



Environmental Engineering and Management Journal

Founding Editor: Matei Macoveanu

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***Leading the Ecological Transition
ECOMONDO 2023***



“Gheorghe Asachi” Technical University of Iasi

Environmental Engineering and Management Journal

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EDITORIAL

Leading the Ecological Transition

ECOMONDO 2023

The Green Technology Expo

The papers collected in this special issue of *Environmental Engineering and Management Journal* were presented as lectures or posters at the scientific and technical conferences hosted by *Ecomondo 2023* held from the Italian Exhibition Group headquarter in Rimini, Italy, during 7-10 November 2023. (<http://en.ecomondo.com>).

Ecomondo is one of the largest European exhibitions in the field of *Green and Circular Economy*, hosting annually over than 100,000 delegates from more than 120 different nations along with 1,600 industrial exhibition stands in 160,000 square meters and over than 200 conferences and workshops on policies, research and innovation, innovation funding opportunities, financing, education, communication, and entrepreneurship and international networking and partnership creation.

As with the previous editions, the aim of *Ecomondo 2023* was to explore and share recent industrial advances and opportunities in: industrial eco-design; industrial symbiosis, renewable and critical resources; industrial technical waste production reduction, recycling and exploitation in the frame of the plastic, packaging, textile, electronic and electric, steel and construction industries; sustainable agrifood and wood chains, biowaste collection and exploitation via integrated biorefinery schemes, with the production of biobased chemicals, materials and biofuels, including methane; wastewater treatment and valorization with nutrients recovery and water reuse; water resources monitoring, protection and sustainable use in the civil and agrifood sectors; marine resources protection and sustainable exploitation and port regeneration; sustainable remediation of contaminated sites and marine

ecosystems; indoor and outdoor air monitoring and clean up; and circular and healthy cities.

Some of the international workshops saw the participation of the EU commission and the EEA and were focused on the emerging trends in the circular economy domains of electronic and electric products, construction and demolition, packaging materials and textile and fashion. A special focus was on the health observation complemented with events organized with EU commission, FAO and OECD on the soil and coastal restoration, the plastic waste and the monitoring, prevention and mitigation of marine litter. Finally, *Ecomondo 2023* also hosted events on the priorities of the Mediterranean macro-region, organized with the EU commission, Union for Mediterranean, and in particular the water scarcity of the area, the Mediterranean Sea contamination (also due to marine litter).

Ecomondo 2023 conferences hosted more than 750 oral communications, about 70 of them were then selected for full paper publications on this journal, the *Procedia Environmental Science, Engineering and Management* and the *Ecomondo proceedings*. This special issue contains some of such papers and provides some of the key information presented and discussed in the frame of some of the mentioned technical and scientific conferences of *Ecomondo 2023*.

We believe that this collection of papers will be useful to people who could not follow the edition of *Ecomondo 2023*. It is primarily towards them but it also aspires to provide permanent records in the promotion, adoption and implementation of the major priorities and opportunities of the green and circular economy in

Europe and in the Mediterranean basin, with the conversion of some of the key local environmental challenges into new opportunities for a green and

sustainable growth of the territories. For additional info, please refer to the following link: <https://en.ecomondo.com>

Guest Editor:

Professor **Fabio Fava**, Alma Mater Studiorum - Università di Bologna, Italy



Fabio Fava, born in 1963, is Full Professor of "Industrial & Environmental Biotechnology" at the School of Engineering of University of Bologna since 2005. He published about 250 scientific papers, 220 of which on medium/high IF peer-review international journals of industrial and environmental biotechnology and circular bioeconomy. He has more than 12000 citations, a H-index of 62 and an i10 index of 150 (Google Scholar) along with 220 papers quoted by Scopus. He is actively working in the fields of environmental, industrial and marine biotechnology and of the circular bioeconomy in the frame of a number of national projects and collaborative projects funded by the European Commission. Among the latter, he coordinated the FP7 collaborative projects NAMASTE, on the integrated exploitation of citrus and cereal processing byproducts with the production of food ingredients and new food products, and BIOCLEAN, aiming at the development of biotechnological processes and strategies for the biodegradation and the tailored depolymerization of wastes from the major oil-deriving plastics, both in terrestrial and marine habitats. He also coordinated the Unit of the University of Bologna who participated in the FP7 collaborative projects ECOBIOCAP, ROUTES, MINOTAURUS, WATER4CROPS, ULIXES and KILL SPILL.

Fabio Fava served and is serving several national, European and international panels, by covering, among others, the following positions:

- Member of the Scientific Committee of the European Environmental Agency (EEA), Copenhagen, for the "Circular economy and resource use" domain (2021-);
 - Italian Representative in the "European Bioeconomy Policy Forum" and the "European Bioeconomy Policy Support Facility" of the European Commission (2020-);
 - Senior Expert of the Italian delegation to the Programming committee Horizon Europe, Cluster VI: Food, bioeconomy, natural resources, agriculture and environment (European Commission, DG RTD)(2020-);
 - Italian Representative and elected vice chair in the "States Representatives Group" della Public Private Partnership "Circular Biobased Europe" (CBE JU), Brussels (2021-);
 - Italian Representative in the "Working Party on Biotechnology, Nanotechnology and Converging Technologies" of the Organization for Economic Co-operation and Development (OECD, Paris) (2008-);
- Finally, he is the scientific coordinator of the International Exhibition on Green and Circular economy ECOMONDO held yearly in Rimini (Italy)



“Gheorghe Asachi” Technical University of Iasi, Romania



AN INTEGRATED AND ECO-EFFICIENT PROCESS FOR MATTER RECOVERING BY WEEE RECYCLING: PINECOR PROJECT

Maria Chiara Canu¹, Roberto Giovanardi¹, Fernanda Andreola¹, Isabella Lancellotti¹, Cristina Leonelli¹, Paolo Pozzi¹, Teresa Sessa², Bibiana Ferrari², Arturo Sommariva³, Giovanni Modica³, Luca De Benedittis³, Giovanna Ferraroli⁴, Sergio Ferraroli⁴, Laura Borghino³, Andrea Gubiani³, Luisa Barbieri^{1*}

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Abstract

The scarcity and supply difficulties of strategic elements for industrial ecosystems make the development of processes/technologies for their recovery from existing resources crucial. In this perspective, the recycling and collection of WEEE (Waste from Electrical and Electronic Equipment) are essential for the circular economy. In Italy, the e-waste stream is divided into two channels (domestic and professional) and five groupings. In 2023, 72% of the waste managed by recycling companies (around 367 ktons) came from the domestic sector, while 28% (around 144 ktons) came from the professional sector. From 2018 to 2023, the sequence of WEEE collected from the highest to the lowest is represented by groupings: R2 (Large Household Appliances), R1 (Cooling and Freezing Appliances), R4 (Consumer Equipment), R3 (Screen and Monitors) and R5 (Lighting equipment). R1, R2 and R4, which seem to represent the categories that would bring more environmental benefits (i.e., tons of CO₂ equivalent avoided) if properly processed and recycled, are the target secondary sources for the PINECOR project. The aim of such a project is to develop an integrated system, based on innovative, multifunctional and eco-efficient technologies for the recovery of glass, siliceous fraction, plastic and metal from WEEE. This paper presents the first results obtained in the project both in terms of separation processes developed to obtain high-purity recycled fractions, and in terms of techniques for the extraction and recovery of secondary raw materials, with particular attention to their application in the most common sectors and those in growth.

Key words: elutriation, glass, metals, plastics, secondary materials, separation procedures, WEEE

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1. Introduction

The amount of Waste of Electrical and Electronic Equipment (widely known as WEEE or e-waste) generated each year in the EU and around the world is increasing rapidly. With an annual growth rate of 2%, it is now one of the fastest growing waste streams (European Commission, 2020). It includes a wide range of equipment such as mobile phones, computers, televisions, refrigerators, household

appliances, lamps, but also medical devices and photovoltaic panels (Chesmech et al., 2023; Khan et al., 2022). E-waste contains a complex mixture of materials, some of which are hazardous. This can cause major environmental and health problems if the discarded equipment's are not properly managed. Modern electronics also contain rare and expensive resources, including critical raw materials (Li, Co, Pt, etc.). At the end of its working life, Electrical and Electronic Equipment (EEE) must be recycled and

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therefore sorted into different groupings for collection. That is why WEEE is identified by the crossed-out wheeled bin symbol. All WEEE have specific tasks to perform to ensure the environmental sustainability of the system and thus contribute to the protection of the environment.

If managed effectively, this waste can be recycled and reused. In addition, improving the collection, treatment and recycling of the electronic equipment at the end of its life can increase resource efficiency and support the transition to a circular economy. It can also contribute to the security of supply of critical raw materials, ultimately strengthening the EU's strategic autonomy (Serpe et al., 2024).

In Italy, the collection and recycling of WEEE governed by Italian Legislative Decree 49/2014, which defines the direct liability of many different players: the producers of EEE, suppliers, local authorities and consumers. The Italian collection system divides the e-waste stream into two channels (domestic and professional) and five groupings. In 2023, the 72% of waste managed by the Recyclers (about 367 ktons) came from the domestic sector, while 28% (about 144 ktons) came from the professional sector. From 2018 to 2023, the sequence of WEEE collected from the highest to the lowest is represented by groupings: R2 (large household appliances), R1 (cooling and freezing appliances), R4 (consumer equipment), R3 (screens and monitors) and R5 (lighting equipment) (CDC RAEE, 2018 2019, 2020, 2021, 2022, 2023a, 2023b).

As mentioned above, WEEE is a problem, but also a great opportunity to recover very valuable fractions and therefore the collection, management and recycling phases are strategic. The target of a minimum collection rate of 65% for WEEE is an ambitious goal for European countries due to the lack of skills, scarcity of effective technologies and gaps in the collection phase. Furthermore, according to the European Commission, the design of products determines up to 80% of the environmental impact of their life cycle: most of the products currently placed on the market are designed without taking into account, or insufficiently taking into account, their recyclability and the end-of-life phase (European Commission, 2024).

With almost one million tons/year of electrical and electronic equipment entering the Italian market (continuously growing), just over 350,000 tons/year of WEEE are separately collected, or 40% (5.8 kg/inhabitant) compared to a European target of 65% (9.8 kg/inhabitant). These quantities are recycled at a rate of 89% (EERP R, 2024).

There is undoubtedly a gap at the collection phase, largely due to the behaviour of citizens who do not manage end-of-life properly. However, it should be borne in mind that, at present, some fractions of separate collection are exported to other European countries, due to the lack of dimensional and technological facilities, and that in the coming years

we will have to increase separate collection by at least 200,000 tons/year (EERPR, 2024).

There is a clear need for financial support for the construction of the plant infrastructure required to take advantage of this industrial opportunity. In order to improve waste management and the circular economy, to strengthen the infrastructures for separate collection, to modernize or build new waste treatment plants, and to bridge the gap between the regions of the North and those of the Center-South (today about 1.3 million tons of waste are treated outside the region of origin), important financial support policies have been implemented in Italy. One of them is the National Resilience and Resistance Plan (NRRP), established by the Government in 2021 with Legislative Decree no.59. The funds earmarked for the private sector in the waste cycle, identified by Investment Line 1.2 "Lighthouse projects for the circular economy", have been allocated to innovative projects for the treatment and recycling of waste from the four strategic supply chains identified by the Circular Economy Action Plan promoted by the EU, namely: Plastics, Paper and cardboard, WEEE (including photovoltaic panels and wind turbines) and Textiles. The total amount of investment has been divided into four equal parts, i.e. 150 million euros for each line of intervention. The objective of these interventions is to support the achievement of recycling targets in the sectors identified by in the Circular Economy Action Plan for each supply chain financed by the NRRP. In particular, the targets set in Italy for WEEE are 55% recycling (AssoAmbiente, 2023). In terms of financial resources dedicated to research and innovation projects, the most important action is the one led by the Italian Ministry of the Environment and Energy Security (MEES) with the Call for co-financing of WEEE Research Projects, approved in 2020 by the Ministerial Decree of the Ministry of the Environment of 9 December 2020, prot. n. 74, with a budget of 900,000 euros. The 2020 edition of this Call also includes the PINECOR project.

In most WEEE treatment facilities, after sorting and pre-treatment (usually mechanical), processing intermediates are obtained that are not market-ready: to become end-of-waste/products they require further purification and separation processes. Often these processes are not only complex, energy-intensive and focused on quantitative rather than qualitative yields, but are also specialized in only one type of "key-fraction" or "valuable fractions" (e.g. the process for recovering recycled plastic is different from that for recovering base metals). In this context, PINECOR aims to provide a multifunctional solution, capable of working as an integrated process to recover glass, siliceous fraction, metal and plastic simultaneously. In fact, despite the type of treatment and the recyclable materials and residual fractions, which vary according to the grouping treated, PINECOR is a flexible and adaptable process capable of efficiently treating three different groupings of

WEEE (R1, R2, R4) with the highest Italian collection rate.

2. Materials and methods

2.1. PINECOR project: context, process and materials

PINECOR (*ECO-efficient and Integrated Process for WEEE Recovery*) is an Italian research project co-funded by the Ministry of the Environment and Energy Security ("Ministero dell'Ambiente e della Sicurezza Energetica" - MASE), which aims to develop a **P**rocess based on an **I**Ntegrated and **E**CO-efficient system capable of covering the entire WEEE **R**ecycling value chain, from the identification and collection of WEEE (and/or its target components) and the associated pre-treatment processes, separation, purification and recovery processes, to industrial applications through the creation of prototypes. Based on innovative, multifunctional and eco-efficient technologies, the main objective for the PINECOR process is the recovery of the glass, siliceous fraction, plastics and metals from WEEE.

PINECOR focuses on the recovery of secondary raw materials from specific groupings of WEEE: R1, R2, R4. For small household appliances (R4), two processes have been tested for the separation of the product fractions (mainly metals and plastics): different types of dense homogeneous liquids and elutriation equipment.

The recovery of Cu through a chemical process of leaching and galvanic electrodeposition was also studied, using the same chemical solution for both phases. With regards to categories R1 and R2 the work focused on the recovery of glass from refrigerator shelves and washing machine portholes, introduced in the formulation of frits for ceramic glazes and in ceramic supports, with parameters comparable to standard formulations.

The collected and selected WEEE will be treated using an integrated approach, with innovative multifunctional eco-efficient technologies, for glass, plastic and metal recovery. To "close the loop", promising value chains will be developed through a qualitative study of eco-sustainable applications in specific industrial sectors, from the most common (i.e. ceramics, construction etc.) to those in expansion (e.g., green building). Groupings R1, R2 and R4

represent the growth drivers of annual e-waste collection volumes at national level (Table 1).

Last year, there was a decline in grouping R3 – TV sets and monitors which, after the exponential growth in 2021 and 2022 linked to the TV bonus, continued in the physiological downward trend and recorded a significant decline in 2023. Contrary to the trend for category R3, all the other four groupings show a positive growth instead. R1, R2, R4 could represent, within the WEEE groupings, the categories that would bring more environmental benefits (i.e., tonnes of CO₂ equivalent avoided) if properly processed and recycled: up to 1.64 tons of CO₂ equivalent avoided by the proper recycling of the R4 grouping, 1.40 tons for R1, followed by 1.05 tons for R2 (Remedia and IEFE-Bocconi University, 2017).

The WEEE pre-treatment stages within PINECOR are shown in Fig. 1: the input stream was sent to specific pre-treatment lines for WEEE grouping; the first phase was manual dismantling to extract the main components and sorting to separate and store the hazardous components, then the main components were sent to coarse grinding (up to 4 cm) to obtain the target fractions for the recovery processes. Five fractions, organized into samples, were obtained from the WEEE pre-treatment process carried out in the operating sites of the project partner *Treee*, according to their already standardized procedures.

The R1 and R2 streams glass from refrigerator shelves *TRE_PIN01* and washing machine portholes *TRE_PIN02*. A heterogeneous yielded mixture constituting mainly of plastics and metals, called *TRE_PIN03*, was extracted from small household appliances from grouping R4. Finally, after manual dismantling, shredding, magnetic separation and sieving of end-of-life photovoltaic panels (belonging to grouping R4), two fractions were obtained: *TRE_PIN04-BATCHA*, consisting mainly of plastics, glass fragments and metal residues, and *TRE_PIN04-BATCHB*, consisting mainly of glass from end-of-life photovoltaic panels. All these materials were then subjected to additional optimization processes of size reduction (0 - 0.5 mm for the glasses and 0 - 1 mm for the mixture by small household appliances) carried out by an external company in order to better prepare the outputs of the pre-treatment for the subsequent separation/valorisation steps.

