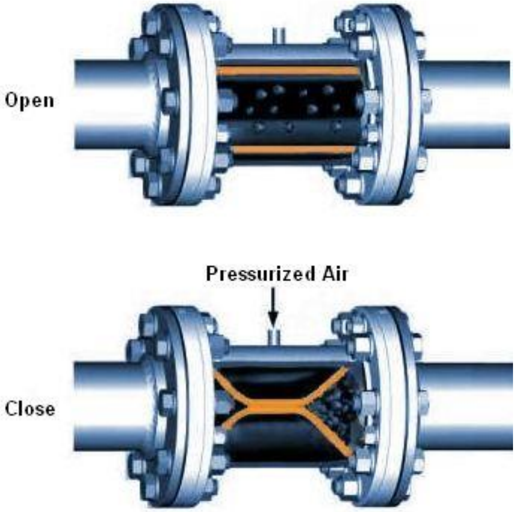


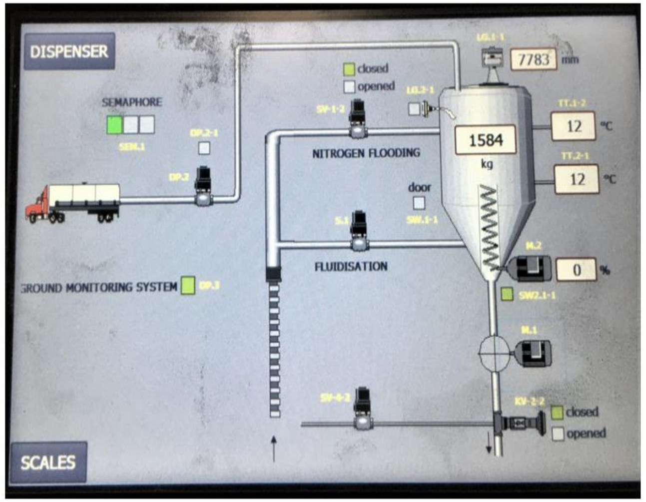
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Plastic recycling in the Steel sector – healthy and safety issues

Plastic grains charging system

- The piping has been designed for pneumatic conveying with special focus to grant the abrasion due to the material.
- The filling tube is coupled to a safety flanged joint.
- A pneumatic pinch valve open and close the tube by air pressure during the filling at any time.
- To obtain the safest process, multiple strategies have been implemented. The safety PLC has the task of managing all the control components and guaranteeing the safe loading of the plastic grains.





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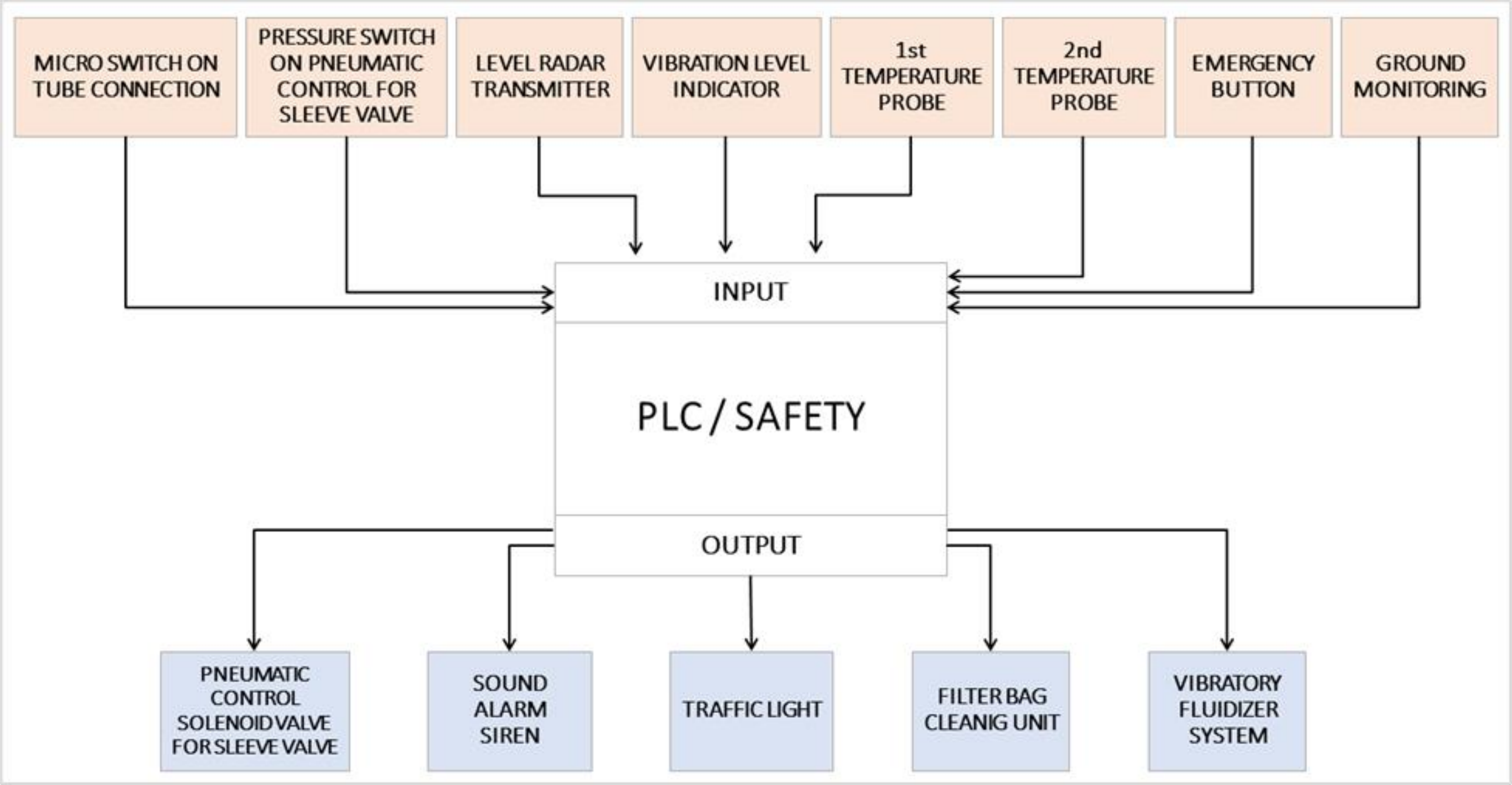
Plastic recycling in the Steel sector – healthy and safety issues

<u>*Safety PLC (programmable logic controller)*</u>

- Industrial computer system that monitors the state of input devices and control the state of output devices.
- Representative diagram of the signals managed by the PLC :



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Plastic recycling in the Steel sector – healthy and safety issues

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Safety PLC (programmable logic controller)

The start of material loading (which occurs with the connection of the loading pipe) must take place on condition that:

1. **No alarm condition** is active (system emergency circuit).
2. The **traffic light** has **GREEN LIGHT** on (silo material request condition).
3. The PLC must activate the GREEN light of the traffic light when it receives the signal from the weighting system (weight less than 10 tons). On the top of the silo, there is a **level indicator** (vibration type) that will allow continuous measurement intervening in exceeding the maximum containment level planned. As a redundancy level, there is also a **radar transmitter** that detects the achievement of the MINIMUM LEVEL (estimated about 500 mm about the silo discharge cone). The task of that radar transmitter is to check the content level within the Silo. The transmitter will provide the PLC with information on the content level present inside the SILO. Two different **temperature probes** are installed on the top of the silo. The probes are Thermocouples complete with on-board electronics. If, one or more probes, detect an internal temperature greater than a defined threshold (i.e. ≥ 100 ° C) they must cause so that the system activates an audible and light alarm signal.
4. When ground is confirmed on ground unit, the operator **pushes the button "START FILL"** and this sends the command of opening the pinch valve.
5. The PLC will issue the **power supply command** for the solenoid valve present in the pneumatic valve control with pinch valve which will allow the compressed air to open the pinch valve.

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Plastic recycling in the Steel sector – healthy and safety issues

Safety PLC (programmable logic controller)

6. At the same time, the PLC must enable the cleaning unit which will automatically take care of activating the **cleaning cycles of the filter bags**. This happens because the plastic material is transported by the air supplied by the silo truck. The bag filter, installed at the top of the silo, with cleaning cycles takes care of the outflow of this loading air.
7. A **weighing system** with load cells is provided to check the content of the material present inside the silo. It sends the weight value to a weight transmitter, installed on the leg of the silo. When the level of the loaded content reaches **80%** of the max allowed weight (Parameter in HMI), the PLC will provide a **signal** that activates the **ORANGE light** in the traffic light. When **100%** weight is reached pinch valve will close and the light switch to **RED**.
8. To manage the **unexpected loading**: Max level switch will close the pinch valve in any situation and light with turn RED or in case of silo filter failure or pinch valve pressure switch not working will switch FLASHING RED.
9. At this point, the person in charge of the load must **stop the pneumatic load and remove the loading pipe** from the connection.
10. The **disconnection** of the loading pipe.

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Plastic recycling in the Steel sector – healthy and safety issues

Silo equipment

Multiple strategies have been implemented for monitoring the silo during loading and usual working conditions.

- On the silo, there is an **inspection door** equipped with a safety switch. If the door is opened, the relative switch will act on the emergency circuit of the switchboard activating the total stop of the system with activation of one acoustic / light signal to be provided on the panel. As mentioned before, the silo is weighted using load cells and using radar transmitter a checking of the content level within it. Instead, the internal temperatures on the silo are monitored using two PT100 probes.
- For guarantying the highest level of security, FENO have implemented a **Nitrogen flooding system** for the silo to prevent fire trigger.
- The input medium is changed from **compressed air to nitrogen** by changing state of a valve for 15 min. If the pressure became to decrease, alarm arises and the system is switched back to compressed air.
- Beside this solution FENO adopted **fire prevention tools**: temperatures in the silo as well as in the pressure vessel are monitored by double channels. When any of the temperatures exceeds max allowed value (setting screen locked by password) the system is switched to nitrogen. The silo and the pressure vessel are filled by nitrogen by opening the dedicated valves.
- The **fire-fighting system** consists of 4 levels each with 8 water nozzles and will be full cone. When the total stop is received all valves turn to their normal status without power. It means that pressurization is stopped, and vessel pressure is vented.

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Plastic recycling in the Steel sector – healthy and safety issues

Injection dispenser and injector

- The dispenser includes **pressure vessel** (PED certified) mounted to a self-supporting dispenser frame. The dispenser includes **knife gate valve** of flap for refilling, sealing butterfly valve, pressure transmitters, pressure regulator, venting valve, and safety pressure release valve. The vessel is rested on **3 load cells** to provide information about vessel weight and injected amount.
- Scales and load cells are **Siemens Siwarex type**. Material is discharged from the vessel via a pneumatic knife gate valve to the mixing part to blend injected particles with transport air. Transport air is measured by flowmeter and controlled by proportional flow control valve. All valves are pneumatically actuated, and instrumentation is connected to electrical cabinet installed at the vessel frame. The vessel volume is 3 m3.
- The **line back pressure** is monitored for auto-detection of possible material blockages. Even in this case, all components and complete equipment including control cabinet are for ATEX Zone 22 due to potential use of volatile and fine material.
- **System operation is fully automatic**. Setpoints of material flow will be received from remote HMI system and accepted by local PLC cabinet located directly on dispenser frame.
- The dispenser has a dedicated **electrical cabinet** where all signals will be wired to. The cabinet is equipped with local IO and CPU module Siemens ET200SP CPU 1512SP-1 PN so that the external wiring is limited only to power supply and data communication (PROFINET). The local cabinet is connected via PROFINET to the furnace HMI.

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Plastic recycling in the Steel sector – healthy and safety issues

Injection dispenser and injector

- Also, there is a **local service HMI Touch panel Siemens KTP400** directly on machine cabinet. Local HMI panel enables the control of all valves and actuators in service mode as well displays all analog values such as pressures, flow, and material weight.
- **PLC software** (Siemens TIAP) enables manual or remote control based on flow/pressure/mass flow set points received from HMI.
- The automation can manage the **auto refill of the vessel** when one of these two situations occurs: the furnace starts tapping and vessel weight is below “Max vessel weight” or when the material weight in the vessel drops below “Min vessel weight”.
- The new injection system is a **prototype installation**, but it is fully **representative of an industrial component**, and it will be used during regular production for long term industrial trials.

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Thank you for your attention

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ANEX B. STANDARD PRESENTATIONS

GENERAL PRESENTATION FOR POLYNSPIRE FINAL EXPLOTATION WORKSHOP & EVENT

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Innovative technologies for plastic recycling

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Welcome!

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Agenda and dynamics for today

polynSPIRE

09:30	Welcoming and project presentation [CIRCE]

[CIRCE-Mr. Alberto Frisa & Ms. Marina Cárdenas] Project coordinators

circe
RESEARCH CENTRE
FOR ENERGY RESOURCES
AND CONSUMPTION

CIRCE Foundation
Zaragoza, Spain
https://www.fcirce.es/
polynSPIRE coordinator

Mr. Alberto Frisa
polynSPIRE Technical Coordinator
Industrial Processes Department
CIRCE Foundation
afrisa@fcirce.es

Ms. Marina Cárdenas
polynSPIRE Project Manager
Public Programs Unit
CIRCE Foundation
mcardenas@fcirce.es

Contact us for any request regarding polynSPIRE!

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Agenda and dynamics for today

10:00 Demos presentations & Project advances

3 round tables approaching the following topics:

- **[10:00-10:40] Chemical recycling: Depolymerization assisted by MW and SMM**

  Chaired by Ms. Seda Araci (KORDSA). Speakers:

  [NIC] Mr. David Pahovnik

  [CIRCE] Mr. Alberto Frisa

  [IONIQA] Ms. Katarina Babić

- **[10:40-11:10] Mechanical recycling: Additivation of recycled material with specific additives**

  Chaired by Mr. Carlos González (CIRCE). Speakers:

  [AITIIP] Mr. Julio Vidal

  [BADA] Mr. Víctor Peinado

  [TUE] Mr. Hans Heuts

- **[11:10-11:40] Valorisation of plastic residues in the steel sector**

  Chaired by Mr. Alberto Frisa (CIRCE). Speakers:

  [CSM] Mr. Filippo Cirilli

  [HTT] Mr. Jaroslav Brhel

  [I.BLU] Ms. Elisa Marchesan

  [FENO] Mr. Daniele Gaspardo

11:40 Coffee break

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Agenda and dynamics for today

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12:00	Brief presentation of R&D sister projects related to circular economy/plastics recycling
12:00	[MICHELIN-Ms. Margarita Dorado]- **BLACKCYCLE** *A major European project for recycling end-of-life tyres into new tyres.*
12:15	[ITENE-Mr. José Luis Dieguez]- **MERLIN** *Increasing the quality and rate of MultilayER packaging recycLINg waste.*
12:30	[CENTEXBEL- Ms. Madeleine Wéry] **CISUFLO** *Route to circular & sustainable floor coverings.*
12:45	[AZCATEC- Mr. Luis Azaña] & [CSM-Mr. Filippo Cirilli] **PLASTICE** *New technologies to integrate PLASTIC waste in the Circular Economy*
13:00	Cocktail

BLACK CYCLE
Move to the green revolution

MERLIN

CISUFLO
CIRCULAR SUSTAINABLE FLOOR COVERINGS

PLASTICE

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Agenda and dynamics for today

polynSPIRE

14:00 Cocktail and networking session

Matchmaking event - How does it work?

A matchmaking event is a quick and easy way to meet potential cooperation partners. 15 minutes run fast, but they are enough to build first connections before the meeting ends and the next talk starts.

1) Registration

Register via the Register button.

2) Publish a business profile to showcase your needs

Create a clear and concise business profile to raise your visibility on the b2match platform.

Your profile should describe who you are, what you can offer to potential partners, and who you want to meet. A good profile will generate significantly more meeting requests.

FLOWCODE

PRIVACY.FLOWCODE.COM

https://polynspire-exploitation-workshop.b2match.io/login

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Agenda and dynamics for today

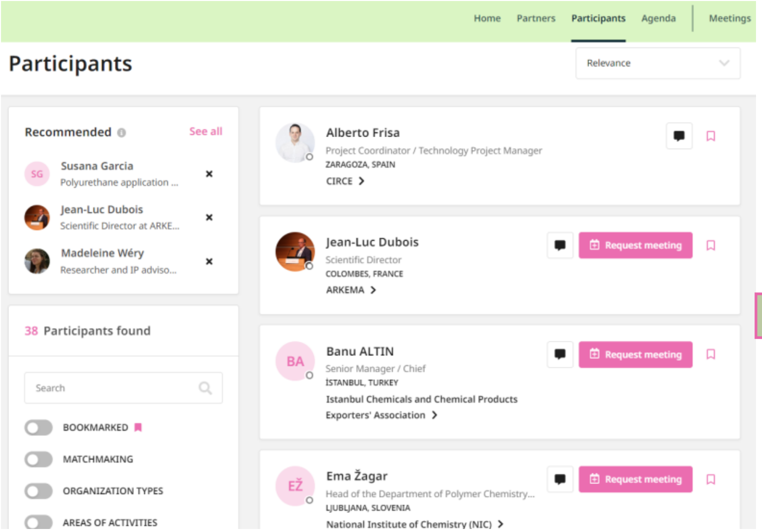
14:00 Cocktail and networking session

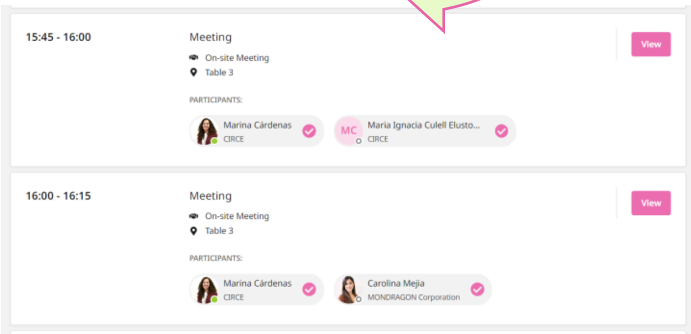
3) Browse profiles of attendees

Go to the participants list to find out who is offering interesting and promising business opportunities.

4) Send & receive meeting requests

Browse published participants profiles and send meeting requests to those you want to meet during the event.





Check participants & request meetings now!

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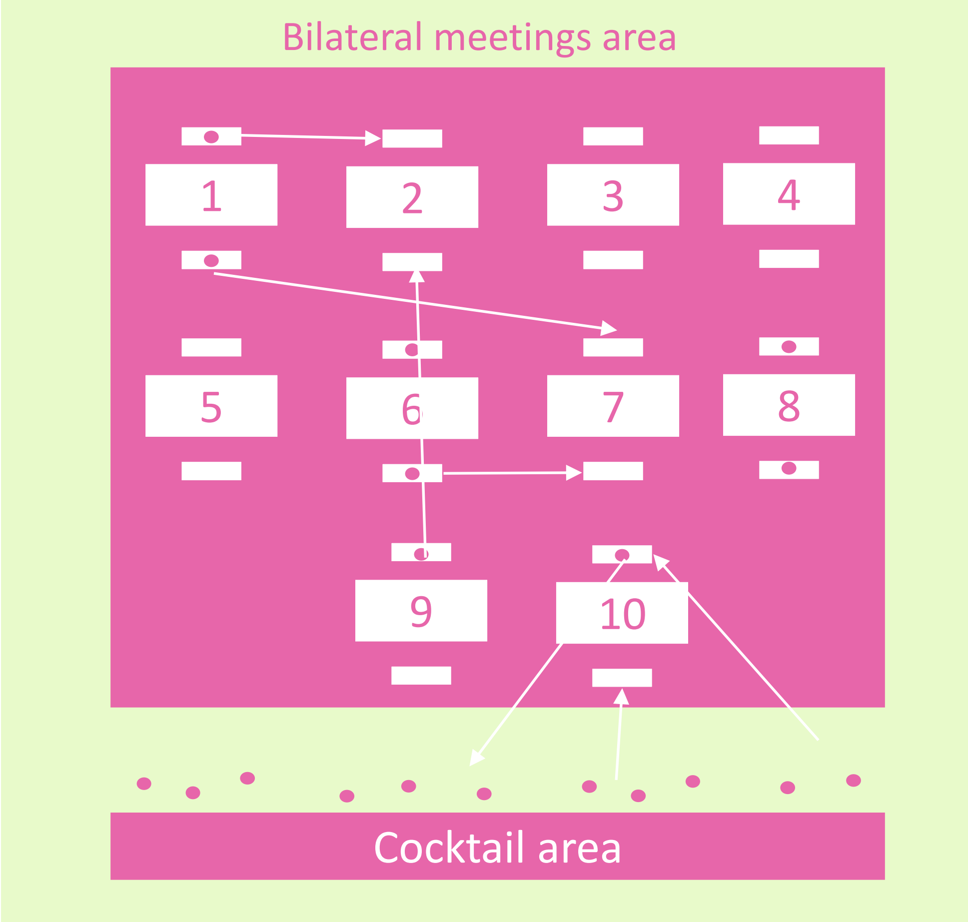
Agenda and dynamics for today

14:00 Cocktail and networking session

5) Matchmaking Event

Access your meetings in My Agenda or Meetings, where you have your complete schedule for the event and the list of your meetings.

Check your agenda and don't be late!



17:00 Closure of the event

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Innovative technologies for plastic recycling

Happy event & matchmaking!

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- ✓ Main Objective
- ✓ Facts & Figures
- ✓ Consortium Layout
- ✓ Goals, challenges and solution

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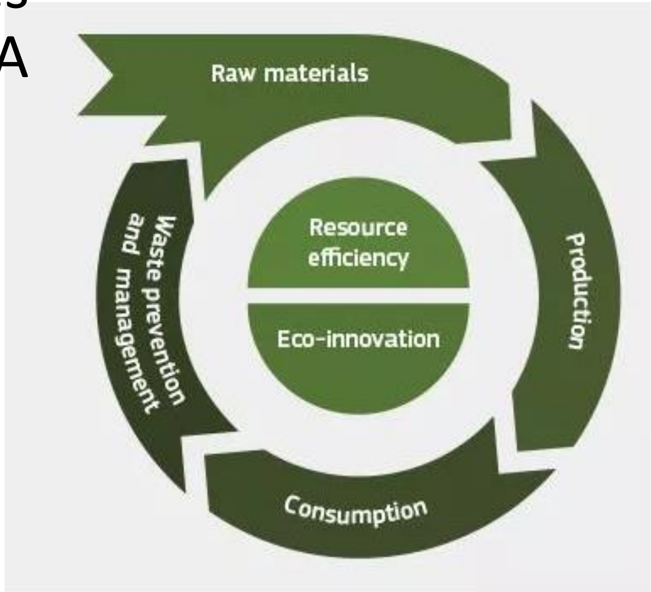
Main Objective

Demonstrating at TRL 7 a set of **innovative, cost-effective and sustainable solutions with an aim to improve the energy and resource efficiency** of plastic recycling processes for materials containing at least 80% plastic focusing on PA and PU.



Source:http://ec.europa.eu/environment/green-growth/index_en.htm

The project is focused on following plastic containing materials:

- **postconsumer** (after products' end of life)
- **post-industrial** (produced during transformation processes from raw materials to final product)

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PolynSPIRE: Facts & Figures

Full title: Demonstration of Innovative Technologies towards a more Efficient and Sustainable Plastic Recycling

Start date: 01.09.2018

Duration: 54 months

Number of partners: 21 including research/academic institutions, governmental organization, industries and SMEs

Budget: €9.95 Million

EU contribution: €7.94 Million

TRL at the end: TRL 7 (system prototype demonstration in operational environment)

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Consortium

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The project involves all relevant actors along the value chain

Technology Developers	circe (Research Centre for Energy Resources and Consumption); aitiip centro tecnológico; ioniqa; TU/e Technische Universiteit Eindhoven University of Technology; RINA
Waste Managers and Providers	I.BLU Gruppo Iren; Bada
End Users and Validators	NOVAMONT; NUREL; KORDSA; ARKEMA (Innovative Chemistry); FERRIERE NORD; REPSOL; MAIER
Technology Providers and Manufacturers	:fm; CPPE (Chemical & Pharmaceutical Process Engineering); HTT
Dissemination, Communication and Standardisation Experts	DS; EUPC; IKMIB

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Our Approach

- ✓ The problem
- ✓ Challenge
- ✓ polynSPIRE Solutions
- ✓ Business Plan
- ✓ Expected Impact

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Plastic: Where is the problem?

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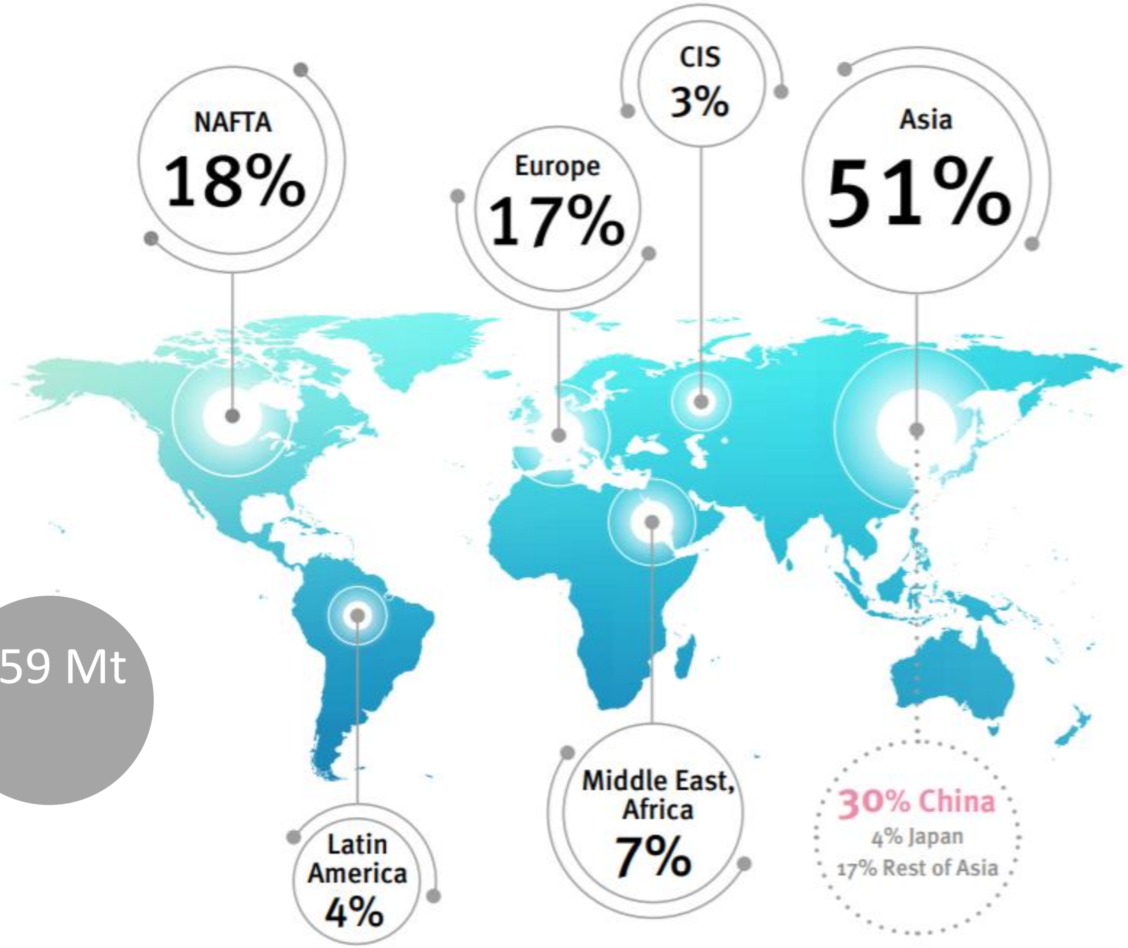
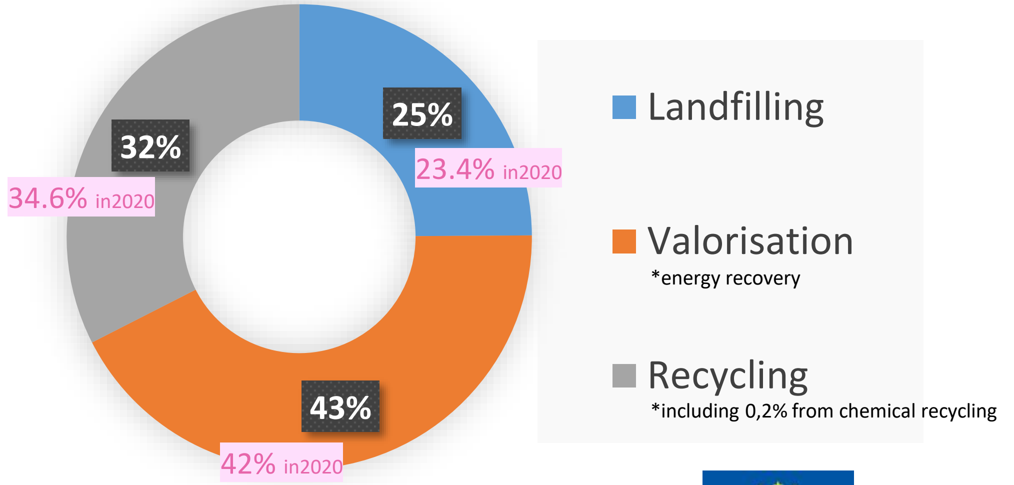
359 Mt



Worldwide production
(Source: Plastics the Facts 2019, PlasticsEurope)

Consumption in Europe: 51.2 millions of tons (17% of worldwide production)