Table 1. Percentage of WEEE collected from 2018 to 2023 per group and average weight of the total amount (source Annual Reports by CDC RAEE)

WEEE Groups	2018 (%)	2019 (%)	2020 (%)	2021 (%)	2022 (%)	2023 (%)	Average values
R1	27.1	27.2	26.5	25.9	27.4	28.9	27.2
R2	32.8	33.6	34.3	33.6	32.5	34.9	33.6
R3	19.2	17.4	17.0	19.8	19.7	13.8	17.8
R4	20.3	21.2	21.4	20.1	19.8	21.9	20.8
R5	0.6	0.6	0.7	0.7	0.7	0.5	0.6
tot	100.0	100.0	100.0	100.0	100.0	100.0	100.0

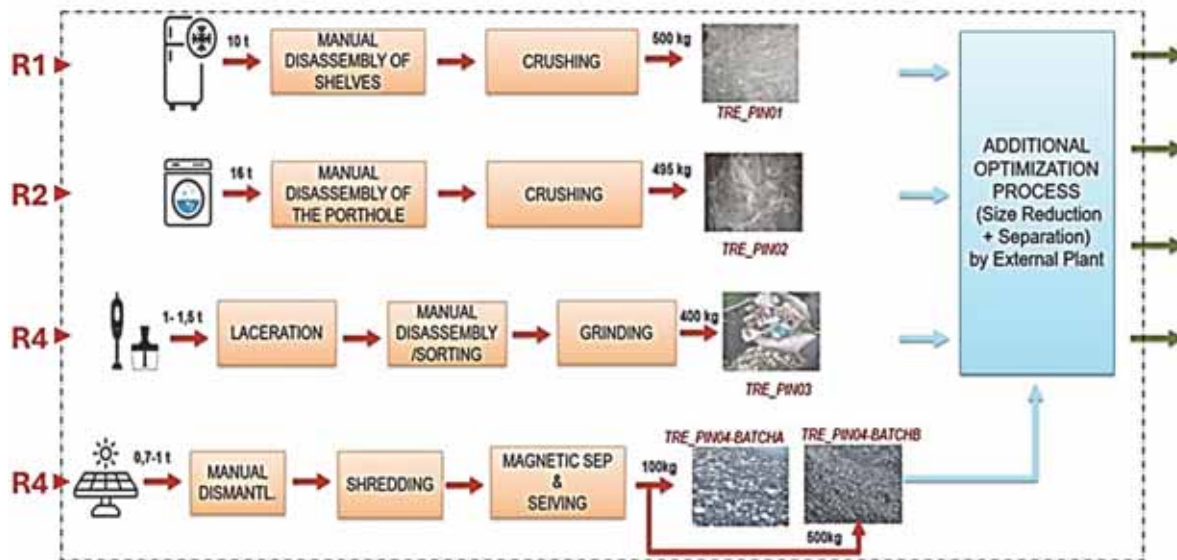


Fig. 1. Pre-treatment of WEEE target stream in PINECOR

2.2. Pinecor methods: separation and extraction process

Given to the heterogeneous nature of the starting material from *TRE_PIN03*, it was first necessary to develop a process for separating metal and plastic, using two approaches.

Firstly, a well-known separation method, the use of homogeneous dense liquids, which is now used in other fields, was adopted and adapted to this specific need to separate the two different fractions mentioned above. First of all, the real densities of the different plastics that made up the sample under investigation, as well as the densities of the metal fractions, were researched in the literature. It is obvious that the density of the latter was significantly higher than that of the plastics. Following the same principle, the densities of the most common saline solutions - potassium carbonate, soda, etc. - were researched in the literature until a saline solution was found with a density between that of the heaviest plastic and the lightest metal. Using the chosen saline solution, the metals (from the lightest to the heaviest) tended to sink during the application of this method, while the plastics (from the heaviest to the lightest) tended to float, allowing them to be separated. The solutions selected and tested were: NaOH 30% (density 1.31 g/cm³), NaOH 50% (density 1.50 g/cm³), K₂CO₃ 50% (density 1.54 g/cm³), ZnCl₂ 70% (density 1.88 g/cm³). Five g of *TRE_PIN03* are added to a beaker containing 200 g of each of the above solutions, the sample is for approximately 15 minutes using a mechanical stirrer and once the stirring is stopped, the separation obtained (metal on the bottom and plastic on the top) can be observed.

The second method tested, more performing, is separation by elutriation (Fig. 2). The laboratory apparatus (elutriator) separates the different particles of the sample according to their size, shape and density, using a flow of water in the opposite direction to that of sedimentation. The elutriator is equipped

with a hole at the bottom for the introduction of the water flow and 3 holes placed at different heights to separate materials with different specific weights (Fig. 2a). A constant flow of water is introduced from the bottom of the apparatus and the material is separated into four fractions according to its density. In detail, the experiment takes place in several steps: initially, the first hole from the top is used, from which the material with the lower density comes out, then the other two holes are used, from which material with a progressively higher density comes out. Finally, the material remaining at the bottom is the one with the highest density. The material is first weighed, then moistened with denatured ethyl alcohol to increase its wettability, and then fed into the machine. Smaller or lighter particles are carried to the top and higher density particles remain at the bottom and are then extracted. In the end, 4 samples are obtained, which are then characterized.

Plastics are dried and characterized by FT-IR, DSC and Melt Flow Index (MFI), and metals by Inductively Coupled Plasma (ICP). DSC and FT-IR are the most commonly used identification techniques for polymeric materials. DSC is mainly used for semi-crystalline materials because the melting point is visible, IR is used for all polymers, but in this case it is necessary to identify any amorphous ones and to integrate the results of the two techniques. As for FT-IR, 5 polymer particles per color extracted from each sample were analyzed in order to identify the different polymers constituents of the mixtures. As for DSC and MFI the mixtures coming from the three holes of the elutriator were ground in order to obtain a homogeneous material. Five replicates were carried out for each mixture.

One of the main objectives was to recover the metal fraction separated by elutriation (as an unseparated bottom body), following a process of chemical leaching and galvanic electrodeposition using the same solution consisting of hydrochloric acid (HCl) and copper chloride (CuCl₂). The recovery

of Cu took place in two steps: 1) Chemical leaching in an acidic environment: ca. 0.5 g of the *TRE_PIN03* metal fraction is placed in a 100 mL beaker together with about 50 mL of leaching solution. The beaker is placed on a magnetic stirrer with a heating table. The sample is kept stirred at a temperature of about 50°C, for a variable time; 2) Galvanostatic electrodeposition of Cu was carried out using an electrochemical cell with three electrodes: copper working electrode (WE); platinum counter electrode (CE); Silver – Silver Chloride, SSCE (Ag / AgCl / KCl_{sat}), reference electrode (RE). In particular, two electrochemical cell designs were tested (Fig. 3a-b). The copper working electrodes were analyzed by SEM-EDS to evaluate the composition and morphology of the electrodeposited metal. The aim of the final phase of the experiment was to recover the siliceous phase by treating the ground glass fractions belonging to the groupings R1 (glass from refrigerator shelves), R2 (glass from

washing machine portholes), and R4 (glass from photovoltaic panels) with a technology that would help to reduce energy costs. For these reasons, in order to extract the siliceous fraction from the selected target materials, a semi-industrial scale pilot plant has been used, which includes a reactor in which intense cavitation is applied to the reactive mass.

The innovation of this reactor lies in the fact that cavitation is generated by the rotation of a rotor with an ad hoc geometry inside a specific rotor. In this apparatus, there are no piezometric or magnetostrictive transducers, bringing advantages in terms of equipment simplification. The modulation of the cavitation intensity is achieved by varying the rotor speed and the operating pressure. This apparatus allows for the activation of the glass dissolution reaction in an alkaline medium starting from 50°C.

The system designed and implemented as a pilot plant (Fig. 4) consists of:

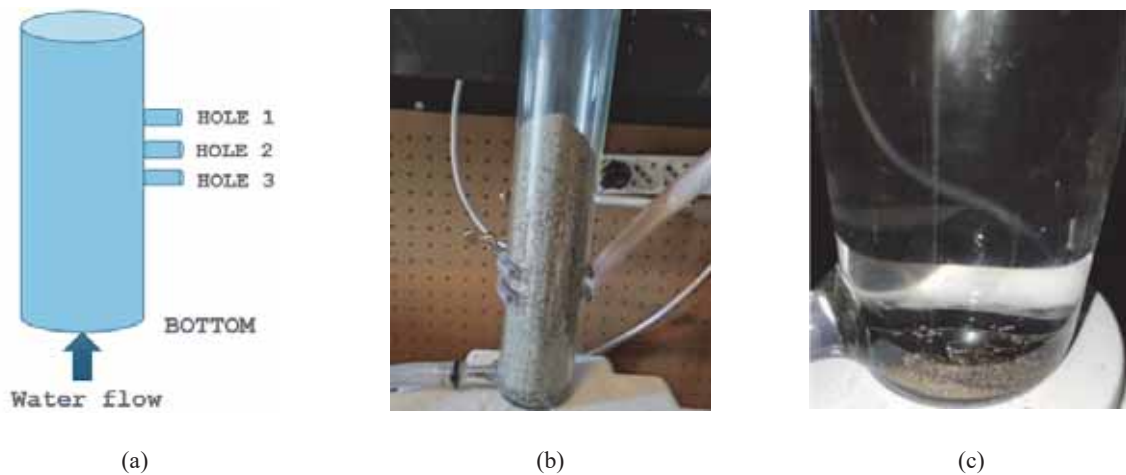


Fig. 2. Elutriator: (a) schematic representation; (b) before the separation test; (c) separated metal fraction on the bottom at the end of separation test

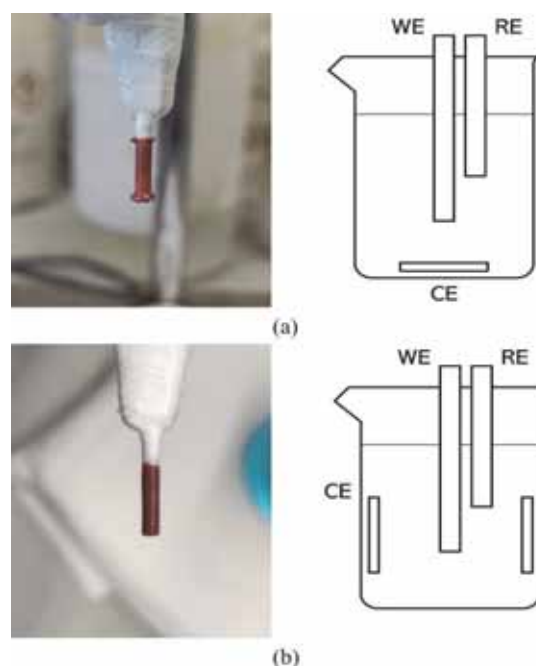


Fig. 3. First (a) and second (b) electrochemical cell configuration

- A thermostatic reactor with associated auxiliary circuit for temperature maintenance and a mixer to prevent sedimentation of the solid fraction during the process;
- Piping and pumping system for solution recirculation;
- Shock Power Reactor (SPR) cavitation system;
- Automatic control and command system via Siemens PLC.



Fig. 4. Pilot Plant for the recovery of the siliceous phase

The plant is equipped with sampling ports and can be operated in semi-automatic or fully manual mode. The main parameters considered during the test set-up phase were:

1. Type of glassy matrix and its composition;
2. Mass ratio: glassy matrix/water/NaOH in the reacting mixture;
3. Temperature inside the reactor;
4. Pressure in the recirculation circuit;
5. Residence time in the cavitation chamber;
6. Rotation speed of the SPR.

The tests were carried out by sequentially loading the liquid fraction (water + NaOH in the chosen proportions) into the reactor, and after starting the plant, the solid fraction was introduced. It was decided to progressively introduce small quantities of glass to allow the system to circulate the suspension gradually, in order to mitigate the high abrasive capacity of this type of matrix on surfaces (including AISI316 steel). This phenomenon is accentuated inside the SPR reactor.

3. Results and discussion

3.1. Separation and extraction process results

Figure 5 shows the results of the separation of the metal and polymer fractions of small household appliances of sample *TRE_PIN03* by dense homogeneous liquids, namely NaOH 30% (density 1.31 g/cm³), NaOH 50% (density 1.5 g/cm³), K₂CO₃

50% (density 1.54 g/cm³) and ZnCl₂ 70% (density 1.9 g/cm³). ZnCl₂ gave the best result due to its higher density: copper wires are deposited at the bottom, the polymer fraction appears at the surface and there is no material in the middle. This method was not applied to R4 photovoltaic panels.

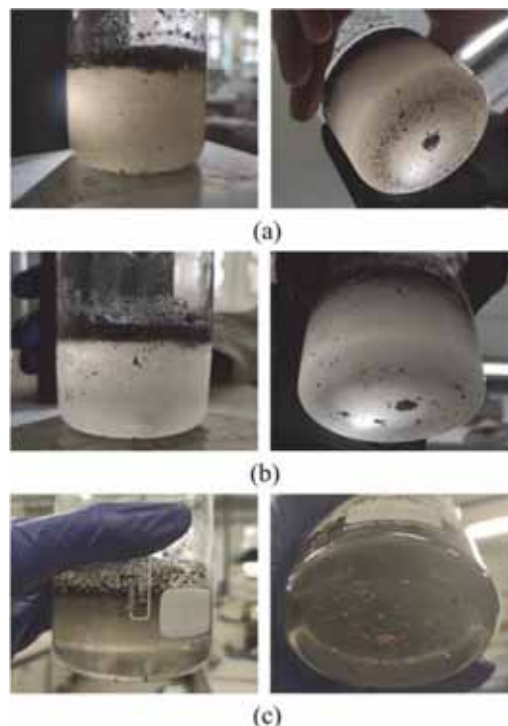


Fig. 5. *TRE_PIN03* size 1 mm (5 g) in 200 g of: (a) NaOH 30%; (b) NaOH 50%; (c) ZnCl₂ 70%

Table 2 shows the chemical composition of the metals recovered from the elutriation process, which allowed the separation of 28% of light plastics (such as polypropylene), 34% intermediate plastics (such as ABS), 10% heavy plastics (such as polycarbonate) and 4% mainly metals, with an in-process material loss of 24%. The percentages were calculated on the basis of the incoming sample weight. The most abundant metal is Cu followed by Fe, Pb, Sn and Al.

As regards plastic fractions obtained by elutriation, DSC analysis (Fig. 6), FT-IR analysis and MFI values are reported (Table 3). The DSC melting peaks identified are at 112 and 122°C corresponding to LDPE and HDPE; at 167°C associated with polypropylene (Fig. 6a). The T_g (glass transition) can be attributed to both polystyrene, and some blends such as SAN (styrene-acrylonitrile) and ABS (acrylonitrile-butadiene-styrene). In Fig. 6b polypropylene (melting peak at 162°C), PA6 polyamide (melting peak at 219°C) and ABS (T_g at 103.6°C) are identified. In Fig. 6c, the melting peak (164°C) is attributable to polypropylene or polyoxymethylene (acetal) (POM), while that at 217°C to PA6. The results of the thermal analysis are compatible and correlated with the MFI values. The MFI values range from about 3g/10min (high viscosity) to about 15 g/10min (low viscosity) (Table

3). The MFI value implies different processing conditions of the polymer, therefore the MFI values can be used to identify the technology of use of the material and provide information on heterogeneous polymeric masses. Low MFI values favour the use of extrusion technology, while high MFI values favour injection molding. The MFI is also an indirect qualitative measure of the molecular weight of the polymer and is also dependent on the degradation of the material. As the thermal degradation of the polymer increases, an increase in the MFI is also observed (due to the decrease in the molecular weight and therefore in the viscosity). In this case, the MFI values obtained identify a molded material that can be remolded. The FT-IR analysis was carried out on 5

different polymer particles chosen based on their color. Table 3 summarises the different polymers identified in the three fractions obtained from the elutriation process.

Table 2. ICP Chemical analysis of the metal fraction at the bottom of the elutriator from TRE_PIN03 sample

Metal	Concentration [mg/kg]
Cu	560.183
Al	8.008
Pb	22.232
Ag	2
Au	< 12.5
Sn	15.006
Fe	82.897

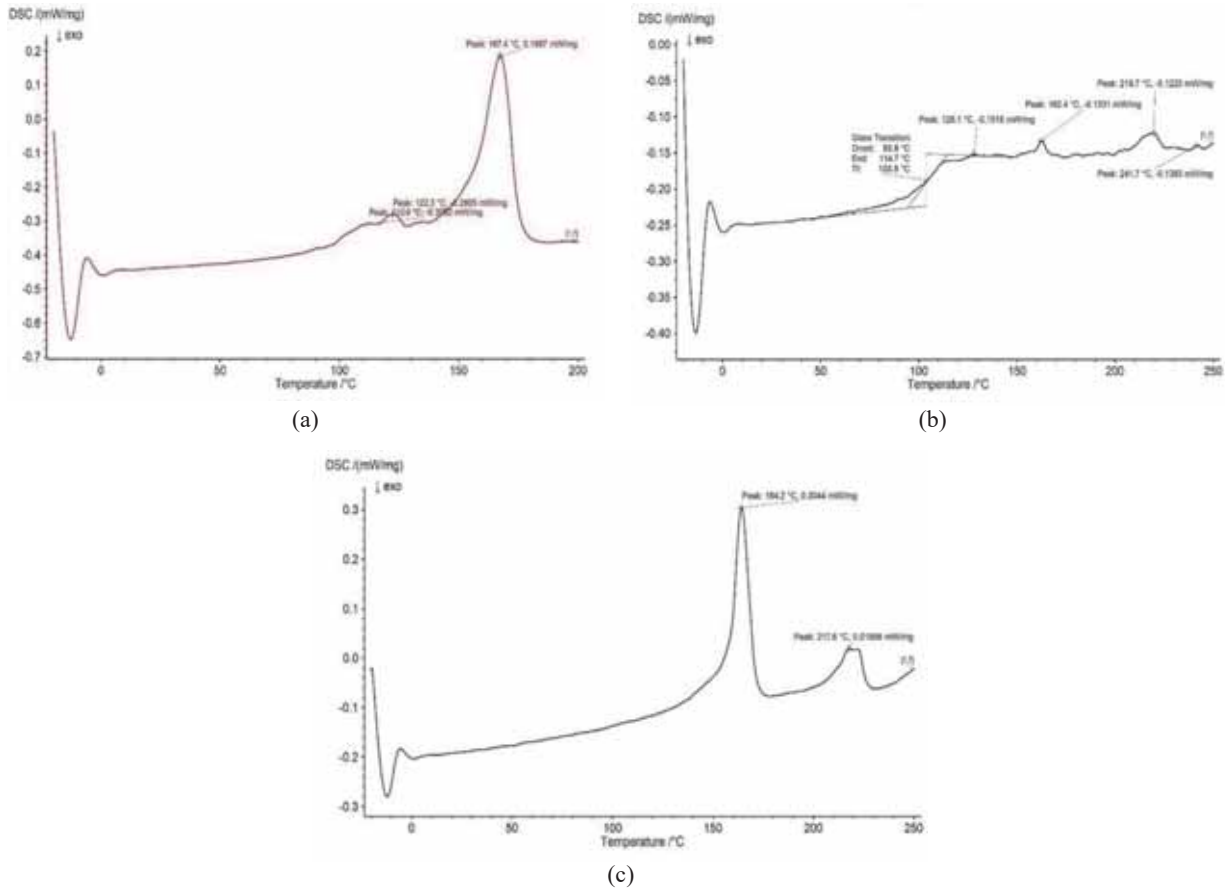


Fig. 6. DSC analysis: (a) light weight fraction (hole 1) of polymers separated through elutriation process; (b) medium weight fraction (hole 2) of polymers separated through elutriation process; (c) heavy weight fraction (hole 3) of polymers separated through elutriation process

Table 3. Main polymers found per hole according to the FT-IR Analysis and MFI values measured on the fractions

Hole 1 – lightweight plastic *MFI 15.3g/10min	Hole 2 – medium weight plastic *MFI 7.5g/10min	Hole 3 – heavy plastic *MFI 3.2g/10min
Polystyrene (density about 1.050 g/cm ³)	Polypropylene (PP) (density about 0.855 g/cm ³ - 0.946 g/cm ³)	Polycarbonate (PC) (density about 1.200–1.220 g/cm ³)
Polypropylene (PP) (density about 0.855 g/cm ³ - 0.946 g/cm ³)	Acrylonitrile butadiene styrene (ABS) (density about 1.060–1.080 g/cm ³)	Polyoxymethylene (acetal) (POM) (density about 1.410–1.420 g/cm ³)
Styrene acrylonitrile (SAN) (density about 1.080 g/cm ³) Low-density polyethylene LDPE (density about 0.94 g/cm ³) High-density polyethylene HDPE (density about 0.96 g/cm ³)	High-density polyethylene HDPE (density about 0.96 g/cm ³)	Polyamide (PA 6) (density about 1.120 g/cm ³ – 1.500 g/cm ³)

Regarding the copper recovery, careful work was carried out to optimize the etching and deposition parameters in order to obtain a controlled morphology and composition of the deposited copper. In particular, the second electrochemical cell configuration tested made it possible to obtain a more homogeneous and smooth morphology (compare Fig. 3 a-b). The optimization of the concentration of the leaching solution and electroplating current density allowed to obtain the deposition of pure copper without chlorine contamination with a high faradaic yield (45%). The absence of chlorine in the deposit is an important result considering the high concentration of chloride ions, which strongly complex the copper ions in the leachate. The optimization work made it possible to achieve the rate of 50 mg/h for leaching and 10 mg/h for galvanic electrodeposition with homogeneous morphology. The results of the SEM-EDS (micrography and chemical semi-quantitative analysis) are shown in Fig. 7.

For the extraction of the siliceous fraction from the glasses of refrigerator shelves *TRE_PIN01*, washing machine portholes *TRE_PIN02*, and photovoltaic panels *TRE_PIN04-BATCHB*, the effects of different parameters on the process outcome were

analyzed:

- Glass matrices: no different reactivity is observed depending on the type of glass fraction treated;
- Effect of the glass matrix/water/NaOH mass ratio in the reaction mixture: the role of water is mainly to facilitate the transport of the mixture, while the minimum amount of NaOH to activate the process is around 20% by weight. The optimum mass ratio of glass to reaction liquid is approximately 1:2;
- Effect of temperature: tested temperatures ranged from 25°C to 65°C. This variation does not significantly affect the reaction yields, indicating that temperature is not the critical process parameter;
- Effect of pressure in the recirculation circuit: the effect of pressure was tested with a variation between 2.5 bar/6 bar, with slight evidence of increased yield with increasing pressure;
- Effect of the residence time in the cavitation chamber: the experimental data obtained show that the reaction initiation is rapid (a few seconds of contact in the cavitation chamber are sufficient for initiation) and that the maximum glass solubilization state is quickly reached. Once this concentration limit is reached, the residence time becomes irrelevant for further increase;

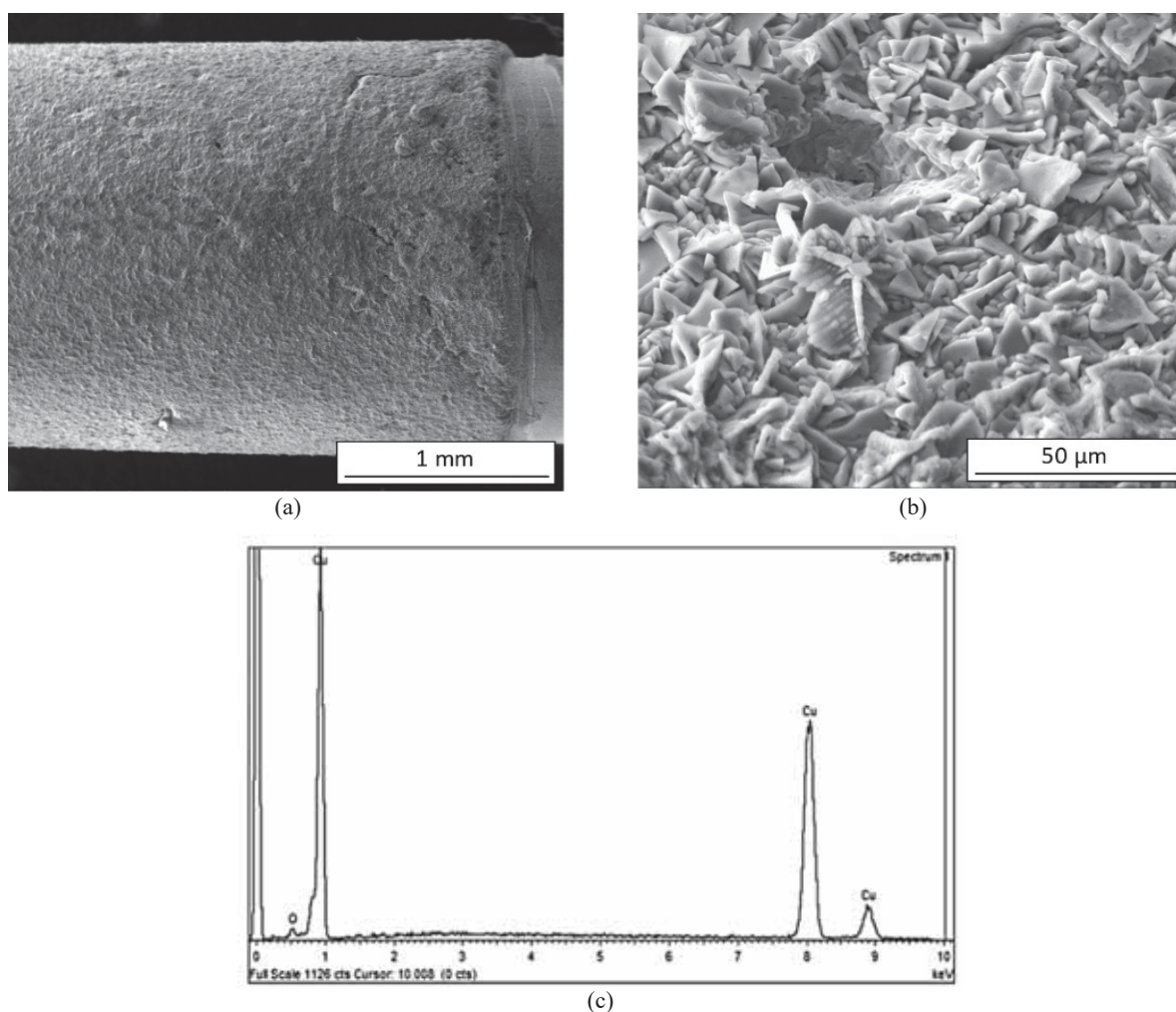


Fig. 7. SEM-EDS results: (a) and (b) Micrographs at different magnification of electrodes subjected to galvanic copper plating in a direct current procedure; (c) EDS spectrum of the electrode subjected to direct current electroplating

- Effect of the SPR rotation speed: both the ignition speed and the reaction yield depend on the speed of the cavitation rotor. Below 3000 revolutions per minute, rpm, the reaction does not take place.

During the pilot plant phase, various semi-quantitative analytical routes were also tested to speed up the identification of the sodium silicate fraction in the solution. In addition, the experiment is verified that the reaction appears to be "blocked" after an initial glass solubilisation phase. It was found that the soluble glass fraction increases over time without the application of any thermal or mechanical stimulation.

This seemingly inexplicable phenomenon is explained by interpreting the mechanism of glass dissolution in sodium hydroxide solution. Glass is a polymeric structure obtained from the reaction of silica, boric anhydride, and alumina with alkali and alkaline earth metal oxides at a temperature of at least 1350°C. Attack of this structure by sodium hydroxide progressively removes the $(\text{SiO}_3)^{2-}$ anion to form soluble silicate. The remaining solid polymeric structure becomes unbalanced and attempts to rearrange itself by expelling more silica and forming new bonds with the ions present. The system operates at a low temperature, around 50°C, which makes the rearrangement kinetics very slow. In any case, glass treatment with NaOH in the SPR reactor is worthwhile, even though all the process parameters still need to be optimized. The "standard" treatment of glass with NaOH to obtain soluble silicates is carried out at a minimum temperature of 220°C with a minimum contact time of 2 hours at a pressure of 25 bar. This treatment is carried out in internationally industrialized processes. The reaction results in a mass composed of: a water-soluble liquid fraction, an intermediate fraction of gelatinous consistency and a solid fraction composed mainly of a white compound with residues of unreacted glass.

Finally, sodium silicate is present in the soluble fraction obtained from the glass treatment. Its presence and quantity have been indirectly verified by precipitation through hot treatment in the presence of a safe excess of sulfuric acid. Sodium silicate is converted into silica and sodium sulphate. Any other cations present, bound to the silicate anion, are also converted to sulphates. Washing removes salts from the silica. Precipitated silica was analysed by SEM-EDS before and after the S removal purification process (Table 4). The unpurified silica precipitate

contains S residues derived from sulfuric acid (Fig. 8). The analysis shows the successful removal of S by washing and the prevalence of SiO_2 .

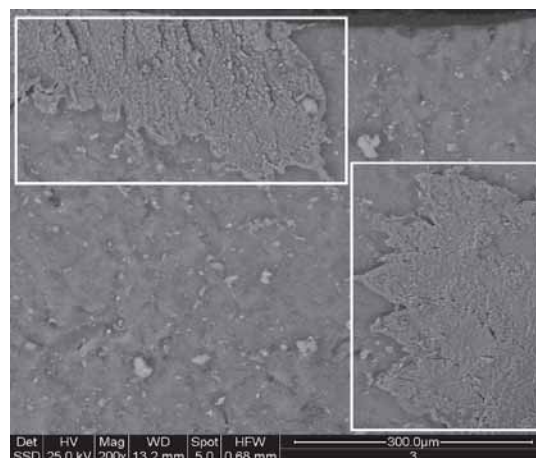


Fig. 8. Micrographs acquired by ESEM scanning electron microscope of precipitated silica before purification. The highlighted areas at the top left and bottom right are rich in S (approx. 22 weight%), the remaining area contains trace amounts of S (approx. 2 weight%)

3.2. Applications in ceramic sector of glass fractions

One of the most promising sectors for glass recovery is the ceramics sector. In fact, the sales of tiles in Italy in 2022 are estimated at 458 million square meters and this important market may incorporate a large amount of WEEE glass (Endhoven, 2023). In order to better address the research on the use of glass in the ceramic sector (both glaze and support), *TRE_PIN01* and *TRE_PIN02* were characterized by chemical (XRF and ICP), thermal (DTA and heating microscope), thermomechanical (Dilatometry) and mineralogical (XRD) point of view.

Table 5 shows the chemical composition of the glasses and Table 6 summarizes the characteristic temperatures and the expansion coefficients (EC) of the glasses that were identified by DTA, heating microscope and dilatometric analysis. From the chemical analysis it is clear that the refrigerator shelf glass (*TRE_PIN01*) is richer in alkaline earth metal oxides than the washing machine portholes glass (*TRE_PIN02*) and both glasses have a low content of chromophore components. Moreover, sample *TRE_PIN02* contains a small amount of B_2O_3 .

Table 4. SEM-EDS Chemical analysis of precipitated silica from washing machine portholes *TRE_PIN02* glass pre and post purification

Element	<i>Precipitated silica before purification</i>	<i>Precipitated silica after purification</i>
	Weight%	Weight%
O	46.97	59.57
Na	22.36	2.44
Al	0.32	1.08
Si	11.39	36.60
S	18.96	0.31
Totals	100.00	100.00

DTA analysis show that the two glasses have similar thermal behavior, they are stable glasses that do not crystallize (absence of exothermic phenomena). XRD analysis shows that the two glasses are completely amorphous, with no crystalline phases present.