29.1 Mt of plastic post-consumer waste were collected



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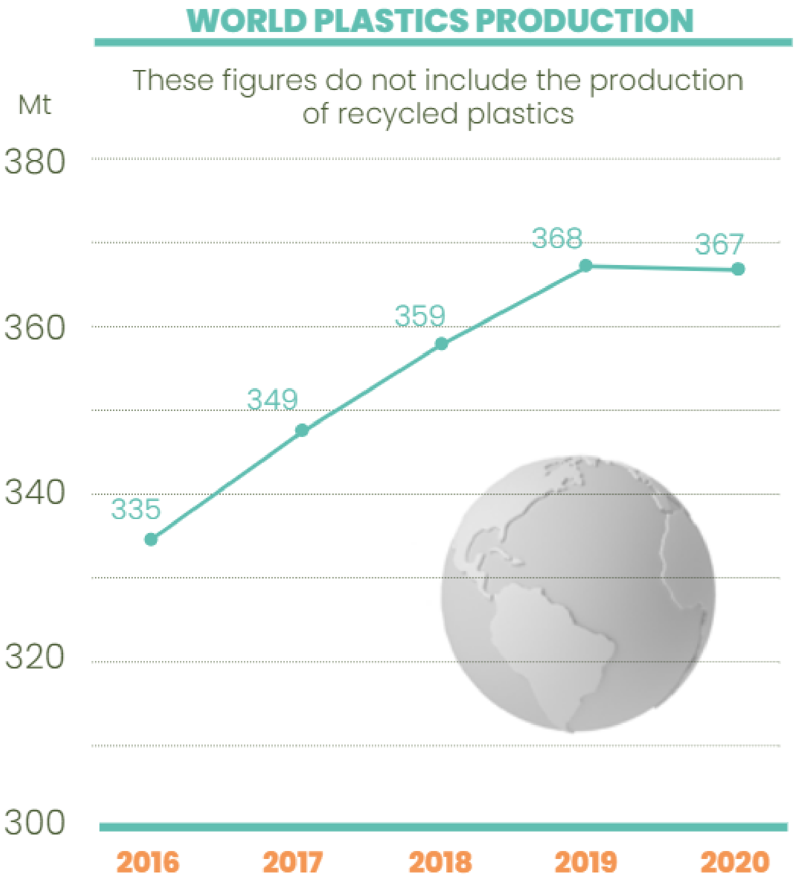
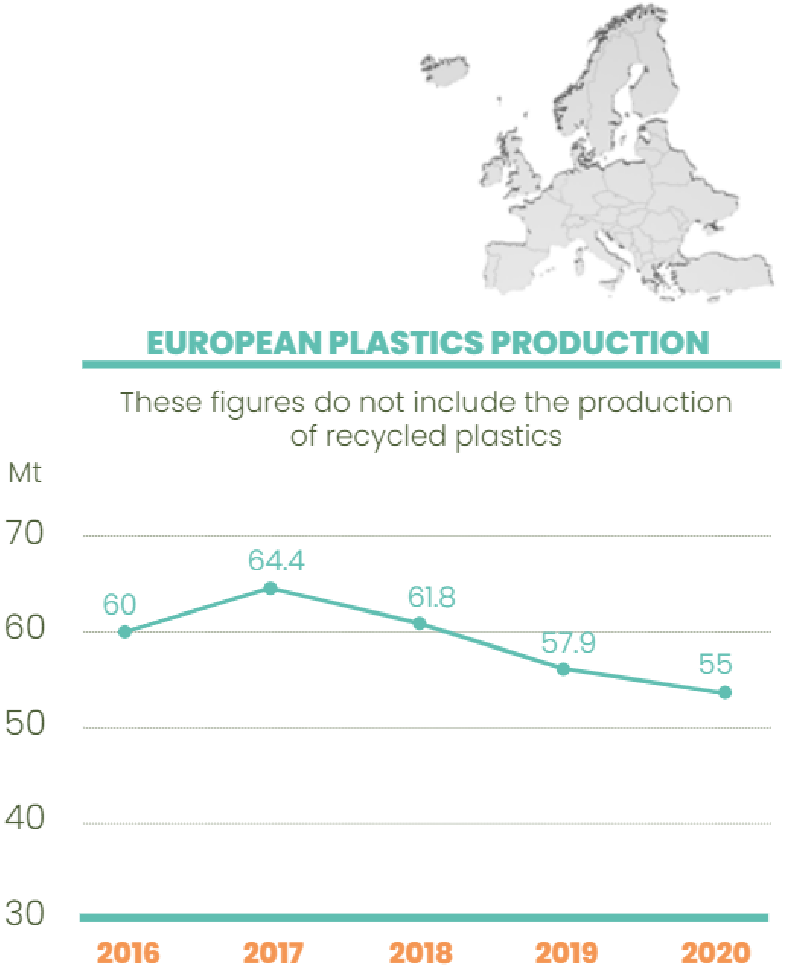
Plastic: production evolution





(Source: Plastics the Facts 2021, PlasticsEurope)

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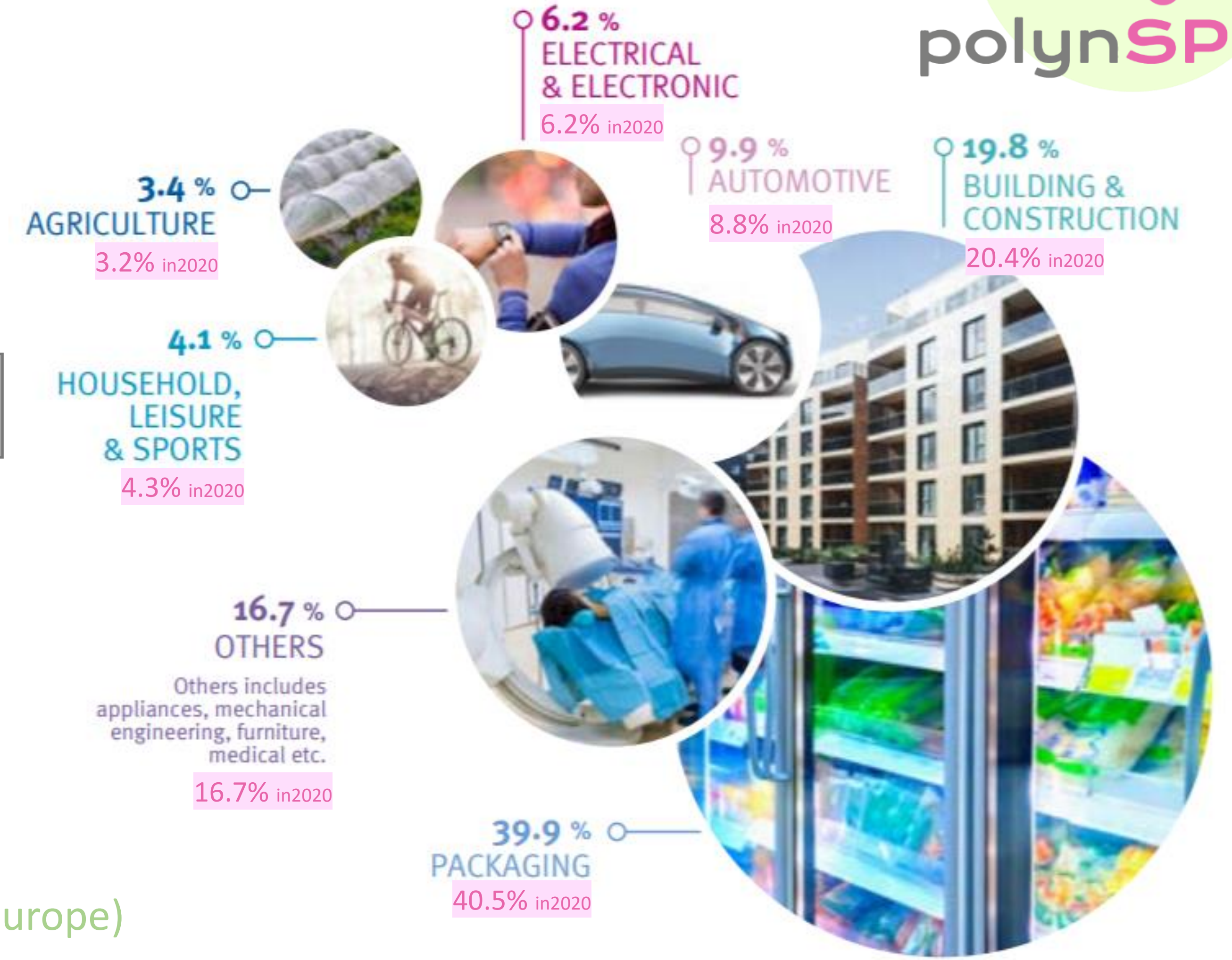
Plastic: Where is the problem?

Plastics use per sector



(Source: Plastics the Facts 2019, PlasticsEurope)

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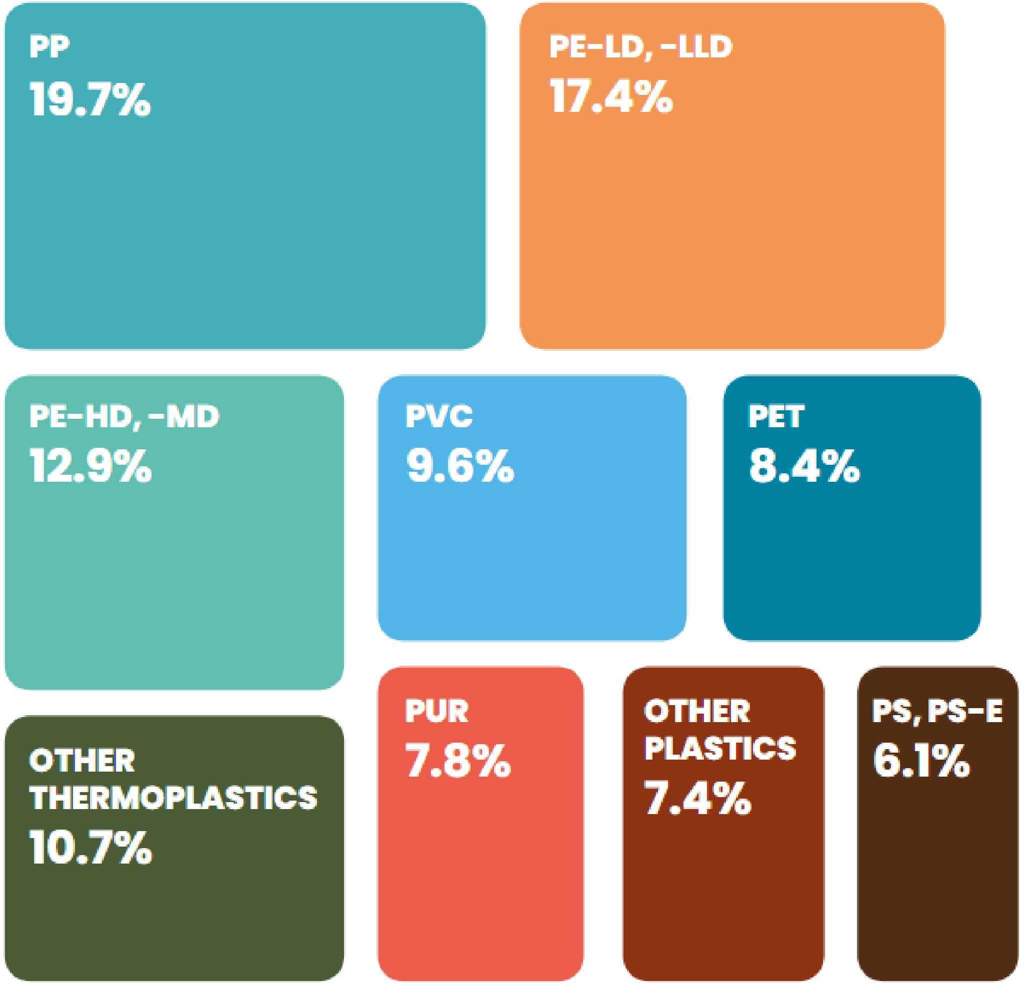
Plastic: Distribution by resin types



(Source: Plastics the Facts 2021, PlasticsEurope)

PP	Food packaging, sweet and snack wrappers, hinged caps, microwave containers, pipes, automotive parts, bank notes, etc.
PE-LD, -LLD	Reusable bags, trays and containers, agricultural film, food packaging film, etc.
PE-HD, -MD	Toys, milk bottles, shampoo bottles, pipes, houseware, etc.
PVC	Window frames, profiles, floor and wall covering, pipes, cable insulation, garden hoses, inflatable pools, etc.
PET	Bottles for water, soft drinks, juices, cleaners, etc.
PUR	Building insulation, pillows and mattresses, insulating foams for fridges, etc.
OTHER PLASTICS	Includes other thermosets such as phenolic resins, epoxide resins, melamine resins, urea resins and others.
PS, PS-E	Food packaging (dairy, fishery), building insulation, electrical & electronic equipment, inner liner for fridges, eyeglasses frames, etc.
OTHER THERMOPLASTICS	Hub caps (ABS); optical fibres (PBT); eyeglasses lenses, roofing sheets (PC); touch screens (PMMA); cable coating in telecommunications (PTFE); and many others in aerospace, medical implants, surgical devices, membranes, valves & seals, protective coatings, etc.

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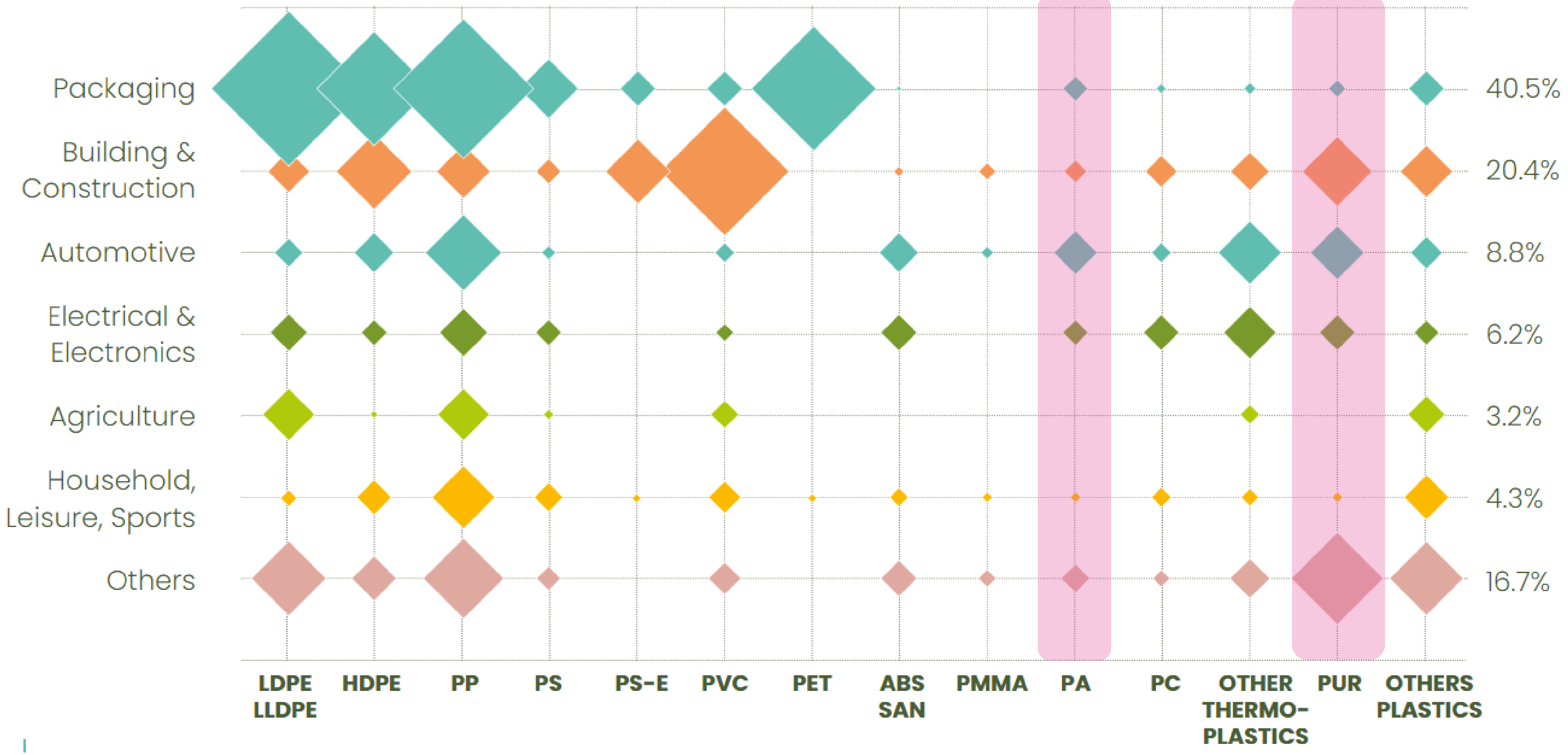
Plastic: By segments & polymers



(Source: Plastics the Facts 2021, PlasticsEurope)
*demand estimations only refer to virgin plastics

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How can we solve the problem?

Some open questions to think about

- Is it possible to built a world without plastic?
- For the engineering and automotive sector…
  - How many lives has plastic use saved?
  - What is the relationship between weight and fuel consumption?
  - What advantages do smart plastic materials bring in comparison to conventional ones?
- If the use of plastic is eliminated for all the applications, what do we do with the millions of plastic generated?

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polynSPIRE and Circular Economy Package

- In 2018, the European Commission adopted a new set of measures, that will help transform Europe's economy to become more sustainable and that will support existing Circular Economy Action Plan
- The new measures include a wide EU Strategy for Plastics in the Circular Economy that will help to transform a smart, innovative and sustainable plastics industry, where design and production fully respects the needs of reuse, repair and recycling.
- polynSPIRE is supporting this effort by introducing a set of novel approaches to recycling and usage of raw materials

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Challenges

- **Recycling and redesigning the plastics value chain are essential in reusing plastic waste material and avoiding landfill**
- **Demand of recycled plastic: 6%**

Technological barriers

- The existing sorting and waste management systems not able to separate plastics blends and composites
- The lack of efficient and flexible valorisation technologies
- The heterogeneity of plastic difficult the mechanical recycling of these plastic materials

Non-technological barriers

- Plastic waste is generated at different points of the value chain
- Existing standards are not homogeneous along Europe (e.g. Waste Directive and End of Waste Criteria)

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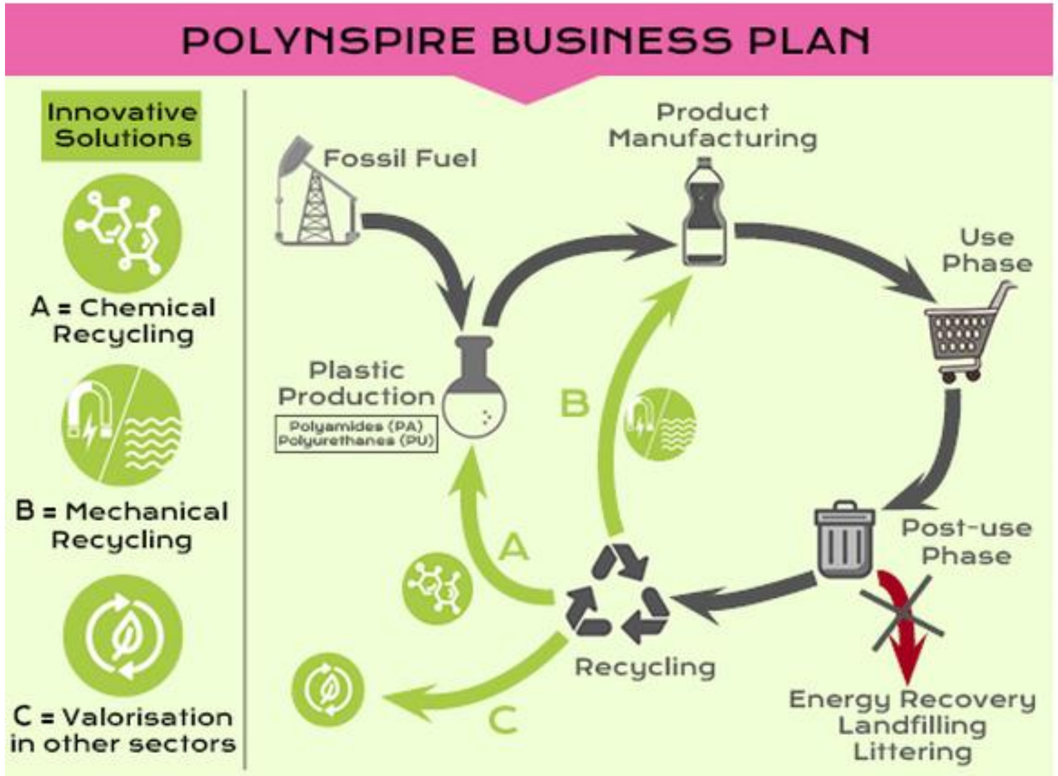
Our Solution

- A cost-effective transformation of plastic waste into raw materials through its recycling and valorisation
- polynSPIRE contains three innovative solutions:
  - ➢ Chemical recycling (Innovation A)
    - ➢ A.1. Via assisted by microwaves
    - ➢ A.2. Via assisted by SMM
  - ➢ Mechanical recycling (Innovation B)
    - ➢ Vitrimers
    - ➢ Enhancement by high-energy irradiation
    - ➢ Integrating additives
  - ➢ Valorisation (Innovation C)



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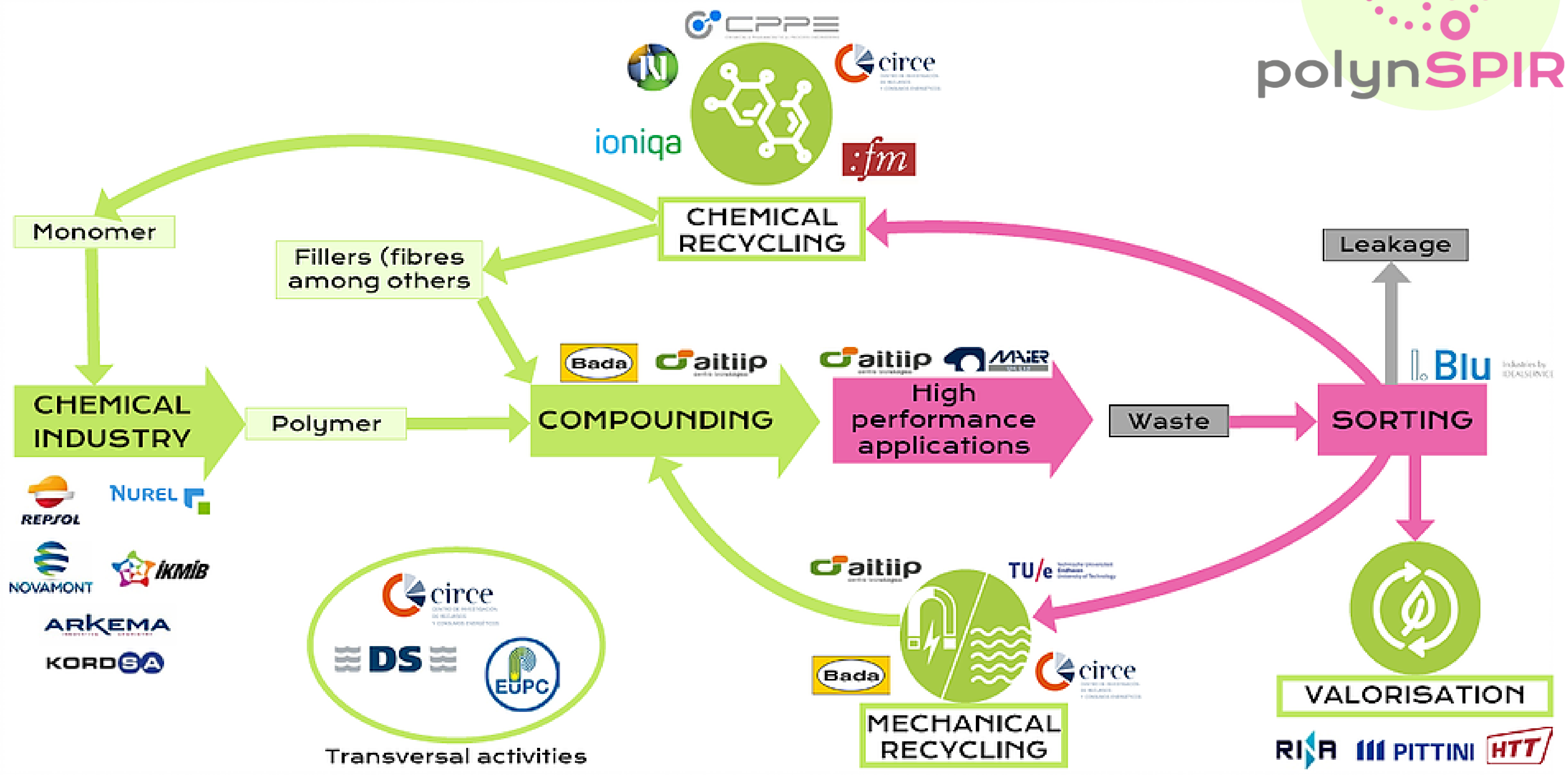
New polynSPIRE plastic value chain



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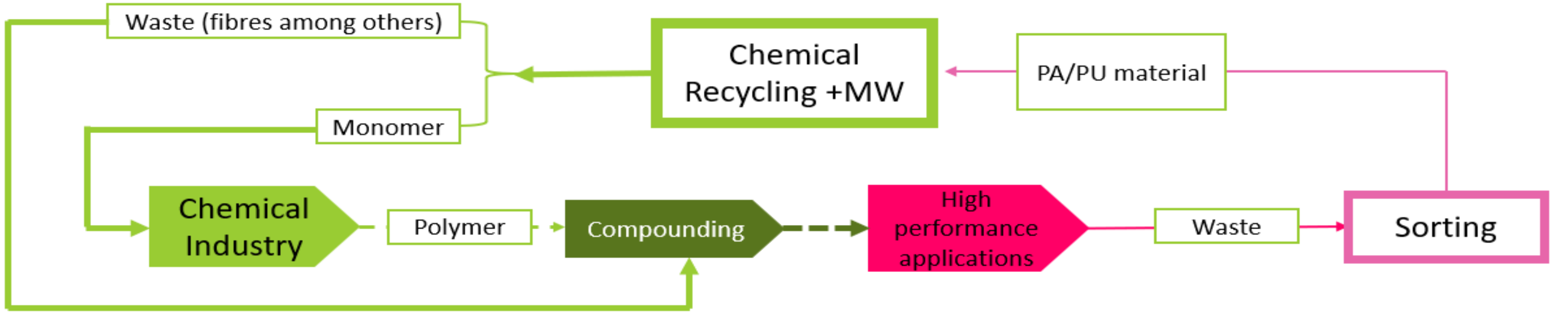
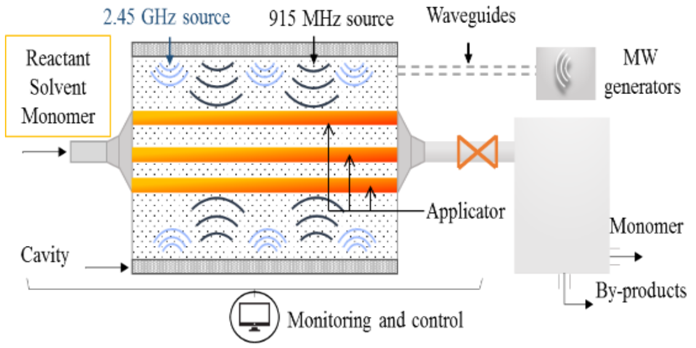
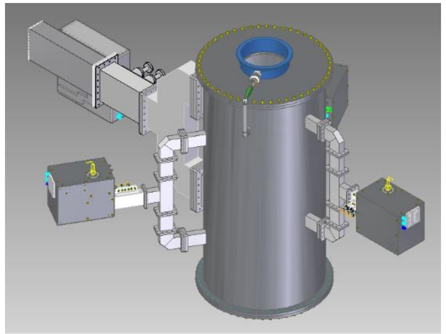
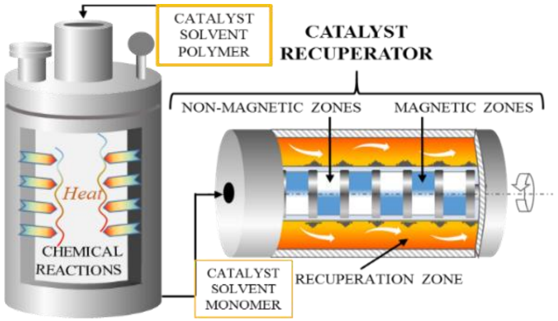
Innovation A

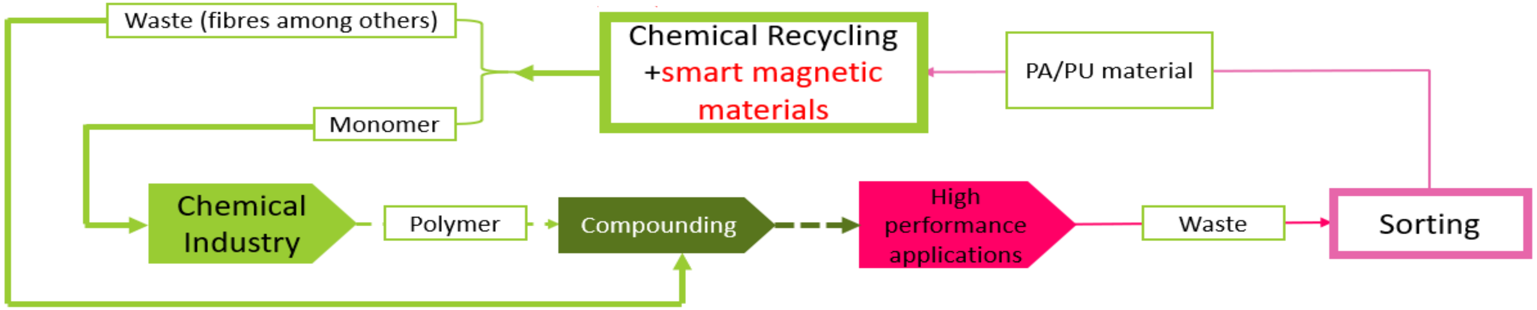


- Shorter times
- Lower energy requirements









- Higher selectivity
- Milder conditions
- Catalyser recovery

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Innovation B

1. Vitrimers

Additives (vitrimers)

Sorting → PA/PU + other material → Physical recycling → Compounding → Other Industries

Collected Polymer | Compound/ Additive → Reactive Extrusion — Vitrimer / CAN

- Better mechanical properties
- Reversible – not affecting future recycling

2. High Energy Radiation

Sorting → PA/PU + other material → Physical recycling → Compounding → Radiation (gamma, electron beam) → Other Industries

Additives → Compounding

- Better mechanical properties
- Compatible with existing equipment

3. Compatibilizing Additives

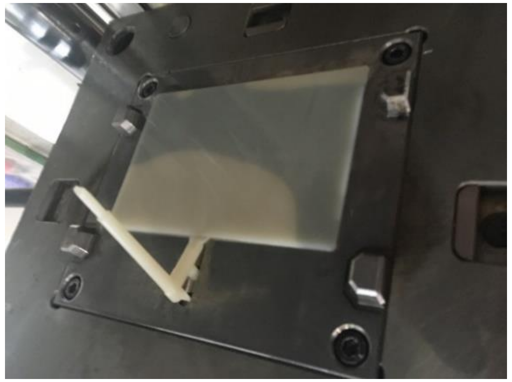
Compatibilizing Additives

Innovation Pillar B.2

Sorting → PA/PU + other material → Mechanical recycling → Compounding → Other Industries

Innovation Pillar 1

Sorting → PA/PU + other material → Chemical Recycling + MW → Fillers (reinforcement fibres among others) / Monomer → Other routes



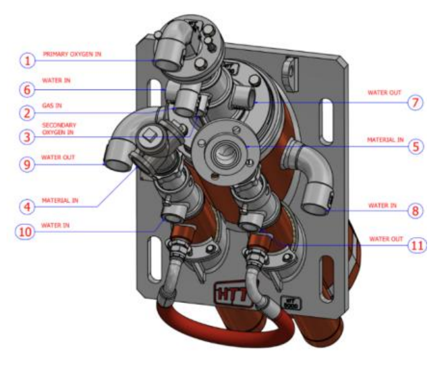
- Improved properties

Innovation C

Enhance valorisation of low-grade plastic wastes by using them as carbon source in steel industry and reduce mineral ore. These wastes promote foaming of the slag, thus improving the energetic and environmental performance of the furnace.