The research was based on replacing a percentage of the atomized mixture with WEEE glass, rather than replacing a single raw material in the mixture. This is a valid solution especially for factories that do not have a complete ceramic cycle, but start directly from the atomized product prepared by third parties. The percentages of substitution tested were: 2.5%, 5%, 10%, 15%, 20% respect to the solid content. In particular, the study discusses the results on the sintering process (total porosity) and on the technological properties (WA%, LS%).

The products obtained were characterized and compared with the standard (atomized without replacement) to verify the variability of the physico-mechanical properties and the applicability of the processes (Table 7). The tests were carried out on samples prepared by tile-making process on a laboratory scale, but the firing was performed in a ceramic roller kiln with the industrial cycle (1200°C, 48 min). The effect of the amount of WEEE glasses into the porcelain stoneware body does not affect the WA%, in fact there are no significant variations with respect to the reference (standard support) and in any case all the compositions are below the 0.5% limit corresponding to highly sintered ceramic tiles (Group Bla).

As for the linear shrinkage, it increases by about 2.6% in the composition with *TRE_PIN01* glass (15 wt%) and 11% with *TRE_PIN02* glass up to 10 wt%, then it drops to values lower than the standard, indicating a decrease in densification, certainly due to an increase in the glassy phase present. The effect on porosity is positive up to 10 wt% of inserted glass for all two glass-based samples. Mechanical tests were carried out according to UNI EN ISO 10545.4 and the values reported are the average of five samples.

Although the samples containing glasses have lower values than the reference, their mechanical properties are higher than those prescribed by the EN

ISO standard for highly sintered commercial tiles (Bla group: $WA \leq 0.5\%$, $MOR > 35$ MPa) and can be considered as porcelain stoneware. The data of the compositions with the highest (20 wt%) glass content are not available (n.a.), as these samples showed very high porosity values compared to the standard. As porosity affects the mechanical properties, a further reduction in the value of the modulus of rupture was to be expected.

Other experiments investigate the effect of *TRE_PIN01* and *TRE_PIN02* in frits used for different ceramic tiles glaze formulations (glaze 1 and glaze 2). These formulations can contain up to 30% by of *TRE_PIN01* and *TRE_PIN02*.

In order to compare the effect of the presence of *TRE_PIN01* and *TRE_PIN02* in the glaze compositions with respect to the industrial standard formulation, solar reflectance (SR) measurements were carried out on the finished glazed tiles and characteristic temperatures were determined using a heating microscope. The expansion coefficient (EC) was determined by dilatometric analysis. The results are shown in (Table 8).

In both cases (incorporation of WEEE glass in support and glazes), the characterizations demonstrated the feasibility of the process and the substantial equivalence of the properties of standard and glass-based products. WEEE is a problem, but it is also a good opportunity to contribute to the self-sufficiency of the economic policy of those countries, such as Italy, that are poor in virgin or strategic raw materials.

This waste can be recycled and reused if it is managed effectively. Furthermore, improving the collection, treatment and recycling of the electronic equipment at the end of its life can increase resource efficiency and support the transition to a circular economy. It can also contribute to the security of supply of critical raw materials and ultimately strengthen the EU's strategic autonomy. The "PINECOR – ECO-efficient Integrated Process for WEEE Recovery" project aimed to develop innovative solutions capable of optimizing the recovery of glass and metal and siliceous fractions deriving from WEEE recycling.

Table 5. Chemical composition (wt% oxide) of *TRE_PIN01* and *TRE_PIN02* glasses

	<i>SiO₂</i>	<i>TiO₂</i>	<i>Al₂O₃</i>	<i>Fe₂O₃</i>	<i>MgO</i>	<i>CaO</i>	<i>Na₂O</i>	<i>K₂O</i>	<i>SO₃</i>	<i>B₂O₃</i>
TRE_PIN01	73.11	0.02	1.13	0.05	3.37	8.86	13.20	0.37	0.20	-
TRE_PIN02	72.52	0.02	2.06	0.04	0.79	8.34	12.11	1.05	0.15	2.81

Table 6. Characteristic temperatures and expansion coefficient (EC) of *TRE_PIN01* and *TRE_PIN02* glasses

	<i>Glass transition (°C)</i>	<i>Sintering (°C)</i>	<i>Softening (°C)</i>	<i>Sphere (°C)</i>	<i>Half sphere (°C)</i>	<i>Fusion (°C)</i>	<i>EC (1/K)</i>
TRE_PIN01	573	708	749	1028	1049	1079	$10.6 \cdot 10^{-6}$
TRE_PIN02	574	709	837	978	1064	1107	$10.5 \cdot 10^{-6}$

Table 7. Experimental values of water absorption (WA %), linear shrinkage (LS %), total porosity (TP %) and Modulus of Rupture (MOR, MPa) of WEEE glass containing support and standard support

	WA %	LS %	TP %	MOR (MPa)
Standard Support	0.003	7.49	7.41	64.16
2.5% TRE PIN01	0.002	7.50	7.20	55.62
5% TRE PIN01	0.001	8.02	7.45	50.39
10% TRE PIN01	0.001	7.99	8.15	46.77
15% TRE PIN01	0.0005	7.69	9.84	47.10
20% TRE PIN01	n.a.	n.a.	n.a.	n.a.
2.5% TRE PIN02	0.003	7.50	7.50	57.84
5% TRE PIN02	0.001	7.86	7.83	53.66
10% TRE PIN02	0.001	8.32	7.99	54.41
15% TRE PIN02	0.001	7.19	8.90	47.76
20% TRE PIN02	n.a.	n.a.	n.a.	n.a.

Table 8. EC, Characteristic Temperatures, Solar Reflectance (SR) values of standard formulations (STD) and respective WEEE-glass containing ones. Solar reflectance (SR) was measured according to ASTM C1549

	EC 10 ⁷ 1/K	Softening temperature °C	Sphere temperature °C	Half sphere temperature	SR
STD glaze 1	66	830	1030	1130	0.856
TRE PIN01 containing-glaze 1	82	951	999	1084	0.853
STD glaze 2	66	830	1030	1130	0.740
TRE PIN02 containing-glaze 2	65	828	1031	1127	0.754

The aim was to reduce waste and increase the recovery of secondary raw materials coming from the "dry mechanical treatment" of WEEE rich in glass free of toxic metal oxides, with high reuse potential, collected in groupings R1 (cold and climate), R2 (large whites), R4 (small appliances and other, including photovoltaic panels). The process used is integrated and eco-efficient, combining mechanical and chemical approaches to promote the reuse not only of the main fractions obtained from the recycling of end-of-life products, but also of the residues which are currently sent for disposal, which represents an additional cost. The partnership between the world of research (UNIMORE) and industry (TREEE srl, the leading Italian group in the WEEE sector, and TRE EFFE Forniture Idrauliche Industriali srl), and with the involvement of companies that have shown interest in the project, has allowed a continuous comparison between the academic and industrial worlds, which has materialized in the development of prototype of processes and products illustrated below.

The results obtained confirmed the multifunctional character of the PINECOR process; certainly, from a PINECOR perspective, shredding is a phase that should be included in the standard pre-treatment processes for WEEE. Furthermore, some intermediate fractions of the pre-treatment (such as the mixed plastics and metals from R4) require further research on the technology to reduce them to millimetres.

Glass from fridge shelf (*TRE_PIN01*) and washing machine (*TRE_PIN02*) has been used as a raw material both in the formulation of glazes and traditional ceramic supports and in the production of tiles for cool roofs (cold roofs with high reflectivity), which are designed to keep the building cool, counteracting the phenomenon of the so-called island

of heat, as evidenced by the development of laboratory demonstrators. Semi-industrial trials are currently underway.

More problematic was the management of the mix of small household appliances (*TRE_PIN03*), a very heterogeneous sample which required a study of the separation of the different product fractions as a preliminary phase for the development of parallel recovery processes for the different product fractions obtained, in this case plastics and metals. Two physical separation processes were tested: separation by dense homogeneous liquids and elutriation. With regards the first, solutions of ZnCl₂, K₂CO₃ and NaOH were used to separate the materials by density difference: the ZnCl₂ 70% solution, in contact with 1 mm ground *TRE_PIN03*, gave the best results in terms of separation. However, the most effective process was elutriation, which allowed the separation of four different fractions of plastics and metals based on size, shape and density. The isolated metal fraction was then used to develop a process for the recovery of copper, a material for which this process is economically justifiable since it is present in non-negligible quantities in WEEE, through the development of an eco-sustainable chemical leaching process that does not use toxic or carcinogenic reagents and followed by deposition in the solid state through a galvanic process that can be carried out in the same leaching tank, thus maintaining the same chemical solution for both phases.

Regarding the plastic fraction, the following three fractions have been identified: light plastics for deep hole drilling such as polystyrene (PS), polypropylene (PP), polyethylene (LDPE and HDPE), styrene acrylonitrile (SAN); medium molecular weight plastics such as polypropylene (PP), acrylonitrile butadiene styrene (ABS); heavy plastics

such as polycarbonate (PC), polyoxymethylene (POM), polyamide 6 (PA6).

With regards to the experimental phase for the recovery of the siliceous matrix, the initial project was aimed to use a widespread technology, considered to be "standard", which consists of treating the glass with sodium hydroxide using a pressurized reactor (25 bar), bringing the product to a minimum temperature of 220°C, with a minimum contact time (only in the peak phase of the reaction) of 2 hours. This type of treatment is already used in processes that are now industrialized at an international level.

Therefore, during the design phase, it was considered to test other less known methods, but more in line with the ecological transition now necessary aimed at researching more sustainable and safer technologies for both operators and the environment. The operational phase focused on pilot-scale tests carried out on an SPR (Shock Power Reactor) plant using the cavitation principle. Although the objective set was not fully achieved during this experimental phase, despite the diversification of the tests carried out, the treatment of glass with soda in the SPR reactor is a system on which studies and analyzes of the recovery variables of glassy matrices of WEEE origin will continue. However, the PINECOR project has made it possible to study many details and aspects that are inevitably an integral part of a technological evolutionary process.

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PHOTOVOLTAIC PANELS RECYCLING TO CREATE SILICON VALUE CHAIN: PARSIVAL PROJECT

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Abstract

Apulia in Italy and Extremadura in Spain are regions characterized by an intensive installation of photovoltaic (PV), which are expected to generate a large amount of PV waste in the next 15 years (~300k tons in Apulia and ~380k tons in Extremadura) but there are no dedicated PV recycling plants in these areas. PARSIVAL aims to solve this problem by proposing PV refurbishment and End-of-Life (EOL) PV recycling technologies.

The PARSIVAL technology for recycling, one of the most advanced in Europe, is able to recover all the valuable materials contained in PV panels (aluminium, glass, copper ribbons, silver, silicon PV cells), but further research is needed to refine the recovered materials in order to commercialize them and, in particular, to find a final market for the recovered PV cells. The latter contain mainly silicon that is a Critical Raw Material, but the presence of paste of aluminium and silicon nitride hinders its reuse. Therefore, the project is also investigating the most profitable ways to valorise silicon, which can be recovered from PV waste, in three different applications (Li-ion batteries, ferroalloy, and aluminium industry). In addition, PARSIVAL is evaluating the feasibility of a refurbishing and recycling plant in Apulia and the replicability of the results in Extremadura contributing to the creation of refurbishment and recycling networks and professionals, through Higher Education Institutions (HEIs) in these areas. The project results were promising: both refurbishment and recycling processes have been validated and the recovered silicon was successfully tested in the addressed applications.

Key words: photovoltaic, recycling, refurbishment, silicon

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1. Introduction

The world's total installed PV capacity exponentially increased since 1990: it reached 1 TW by the end of 2022 (IRENA, 2023) and is expected to rise further to 1.6 TW by 2030 and 4.5 TW by 2050 (Weckend et al., 2016). The European Photovoltaic Industry Association predicted that PV energy might contribute up to 12.6% of the world's electricity needs by 2040, implying that significant amounts of PV modules will be installed in the future.

Taking into account that the mean lifetime of PV panels is 25 years and considering the dramatic growth experienced in the PV industry since 2000 (Chowdhury et al., 2020), significant quantities of photovoltaic panel waste will be produced within a few years and will continuously increase in the future. It has been predicted that the worldwide solar PV waste could unlock between 1.7 and 8 million tons of raw materials (glass, Si, Ag, Cu, Al) by 2030, rising further to about 78 million tons by 2050 (Weckend et al., 2016). Therefore, recycling PV waste for recovering valuable materials, while minimizing the waste generated, is becoming relevant for the solar energy industry. Furthermore, in recent years, recyclers have noticed that the real lifetime of PV modules is even less than 15 years (Artas et al., 2023; Libra et al., 2023).

The PV module performance can degrade due to several factors, such as UV irradiation, temperature, humidity, mechanical shock, local pollutants and toxic fumes, and this degradation leads to premature replacement of the modules (Ndiaye et al., 2013; Saneie et al., 2024). Another problem is that around 40% of photovoltaic modules, although functioning, are replaced due to poor efficiency or only due to the presence of small, easily repairable damages. All these factors contribute to the generation of huge volumes of PV waste.

The disposed PV panels are typically landfilled although these devices could keep working if properly refurbished. At the moment, refurbishment is never performed and only 10% of EoL PV panels are recycled worldwide: this causes a significant environmental impact (Lunardi et al., 2018). Moreover, the only recycling approach for EoL PV panels at industrial scale is a shredding-based mechanical treatment, which recovers aluminum from frames, copper from wires and glass granules from shredded mixture sieving. This downcycling of PV waste causes the loss of important raw materials like silver and silicon contained in PV cells. These two materials represent over 60% of the total economical value of the material in a photovoltaic module (Peplow, 2022), and therefore with mechanical shredding, only 35% of the theoretical economic value can be extracted.

Many EU countries like Germany, Italy, France and Spain, where the installation of PV is concentrated, are starting to address the problem of PV recycling. This problem will become even more acute

in the next few years, especially in some regions of RIS (Regional Innovation Scheme) countries, where many photovoltaic systems have been installed without the presence of advanced recycling plants.

Apulia (South of Italy) is the second region in Italy for installed PV power capacity (2.9 GW, 13.4% of the national installed capacity) and it is a RIS region where the issue is particularly relevant: ~300k tons of PV waste will be collected in the next 15 years. The case of Extremadura, with 3.9 GW of installed capacity (23.4% of the PV solar energy of the country), is similar. In both regions, there are no recycling plants yet, so there is an urgent need to establish a structured value chain to recycle and reuse the valuable materials contained in PV.

The expertise and technologies created by the PARSIVAL consortium can address this issue. The project aims to: i) assess the feasibility of establishing a waste PV refurbishment and recycling plant in Apulia (IT); ii) close the silicon value chain by the analysis of applications for the silicon recovered from PV; iii) build up a solid network able to support PV panels recycling; iv) exploit results in Extremadura; and v) increase the skills of students in Apulia on the themes of recycling, recovery, reuse and substitution of CRM. The novelty of this work is significant, as PARSIVAL is not only exploring two innovative technologies for refurbishment and recycling, but it is also one of the few studies investigating the specific applications for PV cells recovered from end-of-life PV panels.

In this work, the results of the first year of the project are presented: the analysis of PV waste stream in Apulia and Extremadura; tests with refurbishment and recycling technologies; application of recovered silicon in aluminium industry; production of ferroalloys and anodes of batteries with recovered silicon.

2. Material and methods

2.1. Value Chain analysis of PV panels in Apulia and Extremadura

2.1.1. Installation analysis in Italy and Apulia

Information regarding the consistency of installed photovoltaic systems in Italy is scattered. The most reliable sources include data collected and published by the Gestore dei Servizi Energetici (GSE) and made public through the Atlaimpianti web interface. Reconstructing the historical series of installed systems is achievable using data from Terna's Gaudi portal, which is accessible thanks to sector associations. The data collected and used for the reconstruction ranges from 2005 to 2021, and takes into account information such as the number of systems and nominal installed power.

2.1.2. Installation analysis in Spain and Extremadura

The analysis of PV installations in Spain and the Spanish region of Extremadura is based on a

bibliographic study, but also on the Pretor online tool, developed by the Spanish Ministry for Ecological Transition. This tool allows to identify all renewable energy licenses in Spain by region. In the specific case of Extremadura, the year of installation has been identified, but not in other regions. As an alternative, the data for the whole of Spain were obtained through several bibliographies (Heras-Saizarbitoria et al., 2011).