Sorting → Plastic post – consumer residues: PA/PU and polyolefin → Product adaptation → Valorisation in the steel sector → Steel Industry - EAF

Injector installed on the water-cooled side wall of the furnace



Drawing of the injector showing the connections to the utilities

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Expected Impact

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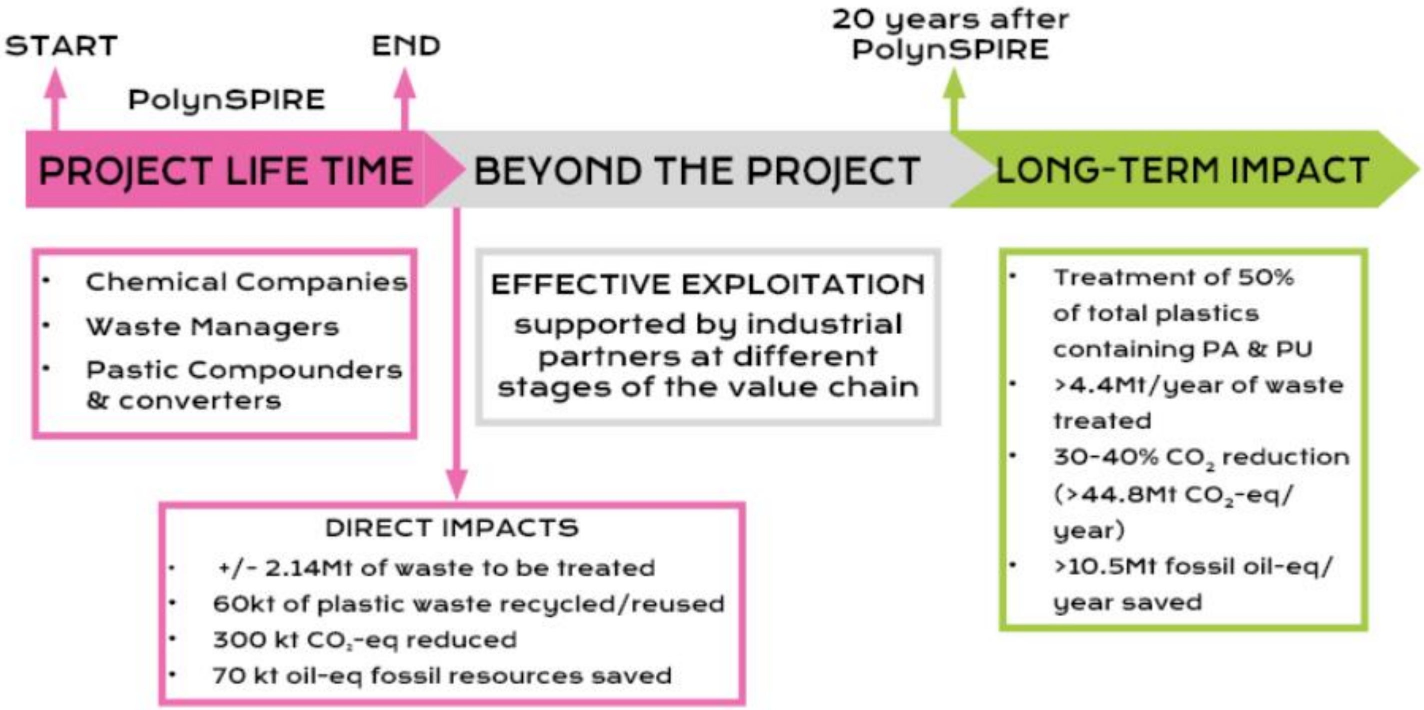
The project address 100% waste containing streams ensuring the recycling of **at least a 50% of total plastics containing PA and PU leading to a reduction of CO_2 equivalent emissions between 30% and 40%**.

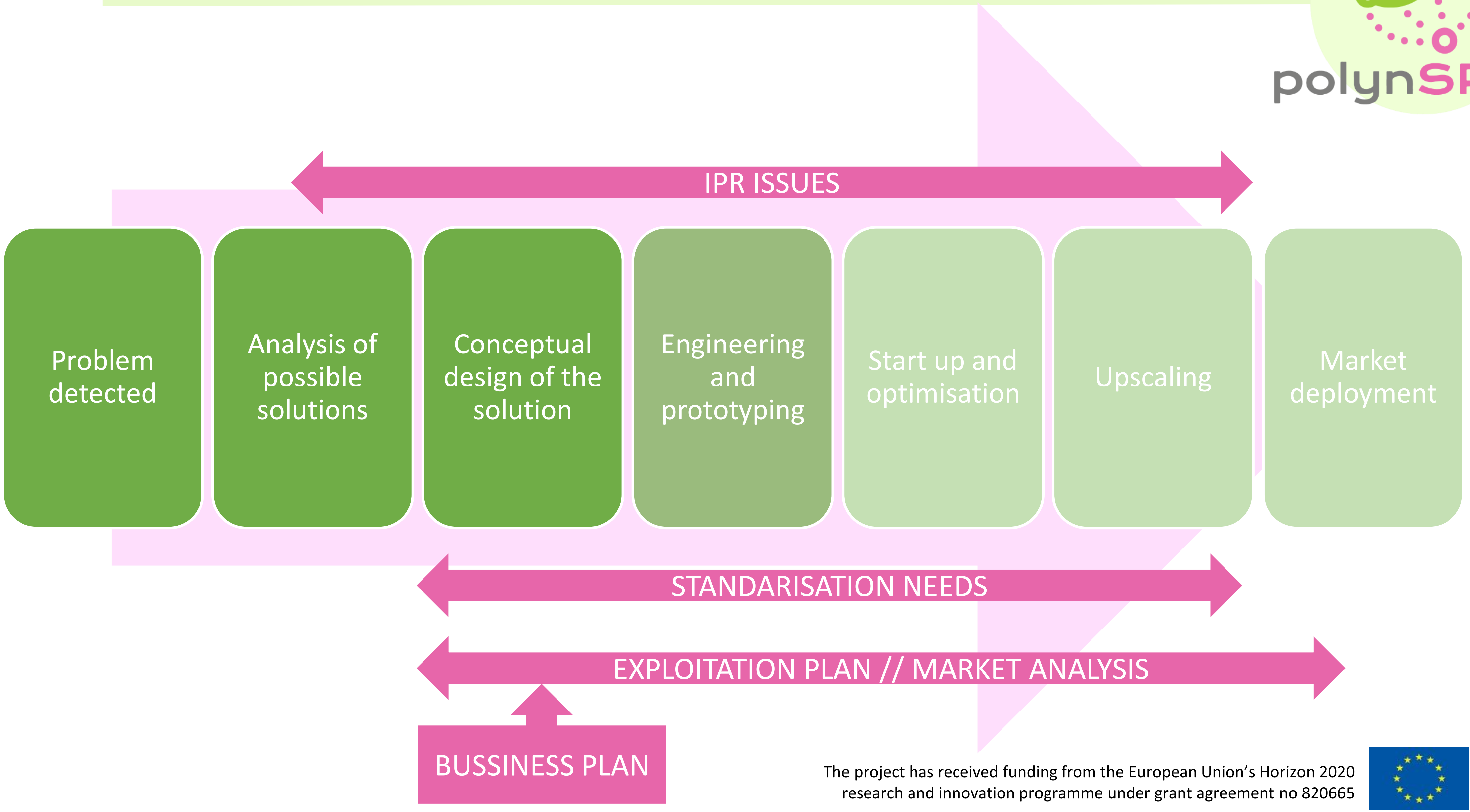


Our compromise:

- +60-70% energy efficiency increase in chemical recycling
- +10 times of reacting time reduction
- 10-20% of increase in mechanical
- Up to 80% of coal substitution in electric arc furnaces
- New business models

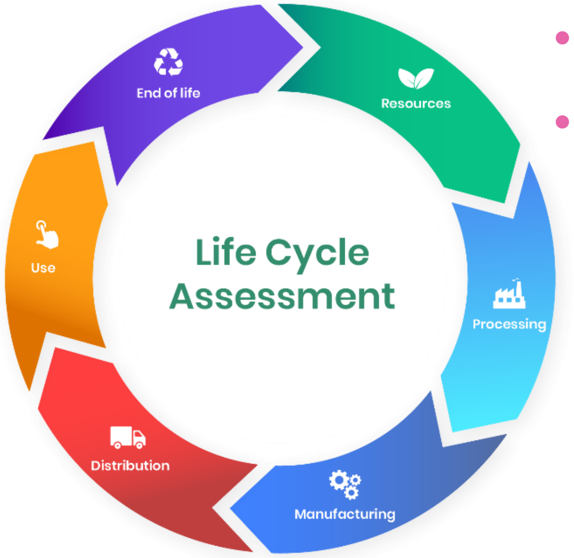
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Lifetime of polynSPIRE innovations

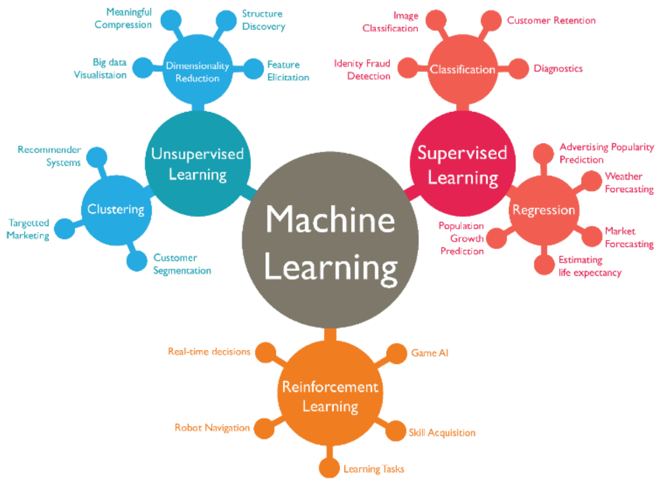


The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Transversal activities



- Life Cycle Cost
- Thermoeconomic analysis



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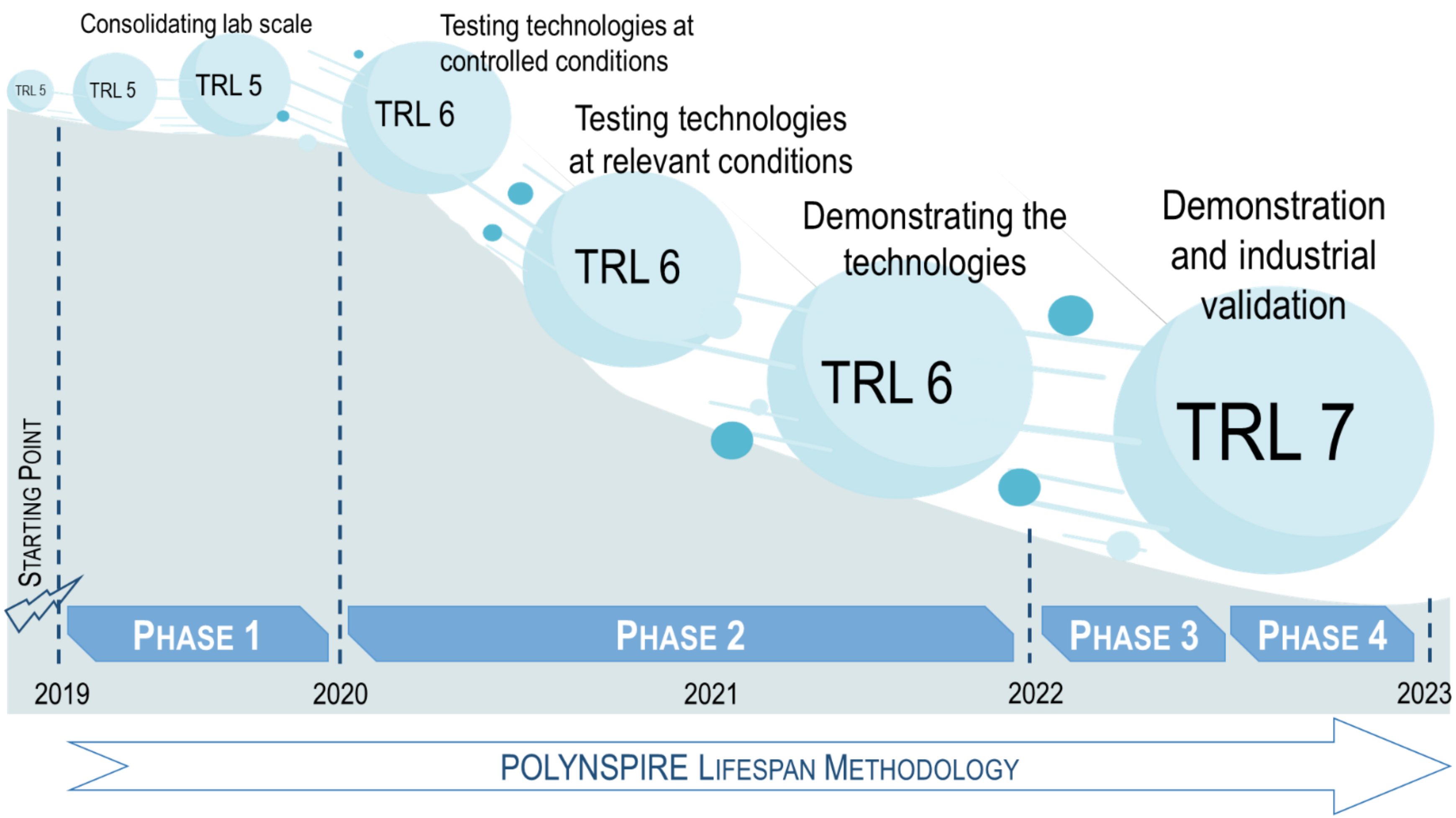
Assess and validate plastics recycling from different strategies:

- Life Cycle & Sustainability assessments
  - Compare with the use of virgin material
- Machine learning algorithms:
  - Use process data to get the optimal parameters
- Health and safety:
  - Creating safe products and processes
- Standardization:
  - Taking part in CEN/TC 249/WG 11 group

Demonstrate how much better it is to use recycled plastics than new ones

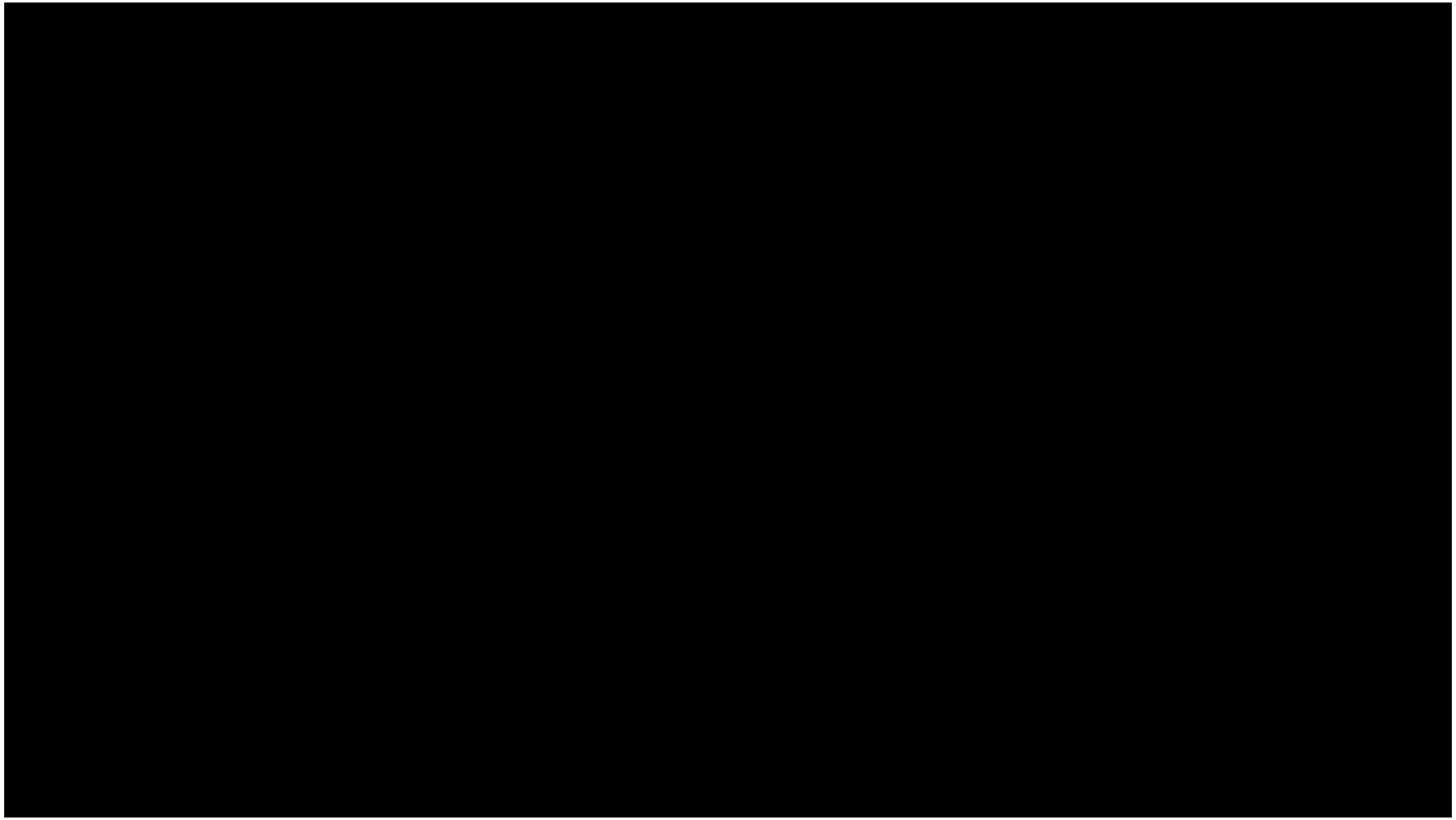
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Project path: 4 months remaining



The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main Objective



The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

It's in our hands to make a difference…

polynSPIRE

Innovative technologies for plastic recycling

Would you like to join us?

www.polynspire.eu

afrisa@fcirce.es

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

STANDARD PRESENTATION OF CHEMICAL RECYCLING

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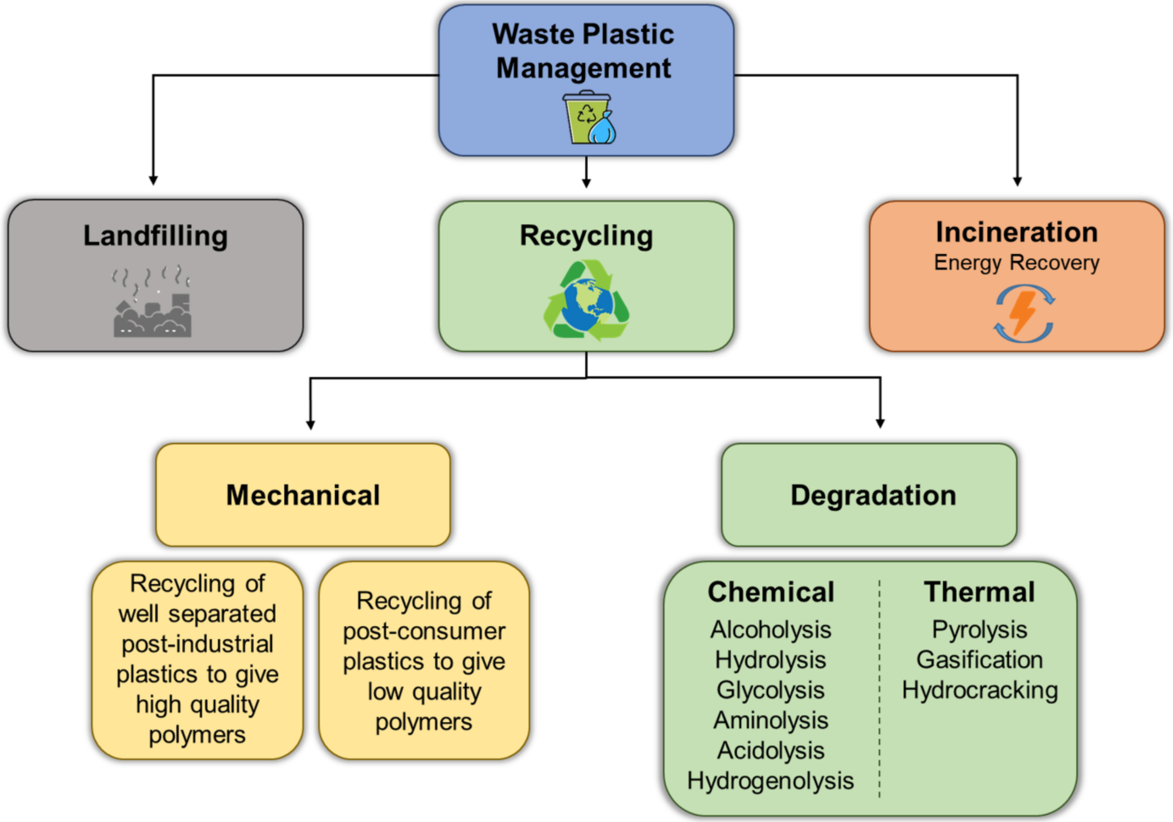
Innovative technologies for plastic recycling

Chemical recycling assisted by microwaves

Chemical recycling assisted by smart magnetic materials

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Polymer Recycling

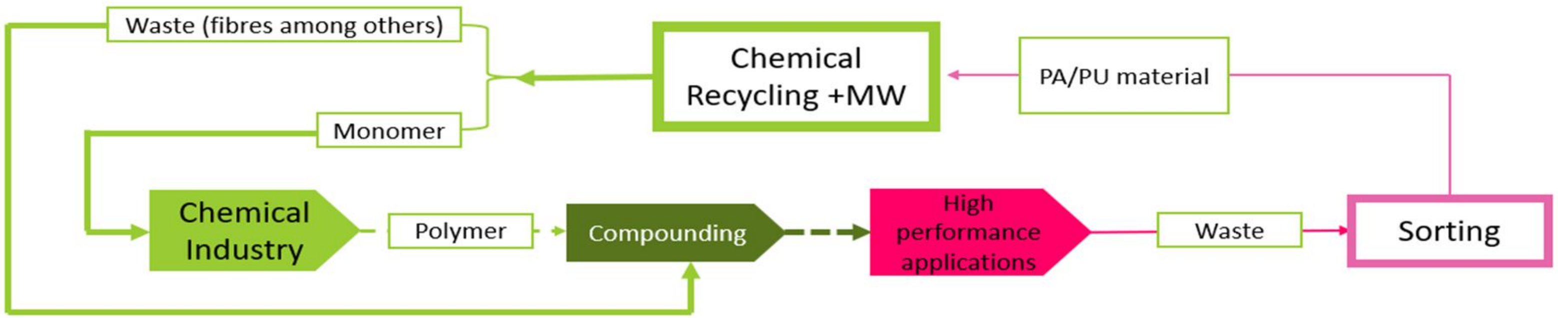


The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

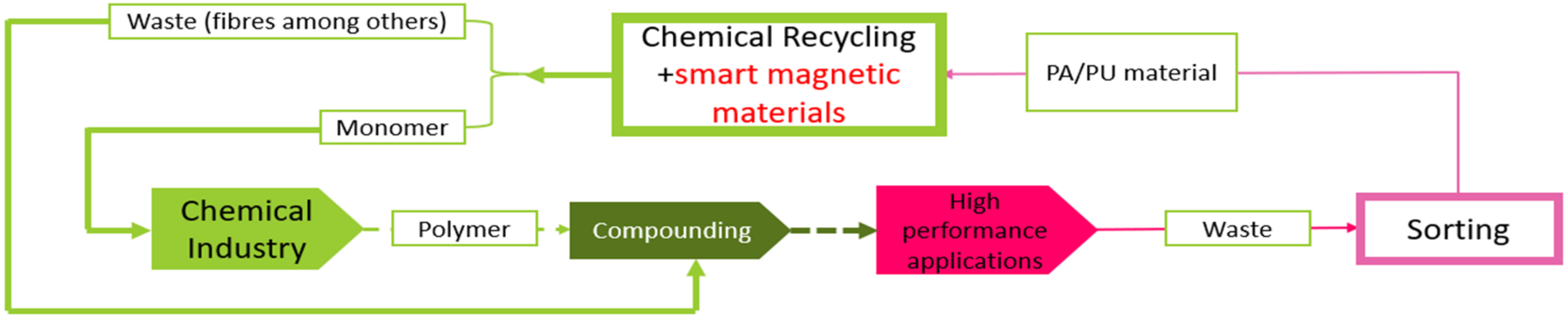
Chemical recycling

- The following technologies are used to depolymerize plastic materials to obtain their monomers (raw materials for polymer production)

Chemical recycling assisted by microwaves



Chemical recycling assisted by smart magnetic materials



The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

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Innovative technologies for plastic recycling

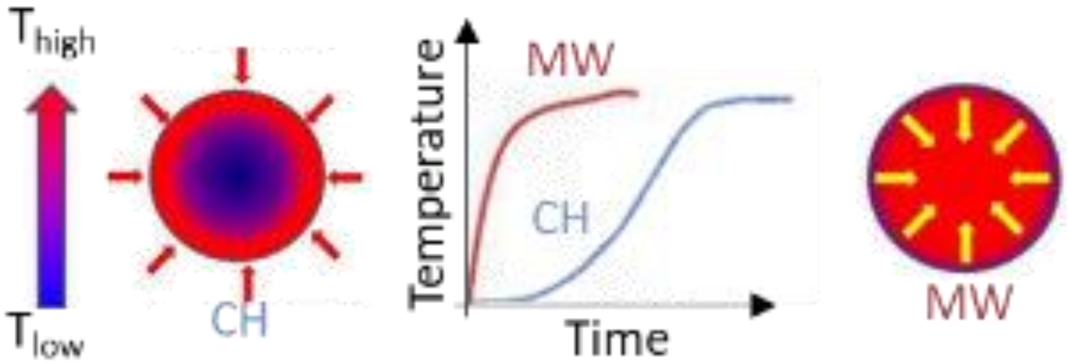
Chemical recycling assisted by microwaves

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

Microwave heating for plastics recycling

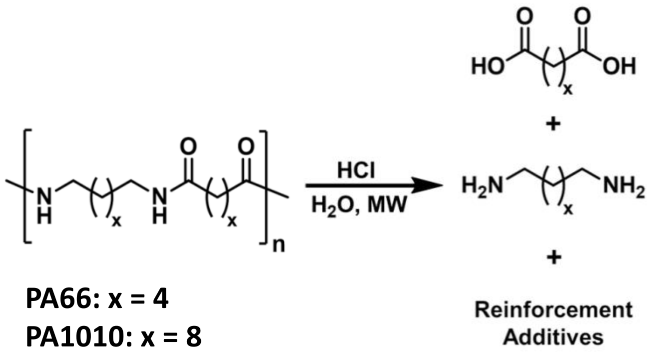
- *Microwaves are electromagnetic radiation with wavelength ranging from 1 mm to 1 m in free space with a frequency from 300 GHz to 300 MHz*
- *In MW heating, energy is delivered directly to materials through molecular interactions with electromagnetic field.*
- *Less temperature gradient compared to conventional heating.*
- *Selective and volumetric heating. Improvement in process control.*
- *High energy efficiency.*
- *Rapid operating times.*
- *Electrification: operation driven by renewables. CO_2 footprint reduction.*
- *Microwave heating (MW) allows efficient recycling of different kind of plastics (PA, PU) to recover valuable secondary products (monomers, polyols) for synthesis of new products with comparable or the same characteristic.*

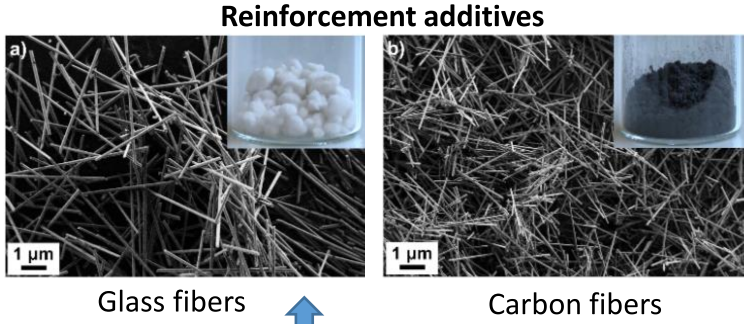


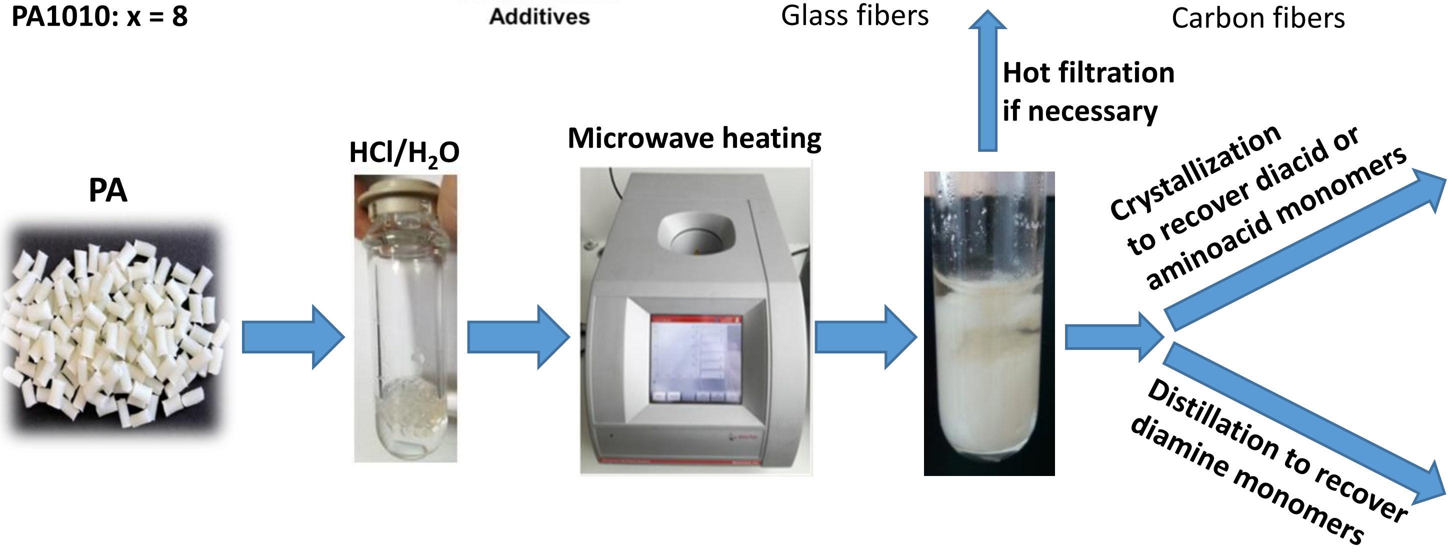
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

MW-assisted polyamide depolymerization to recover the recycled diacid and diamine monomers (PA66, PA1010) or amino acid monomers (PA11, PA12)







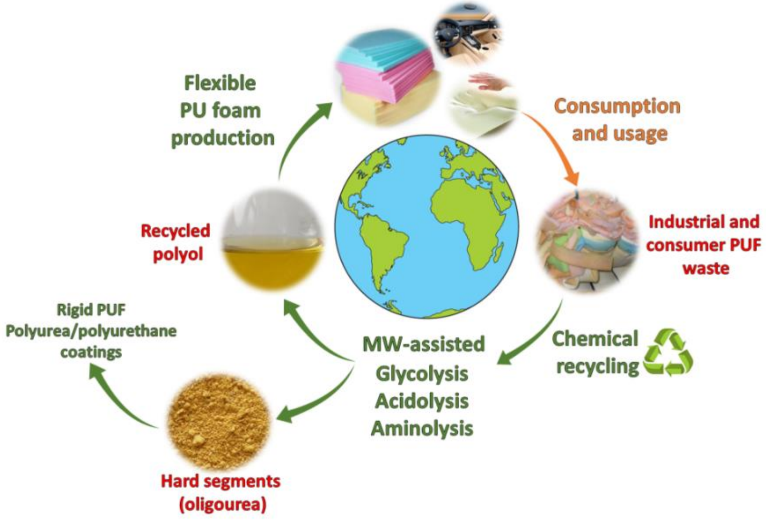
Sample	n(HCl)/n(amide)	MW conditions T (°C)	MW conditions t (min)	Recovered monomer/ Reinforcement additive	Yield (%)	Purity* (%)
PA66	1.25	200	10	AA HMDA	90 86	100.6 ± 0.2 100.1 ± 0.3
PA66-GF$_{35}$	1.25	200	15	AA HMDA GF	83 81 97	100.7 ± 0.2 100.1 ± 0.4 /
PA11	2.5	200	10	11-AUDAxHCl	93	100.1 ± 0.2
PA11-GF$_{30}$	2.5	200	15	11-AUDAxHCl GF	71 97	99.8 ± 0.1 /
PA11-CF$_{30}$	2.5	200	20	11-AUDAxHCl CF	72 99	99.3 ± 0.2 /
PA12	2.5	200	10	12-ADDAxHCl	97	99.9 ± 0.5
PA12-GF$_{50}$	5	200	15	12-ADDAxHCl GF	77 97	97.1 ± 0.3 /
PA1010	2.5	200	17	SA 1,10-DDA	89 78	100.4 ± 0.8 100.0 ± 0.2

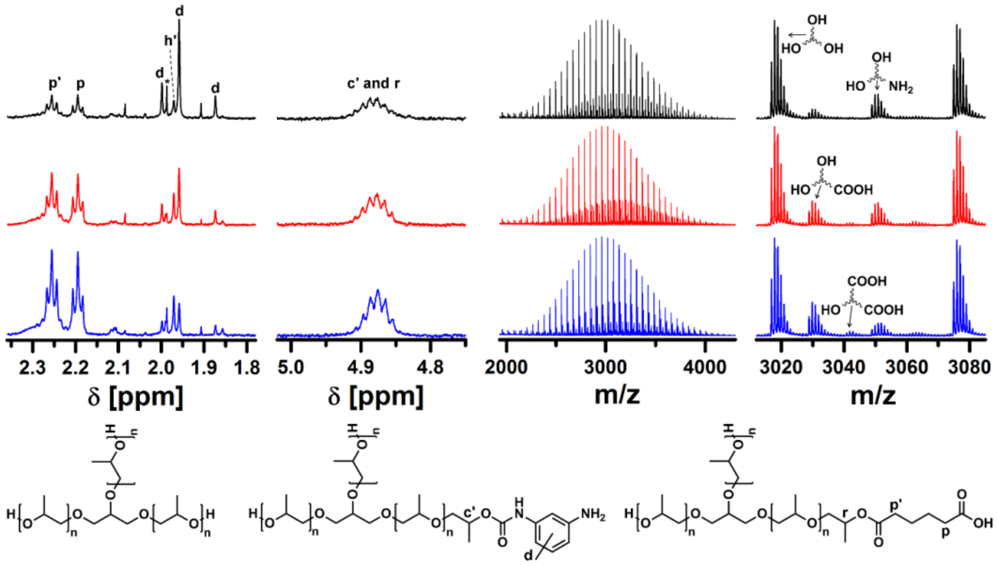
U. Češarek, D. Pahovnik, E. Žagar *ACS Sustain. Chem. & Eng.* **2020**, *8*, 16274

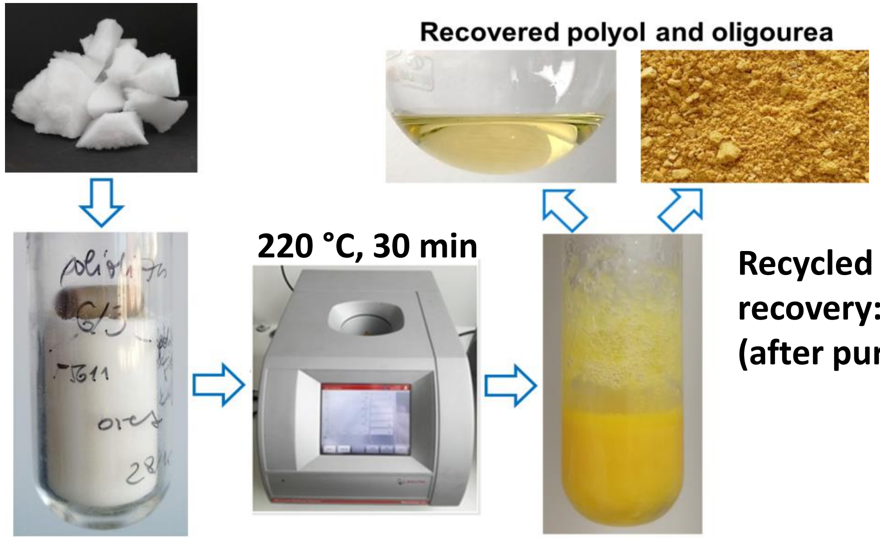
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

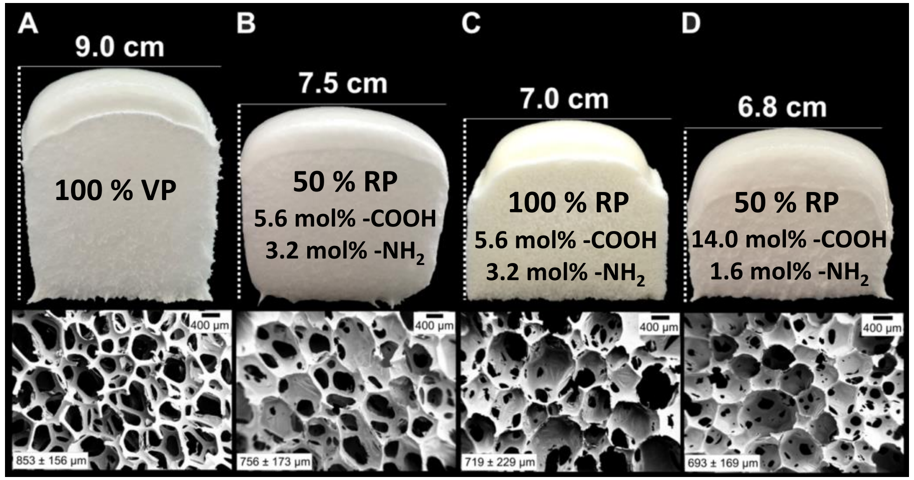
Chemical recycling of flexible polyurethane foams (PUFs) by acidolysis







Recycled polyol recovery: over 85% (after purification)

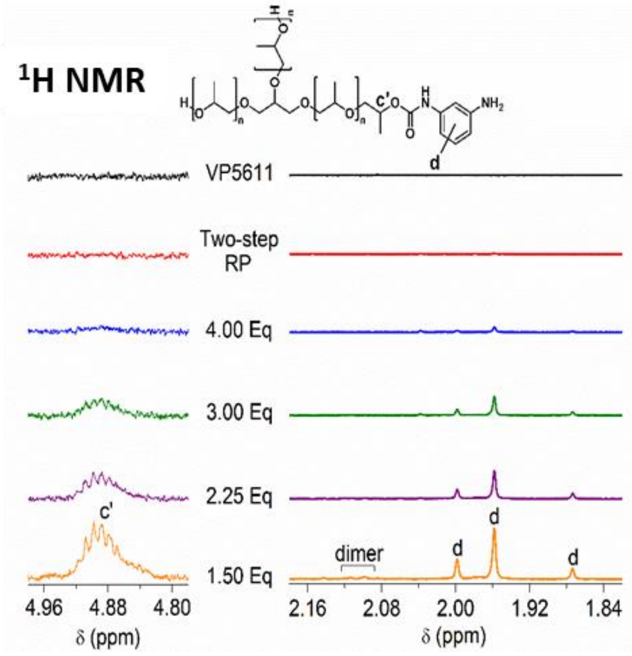


M. Grdadolnik, A. Drinčić, A. Oreški, O.C. Onder, P. Utroša, D. Pahovnik, E. Žagar
ACS Sustain. Chem. & Eng. **2022**, *10*, 1323

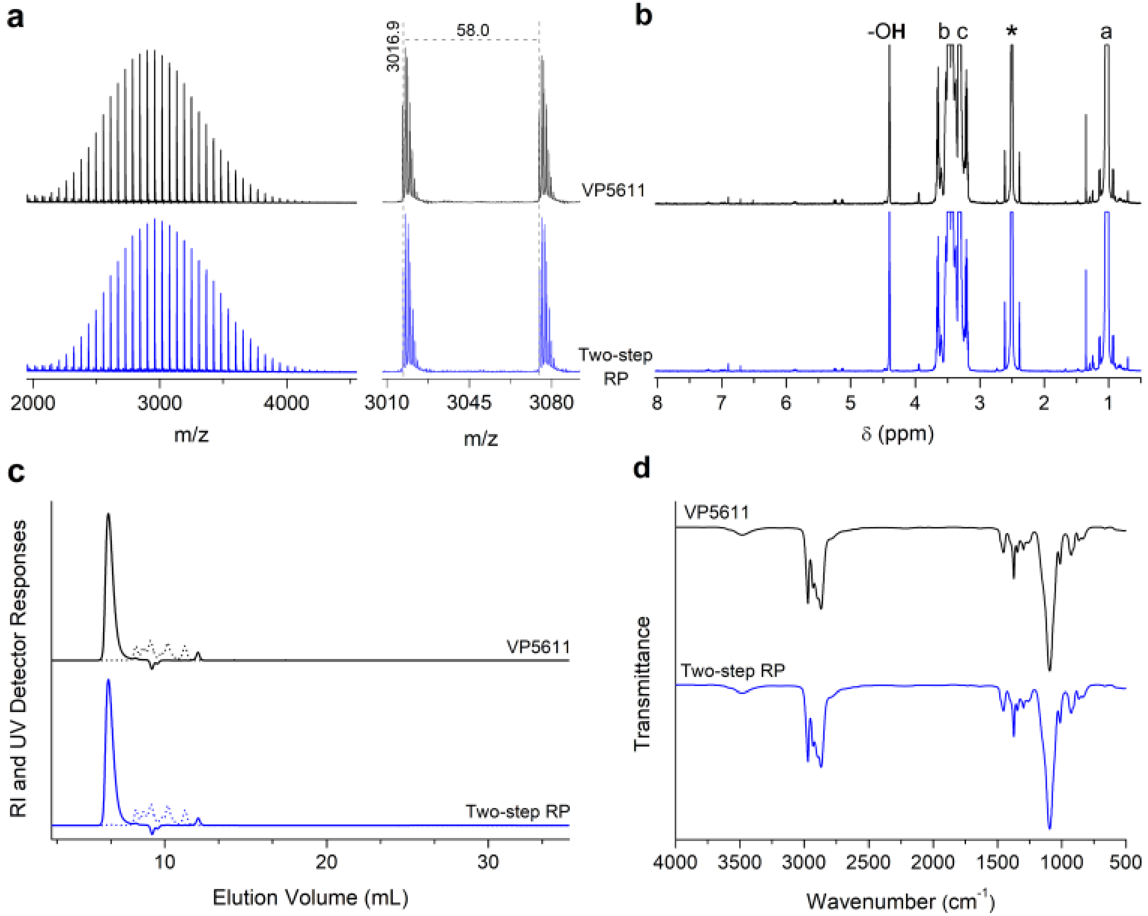
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

Chemical recycling of flexible polyurethane foams (PUFs) by aminolysis



Recycled polyol recovery: over 85% (after purification)



0 20 50 100

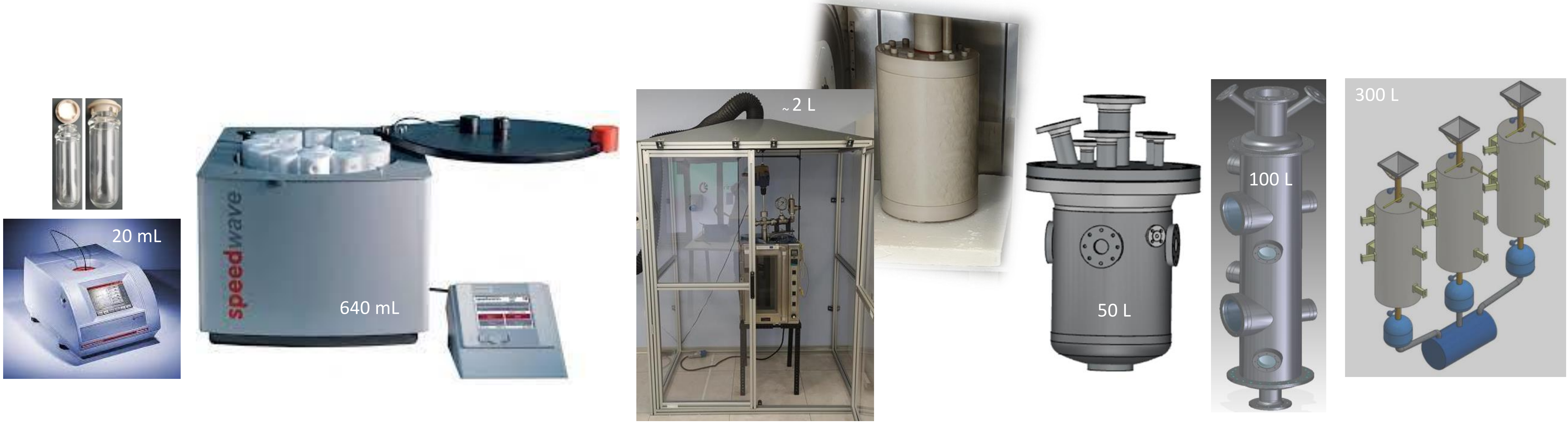
Content of recycled polyol in PUF formulation in wt%

Patent application No.: LU501979

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project

Upscaling of polyamides (PA) and polyurethanes (PU) MW-assisted depolymerization processes.



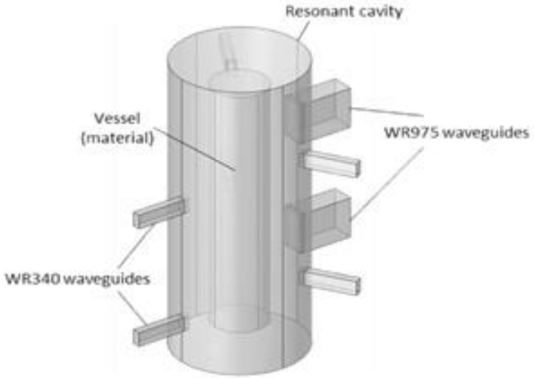
Processing capacity

The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

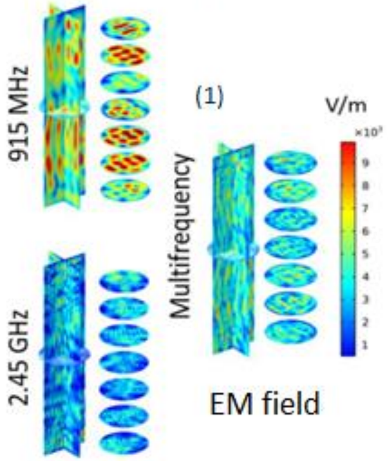
Main results of the project

MW-depolymerisation scale-up pilot: materials and configutations

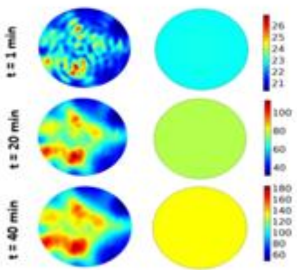
PEEK stirred tank
(T < 300ºC, P < 20 bar)



Initial multifrequency configuration

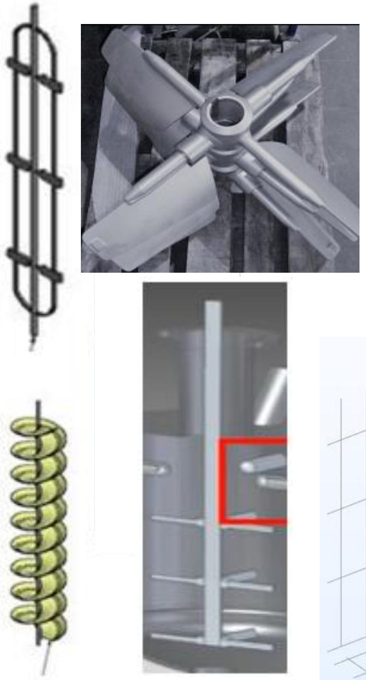


EM field

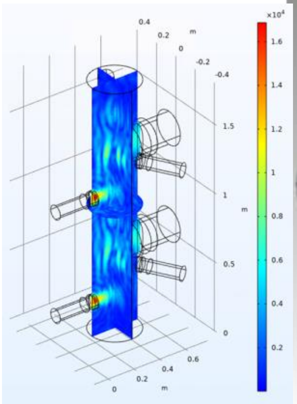


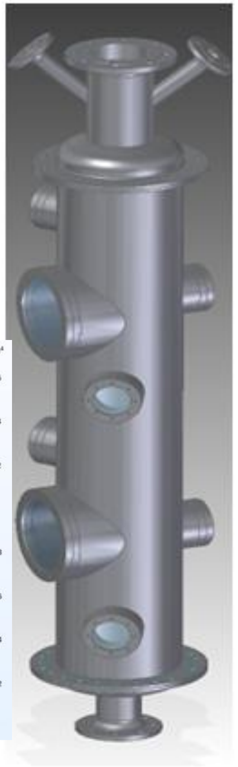
Temperature distribution without/with stirring
(1 m away from reactor bottom)

(1) A. Frisa et al. Cleaner Eng. Technol. 5 (2021) 100297



Mechanical stirrer configurations (axial)





100 L multi-frequency oven (waveguide)

Numbering-up strategies
(basic engineering design)

Teflon-lined SS
(rectangular waveguide)

50 L inconel tank (antenna)

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

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Innovative technologies for plastic recycling

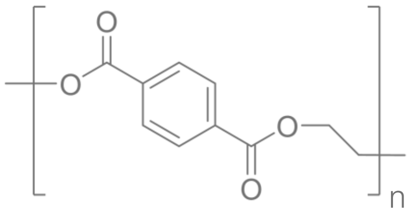
Chemical recycling assisted by Smart Magnetic Materials
WP4

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts: IONIQA's technology

IONIQA **developed and scaled-up** recycling technology for **PET**, both for **packaging** and **fiber** post-consumer feedstock

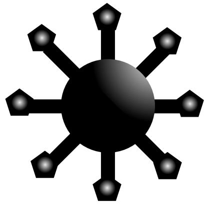
PET



Glycolysis

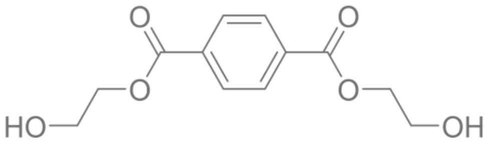
+ Ethylene Glycol (EG)
and

Smart Magnetic Fluid
(SMM)



BHET

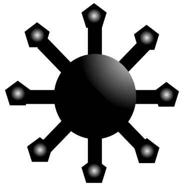
bis (2-hydroxyethyl) terephthalate



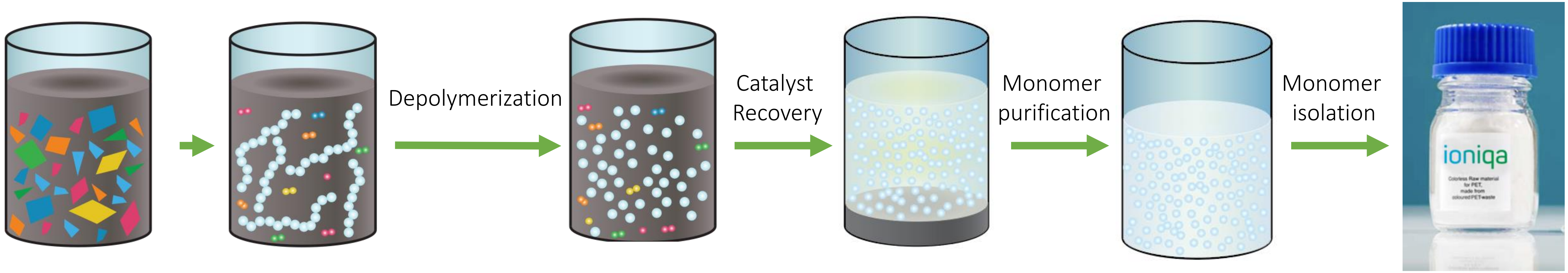
The project has received funding from the European Union's Horizon 2020
research and innovation programme under grant agreement no 820665

Fundamental concepts: IONIQA's technology

Smart Magnetic Fluid → Functionalized magnetic nanoparticles



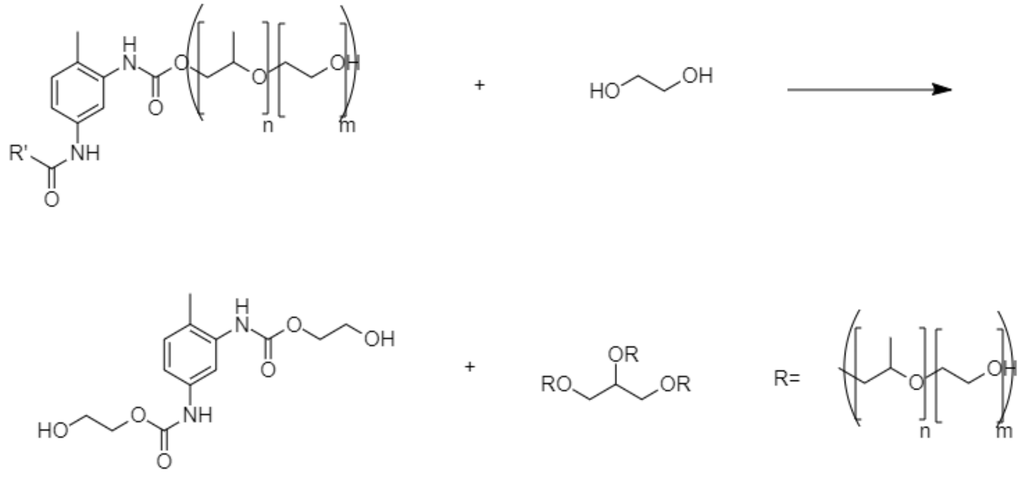
- Better yield and selectivity than benchmark catalysts
- Easily recoverable from the reaction mixture and can be reused



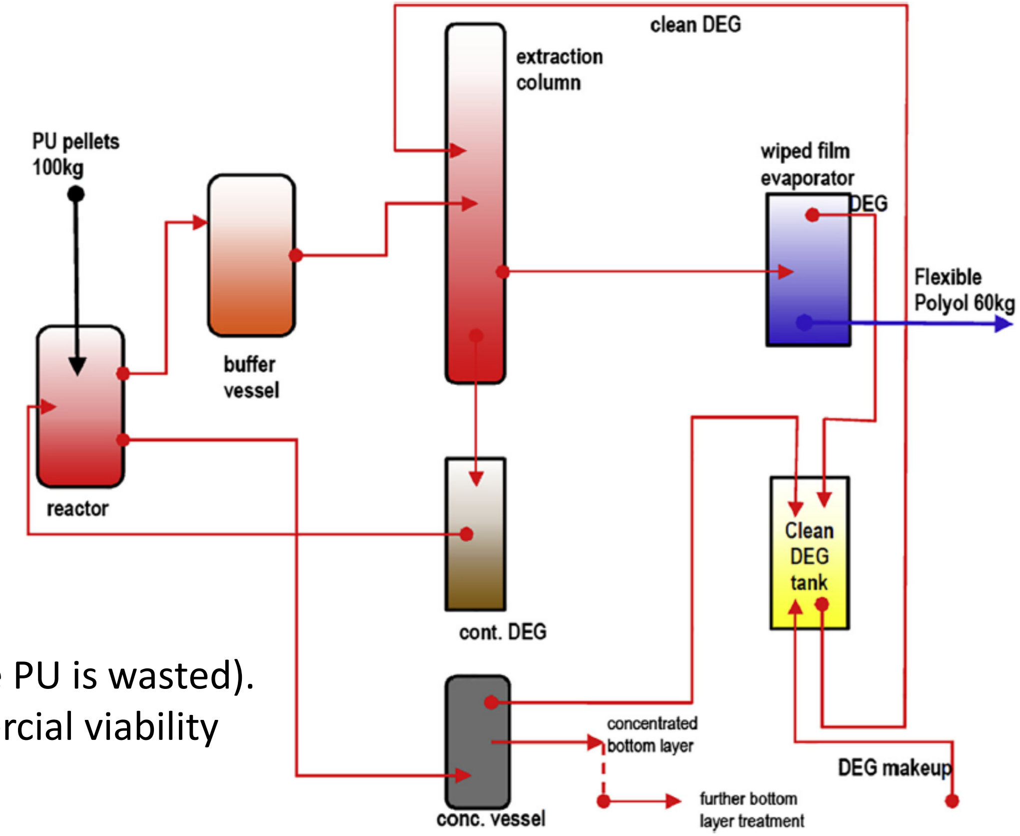
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project: PU recycling

- Process selection based on literature: split-phase glycolysis → minimal by-products (polyol and carbamate), easier product separation



ICI-Huntsman process (according to literature)



Main obstacles include:

- product purification,
- glycol and catalyst recycling,
- product yield and
- production of large amounts of waste (30-40 wt% of the PU is wasted).