Data on self-consumption in Spain are based on the bibliography, but information for Extremadura is lacking due to the absence of official data on self-consumption installations by region. All data for Extremadura and Spain are organized by year, starting from 2004, when PV energy was introduced in Spain, up to 2023, to calculate the cumulative installed power for each year in these region and country. After a detailed analysis of the situation, the main periods of growth and stability caused by specific events, can be identified. This anticipates the behaviour of PV waste production in the study areas.

2.2. Photovoltaic refurbishment and recycling

2.2.1. Refurbishment

The repair and reuse of photovoltaic modules is an important topic because it avoids the production of new modules and therefore helps avoid significant greenhouse gas emissions and the extraction of rare or critical materials. The diagnostic and repair technologies for modules that help extend module life were developed by CEA and tested on several modules. Some PV modules (ref. Solarworld Sunmodule plus SW270 from 2017) were repaired and qualified in semi-industrial configuration.

The following operations were carried out on the different modules:

1. cleaning and visual inspection of the modules;
2. change of the three diodes of the modules;
3. verification of electrical performance by flash test;
4. checking the correct functioning of the cells by electroluminescence;
5. wet leakage test.

The wet leakage current test is an electrical

withstanding test carried out on electrical appliances to evaluate the electrical isolation of the housing. The test is performed by immersing the appliance in water with one cable connected to the appliance's electrical wires and the other cable connected to the water.

2.2.2. Recycling

Photovoltaic modules that cannot be refurbished must be valorized in a different way, and recycling should be the preferred one.

The recycling technology developed by 9-Tech (Mazzi et al., 2024), shown in Fig. 1, was implemented in the PARSIVAL project. This technology can present significant advantages over existing solutions in terms of quality of recovered materials (Cerchier et al., 2022).

During the project, some tests were performed on different PV panels in the TRL6 pilot plant. The process begins with the manual removal of the aluminium frame and junction box, followed by a thermo-mechanical treatment to remove the polymeric fraction, and delaminate the PV panel. Specifically, the PV modules undergo a controlled heating to fully combust the EVA encapsulant efficiently; during this step the temperature of the chamber exceeds 400°C. After the heating treatment, a machine removes the copper wirings, and the material is firstly sieved with a 2 mm perforated sheet. Finally, the mechanical separation of the PV cells from glass is carried out and PV cells are recovered in form of pieces with dimensions of 1-20 cm².

Glass, copper ribbons, and PV cell are the end products of the process. Silicon, the primary material in PV cells, is recovered from PV panels in the form of fragments. These fragments contain not only silicon but also other functional materials such as silicon nitride, aluminium and silver, which are an integral part of the functioning of the cell. While these materials contribute to the functioning of the PV cells, they pose significant challenges in the recycling process as they are considered impurities for further applications of silicon. The PV cells, before their use in the different applications, undergo hydrometallurgical treatment to remove silver from the surface of PV cells and recover it.

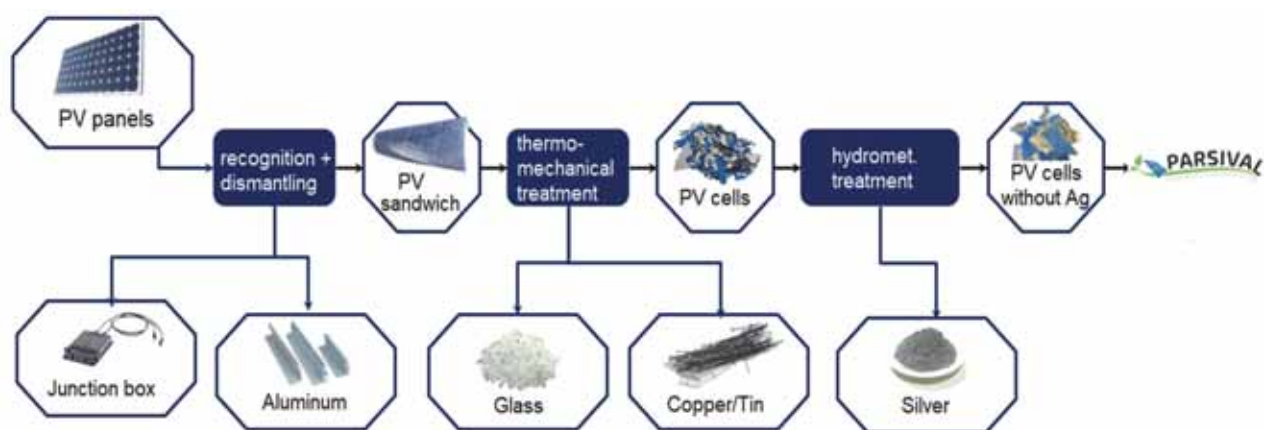


Fig. 1. 9-Tech technology process flowchart

2.3. Reuse of silicon silicon PV cells without silver

2.3.1. Aluminium industry

The use of PV cells has been investigated in aluminium industry, with the advantage that aluminium, commonly present in PV cells, does not represent an impurity. Despite this advantage, the integration of recycled silicon into aluminium alloys, has achieved limited success so far. In fact, previous melting tests reveal poor solubility and yield, attributed to several factors including oxidation, the presence of silicon nitrides which limit wettability, and inadequate mixing with the melt. To address these challenges, experiments have been conducted testing different binders and additives to improve the solubility of silicon in aluminium alloys.

2.3.2. Ferroalloy production with STILLMETAL process

The use of PV cells without silver has been investigated as reagent in STILLMETAL process for the ferroalloy production. STILLMETAL is an upscaling project funded by EIT RawMaterials (2022-2024) and proposes a process to solve the problem of the steel “white” slag, which is considered a waste and must to be disposed of in landfill. STILLMETAL process involves the reaction in the molten state, at high temperatures, between white slag and specific reagents to produce a valuable metal, containing silicon with the composition of a Ferroalloy (FeSi), and a residual oxide slag that can be used in cement industry. The reaction was firstly simulated with Thermodynamic Software Fact Sage 8.2, using FSstel, FTmisc and FToxid databases. Numerical simulations were carried out in order to obtain a FeSi with an amount of Si higher than 40%, and a residual oxide slag with low melting temperature. For the simulation the white slag compositions reported in Table 1 and the following composition of PV cell were used: 90% Si and 10% of aluminium paste.

The results of the simulation were experimentally validated in TRL5 pilot plant (an induction furnace with stirring). The materials were mixed inside a bucket and then put inside a crucible of graphite with the external part of ceramic. During the test the temperature of the system was measured by a pyrometer positioned above the crucible and the data were recorded from the PC connected to the probe. The solidified materials, recovered inside the crucible the day after the tests, were characterized with SEM-EDS analysis using a Cambridge Stereoscan 440 electron microscope equipped with a Philips PV9800 EDS.

2.3.3. Li-ion anode production

In recent years, natural graphite has been extensively utilized as the anode material for lithium-

ion batteries. However, the EU has recently designated natural graphite as a CRM, prompting recommendations for its partial or complete replacement with non-critical or end-of-life materials. Silicon emerges as one of the most promising alternatives to replace natural graphite.

Silicon has the capacity to form lithium alloys, boasting a theoretical specific capacity of 4200 mAh g⁻¹ (Huggins, 1999), significantly surpassing the theoretical specific capacity of graphite (372 mAh g⁻¹) and metallic lithium (3800 mAh g⁻¹).

The primary challenge of utilizing silicon as an anodic material lies in its volumetric expansion, which can reach up to 300% during battery charge-discharge cycles (Rahman et al., 2021). This substantial volumetric variation leads to progressive fragmentation and active material loss, resulting in a rapid decline in accumulated capacity.

Furthermore, the recent categorization of silicon as a strategic material by the EU, coupled with the high environmental impact of silicon production from SiO₂, underscores the importance of its recovery and recycling, particularly from end-of-life products such as photovoltaic panels (Blömeke et al., 2023; https://single-market-economy.ec.europa.eu/sectors/raw-materials/areas-specific-interest/critical-raw-materials_en).

Within PARSIVAL Project, a process has been developed to recover silicon-based powder suitable for reuse as anode material for (lithium ion batteries) LIBs. Two samples of silicon PV cells, obtained using 9-Tech process, were used for the tests. One sample was untreated (fragments of cells without any further chemical treatment) whereas the other one was treated with nitric acid (30% v/v) and sodium hydroxide (30% v/v) solutions by University of Padova in order to remove aluminium and silver. These two samples underwent a mechanical process, which involved a series of grinding and sieving steps to eliminate oxidized components with low electrical conductivity and reduce silicon powder to sub-micrometric dimensions.

This approach aims to achieve a high surface area and minimize volumetric expansion of the anode material during cell operation. Subsequently, the powder was characterized using various diagnostic techniques and mixed with an electronic conductor (nano-carbon) and a polymeric binder to ensure electronic conduction and mechanical stability to the electrode, respectively. This mixture was dispersed in water and deposited onto a copper sheet, serving as a current carrier.

After solvent removal and drying, the electrode, in ribbon form, underwent electrochemical characterization (voltammetry, impedance, and charge/discharge cycles in electrochemical cells) to validate its performance.

3. Results and discussion

3.1 Value Chain analysis of PV panels in Apulia and Extremadura

3.1.1 Installation analysis in Italy and Apulia

Starting from the mid-2000s, the State promoted a regime of incentives aiming at the establishment of photovoltaic energy facilities.

These initiatives successfully met expectations, featuring financial incentives for electricity generation and the option to feed surplus energy into the grid, enabling private entities to sell excess energy for profit. The steady decline in production costs played a pivotal role in advancing this sector. The period between 2005 and 2013 marked a surge in photovoltaic energy adoption in Italy, propelling the country to outperform other nations in global rankings.

From 2010 to 2013, the total installed power capacity surged from less than 4.000 megawatts to 17.000 megawatts. Throughout this golden decade, the sector maintained an impressive average annual growth rate of 63.7%. However, with the conclusion of State subsidies that fueled growth, Italy's progress in photovoltaics experienced a notable slowdown, leading to more modest growth figures. It wasn't until 2018 that the country surpassed the threshold of 20.000 megawatts in installed capacity. As of December 31st, 2022, there are 1.225.431 PV installations in Italy, with a total capacity of 25.064 MW. Installations with a capacity equal to or less than 20 kW make up 93% of the total in terms of quantity and 26% in terms of capacity. The average size of the installations is just slightly above 20 kW.

The graph provided in Fig. 2 presents a comprehensive overview of the progression of both the number and capacity of photovoltaic installations in Italy during the period from 2008 to 2022. Notably, it highlights the initial phase of swift expansion, which was largely propelled by various public incentive schemes, particularly the last three iterations of the Conto Energia. These initiatives played a significant role in fostering the rapid deployment of photovoltaic systems across the country. It is worth noting the surge during the three-year period 2008-2011, which led Italy to become the second country in the world in 2012 in terms of total installed capacity, right after Germany (IEA, 2013). Italy added 9.3 GW in 2011 with the help of such uncapped feed-in tariffs. However, a notable shift occurred in 2013, marking the beginning of a consolidation phase within the Italian photovoltaic industry. During this period, the growth trajectory transitioned to a more gradual and

steadier pace until 2019, the year that marked the beginning of the new incentive scheme FER1.

Italy has firmly established its symbolic milestone achievement of 20.000 megawatts of installed PV power. Surpassing the target with 20.108 megawatts at the close of 2018, this figure continued to ascend, reaching 20.865 megawatts in 2019. Describing the geographical distribution of photovoltaic plants in Italy poses a unique challenge. In contrast to other forms of energy where clear distinctions exist between Northern, Central, and Southern Italy, the landscape of national solar electricity is notably fragmented. Regional (and even provincial) rankings undergo significant variations based on different criteria such as the number of plants and installed nominal power.

3.1.2. Installation analysis in Spain and Extremadura

Spain's historic PV installation is characterized by two periods of growth. The first period goes from 2006 to 2008, and the installed capacity increased from 132 to 3.384 MW. This increase was the result of the subsidy system offered by the Spanish government to encourage the installation of PV panels, aiming to achieve an installed capacity of 371 MW by 2010. In May 2007, the RD 661/2007 was published, providing a subsidy of 0.44 €/kWh for PV facilities (compared to the electricity price of 0.075 €/kWh). This subsidy was reduced to 0.32 €/kWh from September 2008 through RD1578/2008. This promotion system for PV energy ended in 2009 (Fernández-González et al., 2020; <https://www.global-regulation.com/translation/spain/1445573/royal-decree-661-2007%252c-of-may-25%252c-which-regulates-the-activity-of-production-of-electrical-energy-in-special-regime.html>).

PV energy continued to grow moderately until the beginning of the second growing period in 2019, when it increased exponentially again. In 2018, before the second explosive growth period, the PV capacity in Spain was 4.780 MW. In 2023, it reached around 22.256 MW. During this period, the reduction in the manufacturing costs of PV panels directly impacted PV energy production, being the fundamental factor in the growth of PV energy in Spain since 2018. The situation in Extremadura follows the same trend (Fig. 3). Between 2006 and 2008, the installed power increased from 240 to 549 MW.

Since 2018, from 558 to 5.724 MW positioning itself as one of the leaders in energy production in Spain with a contribution of 24% to national generation. Self-consumption started in Spain later compared to other countries.

Table 1. Chemical composition of the white slag

<i>CaO</i>	<i>MgO</i>	<i>Al₂O₃</i>	<i>SiO₂</i>	<i>Fe₂O₃</i>	<i>MnO</i>	<i>Others</i>
49.6	7.9	9.1	31.1	2.0	-	bal
51.9	7.4	14.2	23.8	0.6	0.4	bal
49.6	6.0	29.1	14.7	0.1	0.2	bal

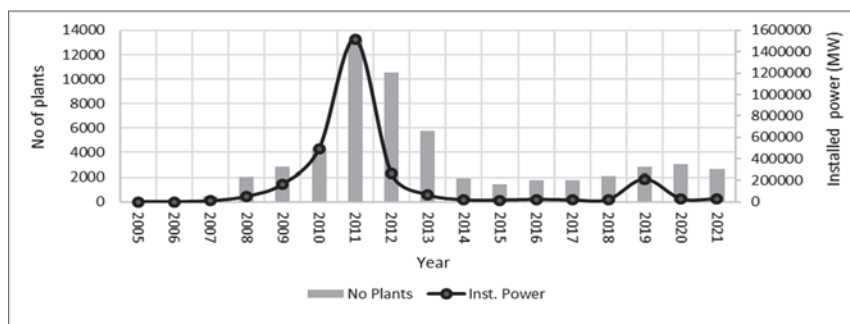


Fig. 2. Installations in Italy overtime 2005-2021. Data source: Terna – Gaudi, 2023

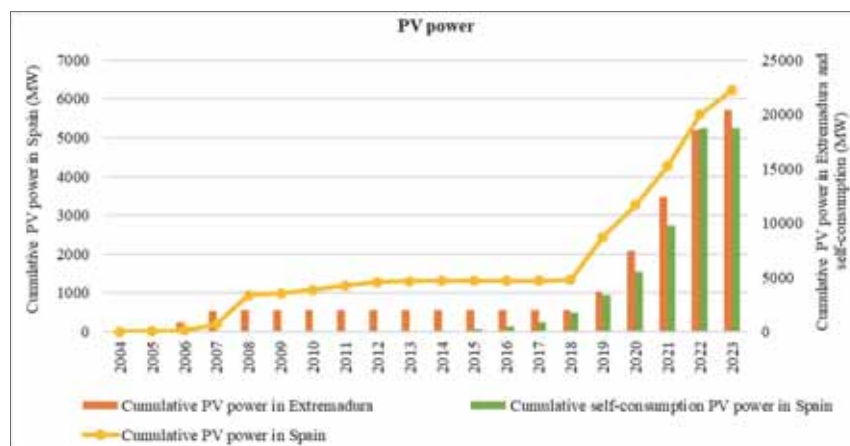


Fig. 3. Cumulative PV power in Spain, Extremadura and self-consumption (Spain) in the period 2004-2023

The key year is 2018 when the installation of PV panels for self-consumption increased exponentially from 484 MW in 2018 to 5.249 MW in 2022. This is due to several factors. The removal of regulatory barriers such as the “Sun Fee” in 2018, the high price of electricity in Spain, the aid provided by the Recovery Funds, the authorization to develop collective self-consumption plants and the regulations established by the European Union to make buildings more energy efficient. According to the bibliography, self-consumption could reach between 9.000 MW and 14.000 MW of installed capacity by 2030. It will enable the achievement of the Self-consumption Roadmap goal of 9 GW in 2024.