If these obstacles can be overcome, the chances for commercial viability are considered to be positive.

D. Simón, A. M. Borreguero, A. de Lucas, and J. F. Rodríguez, "Recycling of polyurethanes from laboratory to industry, a journey towards the sustainability," *Waste Manag.*, vol. 76, pp. 147–171, 2018.

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project: PU recycling by glycolysis

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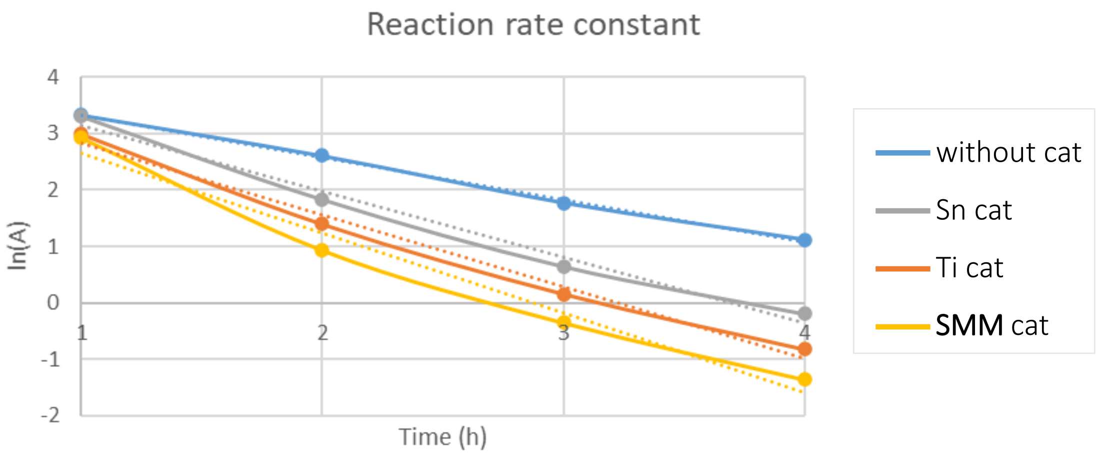
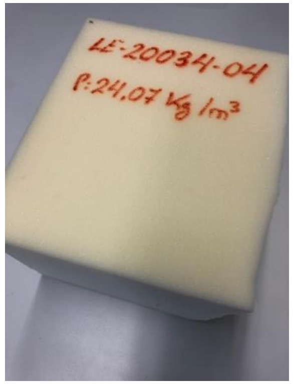
SMM catalyst performs **better** than state-of-the-art catalysts

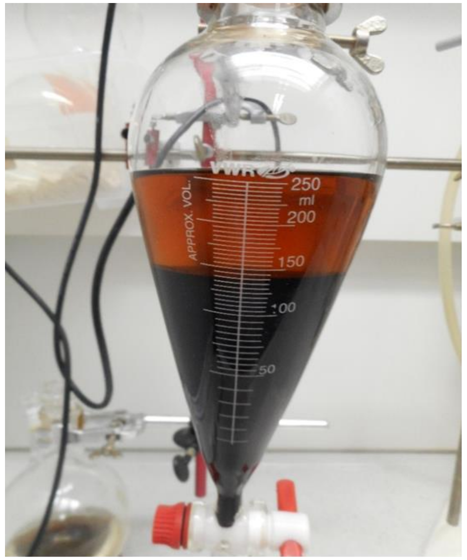


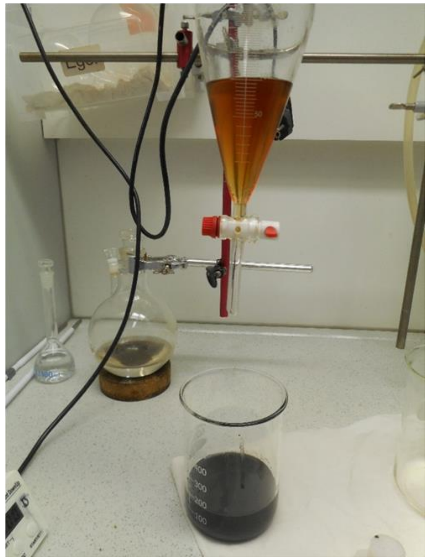
- Higher reaction rate → **faster** reaction!
- Reduced the amount of residual oligomer (and thus, aromatic compounds) in the final polyol product → **better quality** product!
- Heterogeneous → **easier** to **recover** and reuse!



Split-phase glycolysis:

- Glycol solvent and SMM catalyst (bottom layer) is thereby **easily separated** from polyol (top layer)





The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project: PU recycling by glycolysis

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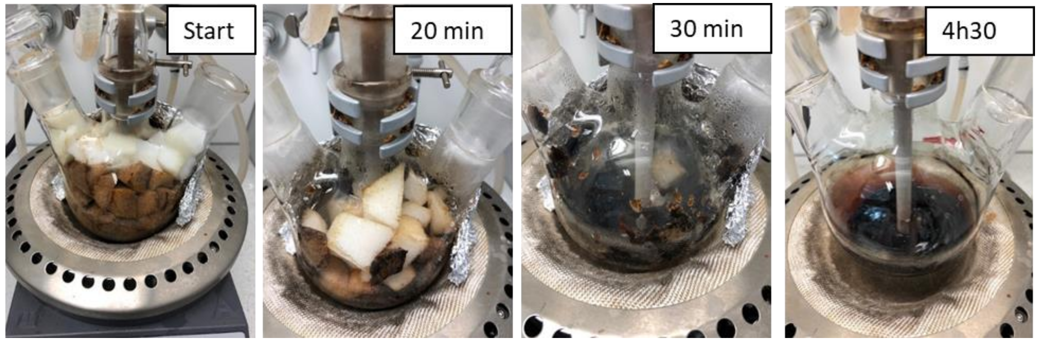
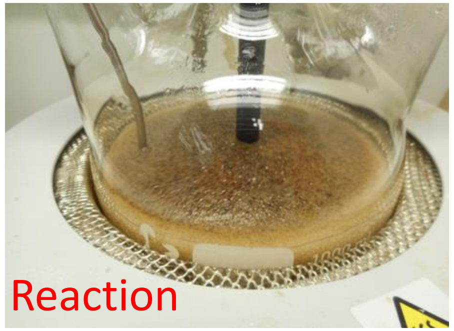
Process with **state-of-the-art catalyst**

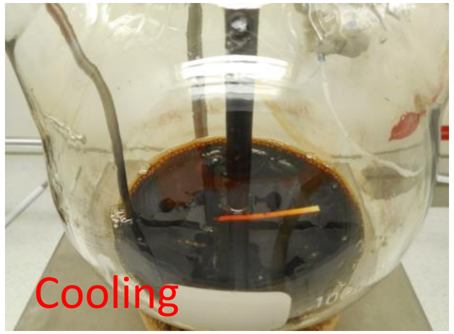
- very poor performance of the initial PU melting
- poor conversion rate
- severe PU degradation resulting in discolouration and side reactions
- catalyst and polyol product cannot be isolated or purified

Process with **SMM catalyst**

Clean reaction mixture, product easy to separate







The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project: PU recycling by glycolysis

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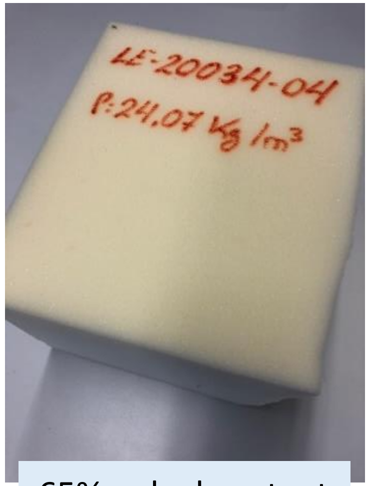
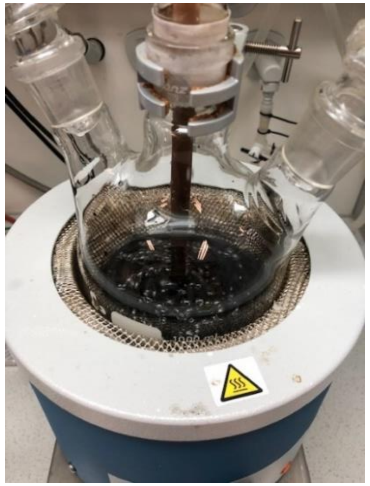
Industrial **PU foam** waste (scrap) from REPSOL



65% polyol content

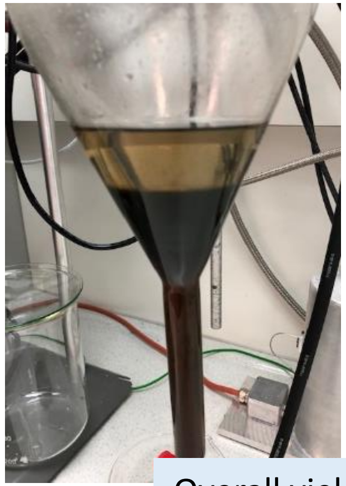
PU glycolysis with SMM

Step-wise addition of PU foam

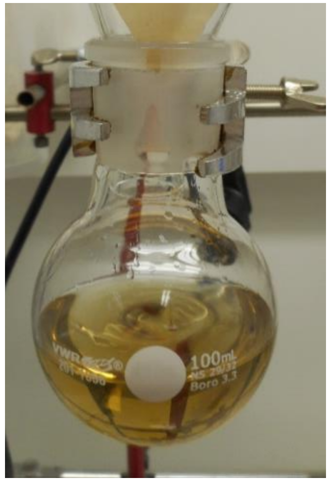


Reaction and cooling

Product **isolation** and **purification** via liquid-liquid extraction



Overall yield: 47.1%
Effective polyol yield: 72.5%



Polyol product after filtration and drying

Next step: **Vacuum distillation** to remove solvent residues

- **Lab-scale** batch: ~15-20 gr of polyol monomer is obtained
- Recycled polyol characterized according to industry ISO/ASTM procedures: recycled monomer close to industry standard – except colour and Si standard
- Si originates from PU foam formulation and needs to be removed to satisfy specs
- Next step: demonstrate technology at **pilot plant scale** (1000L reactor)

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Partners involved

polynSPIRE

- WP leader

:fm ioniqa

- Co-leadership (task leaders apart from WPL)

circe RESEARCH CENTRE FOR ENERGY RESOURCES AND CONSUMPTION

CPPE CHEMICAL & PHARMACEUTICAL PROCESS ENGINEERING

- Other partners involved

NUREL KORDSA REPSOL NOVAMONT ARKEMA INNOVATIVE CHEMISTRY

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Thank you for your attention

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STANDARD PRESENTATION OF MECHANICAL RECYCLING

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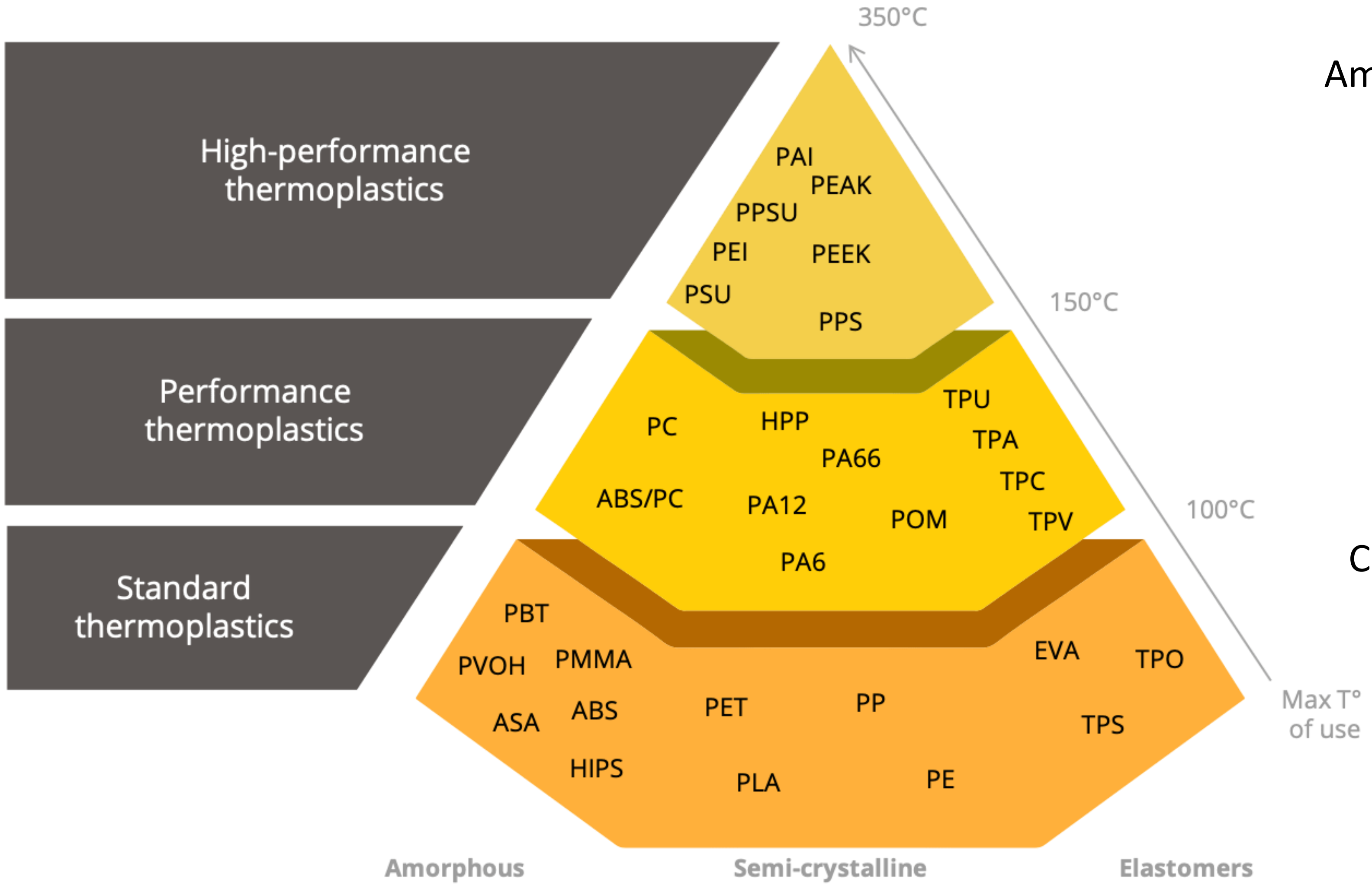
Innovative technologies for plastic recycling

Mechanical recycling.
WP5

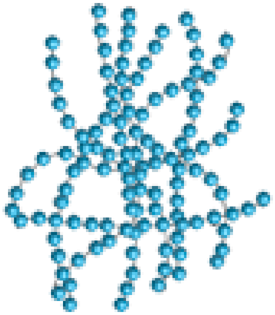
The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

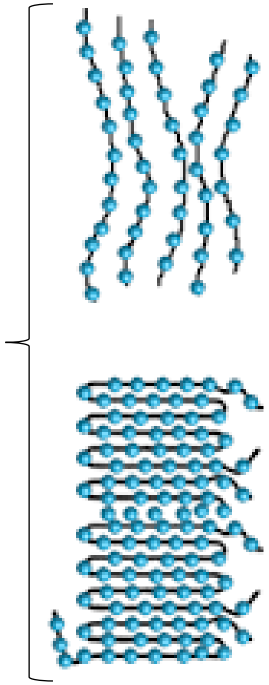
Thermoplastic materials



Amorphous phase



Cristaline phase



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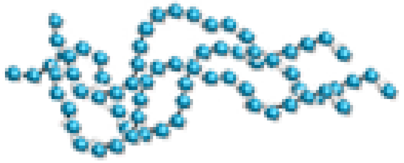
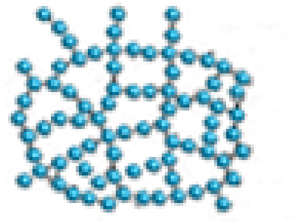
Fundamental concepts

Plastic materials





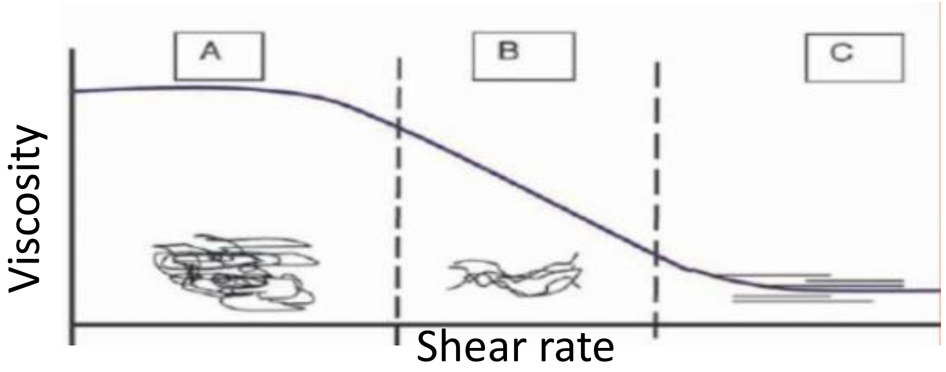
Thermoplastic materials:

- Weak conections among the different polymeric chains
- Can be melted without degrading

Thermoset materials:

- High crosslinking between the diffrent polymeric chains
- It can not be melted without degrading

Thermal treatments breaks the bonds between the different polymeric chains or even within the chain itself, modifying the properties of the material itself. As the bonds and interactions among polymeric chains are reduced, it is easier for the material to flow.



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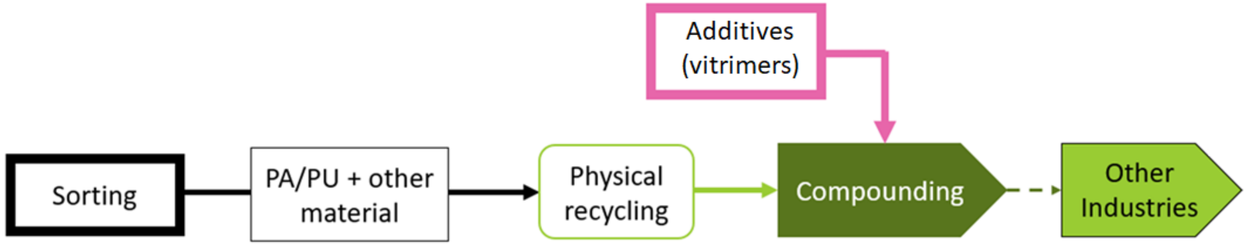
Mechanical recycling

- The following technologies will be used to improve recycling efficiency:

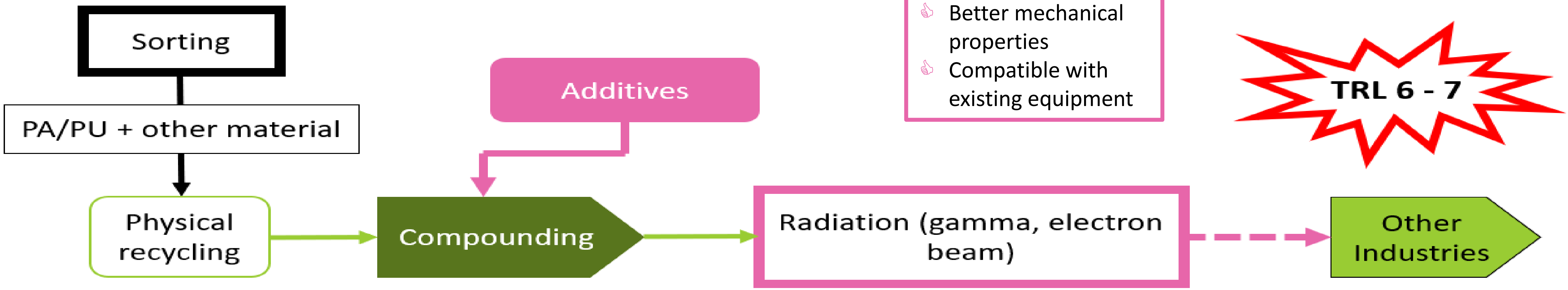
Vitrimers



- Better mechanical properties
- Reversible – not affecting future recycling

High Energy Radiation

- Better mechanical properties
- Compatible with existing equipment



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Innovative technologies for plastic recycling

Development and optimization of vitrimer formulations for enhancing recycled materials

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

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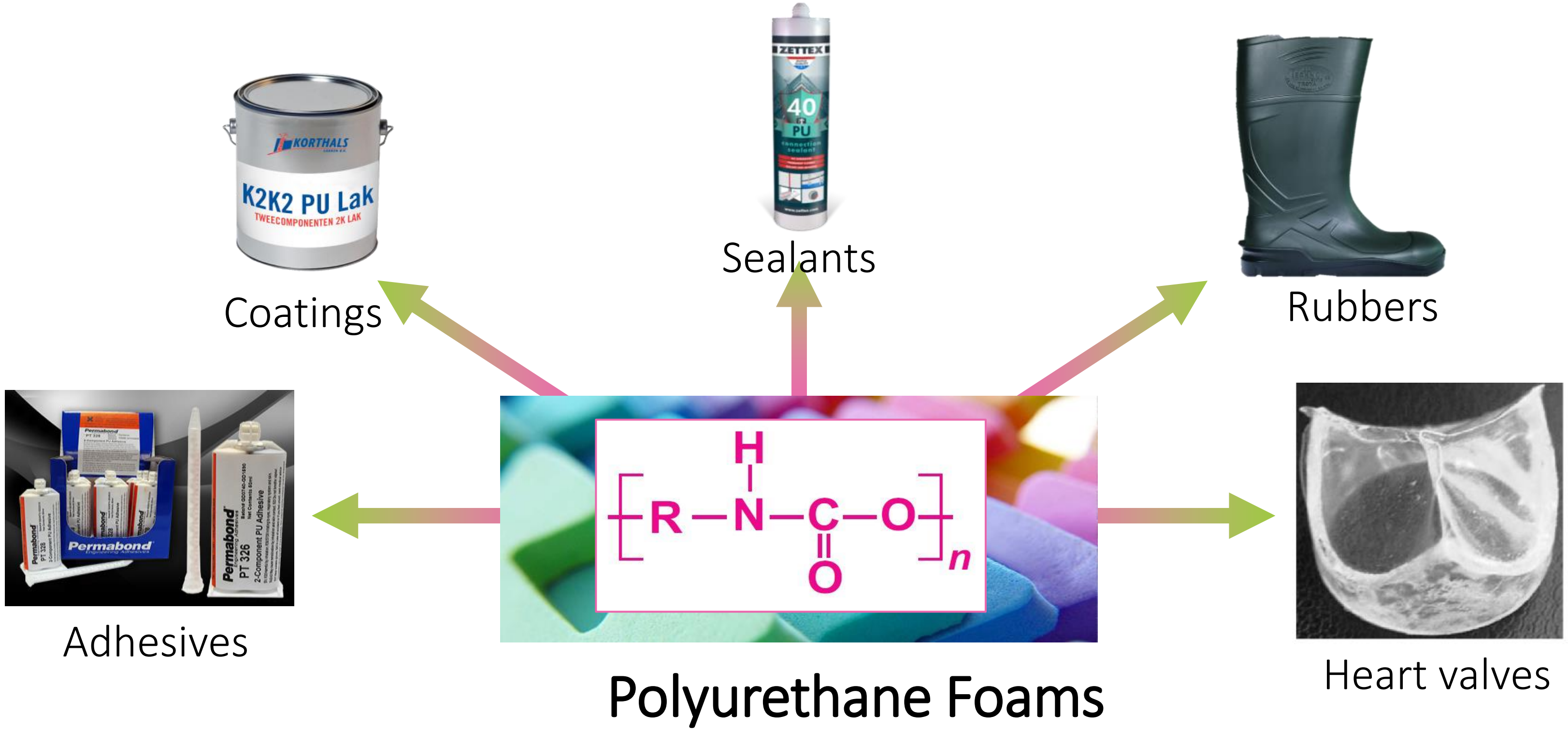
Polyurethanes

- Currently post consumer PU waste is either incinerated (pyrolysis) or ends up on landfill sites, both leading to an increase in waste and greenhouse gas emissions. By carrying out high-fidelity laboratory scale experiments by TU/e on post industrial PU foams, an attempt is made to develop and optimize additive formulations, converting these thermosets into dynamic covalent networks.



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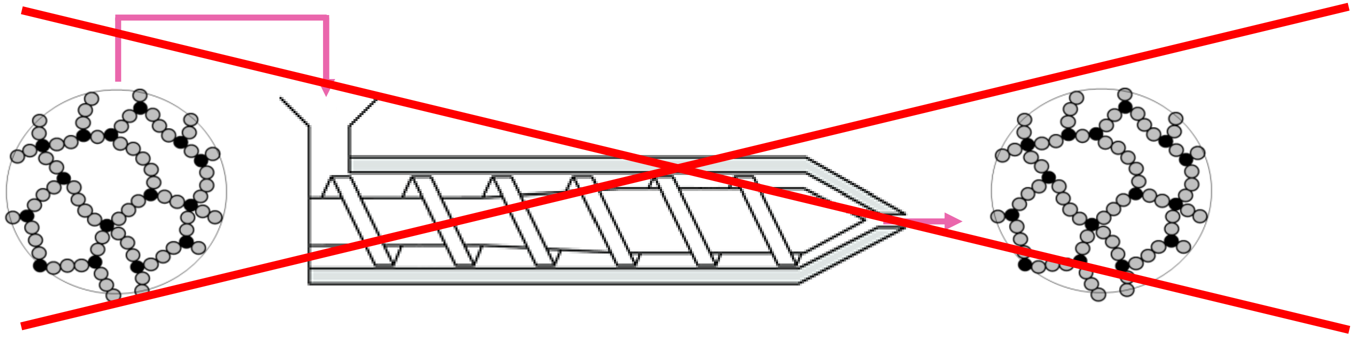
Fundamental concepts

Recycling of polyurethanes

[1]



PU has a 6% global market share [2]
- ≈75 vol% is thermoset PU foam
- Wear and replacement causes a constant stream of waste
- Incineration plants or landfill

Conventional recycling methods
- Melt reprocessing does not work

What does happen?
- Mechanical grinding → use as a filler material
- Chemical recycling → Glycolysis

Need specialised equipment

[1] Ragaert, K.; Delva, L.; van Geem, K. Mechanical and Chemical Recycling of Solid Plastic Waste. Waste Management. Elsevier Ltd November 1, 2017, pp 24–58. https://doi.org/10.1016/j.wasman.2017.07.044.
[2] Kemona, A.; Piotrowska, M. Polyurethane Recycling and Disposal: Methods and Prospects. *Polymers (Basel).* **2020**, *12* (8). https://doi.org/10.3390/POLYM12081752.