However, the self-consumption field still has some challenges, for example, the zero-waste regulation, which states that the surplus of solar installation does not go to the national grid but remains in the production centre. In 2022, self-consumption covered 1.8% of the country’s demand. Furthermore, it could have covered another 0.4%, but the difficulty of accessing the supply network and the zero-waste prevents its providing, and it is replaced by natural gas supply instead.

The development of PV energy will ultimately involve the management of PV panel waste. Under the general assumption that the lifetime of a PV panel is 25 years (Chowdhury et al., 2020) it is expected that the generation of this waste in Spain will increase exponentially in the coming years. However, the exact

waste generation is currently under discussion, and it is expected to reach 0.4-0.8 Mt of PV waste by 2040 (Kastanaki and Giannis, 2022; Santos and Alonso-García, 2018).

3.2. Photovoltaic refurbishment and recycling

3.2.1. Refurbishment

In Fig. 4a the result of a flash test is reported. It can be seen a normal behavior of a repaired module (Voc of 37 V and a Isc of 8.8 A). In Fig. 4b the results of the electroluminescence test are shown. The image reveals regions where cells have defects: as shown in one can identify small cracks that will not make problem for the reuse of the module. The developed process for refurbishment showed to be very efficient in finding defect and guaranteeing proper working of refurbished modules.

However, to create a market, it is important to provide confidence in these products. Also, it is important to guarantee the quality and safety of the repair by carrying out specific tests which will be carried out on all the repaired modules. Finally, repairs must be durable. To do this, it is necessary to ensure that the repairs will allow the module to have a long residual lifespan. This was ensured by accelerated aging tests specific to each repair. Based on this important know-how, the CEA is created the spin-off SOLREED, which aims to be the first company in Europe to carry out repair and reuse on a large scale.

3.2.2. Recycling

During the experimental phase, ended in January 2024, more than 1380 kg of end-of-life PV modules have been treated, and the prototype showed a high recovery yield of around 90% and a raw material purity high enough to be suitable for other applications. Specifically, from 100kg of PV panels, can be recovered: 65 kg of glass, 19 kg of aluminium, 2 kg of junction boxes (mainly composed of plastic and copper, 3 kg of PV cells (mainly composed of silicon), 1 kg of copper ribbons, and 0.05 kg of silver. Focusing on silicon, 9-Tech technology is able to recover up to 95% of it, with a purity higher than 90%. These data were confirmed by certified laboratories that performed several analyses on the materials recovered. In Fig. 5. are shown the materials recovered with 9-Tech technology. Moreover, the process showed low energy consumption, comparable to that of mechanical treatments.

3.3. Reuse of silicon

3.3.1. Aluminium industry

The trials to improve the solubility of silicon in aluminium alloys with the additives have shown promise in enhancing the integration of recycled silicon, potentially leading to better yields. However, the addition of these substances introduces extra costs, complicating the economic viability of this recycling pathway.

3.3.2. Ferroalloy production with STILLMETAL process

Several simulations were performed and the indicative results of a simulation are shown in Fig. 6. In this case, ferroalloy is characterized by an amount of Si higher than 40% (47.9% that corresponds to a FeSi-50) and the residual oxide slag has low melting temperature (lower than 1400°C) and a right composition to be used in cement industry.

In order to validate the results of the simulation, experimental tests were carried out with TRL5 pilot plant (induction furnace) using a crucible of graphite (W215/130 h330mm). The day after the

test, the crucibles were broken to recover the solidified materials inside. In all tests, it was possible to observe two distinct phases: on the top the oxide phase (residual oxide slag), and on the bottom the metallic one (ferroalloy), as can be noted in Fig. 7.

From the SEM and EDS analysis of the ferroalloy it resulted that the composition was homogenous along the height with an average value of Si of about 44%, lower than the expected one of 47.9%. Moreover, the amount of Al also in this case is higher than the one expected (4% instead of 2%). The microstructure resulted homogenous in the different part of the ferroalloy and with also a homogeneous silicon average.

In the ferroalloy four different phases were present (Fig. 8.): the phase 1 richer in Si (53.3%); the phase 2 richer in Fe (23.4%) with 37.2%Si; the phase 3 richer in Al (15.9%) with 43.2%Si; and the small phase 4 richer in Ti (26.0%) with 38.3% Si.

3.3.3. Li-ion anode production

The production of anodes for batteries presents a less demanding environment for the use of recycled silicon, despite the presence of impurities. Metals, in general, can enhance the electrical conductivity of anodes, suggesting that the impurities in recycled silicon might not be as detrimental in this application. This paves the way for utilizing recycled silicon from PV panels, where the strict purity requirements of other applications are relaxed.

Preliminary electrochemical tests performed on anodes prepared using the silicon PV cells powder have demonstrated that this silicon can form alloys with lithium. Thus, the potential for creating high-voltage anodes in lithium-ion batteries is confirmed. Also, the proprietary technology developed by NorcSi for silicon anode production has demonstrated encouraging results.

By refining and remelting recycled silicon, this technology aims to produce high-quality silicon suitable for use in anodes. While preliminary outcomes are promising, further validation is necessary to confirm the efficacy and scalability of these approaches.

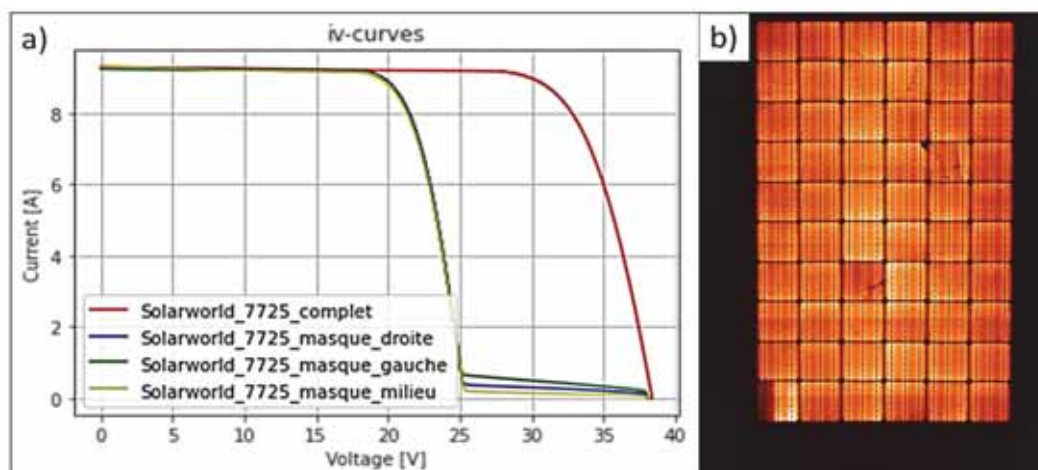


Fig. 4. a) Flash test result showing I-V curve on one module, b) Electroluminescence test showing some small cracks on cells



Fig. 5. Materials recovered in a 9-Tech demo plant that perform thermomechanical recycling from left to right: a) glass, b) copper ribbons, c) silicon PV cells, d) Aluminium, e) Junction boxes

residual oxide slag	ferroalloy
(1400 C, 1 atm, a=1.0000)	(1400 C, 1 atm, a=1.0000)
(51.272 wt.% Al2O3	(2.0293 wt.% Al
+ 3.2846 wt.% SiO2	+ 5.9758 wt.% Ca
+ 39.925 wt.% CaO	+ 43.089 wt.% Fe
+ 8.9784E-08 wt.% FeO	+ 0.52753 wt.% Mg
+ 3.8791E-11 wt.% Fe2O3	+ 0.46510 wt.% Mn
+ 5.5181 wt.% MgO	+ 1.6832E-04 wt.% O
+ 4.3815E-06 wt.% MnO	+ 47.913 wt.% Si
+ 2.4388E-11 wt.% Mn2O3)	+ 2.0990E-05 wt.% MgO
	+ 5.6132E-06 wt.% CaO)

Fig. 6. Results of the simulation with FactSage8



Fig. 7. Cross-section of the crucible coming from one experimental test

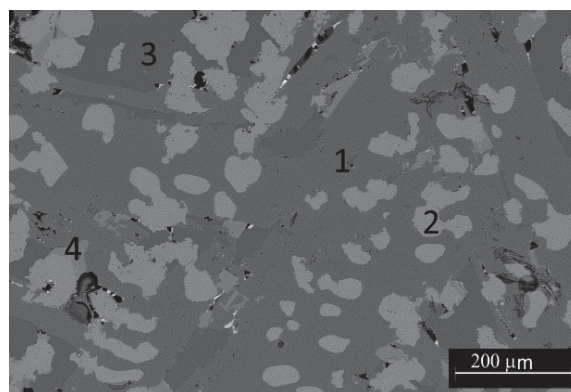


Fig. 8. SEM backscattered electrons images and EDS analysis of the central part of the ferroalloy

4. Conclusions

The PARSIVAL project represents a significant leap toward sustainable photovoltaic panel management. By introducing cutting-edge recycling and refurbishment technologies, the project tackles the impending challenge of PV waste in regions with

intensive solar installations like Apulia and Extremadura. The analysis of installations suggests that million tons of PV waste are expected by 2040.

PARSIVAL showed that refurbishment of PV modules is possible and is a promising solution to increase PV lifetime. Furthermore, the feasibility of the recovery of all the raw materials from end-of-life

panels, in particular silicon, a strategical raw material for the EU was also assessed.

The reuse of silicon recovered from PV panels is a complex process, hindered by the presence of impurities and the challenges associated with meltability and refinement. Despite these obstacles, innovative solutions and technologies are emerging, offering new pathways for the sustainable use of recycled silicon.

Specifically, the exploration of silicon reuse in applications such as aluminium industry, ferroalloys and Li-ion batteries produced promising results and will contribute to more sustainable and circular economy. In detail, the preliminary tests in the aluminium industry production have shown good performance of the recycled silicon, the use in the ferroalloys production allowed obtaining an alloy comparable with the FeSi-50 and the first anodes produced with recycled silicon showed good performances form the electrochemical point of view.

Further research and development are essential to optimize these processes, reducing costs, and enhancing the economic and environmental benefits of silicon recycling in the context of the growing solar energy sector. From a practical perspective, identifying various potential markets for silicon recovered from PV panel recycling will enhance the profitability of the entire process, enabling more widespread recycling of PV waste. This clearly results also in relevant social impact, considering the importance for future generations to recover critical materials and avoid landfill disposal. From the scientific point of view both the re-use of the silicon in the ferroalloys production and in the production of anodes is extremely innovative and the project can result the starting point of future research in the field.

Furthermore the project contributes to several Sustainable Development Goals (SDGs) such as SDG 7, affordable and clean energy by promoting the recycling of PV panels, ensuring access to affordable, reliable, sustainable, and modern energy for all; SDG 9, industry, innovation, and infrastructure, by representing a significant advancement in recycling technology, contributing to the development of sustainable industries; SDG 12, responsible consumption and production, by supporting sustainable consumption and production patterns and by enabling the recycling and refurbishment of PV panels in a manner that maximises the recovery of valuable materials; SDG 13, climate action, by reducing CO₂ emissions and contributing to efficient use of resources to climate action efforts.

PARSIVAL's structured value chain for material recovery holds promise for transforming PV waste management practices and fostering a more sustainable solar industry.

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ENVIRONMENTAL TECHNOLOGY VERIFICATION (ETV): A TOOL TO DEMONSTRATE THE INNOVATIVENESS OF TECHNOLOGICAL SOLUTIONS FOR CIRCULAR TRANSITION

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Abstract

The Environmental Technology Verification (ETV) tool is part of a voluntary programme to facilitate market access for new environmental technologies. It is a verification protocol for innovative technologies based on their environmental performance, impartially certified by an accredited verification body. As part of the LIFEproETV project, an European initiative aimed at promoting market acceptance and recognition of the ETV scheme, ENEA (Italian National Agency for New Technologies, Energy and Sustainable Economic Development) has identified ETV as a key instrument to support innovation in the field of Circular Economy (CE). Indeed, the transition towards circular production and consumption models implies the use and dissemination of new technologies, whose environmental performance must be credible and impartially verified.

The ETV scheme has been successfully applied to verify the performance of 15 innovative environmental solutions with circular applications. These technologies could not be measured or verified using existing regulations, certifications, authorizations, or standards.

In this context, to identify concrete use cases for ETV within Italy's environmental policies and to outline the opportunities and enabling environment for promoting ETV-verified technologies that support the circular economy, ENEA has developed a roadmap. This roadmap defines the potential areas where ETV can be used as a policy tool to help achieve the specific objectives of the National Strategy for the Circular Economy (NSCE). It also explores the value and potential benefits of using the ETV scheme to foster innovation ecosystems, address environmental challenges, and promote sustainable finance.

Key words: circular economy, EN ISO 14034, environmental technologies, industrial symbiosis, green performances

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1. Introduction

The Environmental Technology Verification (ETV) scheme has been conceived with aim of facilitating the demonstration of the environmental performance of new technologies and accelerating their market adoption, as investigated by Merkourakis et al. (2007). Based on internationally recognised verification procedures included in EN ISO 14034 (Environmental Management - ETV and competences confirmed by national accreditation bodies through accreditation for compliance with ISO 17020) for Type A inspection bodies, verification bodies provide

objective evidence that the claimed environmental performance of technologies is valid and supported by quality assured test data, as recently outlined by Hansen et al. (2021).

ETV can operate either as an independent market mechanism or as part of a broader program to support the implementation of environmental policies. In this regard, the European Commission has established and promoted a dedicated EU ETV programme for several years (COM, 2008).

A pilot phase of the Environmental Technology Verification (ETV) programme was launched in 2012 to investigate three key technology

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areas: water treatment and monitoring, energy technologies, and materials, waste and resources. In addition, in view of the expansion of the EU ETV programme from pilot to full scale initiative, four additional technology areas were defined: cleaner production and processes, air pollution control and monitoring, soil and groundwater remediation technologies, and environmental technologies for agriculture. However, the European Commission decided to discontinue its support for the EU ETV programme as of November 2022, following an internal evaluation.

Despite this decision, there remains a strong and ongoing demand among stakeholders for impartial, verified data on environmental technologies. LIFEproETV project aims to fill this gap by promoting and establishing a voluntary scheme to verify the performance of new environmental technologies prior to market entry. In consequence of the closure of EU ETV programme, verification bodies and other entities involved will need to develop a market-driven business model to continue their activities in compliance with ISO 14034. This will require the formation of new partnerships between ETV bodies in Europe and market players. These stakeholders will need to clearly define their roles within the value chains to which they contribute, aligning their capabilities with the specific needs of the industries they serve. Notwithstanding these challenges, the role of ETV in providing objective, credible information on green alternatives remains vital. There is significant potential for ETV to be supported by both market and policy actors moving forward.

In accordance with the Policy Brief published within the LIFEproETV project, the information provided by ETV can also serve as evidence for revision of performance-based regulations for meeting the targets defined in EU Green Deal policies (LIFEproETV, 2022).