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Fundamental concepts

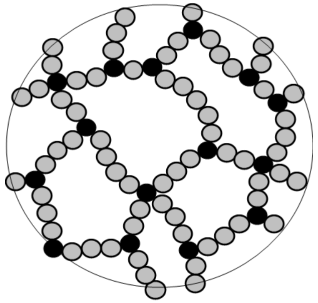
Dynamic Covalent Network (DCN)

Bridging the gap between mechanical and chemical recycling

Mechanical recycling

Chemical recycling

Immobile network

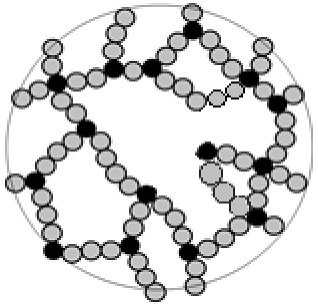


Dynamic Covalent Network

← low temperature

high temperature →

Mobile network



Reactive Extrusion

Using industrial equipment that is already there

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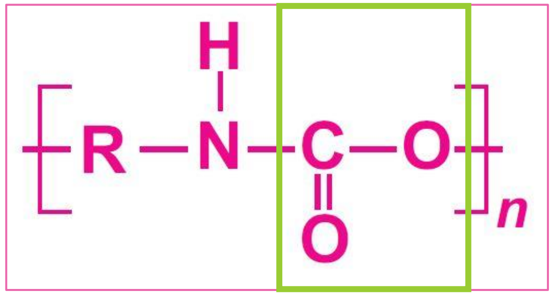
Fundamental concepts

DCN chemistry



Carbamate group

Exchange of the backbone

Transcarbamoylation

By using the reactivity that is already there, by employing the urethane group, effective exchange of the backbone can occur. This allows for mobility of the network

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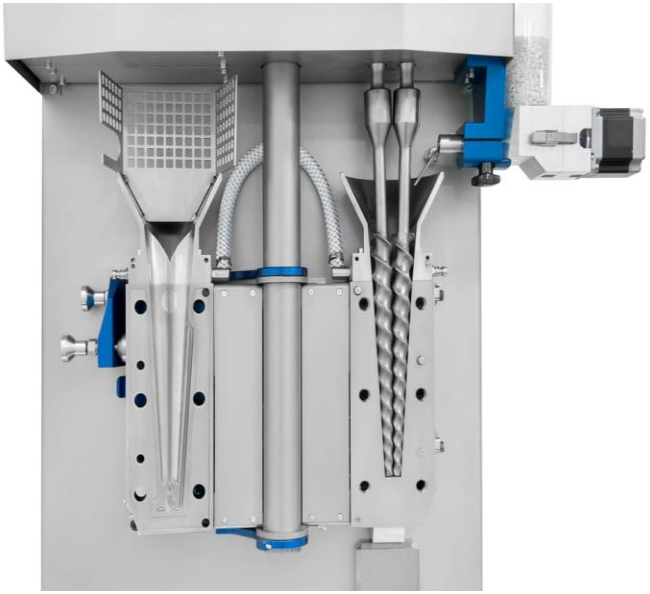
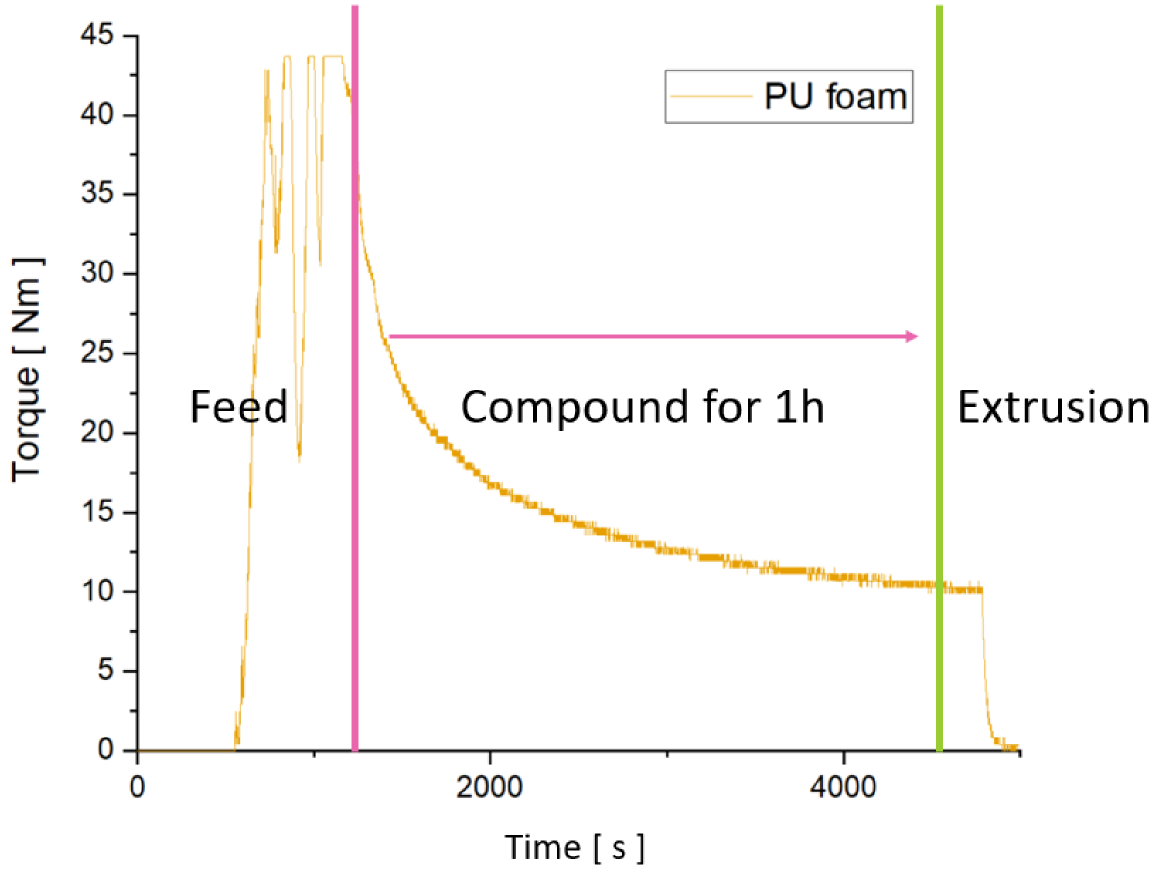
Main results of the project

Reactive Extrusion

Extruder as reactor vessel
- Micro Compounder was used for tests
- Gathering torque data during reaction
- 15 mL volume



Example of torque data



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Main results of the project

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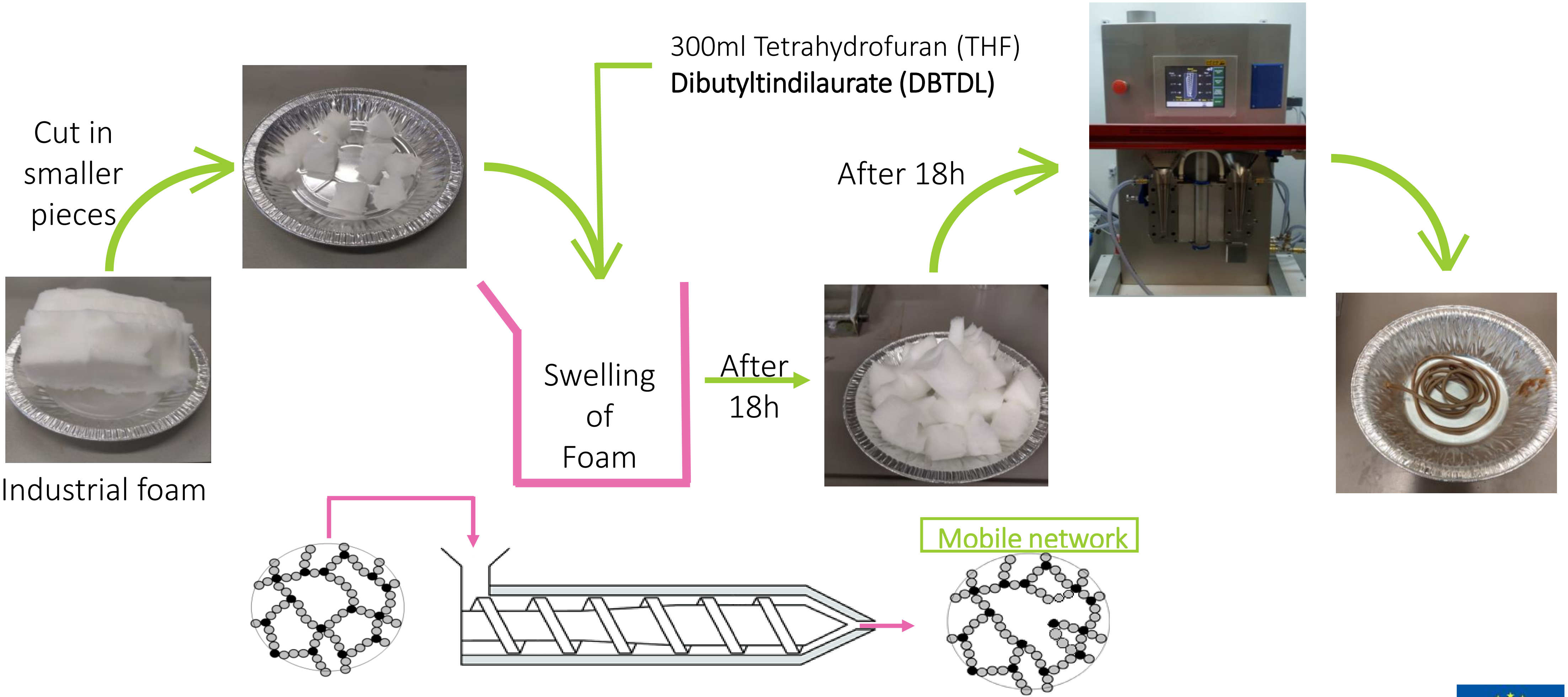
Pre treatment of polyurethane foam (for 15g)



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Main results of the project

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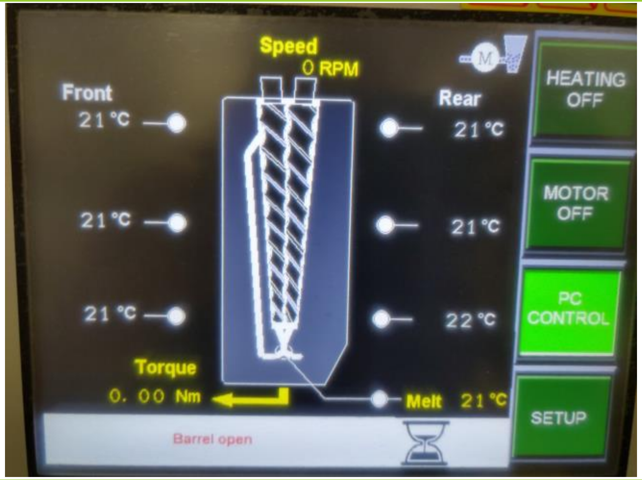
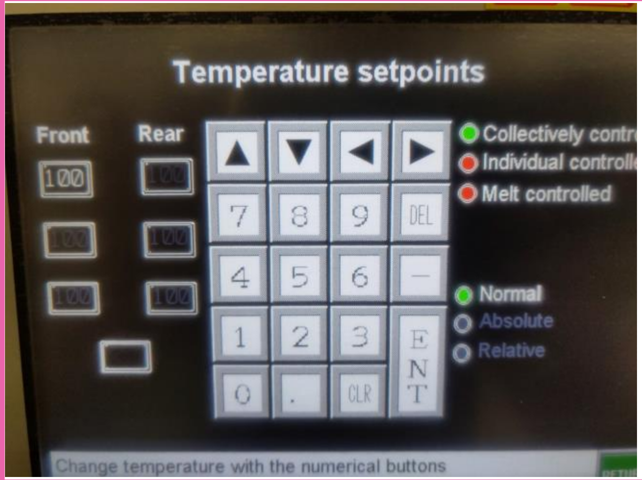
MC-15-HT overview*



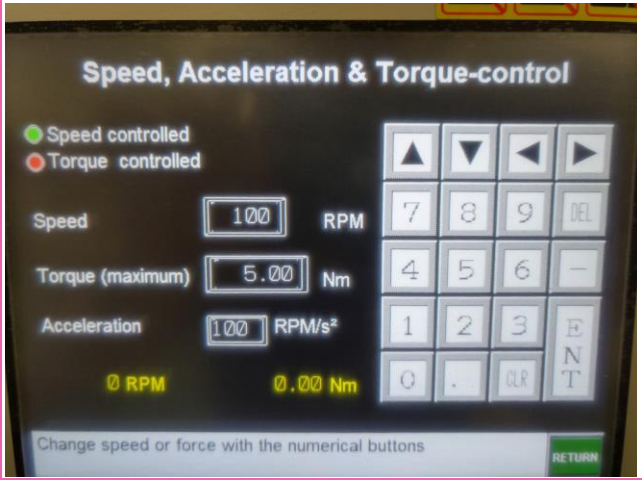
Overview screen

Heating Off – On	Switches the heating on and off
Motor Off – On	Turns the screws on or off. (Only works when barrel is closed)
Pc control	When enabled, settings can be changed via PC software
Setup	Allows access to calibration and software updates

Settings used during experiments



Temperature must be perfectly controlled as it is the main parameter for the reaction



Speed & Torque settings:
As crosslinked polymers are added to the microcompounder, the force required to process them are high.

Set to 50 RPM with a maximum torque of 40 Nm

*Always refer to the manual for complete instructions and safety procedures

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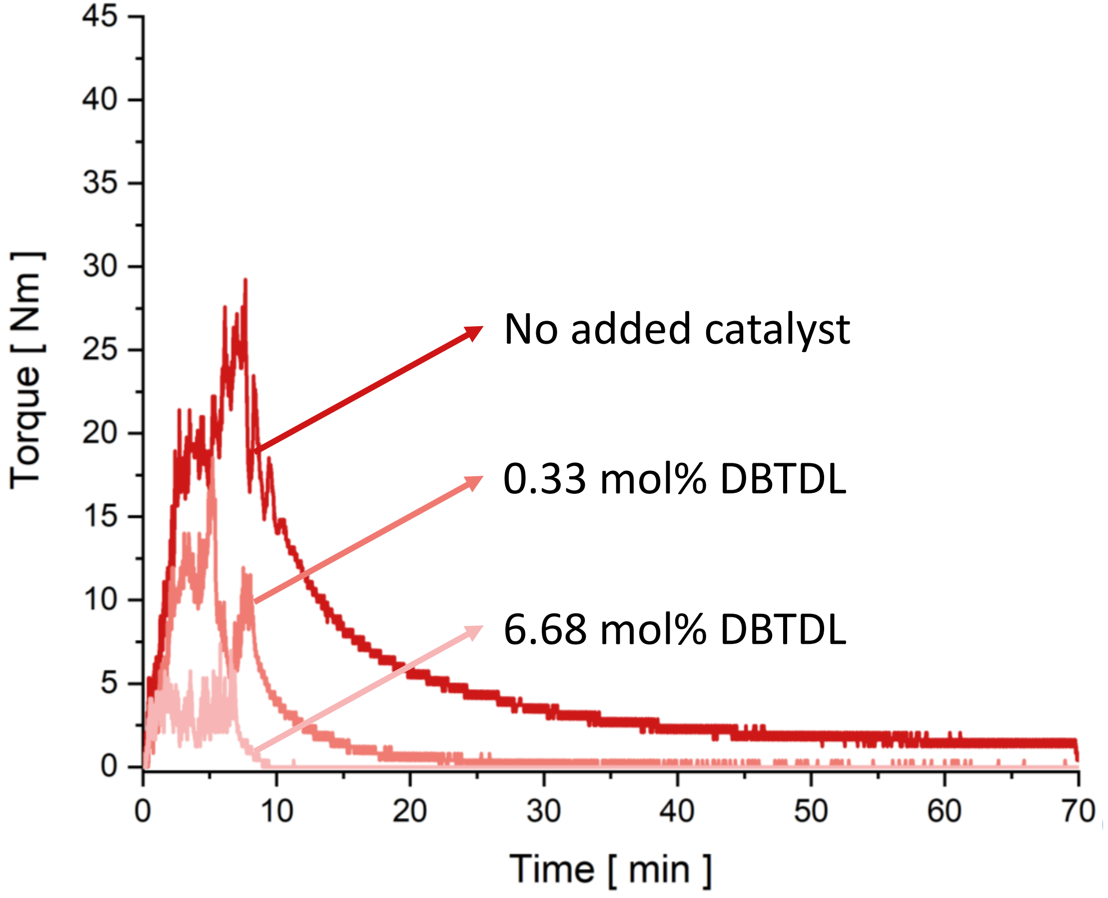
Main results of the project

Extrusion temperature: **180 °C**

Compounding time: 1h



Low residual torque without added catalyst at 200 °C suggests the presence of:

- Uncatalyzed bond exchange
- Reactions due to catalyst present from formation
- Loss of integrity due to thermal degradation

At 180 °C

- Increase in overall torque compared to 200 °C
- For both 0.33 mol% and 6.68 mol% DBTDL Torque signal very low
- Effect of temperature still high

At 160 °C

- Higher, visible, torque values for al catalyst concentrations
- Effect of catalyst is more pronounced

23-2-2023

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Main results of the project

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Comparison and overview of result interpretation

[CAT]

Temperature

	No additional catalyst	0.33 mol% DBTDL	6.68 mol% DBTDL
200°C	 Torque at extrusion 1.2 Nm Gel content 87%	 Torque at extrusion 0 Nm Gel content 87%	 Torque at extrusion 0 Nm Gel content 54%
180°C	 Torque at extrusion 2.9 Nm Gel content 87%	 Torque at extrusion 0.4 Nm Gel content 75%	 Torque at extrusion 0 Nm Gel content 75%
160°C	 Torque at extrusion 18.1 Nm Gel content 88%	 Torque at extrusion 9.9 Nm Gel content 87%	 Torque at extrusion 0.8 Nm Gel content 90%

Gel Content

Gel Content

Gel content indicates a network is still present. But a loss of network integrity can be observed at high catalyst concentration and/or high temperatures

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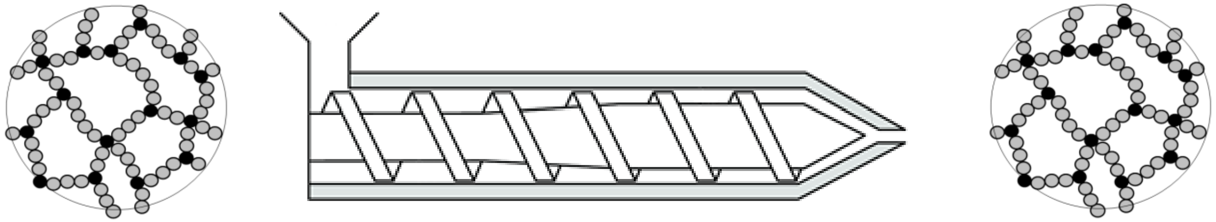
Innovative technologies for plastic recycling

Development and optimization of high irradiation energy as a way for recycling

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Fundamental concepts

Plastic materials



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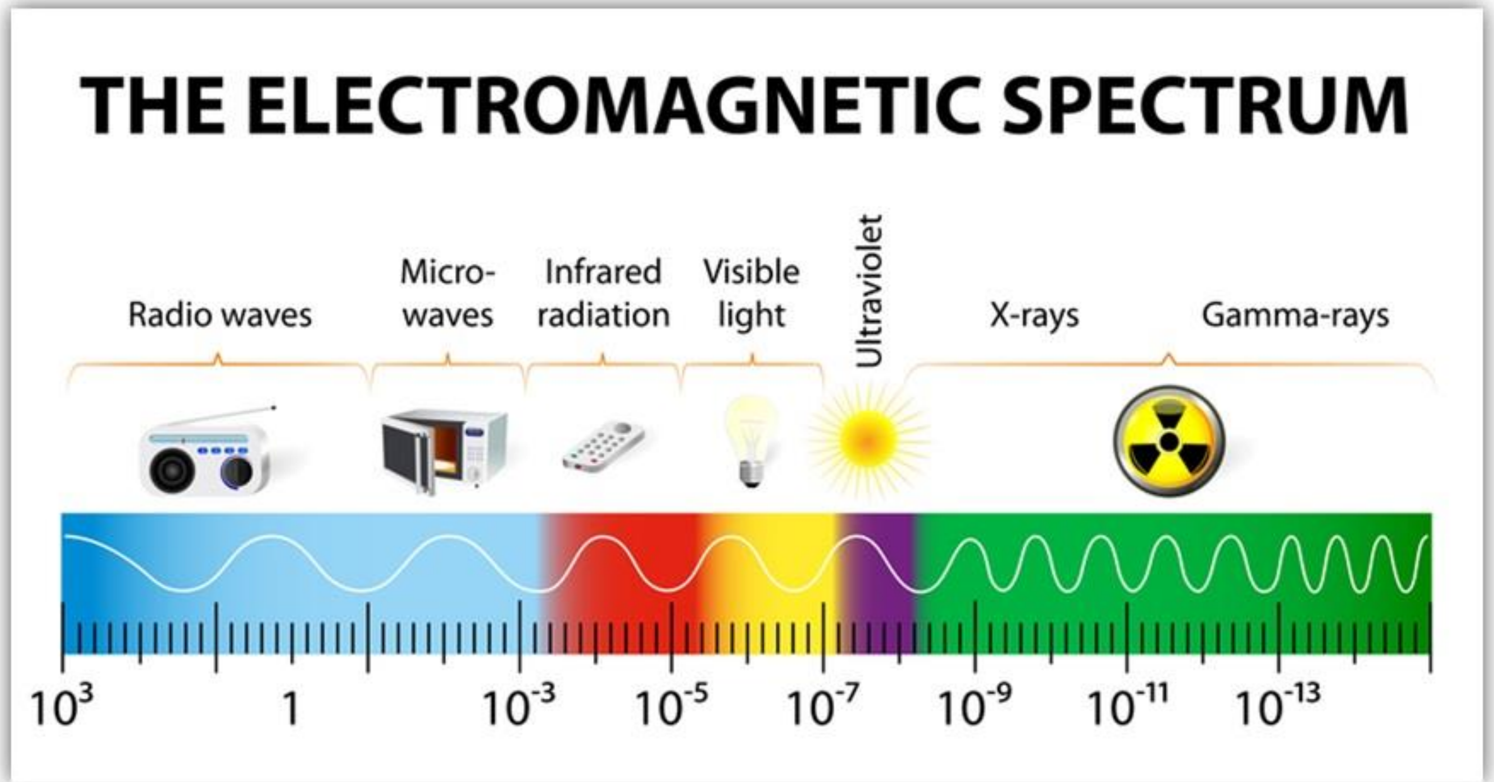
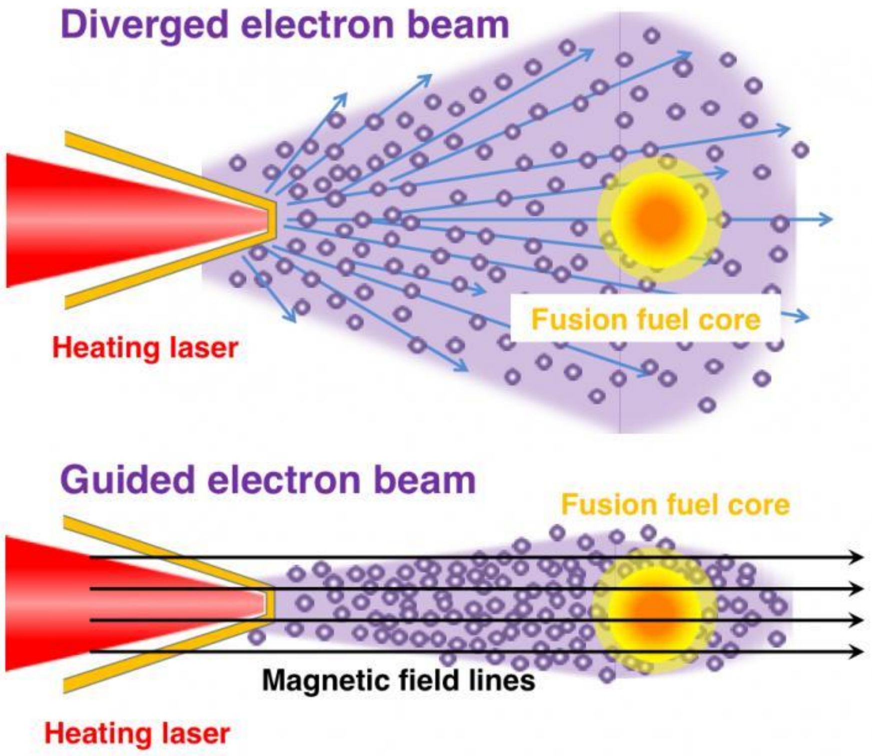
Fundamental concepts

High energy irradiation





Radiations of high energy, those of short wavelenght have the capacity to modify electronically a molecule

Irradiation is measured in Gray (Gy)

$$Gy = \frac{J}{Kg}$$

Guided electron beam has the capacity to charged electronically the molecule, providing the capacity to generate new bonds

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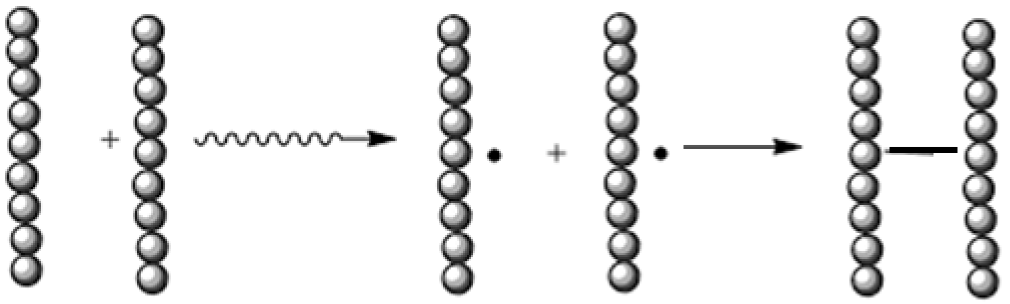
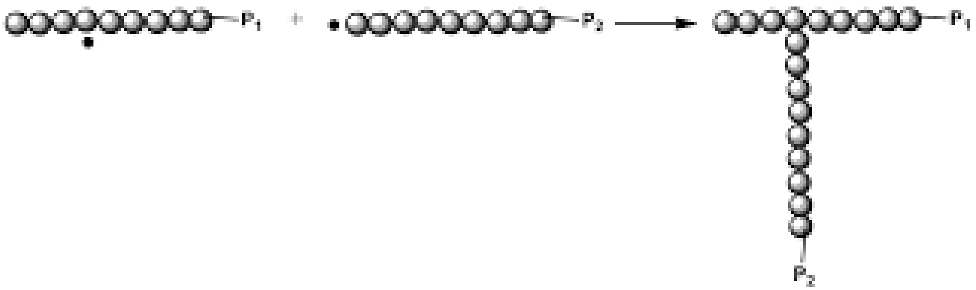
Fundamental concepts

High energy irradiation



The gamma radiation generates free radicals in the polymeric chain.

These free radicals can give differnt scenarios
1- Oxidation of the polymeric chain due to the interaction with the oxygen in the air
2- Generation of a bond between differnt polymeric chains



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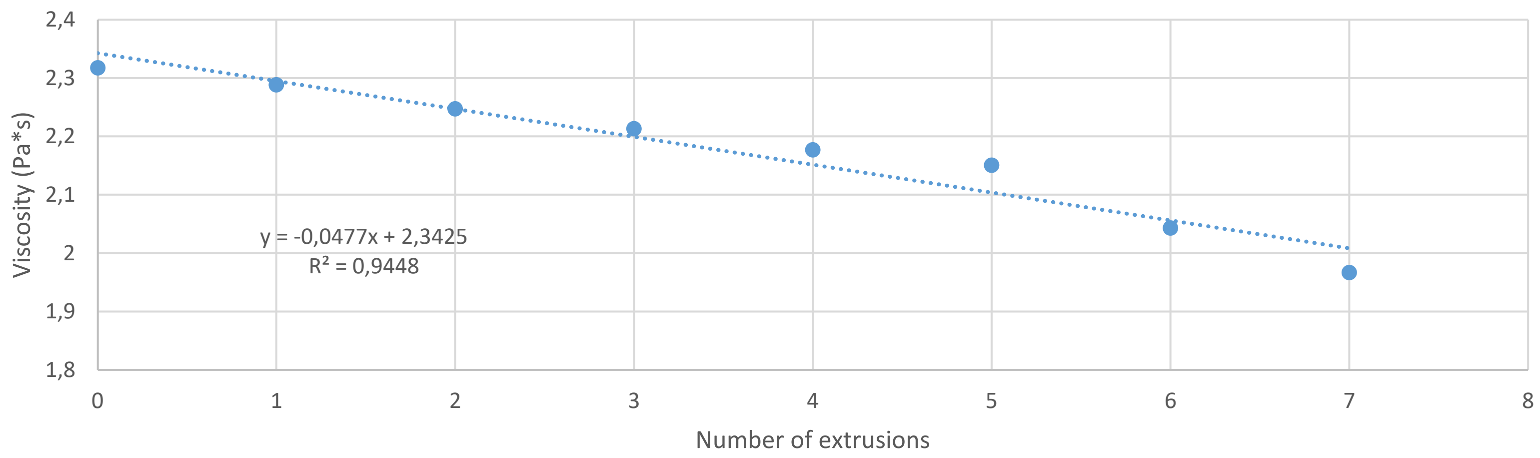
Main results of the project

High energy irradiation

Residue simulated after 7 extrusions



Raw material after irradiation
100 KGy-250 KGy- 500 KGy

Thermally degraded material after irradiation
100 KGy-250 KGy- 500 KGy

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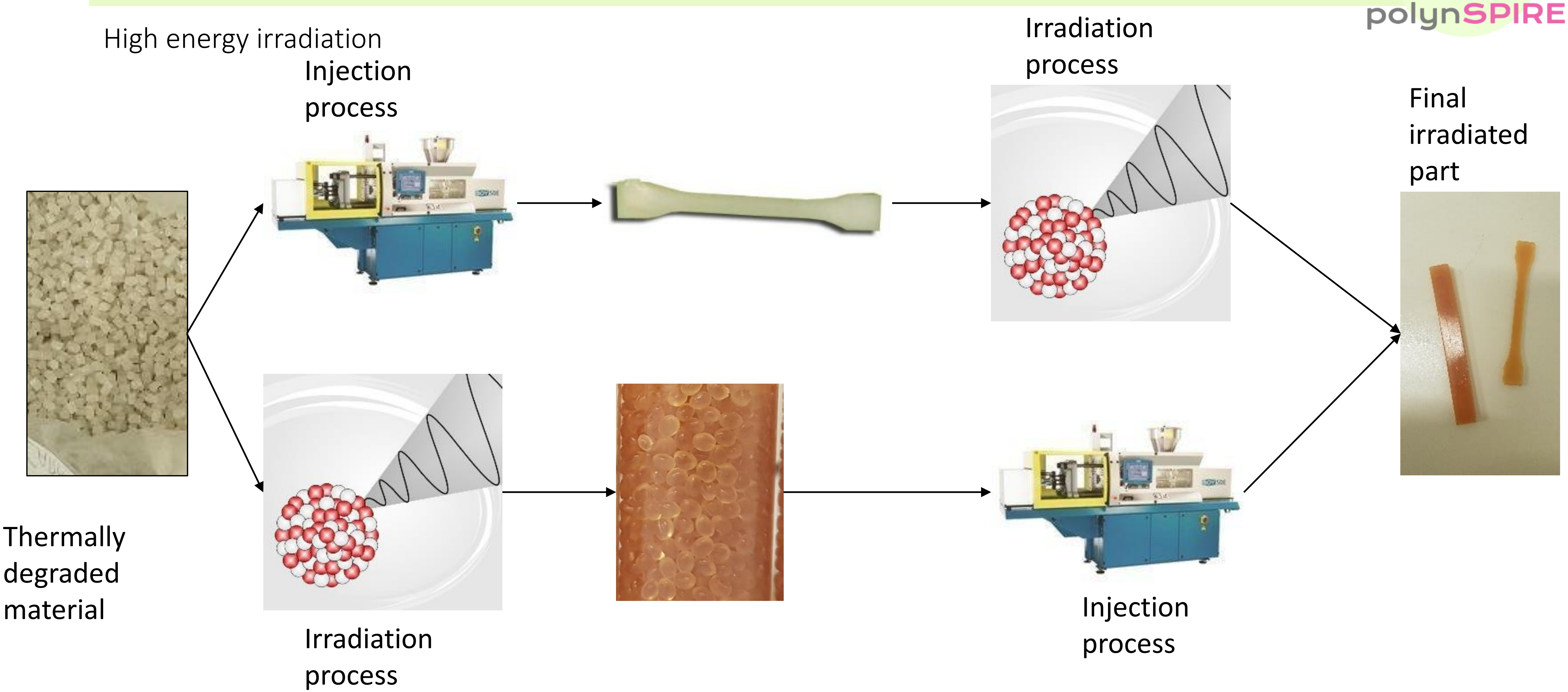
Main results of the project

High energy irradiation



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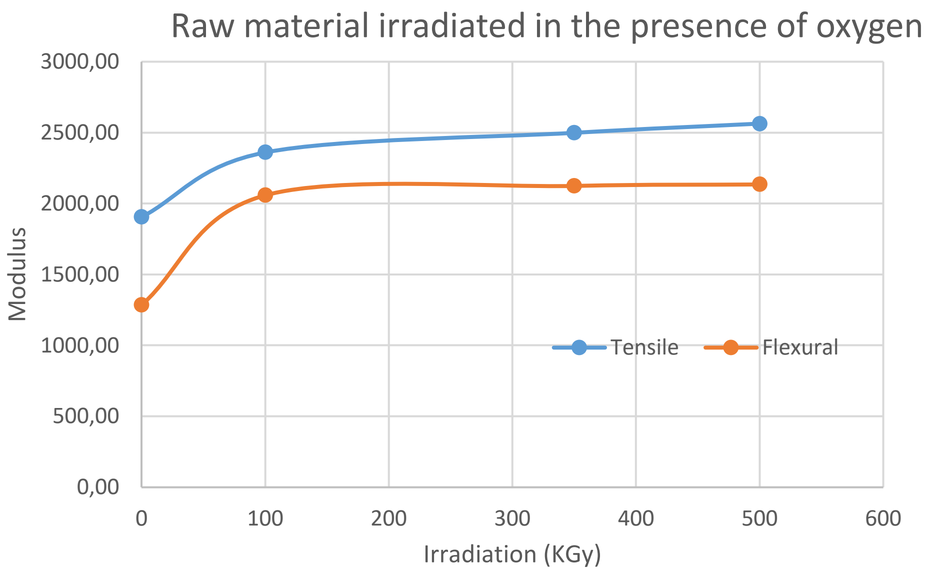
Main results of the project

High energy irradiation



The irradiation process generates crosslinking between the polymeric chains.

The crosslinking affects directly to the mechanical properties. Nevertheless, after reaching a certain point the effect of the irradiation is reduced.

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Partners involved

WP leader

- Co-leadership (task leaders apart from WPL)

- Other partners involved

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Thank you for your attention

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STANDARD PRESENTATION OF VALORISATION OF PLASTIC WASTE IN STEEL FURNACES

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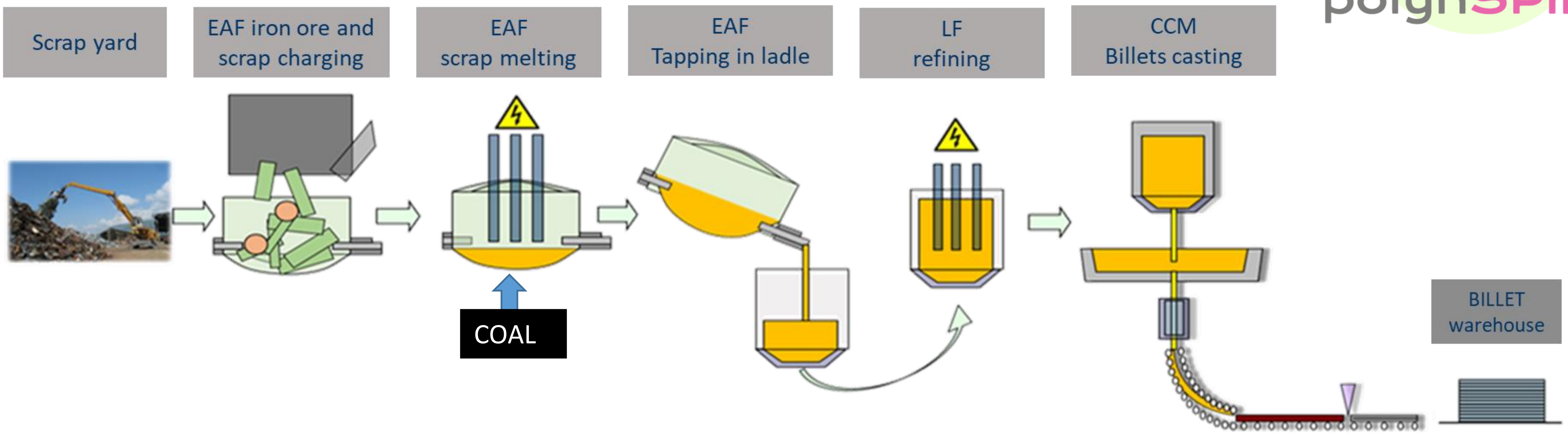
Innovative technologies for plastic recycling

Valorisation of plastic waste in the steel sector

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Plastic recycling in the Steel sector – fundamental concepts

ELECTRIC ARC FURNACE (EAF) STEEL PRODUCTION PROCESS



- Steel production from scrap in **Electric Arc Furnace (EAF)**. Is addressed in the project. EAF requires a relevant amount of coal as reducing agent and trigger foaming in the slag (increasing process efficiency).
- An average EAF produces **500-1000 kt of Steel per year with coal demand of 10-20 kg/t of produced steel**

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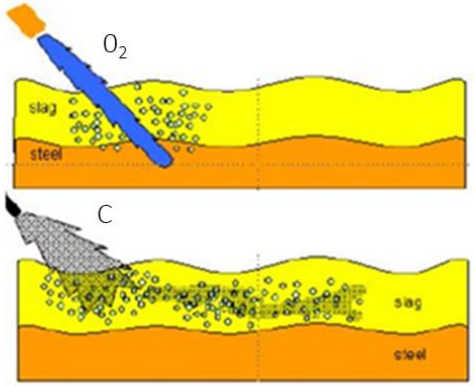
Plastic recycling in the Steel sector – fundamental concepts

Why is coal used in the EAF process?

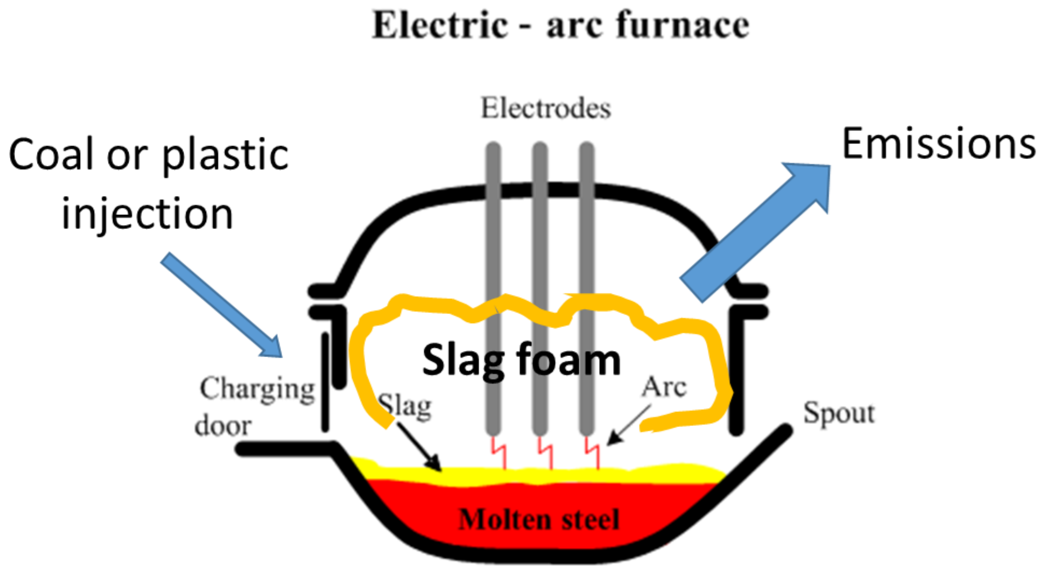
- A layer of protective slag is present in the furnace (slag is formed by CaO, FeO, Al2O3, MgO, SiO2, MnO). The foaming of the slag by CO/CO2 gas bubbles occurs naturally in the process by the oxidation of the carbon in the molten steel by oxides in the slag.
- Foaming process is intensified by the injection of carbon-bearing materials, traditionally fossil coal (used as carburizing/foaming agent and reducing agent). C reacts directly with oxygen and iron oxide forming gaseous CO. The gaseous emission forms a foam with slag, reducing the heat losses, NOx and noise emission from furnace.
- The process of carburization and reduction is fundamental to ensure efficiency and productivity. When using plastic grains, the reduction process is futher promoted by the Hydrogen content.

Reactions:

1) $C + \frac{1}{2} O_2 \rightarrow CO$
2) $FeO + C \rightarrow Fe + CO$
3) $FeO + CO \rightarrow Fe + CO_2$
4) $C + CO_2 \rightarrow 2\ CO$



Basic features of slag foaming



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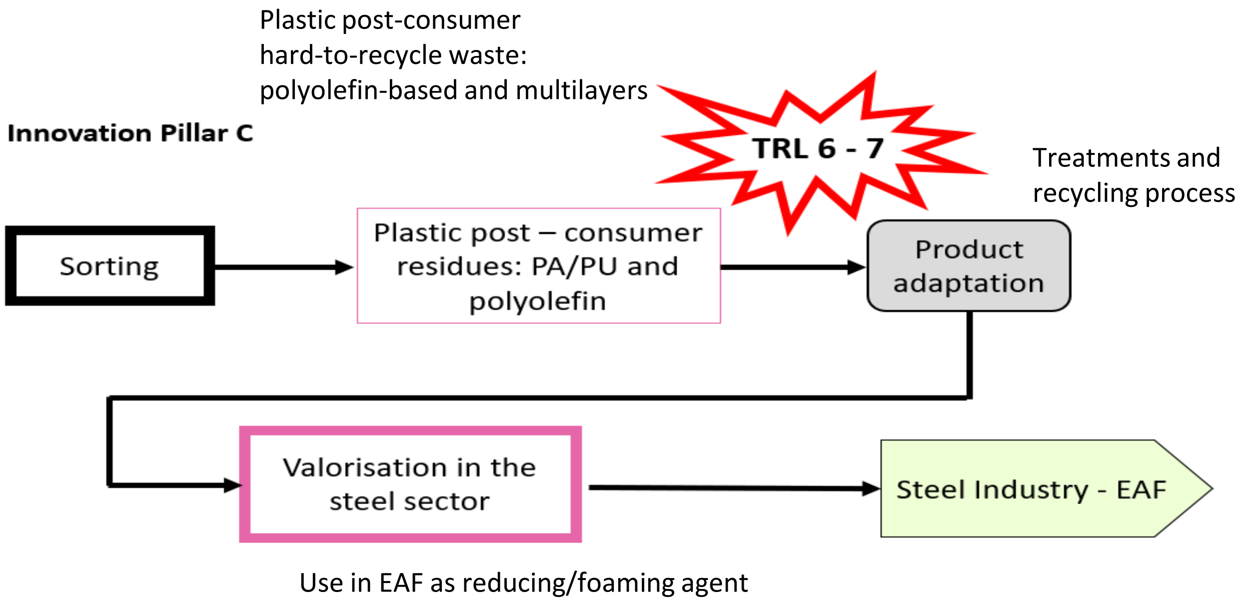
Valorisation of plastic residues

Valorizing-hard-to-recycle plastic waste by **recycling** them and using them as reducing/foaming agents in the **steel-making process**. Thanks to their carbon and hydrogen content, the polymers trigger oxides reduction and the foaming in the slag, thus improving the environmental performance of the furnace.



- Decarbonization thanks to lower coal consumption
- Contribution to EU recycling targets

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Valorisation of plastic residues in the steel sector

The substitution of coal with polymers requires:

- Characterization of the injected coal and of the plastic waste available to be utilized in EAF
- Polymers grains development: define the optimal mixed plastic grains manufacturing to match the steelmaking process requirements
- Design and manufacture an injection system suitable for plastic residues: the current injections devices are designed for coal and must be tailored for polymers
- Long term industrial tests: demonstrate at industrial scale the feasibility of the systems, aiming to reduce carbon consumption in steel industry

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Main results of the project– materials characterization

The material used in the project is a mix of plastics mainly from packaging

parameters	Plastics	Antracite (reference)
Heating value (MJ/kg)	32	26-30
ash (% dry)	5-10	1-10
Cl (% dry)	0.5	<0.01
S (% dry)	0.03	0.5-1.5
H (% dry)	10	0.5-1.5
N (% dry)	1.1	0.2-0.3
C (% dry)	65.0	80-85
Volatile matter (%)	88-90	1-10
C fix(%)	1.5	75-80

Performed characterizations:

- **Chemical analysis**
- **thermogravimetric tests**
- **Melting tests**

parameters	thresholds
Plastic content	≥ 80% dry material
Lower heating value(LHV)	≥30 MJ/Kg
Cl	≤ 2%
Cd	≤ 8 mg/Kg
Pb	≤ 100 mg/Kg
Hg	≤ 0,6 mg/Kg
H2O	Max 10%

The material is manufactured into grains for steel production according to italian standard UNI 10667-17:2018

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Main results of the project– polymers grains development

Sorting and plastic products preparation:

- The plastic product is produced from post-consumer MPW (mixed plastic waste) deriving from the packaging separate collection.
- This mix is recovered at the end of the sorting chain, and it is considered difficult to recycle due to its high variability.
- The transformation of MPW into the product BLUAIR® compliant with the technical standard UNI 10667-17 (addressed as Secondary Reducing Agent or SRA) is carried out through a duly authorised Recycling process (R3) by I.Blu. Its valorisation in the steel sector allows to close the loop of plastic recycling.
- Polymers utilization contributes to decarbonize the steel-making industry.

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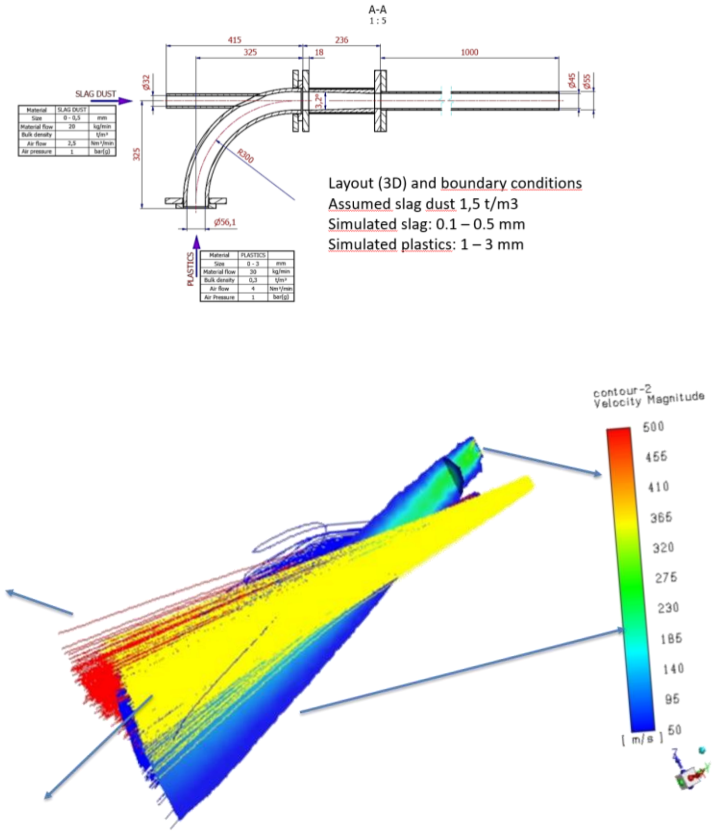
Main results of the project– design of the injection system



Injector design and CFD modelling

A new injector has been designed by HTT and evaluated by CFD modelling and by experimental tests with physical model

The validation permitted to asses the suitability of the new designed device to injected efficiently the plastic grains, promoting steel and slag emulsification and efficient foaming reactions

Injection tests with physical model

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project– Design of the whole pneumatic system for polymers injections

The industrial injection and pneumatic system has been designed and installed by HTT and FENO

The system is designed to be used during regular industrial steel production. The system is composed by following components:

- Plastic grains charging station
- Silos
- Weighting system
- Dispenser
- Injector; purposely designed within this project

Even if not originally foreseen, the system is equipped with ATEX components (protection from explosion), in order to guarantee the highest safety working conditions.

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project– Design of the whole pneumatic system for polymers injections

HTT designed the whole injection system to be installed in FENO

A replica of the pneumatic system has been also realized at HTT premises, in order to perform cold injection trials to check the regular working of the whole system before industrial installation

The testing facility in HTT plant includes

- Dispenser - will be purchased according to specification of previous chapter
- Storage silo with filtration system so material can be injected back to the silo
- Supporting and service platforms to operate testing equipment
- Testing pipe lines
- Nitrogen supply system
- Control system with data logging to record data gained during the test

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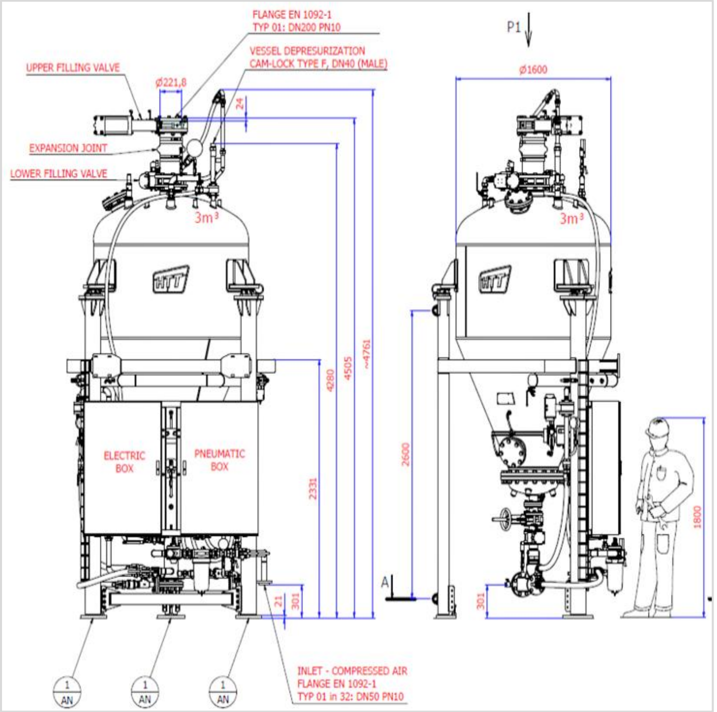
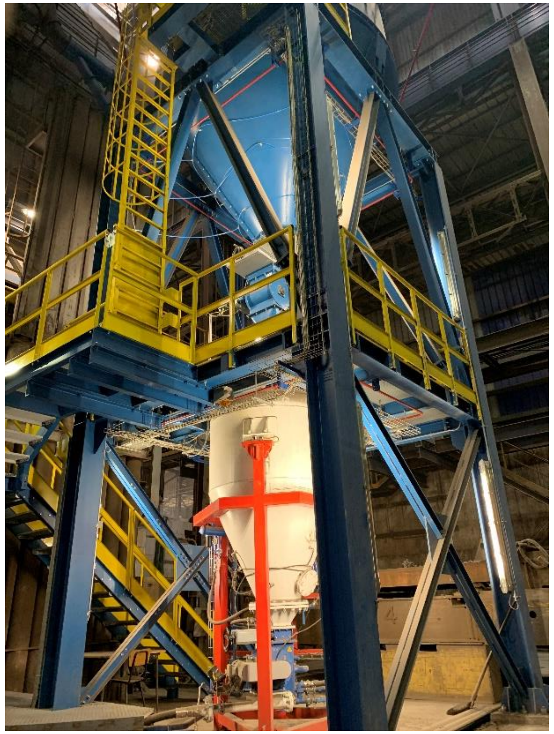
Main results of the project– installation, starting up and operation

Industrial pneumatic and injection dispenser installed at FENO steel factory





Injection dispenser layout

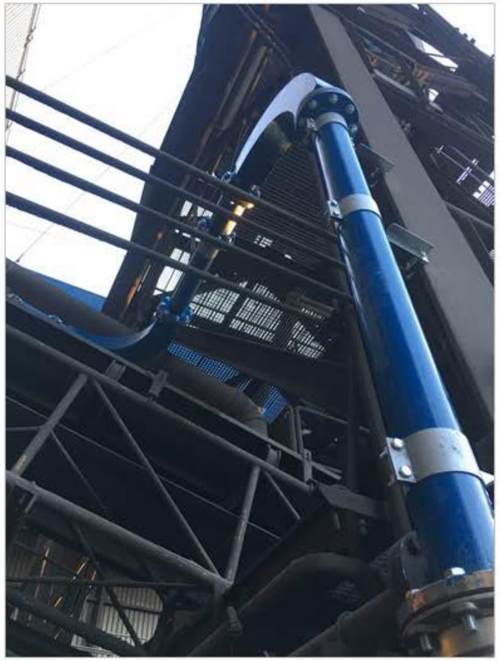
- The silo and pneumatic system has been purposely designed for plastic grain, with possibility to use inert gas.
- Compliance with ATEX Directive.
- The injection system is fully automatized and controlled from the Furnace control cabinet.
- It works with pressurized air.

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Main results of the project– installation, starting up and operation

Plastic grains charging system

- Plant has been designed in order to charge the silo directly from the truck.
- Truck is connected to a loading pipe with a pneumatic pinch valve that opens and closes by air pressure.
- The filling sequence is remotely controlled on the safety PLC (programmable logic controller).



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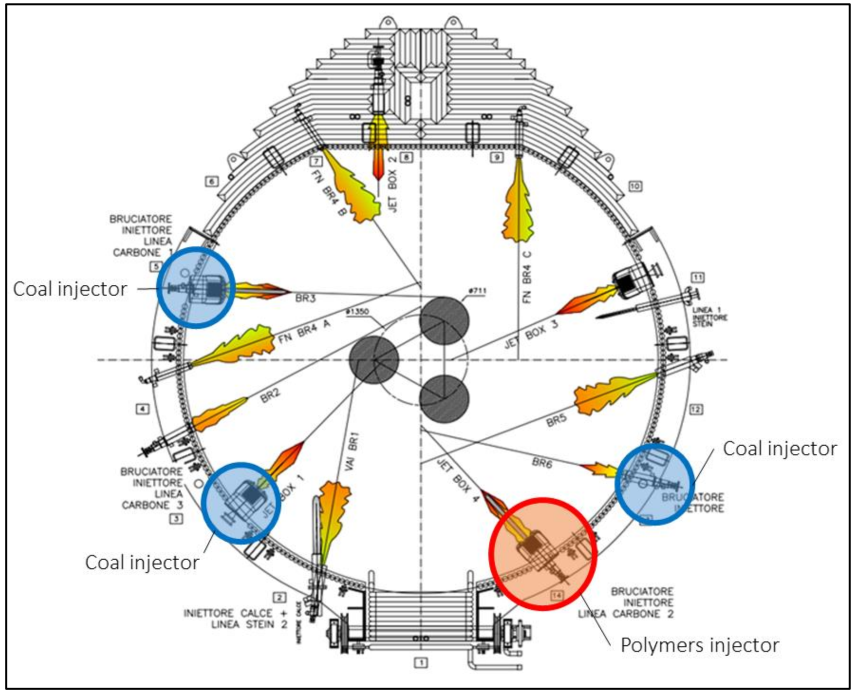
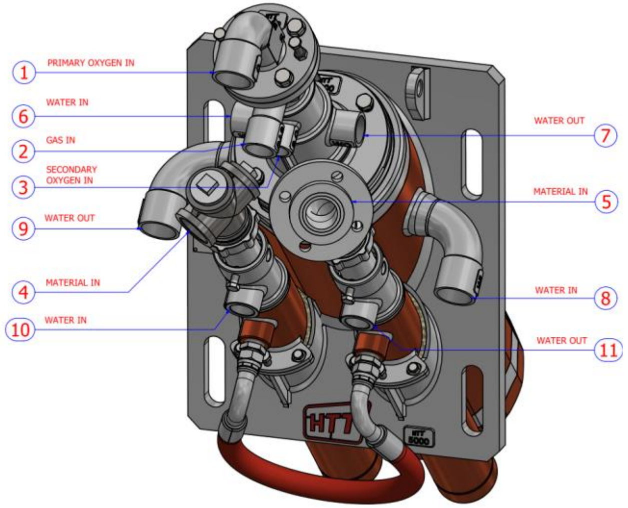
Main results of the project– installation, starting up and operation

Injector and its installation at the EAF

- During standard operation, there are 3 anthracite lances used with a flow of 10 – 25 kg/min.
- One of them has to be switched off and substituted by the new polymer injector (approximately 30% of the injected anthracite is replaced by polymers).
- The injector is a water-cooled lance already equipped for simultaneous utilization of: Plastic grains, oxygen (to promote foaming as explained in previous slide), or other grains (as anthracite, or slagging agents).

Top view of the furnace with the three lances for coal (blue) and the additional one for polymers (red)





The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

Industrial trials

INDUSTRIAL TRIALS

- Plastic injection has been tested in industrial trials
- **The substitution percentage of coal was about 30 % and 50% .**The substitution ratio is about 1:1.
- About two thousand of heats (production batches) carried out (one hundred foreseen in the project)
- More than 40 process variables were considered as performance indicator and the outcomes of the tests have been considered positive: **no production anomalies detected** (average power consumption in agreement with usual values).
- The pneumatic and injection system worked properly, without significant problems
- A measurement campaign at the chimney has been performed both with polymers and without, on the same process configuration. **The results highlighted no differences**.

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Industrial trials

INDUSTRIAL TRIALS

	DELTA USING POLYMER
Anthracite consumption per heat	- 34 %
Calorific value per heat	+ 4,76 %
Total Harmonic Distortion	+ 0,45 %
Sound Pressure Level	- 0,47 %
Average Active Power	+ 0,37 %
EAF Specific Electric consumption [$kWh/t_{liq\ steel}$]	- 1,3 %
Metallic yield	- 1,44 %
O_2 Consumption	- 0,3 %
CO_2 emission per heat	- 5,6 %
Exported power from furnace panels	- 13 %
Exported power from settling chamber and fume treatment	+ 9 %

Results obtained with 30% polymers injection (values expressed as % respect operating practices with 100% coal)

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Industrial trials

MAIN RESULTS

- The tests carried out have demonstrated that a tangible substitution of coal with plastic grains is achievable.
- The Process Parameters did not show critical variations: the main factor affecting process variability is the quality of the scrap.
- The results have demonstrated that at least 30% of anthracite can be replaceable by polymers that led to saving in terms of CO_2.
- A further increase in the quantity of polymers is feasible and desirable.

BENEFITS

- Reduction of dependency from fossil coal.
- Promotion of local economies.
- Valorization of mixed plastic with low market value.
- Closure of the loop of plastic recycling.
- Reduction of impact of transportation of raw materials.
- Reduction of CO2 emissions.

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EXPECTED OUTCOMES AND IMPACT

polynSPIRE

- The technology of polymers injection is not dependant on a specific EAF and can be adopted in all steel factories
- An average size steel factory CO2 savings evaluated at 6% without accounting the percentage of biogenic materials (in the average range 20-30%)
- Polymers demand for average size steel factory may be in the range 3000-5000 t per year
- Possible application in other sectors (e.g. foundry processes and iron alloys production)
- Allow to target 100% plastic recovery

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Partners involved

RINA — WP leader; materials characterizations; support to injector design (CFD); support to material improvement; support to industrial trials

- Co-leadership (task leaders apart from WPL)

I.BLU Gruppo Iren — Recycler and developer of plastic grain product for steelmaking. Production of materials for industrial trials

PITTINI — Cooperation to the design of injector and pneumatic system. Industrial installation of the system. Industrial trials. Evaluation of results

HTT — Design of injector and pneumatic system. Assistance to Industrial installation. Assistance to industrial trials

- Other partners involved

REPSOL — Production of residue to be evaluated for steelmaking utilization

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Thank you for your attention

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ANEX C. GUIDEBOOK OF POLYNSPIRE PROJECT

Guidebook of polynSPIRE project

The project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement no 820665

TABLE OF CONTENTS

GLOSSARY

- CANs: Covalent Adaptable Networks
- DBTDL: dibutyltin dilaurate
- EAF: Electric arc furnace
- EU: European Union
- MW: Microwave
- PA: polyamide
- PU: polyurethane
- PUF: polyurethane foam
- SMM: Smart magnetic materials

1. OVERVIEW OF PolynSPIRE PROJECT

1.1 PolynSPIRE background[1]

Plastics materials are used in a wide range of applications due to their properties, versatility, light weight and price. Due to these advantages, the production of plastics has been growing steadily in recent years, reaching a production of 390 million tons worldwide in 2021 and 57 million tons in Europe.