1.1. ETV supporting CE: current state

The concept of CE represents a cross-cutting theme that encompasses a wide range of issues including the reduction and optimisation of raw materials, the reuse of products, the strengthening of secondary materials value chains, and eco-design. The overarching objective of CE is to facilitate the transition towards more sustainable production and consumption models, focusing both on environmental and economic sustainability. By promoting and reinforcing new business models that lead to significant material savings throughout the value chain and by fostering IS collaborations, the CE create valuable business opportunities. Innovative environmental technologies, products and services are key enablers of these processes (Chertow, 2000). The lack of credible, high-quality information on the performance and outcomes of new environmental technologies often hinders their implementation and hampers the purchasing decisions of final technology

buyers. In this context, ETV has a great potential to meet this need also investigated by Marrucci et al. (2019), detailed study has been conducted to identify technologies that have already been validated through ETV and are directly related to circular economy applications or support the transition towards circular models. An in-depth analysis, conducted as part of the 'Roadmap for building ETV market acceptance and recognition' (LIFEproETV, 2023b), examined technologies with verified environmental performance. Of the 60 technologies verified by ETV, 25% are linked to the circular economy (CE). These are mainly in the technology areas of materials, waste and resources (80%), water treatment and monitoring (13%) and energy technologies (7%), as illustrated in

Fig. 1. The ETV scheme has been successfully used to provide independent, credible and market-relevant information on 15 innovative environmental solutions, listed in Table 1. These solutions have applications in the circular economy, including:

- New technical solutions for waste separation and recycling, energy and resource recovery from waste and water recycling;
- New materials such as bio-based and biodegradable plastics.

As they fall outside the existing regulatory, certification, approval or standards frameworks, their performance characteristics could not otherwise be accurately measured and verified (LIFEproETV, 2024).

1.2. ETV in support of the Italian strategy to guide the transition to the CE

In Italy, nine technologies have been verified through the ETV scheme, all of which are provided by Italian technology providers. These technologies have been validated by two Italian verification bodies, RINA and Certiquality, which together have certified a total of 11 technologies under the ETV scheme, in particular in the field of water treatment and monitoring.

The interest in adopting this tool is evident from the number of Italian technologies verified with ETV, but there are several barriers to its diffusion and adoption. Despite these challenges, the NSCE emphasizes the need to incorporate criteria for assessing and quantifying the innovativeness of technologies, which is critical for advancing the CE. Specifically, the NSCE stresses the importance of defining clear indicators and parameters that can be used to monitor economic circularity and ensure the efficient utilization of resources. By integrating ETV into these frameworks, Italy can better facilitate the transition to a CE and encourage the adoption of innovative technologies that support sustainability goals. The Italian National Strategy for the Circular Economy (MITE, 2022) is a programmatic document launched in June 2022. Its purpose is to establish the framework of institutional policies needed to guide Italy's transition toward circular models of growth and development.

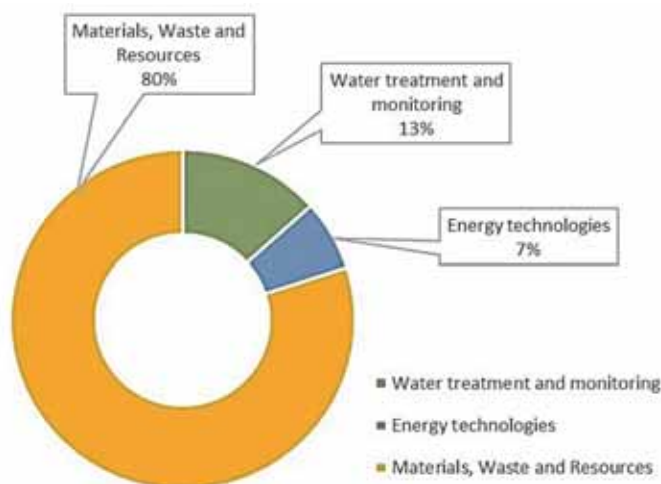


Fig. 1 . Detail of the technological areas of belonging for technologies verified with ETV in circular applications

Table 1. ETV verified technologies for CE applications

<i>Technology name</i>	<i>Technology function</i>	<i>Producer/Provider</i>	<i>ETV Statements of Verification no:</i>
RECYOUEST process	Recycling of contaminated plastics from agricultural environment	Recyouest, France	VN20190036
BIO-COM SYSTEM	Biostabilisation and composting of organic waste	SELMA Sp. z o.o., Poland	VN20150005
ECOGI	Separator for extraction of organic waste from pre-sorted household waste	Komtek Miljø A/S, Denmark	VN20160011
EWA Aerobic Fermenter, model 2020	Aerobic stabilisation of organic waste and sanitation of animal by-products	RSP Ostrava, a.s., Czech Republic	VN20160014
PURROT	Separation of dry matter from liquid waste	PurFil Aps, Denmark	VN20150006
ReStoRe	Transformation of low-value spent refractory waste into high-value products for steelmaking	Deref S.p.A, Italy	VN20210048
BIOMInE® BM-LMI-03	Biobased (80% organic carbon) polymer from PLA	Futuramat, France	VN20170020
BIOPOLYME BP-LXX-06	Biobased (81% organic carbon) polymer from PLA and starch	Futuramat, France	VN20170019
Re-Match Artificial Turf Recycling	Recycling of synthetic turf	Re-Match, Denmark	VN20170025
LIGNO-ENZYM	Enzyme decomposition of organic waste to intensify methane production in biogas plants	CONFORMITY, s.r.o., Czech Republic	VN21210045
Periodic anaerobic bioreactor ANABIOREC	Enhance the energy recovery from separated organic fraction of municipal waste	NOVAGO Sp. z o.o., Poland	VN20190037
Rich Water series 2018	Wastewater treatment for combined irrigation and fertilisation purposes	BIOAZUL S.L., Spain	VN20200042
HYDRO-1	Reclamation of water and nutrients from wastewater for agriculture applications	IRIDRA Srl, Italy	VN20220054
MATER-BI	Biodegradable and compostable bioplastics mainly made from renewable materials	Novamont S.p.A., Italy	VN20150004
Biofibra®	Biodegradable biopolymers made entirely of bio-based carbon materials	FuturaMat, France	BF-LhE-01 – VN20160013 BF-LED-10 – VN20160021

The strategy outlines the specific actions, objectives, and measures that will be promoted to achieve this transition.

Italy began exploring the concept of the circular economy as early as 2017, initiating a series

of consultations with a diverse range of stakeholders, including businesses, industry representatives, and consumers. These consultations were aimed at defining the goals and measures necessary to position Italy as a leader in this. This was done in alignment

with the commitments made within the Paris Agreement on climate change, the United Nations 2030 Agenda on sustainable development, the G7, and the European Union (LIFEproETV, 2023b).

The NSCE incorporates contributions from both throughout the 2017 and 2021 +consultation phases. Its goal is to outline a comprehensive action plan encompassing the strategic directions and measures that must be undertaken to initiate a transition towards innovative, circular economic models. The strategy focuses on the following key themes: eco-design; reuse and repair; end-of-life (EoW); critical raw materials and the development of a secondary raw materials market; Green Public Procurement (GPP) and Minimum Environmental Criteria (MEC); strategic industrial supply chains; IS; Extended Producer Responsibility (EPR); digitisation; and tools to support CE.

While Italy faces challenges in decoupling economic growth from resource consumption, there are positive signs regarding its circular material use. In 2021, Italy's rate of circular use of materials stood at 18.4%, well above the European average of 11.7%, according to Eurostat and the Circular Economy Network (CEN) (CEN, 2023). This marks a significant improvement from previous years, positioning Italy among the leading countries in advancing the CE. Nevertheless, there is a call for both Italy and Europe to expedite their transition towards circular models in order to address environmental and geopolitical challenges. This is crucial in maintaining a pivotal position within the international and European landscape, while also guaranteeing the attainment of progressive improvement objectives.

Innovative environmental technologies, products and services are enabling factors in these processes. However, the lack of reliable, high-quality information on their performance and outcomes often impedes their adoption. Without such information, technology users and buyers are unable to make informed purchasing decisions.

2. Material and methods

The methodology proposed in this study has been developed to create a roadmap to facilitate the development of market acceptance and recognition of ETV as a voluntary environmental scheme. The aim to accelerate the uptake of new environmental technologies in the market. The methodology presented in this study can be used by a wide range of stakeholders to propose a framework for valorizing new environmental technologies in the market, addressing real use cases and challenges, and leveraging the benefits of ETV.

Technology producers and developers can apply this methodology to highlight the environmental benefits of the technologies they develop, specifically within the context of particular environmental, policy, or regulatory challenges. This approach enhances the competitiveness, credibility, and innovative potential of these technologies, making them more attractive to

the market. The methodology is also valuable for potential investors or users, as it provides a clear understanding of the economic and environmental benefits, enabling more informed and responsive decision-making when selecting innovative solutions. Additionally, policymakers can use the methodology to develop strategic innovation pathways, guiding the creation of supportive frameworks for sustainable technology adoption.

In accordance with this approach, five national roadmaps were devised (one for each country: Hungary, Italy, Poland, Slovenia and Spain), delineating explicit objectives for the campaigns and substantiating the business case for ETV. The roadmaps demonstrated the distinctive value proposition of ETV in relation to national or European policies, fostering national or sectoral innovation ecosystems, addressing environmental challenges, and supporting sustainable financing. While the contexts of ETV market acceptance and recognition vary across the aforementioned countries, the ETV use cases and methodology for the development of roadmaps can serve as a source of guidance for other countries, decision-makers, and organisations interested in applying the ETV scheme for their own purposes.

This document outlines a methodology developed as a structured process comprising distinct phases (steps). Each phase is investigated in depth, with a particular focus on defining the individual elements that emerge according to a systematic framework. Fig. 2 provides a general overview of the methodology, highlighting illustrating the aspects addressed in each phase and the unique elements characterizing each step. The phases can be summarised as follow:

- Step 1: ETV use case definition

The main users of ETV are identified and the methods for applying ETV to achieve specific objectives are outlined. Partners of the LIFEproETV Project formulated hypotheses regarding the potential use cases. Following consultation with the main stakeholders, the use case was validated on the basis of positive feedback.

- Step 2: Problem definition

Provides a comprehensive description of the issues, conditions and challenges related to the use case. It identifies discrepancies between the problem and the objective, focuses attention on factors impeding application, defines stakeholders and establishes priorities.

- Step 3: Action plan

A checklist is created to outline the steps needed to achieve set goals. The action plan is the main part of the strategic planning process. In this phase, the actions, subjects, priorities and timing are defined.

With regard to the Italian Roadmap, ENEA focused its attention on the subject of the circular economy, with a view to achieving the specific objectives set out in the national policy. The NSCE constitutes a substantial advance in the transition of the Italian productive system towards circular models.

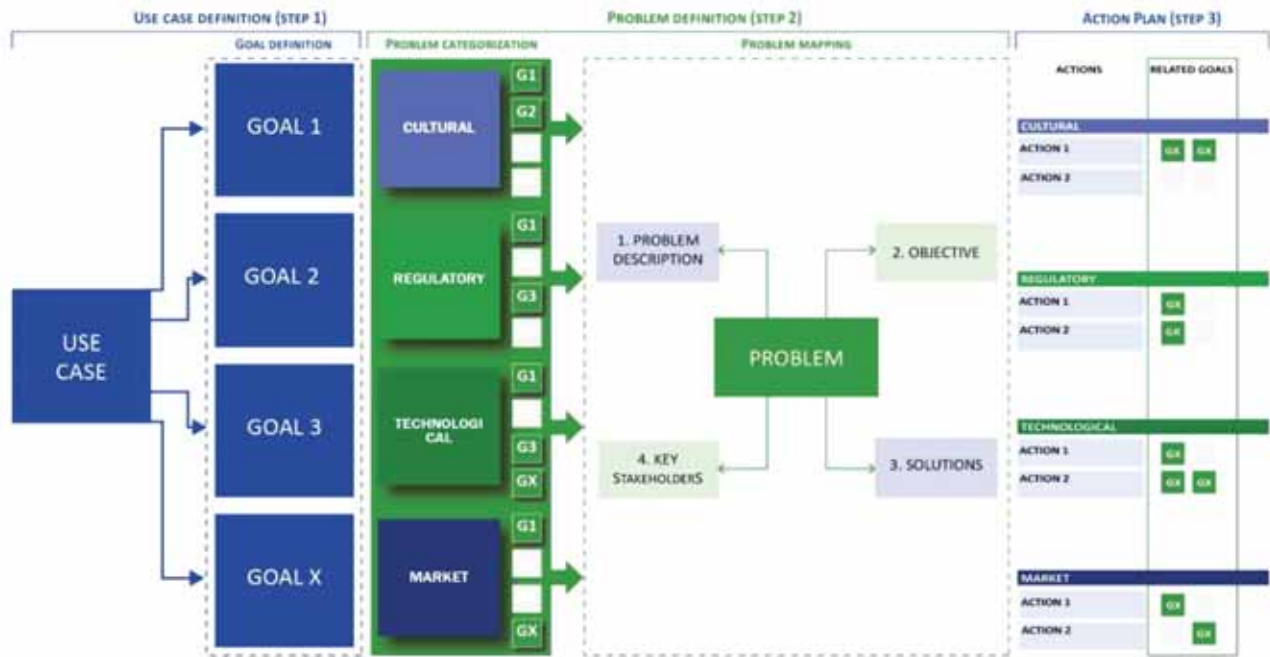


Fig. 2. Methodology diagram (LIFEproETV, 2023a)

It outlines a series of actions to be implemented at defined intervals, providing a roadmap for this crucial transition. In light of the aforementioned reference, ENEA has developed a document entitled "Roadmap for building ETV market acceptance and recognition" (LIFEproETV, 2023b) which was created within the framework of the LIFEproETV project and follows the methodology described above. This document follows the methodology described earlier and aims to define a tangible trajectory for the valorization of ETV. Specifically, it identifies business use cases where an impartial performance audit of innovative environmental technologies can significantly contribute to their implementation and widespread adoption.

The roadmap delineates a series of specific measures to argue the acceptance of ETV in accordance with the identified needs and priorities of the various stakeholders, including policymakers, decision-makers, technology providers, and technology buyers. This accomplished through a comprehensive and active involvement process.

Following the identification of the potential benefits of adopting the ETV as a third-party verification in the CE field, use cases have been defined. The aforementioned use cases have been devised with the objective of facilitating the transition of the ETV from a cost to an added value in the dissemination and marketing of the technology itself.

In this strategic context, it has been determined that the role and utility of the ETV must be defined in relation to the programmatic and strategic objectives of the NSCE, which serve to guide the development of pathways towards the CE. Table 2 presents the selected NSCE-specific objectives. The selection

process took into account those issues where technological innovation and improvements in environmental performance are critical to achieving these objectives. In particular, this included areas related to:

- recycling and reuse of materials;
- resources recovery;
- replacing of virgin raw materials with secondary materials.

Concurrently, it delineates the role that ETV could play in the attainment of these objectives. The definition of use cases is the process of identifying obstacles that hinder the achievement of objectives in support of circularity. This process is followed by the identification of concrete actions, which are then categorised based on priority and effectiveness criteria. The objective of this process is to support the construction of a strategic path for the valorisation of ETV in business scenarios. To facilitate this, a stakeholder map was created to identify and engage the relevant stakeholders.

2.1. Obstacles to objectives implementation

In this context, the obstacles to achieving the selected objectives were identified according to the methodology described above. This identification was carried out on the basis of the information gathered from stakeholders involved and consulted during the meetings, conferences and brainstorming sessions organised in the framework of the LIFEproETV project. Once the main barriers were identified, the obstacles were selected according to their nature and classified into four main categories (problem categorisation):

Table 2. Objectives selected from the specific objectives defined by the NSCE and potential role of ETV

<i>Specific NSCE objective</i>	<i>ETV role</i>
G1 – Provide tools and services to support companies, especially SMEs, in the implementation of technologies, methodologies and approaches aimed at the efficient and sustainable management of products.	An ETV Statement of Verification can serve as a means of substantiating the efficacy of solutions that prioritise innovation in the domain of optimising material and energy utilisation, thereby enhancing efficiency and mitigating the environmental impact of organisations that adopt such solutions. The utilisation of certified solutions can assist companies in investing in efficacious and profitable solutions, meeting their requirements whilst simultaneously mitigating investment risks.
G2 – Create the conditions for a market of SMR that are competitive in terms of availability, performance and costs, acting on the standardization of materials, and on the criteria for removing the qualification of waste from materials ("End of Waste").	The use of ETV allows for the verification of the performance and environmental impacts associated with the production and regeneration processes of SRM-based materials. It demonstrates that new materials or processes do not result in secondary environmental pollution (especially when using hazardous substances) in comparison to conventional processes. The demonstration of a reduction in the environmental impact of materials produced with the use of secondary raw materials can act as an incentive for their use, thereby increasing the market.
G3 – Create the conditions for a market of "by-products" in terms of greater certainty in recognition, availability, acting on standardization for certain supply chains (e.g., residues and by-products of agricultural origin) and on the revitalization of the by-product exchange platform, to concretely support operators in the full implementation of the IS also in the field of bioeconomy.	ETV offers data on the functional and environmental performance of the by-products, thereby guaranteeing that the material aligns with the stated functional technical parameters for subsequent utilisation as a raw material and to obtain an EoW status certification. This may facilitate standardisation, thereby increasing the exchange of resources between companies (IS).
G4 – Set the conditions for the extension of the life of the product through its design inspired by the principles of modularity and reparability.	The ETV has the potential to serve as a supplementary evaluation scheme for the functional performance of products and technologies with business-to-business (B2B) applications that align with the criteria set forth in the Ecodesign Directive. ETV Statement of Verification may be used as a source of information to support compliance with environmental performance systems for products, including the Environmental Product Declaration (EPD), the Ecolabel, the Product Environmental Footprint (PEF), and the Eco-design Directive.