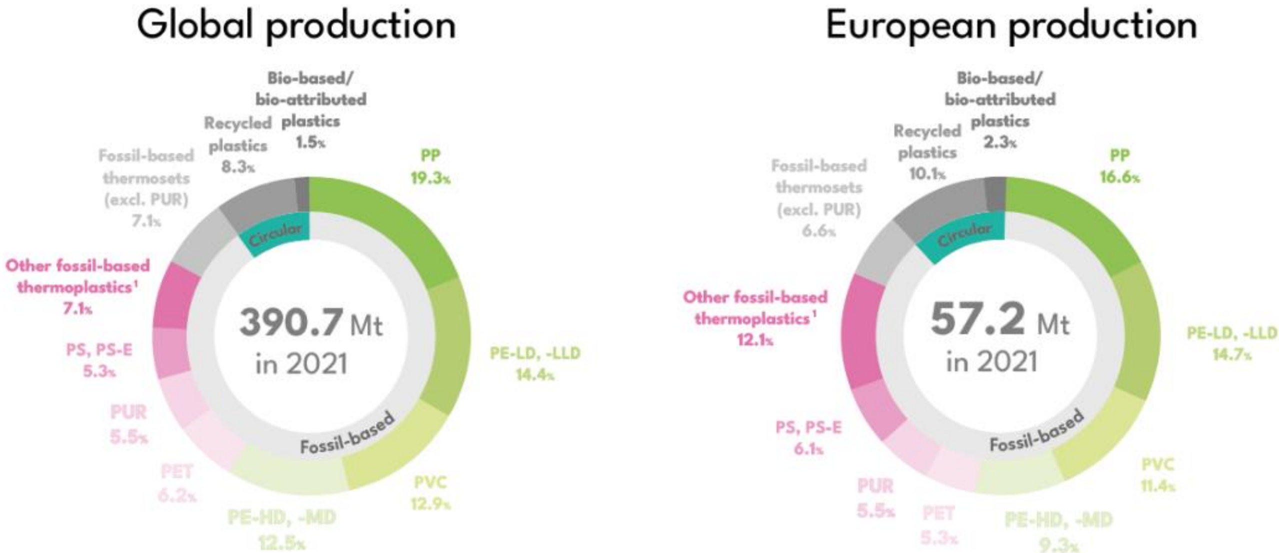

Figure 1. Distribution of the Global and European plastic production by type.

The demand for plastics is continuously increasing, as is the demand for fossil sources, which implies a high dependence on the oil market, as well as highly fluctuating prices. In addition, this extensive use leads to an enormous waste stream. According to Plastics Europe, around 29,5 million tons of plastic waste was generated in 2020 in the European Union. Around 35% was sent to recycling facilities, but more than 23% was still sent to landfill and more than 40% to energy recovery operations.

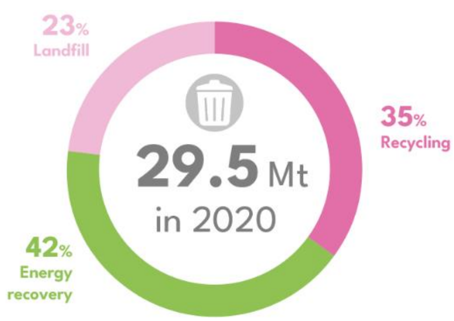

Figure 2. Post-consumer plastic waste management in 2020 (EU27+3).

The packaging sector has high recycling rates, around 80%, thanks to the fact that plastics waste is collected separately. However, post-consumer plastics waste from other sectors reveals a

[1] Plastic Europe. Plastics – The Facts 2022

lower recycling rate. Most of these plastic-containing materials are considered non-recyclable and, of the recyclable part, around 60% is exported outside the EU. One of the main reasons for the low recycling rate of plastic-containing materials outside the packaging sector is their heterogeneity. New trends in plastics production have introduced products with composite materials or reinforced plastics (most of them with fibers, mineral fillers or other performance-enhancing additives) with complex structures to meet specific requirements into the market. Due to this structure, these materials are difficult to reprocess efficiently while maintaining quality requirements.

1.2 PolynSPIRE innovations

In this sense, polynSPIRE will contribute to increase the recycling and valorization rate of widely available plastic containing wastes (having low recycling rate) by means of improving the energy and resource efficiency, reducing the overall environmental footprint and increasing the sustainability of plastic sector.

PolynSPIRE addresses these improvements throughout different recycling and valorization routes, which allow to convert wastes into raw materials that can be used in different steps of the plastic value chain or in other value chains such as the steel sector.

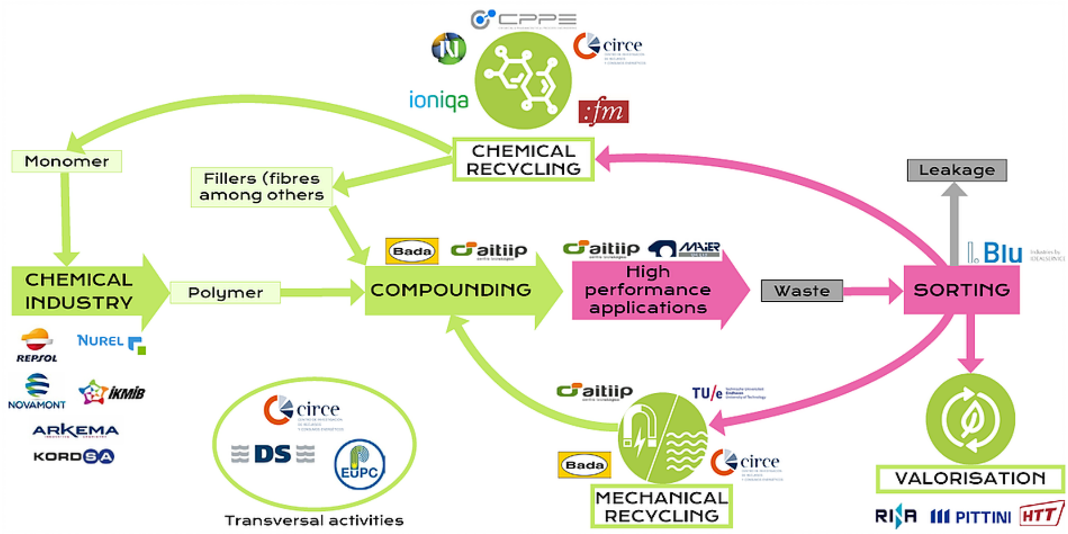

Figure 3. New polynSPIRE value chains.

A comprehensive set of innovative, cost-effective and sustainable solutions have been developed in the project, aiming at improving the energy and resource efficiency of the recycling processes for post-consumer (after product’s end of life) and post-industrial (produced during transformation processes from raw materials to final product) plastic containing materials. The three innovation pillars addressed in the project are:

A. Efficient chemical recycling of engineering polymers:

Focused on chemical recycling as a path to recover plastic monomers and valuable fillers (such as carbon or glass fibers) relying on microwaves-assisted organic chemistry and smart magnetic catalysts. These monomers later can be reintroduced at the beginning of the value chain to reduce the consumption of fossil raw materials.

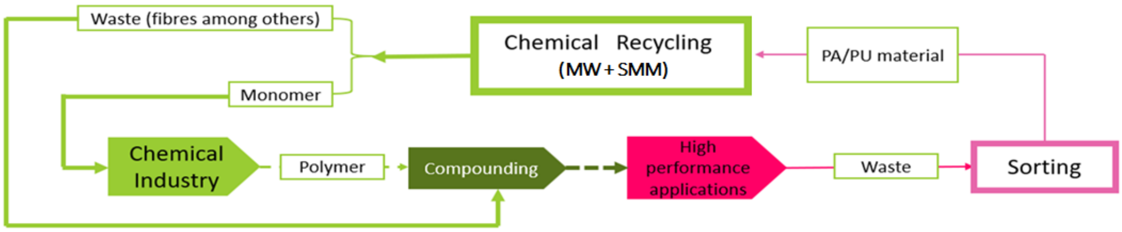

Figure 4. Innovation pillar A.

B. Improvement of mechanical recycling by means of advanced additives and high energy radiation:

Advanced mechanical recycling processes to enhance recycled plastics quality, using vitrimers and high-energy radiation is the aim of this innovation pillar. Thus, plastic wastes are reintroduced at the middle of the value chain fulfilling requirements of high value applications.

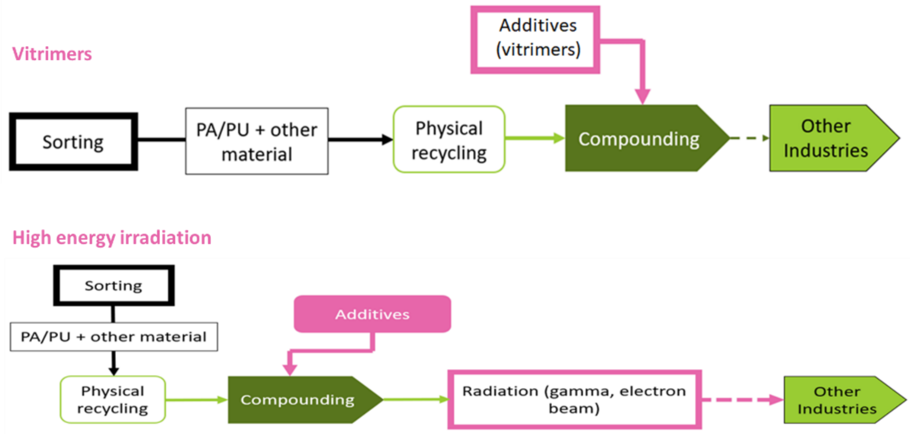

Figure 5. Innovation pillar B.

C. Valorization of plastic residues in the steel sector:

This innovation is focused on the valorization of low-grade plastic waste as carbon source in the steel industry. Plastic wastes are used in the electric arc furnaces (EAF) as substitute of carbon source for iron ore reduction and foaming agent, reducing fossil carbon sources.

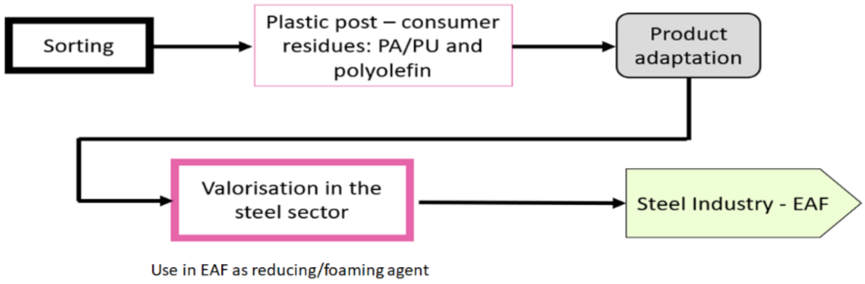

Figure 6. Innovation pillar C.

2. INNOVATION PILLAR A: CHEMICAL RECYCLING

Chemical recycling of plastics via depolymerization process allows to recover recycled raw materials, monomers or oligomers, that can be polymerized again. PolynSPIRE projects has focused on the recovery of monomers from different type of polyamides (PA6, PA66 and long-chain PAs) and oligomers (polyols) from polyurethane foams.

Several chemical routes can be used for the depolymerization of polyamides and polyurethanes. On the one hand, for PU, depending on the reagents used for the degradation of carbamate (urethane) bond, different routes can be applied such as hydrolysis, glycolysis, phosphorolysis, alcoholysis, aminolysis, and acidolysis. Aminolysis, alcoholysis and phosphorolysis are still used on a laboratory scale only, hydrolysis has been developed on a pilot scale, while glycolysis and acidolysis have already been successfully exploited on an industrial level. On the other hand, diverse types of Polyamides can be depolymerized by hydrolysis, aminolysis, or alcoholysis. Although PA66 is beside PA6 the most commonly used polyamides, literature reports on PA66 depolymerization are more limited than for PA6.

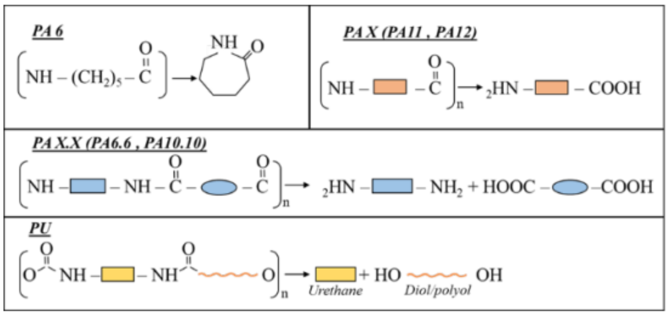

Figure 7. Different type of polymers that can be chemically recycled.

In polynSPIRE project, chemical depolymerization of polyamides and polyurethane has been improved through two innovative technologies: microwave (MW) and Smart magnetic materials (SMM).

2.1 MW recycling

Microwave irradiation is a well establishing technique in organic synthesis, including polymer synthesis and depolymerization. It allows efficient recycling of different kind of plastics (such as PA and PU) to recover valuable monomers or oligomers for synthesis of new products with the same characteristic as the virgin ones. The main advantage of using MW is contactless, instantaneous and rapid heating, resulting in faster reaction and thus shorter reaction time. Moreover, energy is delivered directly to materials through molecular interactions with electromagnetic field, obtaining a high energy efficiency in the process.

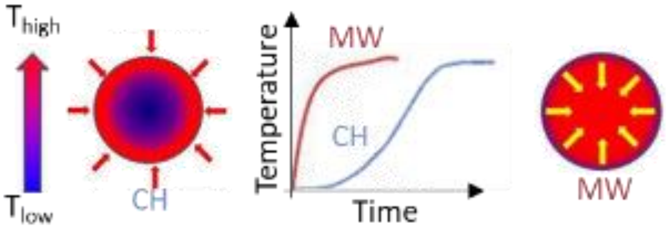

Figure 8. Advantages of the use of microwave heating.

Microwave assisted depolymerization of PA6 is well documented, while the use of MW for depolymerization of PA66 and long-chain PA are scarce. On the other hand, as physical recycling cannot be applied to a majority of PU due to their cross-linked structure and thermostability, chemical recycling has lately been a sought for solution. For this reason, microwave-assisted depolymerization is a good solution for the recycling of these two types of polymers, polyamides and polyurethanes.

In polynSPIRE project, different types of microwave-assisted depolymerization reactions were investigated for PU and different types of PA at laboratory scale. However, aminolysis for PU and acidolysis for PA66 were the reactions with the best results. This depolymerization routes have been tested at medium scale during the project with promising results.

2.2 SMM recycling

The use of Smart Magnetics Materials (SMM) catalysts is the second technology of this innovative pillar. SMM catalyst are composed of a solid support (A), bridging moiety (B) and functionality (C). The solid support may be composed of a magnetic material. The functionality (C) may be composed of a catalyst complex. These SMM catalysts are being produced by IONIQA on large scale.

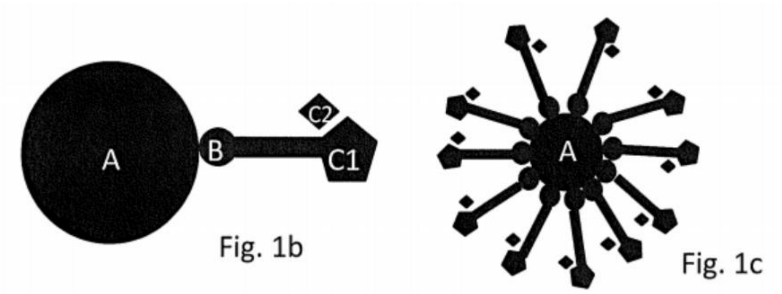

Figure 9. Chemical structure of SMM.

The main advantage of SMM catalysts is that they have better yield and selectivity than benchmark catalysts for polymer depolymerization, resulting in a higher product recovery and a and a significant lower energy usage. In addition, they are easily recoverable from the reaction mixture and can be reused.

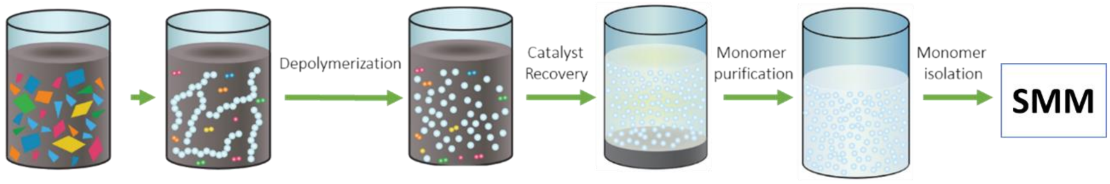

Figure 10. Depolymerization process assisted by SMM.

In polynSPIRE project, different depolymerizations tests for polyamides and polyurethanes were performed with SMM as catalyst. The most promising results have been achieved for the glycolysis of PU.

3. INNOVATION PILLAR B: MECHANICAL RECYCLING

The polynSPIRE project is working to recycle and increase the lifespan of polymeric materials by researching various chemical, mechanical, and economic solutions. The second innovation pillar is focused on mechanical recycling of polyamides and polyurethanes using high-energy radiation to induce chemical crosslinking or the addition of additives to produce vitrimer-like materials. The goal of mechanical recycling is to improve the processability and recyclability of these materials.

3.1 Vitrimers

PolynSPIRE project has shown that it is possible to convert polyurethane foams (PUF) into vitrimer-like materials via reactive extrusion, which opens the way to reprocessing waste PUF in a similar way.

This innovative solution was decided to focus on polyurethane materials as the waste material in question is already cross-linked, making it hard to process with those methodologies used for thermoplastics but making it a prime material for introducing dynamic bonds.

Vitrimers, in stringent contrast to dissociative networks, include Covalent Adaptable Networks (CANs), that are a way to improve the recyclability of polymer networks by incorporating exchangeable chemical bonds during synthesis. These bonds allow for the reprocessing of polymer networks and can be divided into two main categories: associative CANs (only allow for the breaking of a bond if a new linkage is already formed) and dissociative CANs (require the breaking of a bond before a new one can be formed). CANs have been studied as a way to improve the recyclability of polymers, and recently steps have been taken to incorporate these functionalities into commercially available polymers.

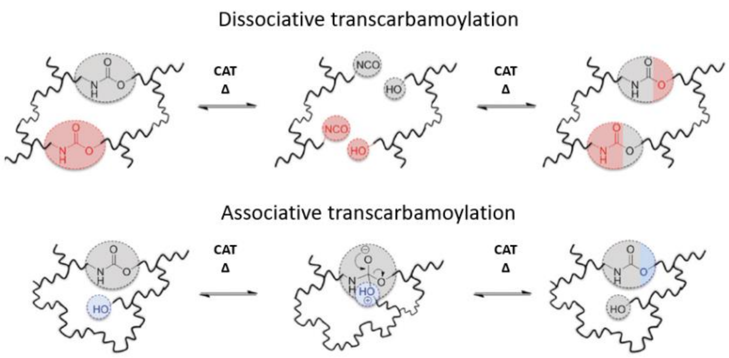

Figure 11. Types of covalent adaptable networks (CANs).

In polynSPIRE project, the focus of the work has been on thermoset MDI-based polyurethane foams, and it is shown that dibutyltin dilaurate (DBTDL) is a very efficient catalyst for the "conversion" of static cross-links into dynamic cross-links causing a significant reduction in torque during extrusion. Increasing the catalyst concentration and/or the temperature leads to greater reductions in torque, and it is shown that good processability of the materials is obtained at temperatures as low as 160°C and catalyst concentrations lower than 6%. At temperatures of around 200°C, the materials show good processability, but suffer from some thermal degradation.

These results clearly show that reactive extrusion is a feasible technology for reprocessing PUF in the presence of DBTDL and scale-up should be possible. Future work will focus on the initial additions of polyols in combination with the catalyst, so as to even further decrease the viscosity initially and to allow for a reformation of the network (potentially in a subsequent step with additional diisocyanates).

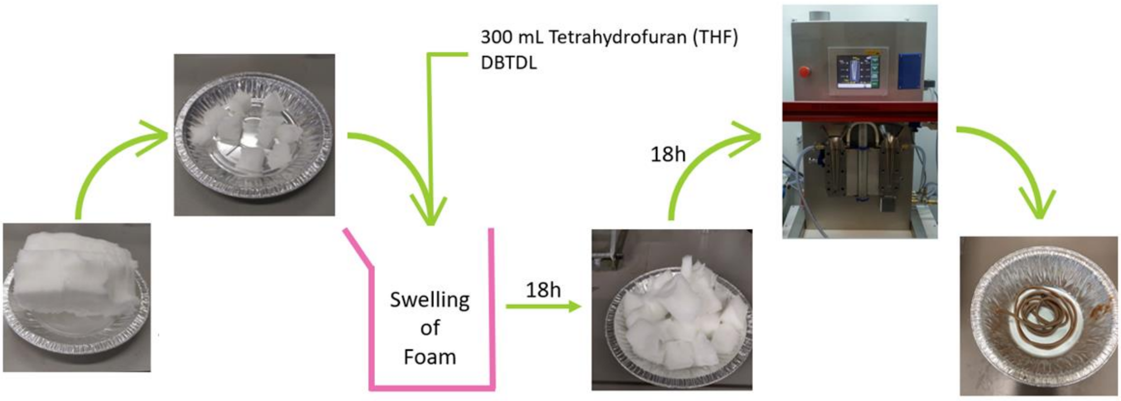

Figure 12. Reprocessing of PUF by reactive extrusion in presence of DBTDL.

3.2 High energy irradiation (gamma radiation)

Polyamides find use in several industrial applications where processing conditions make them subject to thermo-oxidative and mechanical degradation. At the same time, plastic pollution is reaching exorbitantly high levels that must be addressed to avoid a concomitant environmental crisis. It is therefore pertinent to devise strategies that allow to upgrade and recycle degraded plastics, thus contributing to the establishment of a circular economy.

Chemical crosslinking of Polyamides through the use of ionizing radiation may prove a suitable strategy to attain a recycled product whose main chemical and mechanical features resemble to the greatest extent those of the original polymer. Several high energy sources may be used for this purpose, among which both gamma and electron beam radiation are the most popular choices. Out of the two, gamma radiation was chosen to carry out the experiments on crosslinking of PA6, due to its penetration depth and replicability potential.

Gamma irradiation is the energy generated in the decay of radioactive atoms and the disintegration of subatomic particles. High-energy irradiation of polyamides generates free radicals that can subsequently react leading to crosslinking or chain scission phenomena. This method makes it possible to initiate chemical reactions with compounds in the solid state without the addition of an initiator.

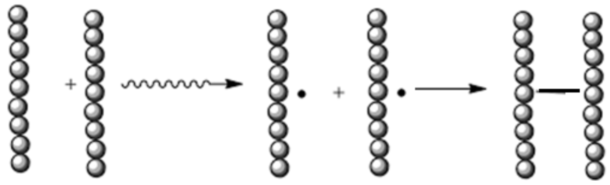

Figure 13. Crosslinking of polyamides due to high energy irradiation process.

PolynSPIRE project has demonstrated that polyamides irradiated at an optimum dose of 100 kGy has an increase in toughness without affecting other mechanical properties. High energy irradiation technology for PA materials is replicable at medium scale with the material used in the industry. Although characterization techniques done to the irradiated materials have shown that the final properties achieved are not in the same range as for raw PA compound, an improvement between the irradiated and the recycled material has been achieved.

In summary, gamma irradiation is a suitable solution to reduce waste and favour the establishment of a circular economy, obtaining promising outcomes for not high demanding applications, opening the door to the application of PA in other sectors and improving the properties of those components. Due to the broad industrial applicability of the sample polymers chosen for the experiments, namely PA6 and PA66, the technique may be replicated in diverse industrial sectors to create efficient business models.

3.3 Additives and fillers

Compatibilizing additives are widely used to enhance interaction between different polymers in blends and to enhance interaction between reinforcement (fillers/fibers) and plastic matrices in composites. A better interaction between the different components (polymer-polymer or polymer-reinforcement) lead to a significant improvement in properties while also to more homogeneous properties in the material.

PolynSPIRE project has demonstrated the possibility of reusing the glass fiber and other additives obtained from the chemical recycling of polymers, especially polyamides. When polyamides are charged with different percentages of glass fibre, the irradiation process does not have the same effect when the glass fibre absorbs part of the. However, if additives are properly integrated in the final compound and allow the matrix absorbs the gamma irradiation, it will generate some crosslinking between the different polymeric chains, regenerating the properties lost by the degradation. In those cases, the recycled material shows the same performance than the raw material.

4. INNOVATION PILLAR C: VALORISATION OF PLASTIC WASTE IN STEEL FURNACES

This innovation aims to valorise low-grade plastic waste from post-consumer mixed plastic packaging waste by using them as carbon source in steel sector. A specific recycling process has been developed during the project to recycle mixed plastic waste (MPW) and prepare them to substitute virgin fossil resources in EAF steelworks.

The electric arc furnace is the most important scrap recycling process. Globally, Electric Arc Furnace steel production accounts for 28% of the total and 46% in Europe. The electric furnace melting process is, in general, a batch process. It can be divided into the following stages:

- Charging of materials (scrap charging by basket)
- Melting phase, which in turn includes the melting of solid materials
- Refining, in which metallic species (mainly Si and Al) and P are removed by oxygen injection and captured by the slag phase covering the liquid bath
- Deslagging, in which slag is removed
- Tapping, in which steel is transferred to a ladle for further production steps up to final composition

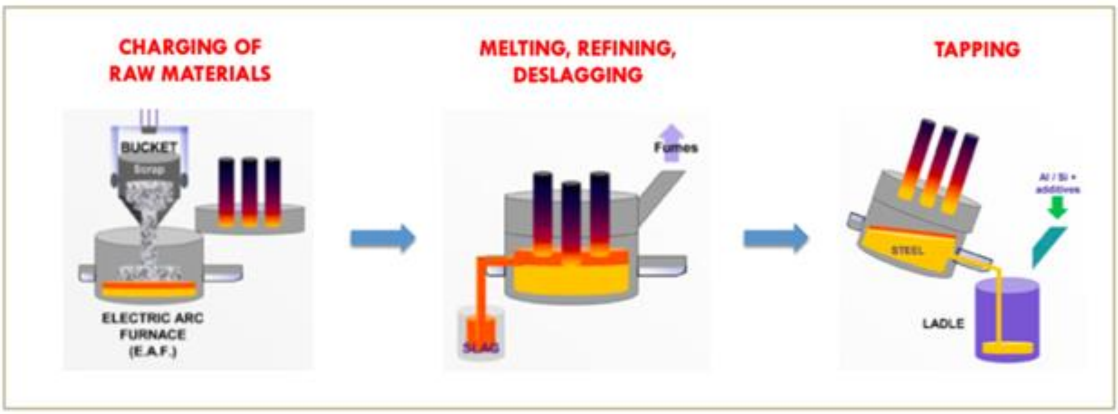

Figure 14. Process stages of EAF.

Electric Arc Furnace uses a relevant amount of chemical energy. Carbon (currently as fossil hard coal) is also used in the process. The current state of the art in EAF steelmaking is to use as much as possible chemical energy, besides electric energy, to accommodate tap-to-tap times to the pace of the downstream continuous caster. The share of chemical energy is in the range 30-50%. Chemical energy is introduced by oxidation reactions which occurs inside furnace and mainly by natural Gas burning.

In the furnace, a layer of protective slag is present (slag is formed by CaO, FeO, Al2O3, MgO, SiO2, MnO). The foaming of the slag by CO/CO_2 gas bubbles occurs naturally in the process by the oxidation of the carbon in the molten steel by oxides in the slag.

Reactions:

1) $C + \frac{1}{2} O_2 \rightarrow CO$
2) $FeO + C \rightarrow Fe + CO$
3) $FeO + CO \rightarrow Fe + CO_2$
4) $C + CO_2 \rightarrow 2\,CO$

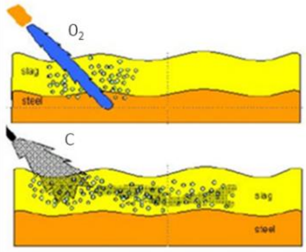

Figure 15. Reactions of the foaming process.

This foaming process is intensified by the injection of carbon, usually fossil coal (a carburizing/foaming agent and reducing agent). Carbon reacts directly with oxygen and iron oxide forming gaseous CO. The gaseous emission forms a foam with slag, reducing the heat losses, NOx and noise emission from furnace.

The process of carburization and reduction is fundamental to ensure energy efficiency and productivity.

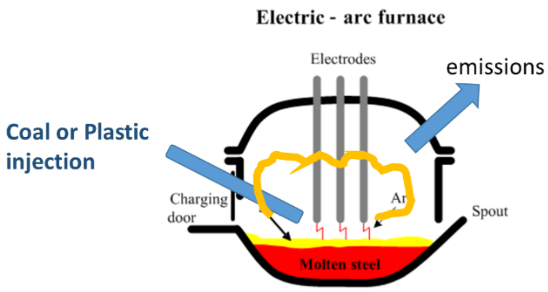

Figure 16. Schematic diagram of the foaming process in the EAF

In polynSPIRE project, post-consumer plastic waste has been used to partially replace fossil carbon injection. These plastics are composed of a mixture of plastic residues resulting from sorting processes, which are particularly difficult to recycle, called Secondary Reducing Agent (SRA). By implementing SRA in the EAF processes, it would be possible to revalorise them, preventing them from ending up in landfills or incinerators. Plastic injection has been tested in industrial trials, demonstrating that substitution of coal by plastic grains is achievable. The results have shown that at least 30% of the anthracite can be replaced by polymers, which means tangible savings in terms of CO_2 and partly in terms of energy, with the hope of reaching 50% polymer in the future.