- *Regulatory problem/barrier:* Lack of a comprehensive framework of tools, standards and policies for the transition to the Circular Economy;

- *Cultural problem/barrier:* Lack of awareness and ability to undertake development paths aimed at supporting the circular economy;

- *Technological problem/barrier:* Lack of effective technologies to implement the circular economy.

- *Market problem/barrier:* Lack of economic feasibility of circular models. A detailed description of the causes contributing to these barriers was developed through a thorough problem mapping process. This exercise led to the following insights for the Italian roadmap;

- *Regulatory:* A significant regulatory obstacle is the absence of a harmonized and clearly defined framework of tools and policies to support the application and use of innovative environmental technologies. To cite one example, the current regulatory framework presents a number of challenges. These include the lack of harmonisation and coordination of test protocols, the scarcity of

standards in the field of by-products, and the absence of regulatory recognition of environmental performance measurement tools (such as ETV) in procedures designed to support GPPs.

In the Italian context, the absence of a comprehensive, organic and standardised framework of rules and tools to support circularity represents a significant barrier to the adoption of circular models. This is primarily due to the absence of mandatory product certification, the lack of clarity in the definition of waste and by-product, and the consequent absence of reference standards.

- *Cultural:* Include those barriers deriving from the uncertainty linked to innovation and therefore from the lack of recognition, acceptance and trust in innovations, especially in the environmental field. This uncertainty hampers innovation and the scaling of new solutions. Examples of this include the uncertainty surrounding the diffusion of new technologies on an industrial scale and the reluctance to invest in innovations that lack concrete evidence of improvement in environmental performance. Furthermore, there is a general lack of knowledge

regarding the benefits derived from such innovations (Chen and Wang, 2023 LIFEproETV, 2023a).

With regard to the CE, the lack of awareness and capacity among companies and the market to pursue innovation pathways is frequently associated with a dearth of knowledge regarding processes or services for the transformation and enhancement of waste.

- *Technological*: However, these technologies often fail to achieve significant market penetration, as potential users remain skeptical about their effectiveness. As a result, many users are hesitant to adopt new technologies until their benefits have been clearly demonstrated. This leads to significant challenges in upscaling innovations, with many technologies remaining at the pilot stage or failing to gain the market traction necessary for widespread adoption.

- *Market*: Economic barriers play a major role in hindering market entry for circular economy solutions. These barriers include the high initial costs of innovation, the scarcity of funding to support new technologies, and the tendency to focus on short-term financial gains rather than the long-term economic benefits of cutting-edge solutions. The lack of economic viability and the necessity for significant investment to stimulate innovation in the utilisation of secondary raw materials or in the preparation of MPS for use in other production processes represent the most prevalent factors impeding the dissemination and adoption of innovative environmental solutions. With regard to the transition towards the CE, one of the principal obstacles is the relatively low price of virgin materials, which acts as a disincentive to the use of recycled materials.

2.2. Solutions and opportunities

In accordance with the methodology, the problem mapping entails the simultaneous development of solutions for its resolution, while also considering the potential opportunities that may arise from it. This phase therefore highlights how ETV can support strategies to overcome barriers in the transition to a CE. From a regulatory perspective, ETV has the potential to become a CE standard for verifying the performance of a number of emerging technologies, including those that facilitate the conversion of waste into resources, as well as secondary raw materials destined for industrial applications and products based on secondary raw materials.

The adoption of ETV as a standard could provide a robust basis upon which to address regulatory gaps related to the performance of technologies and materials. Furthermore, at the national level, ETV, as a reliable system, could be acknowledged by regulators as a means of demonstrating compliance with BAT performance levels.

As ETV provides an impartial assessment of environmental performance, it could be employed as a

methodology for demonstrating compliance with technical specifications in national GPP. ETV verification statements can further substantiate compliance with various environmental performance systems, such as EPD, ecolabel, Environmental Product Footprint (PEF), and eco-design.

With regard to the issue of cultural barriers, ETV can assist in establishing credibility for innovative technologies by verifying performance in relation to resource use. This can assist technology purchasers in mitigating the technological risks associated with investment, and provide companies with a genuine opportunity to embark on IS pathways based on an audit that highlights the benefits. Moreover, ETV can provide certification attesting to the benefits of innovative technologies, namely their capacity to reduce adverse impacts on human health and the environment. It can also facilitate the process by which companies can obtain permits allowing them to transform their waste into valuable by-products or SRM.

To overcome the technological obstacles that hinder the progression of environmental solutions, ETV plays a pivotal role in defining the technological potential and viability of these innovations in addressing environmental concerns. Furthermore, the flexibility in selecting evaluation parameters allows the identification of the most suitable technology to address specific environmental challenges. Furthermore, ETV can facilitate the dissemination and market acceptance of innovative environmental technologies, as it can inform the establishment of appropriate and meaningful incentives for manufacturers, suppliers, buyers, and users of ETV-verified technologies.

Moreover, incorporating ETV into GPP and IP procedures, as a compliance verification tool could be highly beneficial. This would involve aligning ETV with test reports from conformity assessments and establishing a verification statement similar to the Ecolabel. Such an approach could significantly enhance the market acceptance of innovative environmental technologies by providing credible, third-party verified evidence of their performance.

Additionally, focusing on Small and Medium Enterprises (SMEs), which are agile and well-positioned to drive the transition to a circular economy, ETV could help SMEs create non-financial statements to showcase the viability of circular business models. This would promote their adoption of sustainable practices and demonstrate the environmental and economic benefits of circular approaches.

2.3. Action plan

For each of the aforementioned problem category, which encompass regulatory, market, technological, and cultural concerns, a set of defined actions has been established.

i. Regulatory actions:

- Integrate ETV as CE standard: The ETV

scheme should be adopted as the standard for CE practices, providing a market-relevant framework for future legislation. This would address regulatory gaps related to product-integrated environmental protection and performance-based regulations, particularly for technologies and materials involved in circular processes.

- Use ETV for BAT Compliance: ETV should be utilized to demonstrate compliance with performance levels for BAT. By implementing ETV-verified technologies, companies can reduce uncertainties regarding the potential environmental benefits of these technologies.

- Recognize ETV in National GPP: ETV should be recognized within national GPP frameworks. This would offer a systematic methodology for verifying compliance with technical specifications, while also supporting a comprehensive life cycle approach to product and technology evaluation.

ii. Cultural actions:

- Disseminate knowledge of ETV: Efforts should be made to disseminate knowledge about the ETV system through collaboration with various stakeholders involved in innovation support. This will help raise awareness and understanding of ETV's benefits across different sectors.

- Promote ETV to streamline technology scaling: ETV should be promoted as a tool to facilitate dialogue and streamline administrative processes for scaling up innovative technologies at the European and national levels. This will help create a more supportive environment for technological innovation.

- Enhance information access for SMEs and Start-ups: It is essential to improve access to information about ETV, particularly for SMEs and start-ups. This can be achieved by strengthening the role of business support organizations, which can provide guidance on the adoption and benefits of ETV.

iii. Technological actions:

- Facilitate industrial-scale replication: Conditions should be created that allow for the industrial-scale replication of innovative technologies by using the ETV verification process as a formal validation protocol. This will help reduce investment risks and increase the competitiveness of emerging technologies compared to traditional alternatives.

iv. Market actions:

- Incorporate ETV into national innovation policies: ETV should be integrated into national policies aimed at fostering innovation, particularly in SMEs. This can be achieved by providing economic incentives such as tax deductions or subsidies for the development and adoption of new environmental technologies.

- Promote ETV with a Focus on Environmental Benefits: In addition to highlighting the market potential of ETV-verified technologies, emphasis should be placed on the environmental benefits these technologies provide. This will not only help

accelerate market acceptance but also contribute to achieving environmental goals at the local and community levels.

3. Result and discussions

In the overall objective of this study, the aim is to boost awareness, market recognition, and acceptance of ETV scheme. Market acceptance has two aspects: Firstly, it measures the extent to which ETV satisfies stakeholders, in particular technology buyers and providers. Technology buyers must perceive ETV as a valuable support mechanism that simplifies their decision-making processes, clarifies their options, and assists them in overcoming challenges related to purchasing and technology selection. Conversely, technology providers need to see a compelling business case that justifies their participation in the ETV scheme, ensuring that it adds value to their offerings and enhances their market competitiveness. Secondly, market acceptance encompasses a broader adoption process wherein various market actors incorporate ETV into their operations and strategies.

Beyond the core target groups, other stakeholders such as policymakers, research and innovation (R&I) program operators, regulatory bodies, and investors have their unique needs and motivations for utilizing ETV. Each of these stakeholders may adopt the ETV framework to serve their specific objectives, whether it is to promote regulatory compliance, leverage technological innovation, attract investments, or align with sustainability goals. Their engagement is crucial, as it contributes to the overall market uptake of ETV, ensuring that it remains relevant and effective across different contexts and applications.

Market recognition plays a crucial role in the success of a product or service, such as ETV (Environmental Technology Verification). When a technology or service is widely recognized, it significantly influences the purchasing decisions of both technology providers and buyers. The project has therefore developed an innovative methodology for the co-creation of collaborative institutional ecosystems, with the aim of actively involving all key stakeholders in the promotion and adoption of the ETV scheme. This approach is particularly valuable because it is embedded in a concrete national context, allowing environmental challenges to be addressed with strategies tailored to local specificities.

The project not only promotes the adoption of this methodology, but also aims to support long-term sustainable development. The cooperation between the different actors involved enables the integration of skills and resources, the creation of a more favourable regulatory environment and the raising of awareness of the importance of innovative environmental technologies. Based on the guidance provided by the concrete ETV use case, discussed and detailed in the Italian roadmap, the creation of a collaborative ecosystem was successfully initiated. In fact, as

Knowledge Centre and potential national Scheme Owner (responsible for the development and maintenance of a compliant scheme and for the definition and application of rules for the use of the scheme in different technological contexts), ENEA has defined a collaborative ecosystem as detailed in the chart in

Fig. 3, which envisages the interaction with different subjects and stakeholders with whom collaboration has already been initiated.

These activities are currently in place and will continue in the next phase of the After Life Plan, i.e. a strategic plan aimed at ensuring the durability and persistence of the project results over time and the resources required for their application. To date, the construction of the network for the implementation of the ecosystem has seen the active involvement of numerous institutional actors, business associations and individual organisations. ENEA, in particular, has organised and participated in 13 events, including conferences and workshops, at which it has presented ETV to specific target audiences, gauging the interest of various actors in utilising and disseminating the ETV scheme. The promotional campaign for the ETV initiative in Italy, which integrates within the national strategies for circular economic development, has significantly advanced key objectives. The main outcomes can be summarized as follows:

- *Network expansion:* Targeted meetings successfully defined and broadened a collaborative network, identifying two additional prospective knowledge centres for ETV in addition to ENEA.

- *Strengthened partnerships:* The initiative enhanced partnerships with existing circular economy platforms and their member organizations, fostering a collaborative approach to identify innovative circular economy technologies eligible for ETV.

- *Increased awareness:* The advantages of ETV in facilitating the transition to circular economic models were communicated effectively to organizations that promote the adoption of new technologies among businesses. This effort has led to

a deeper and more contextual understanding of ETV's role and its benefits in promoting green technologies.

- *Outreach and education:* Awareness of the ETV initiative surged, aided by the distribution of promotional videos and interviews in specialized journals and magazines focused on the circular economy. Additionally, many small and medium-sized enterprises, along with the scientific research community, received in-depth information about ETV through workshops conducted during the campaign.

These outcomes highlight the effectiveness of the campaign in building a robust framework for promoting ETV as an essential tool for advancing Italy's circular economy objectives and driving the adoption of sustainable technologies.

4. Conclusions

The role and applications of ETV can vary depending on the stage of the product and process life cycle under consideration. It serves to demonstrate the efficacy of the reduction in the utilisation of primary materials and energy, as well as the promotion of the adoption of technologies that have been verified and deemed reliable, which enhance the efficiency of companies while minimising the environmental impact. Furthermore, ETV plays a pivotal role in demonstrating the implementation in the use of SRM to guarantee that new materials or processes do not lead to secondary pollution.

This approach, facilitates the overcoming of obstacles to the development of industrial symbiosis (IS) pathways, a collaborative model involving traditionally separate industries in a collective approach to competitive advantage involving the physical exchange of materials, energy, water and by-products. The key to industrial symbiosis are collaboration and the synergistic possibilities offered by geographic proximity and circular business models while verifying that these processes do not result in environmental harm.

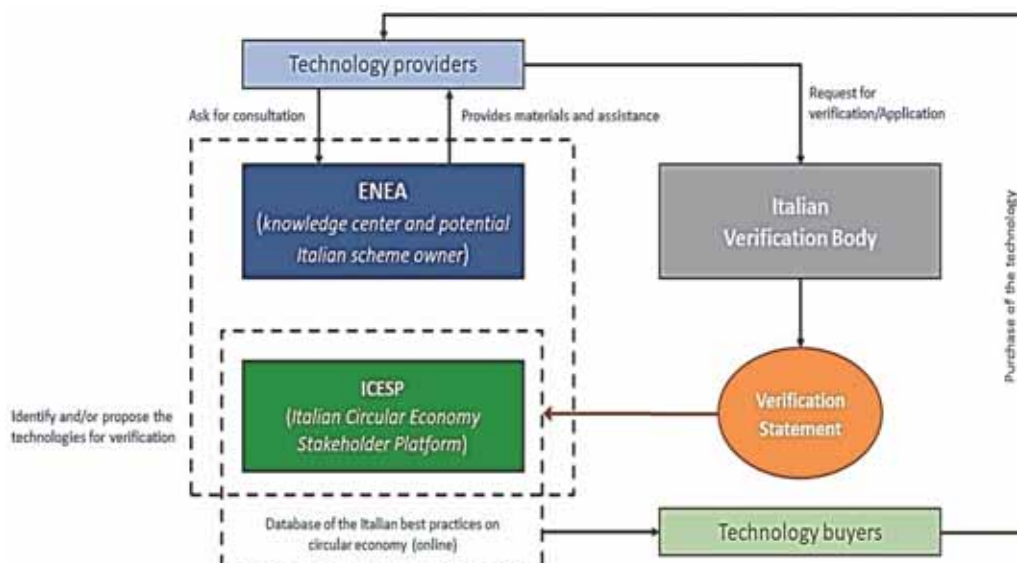


Fig. 3. ETV ecosystem for Italy

Furthermore, ETV can furnish data regarding the functional and environmental performance of by-products and product parts intended for circular applications in industry.

Furthermore, ETV Verification Statements ensure that verified by-products meet the necessary technical and functional criteria for subsequent use, including the issuance of an End-of-Waste. The role of the ETV is of great importance in the promotion of sustainable practices and the fostering of a CE. The role of the ETV in promoting green innovation in public procurement (GPP and IP) could be pivotal, as it serves as a conformity assessment tool and is acknowledged as an equivalent means of proof. Additionally, ETV Statements of Verification can furnish information that is instrumental in guaranteeing that products meet environmental performance standards, including those set forth in the EPD, Ecolabel, PEF, and Eco-design.

To achieve these objectives, a collaborative network was established to create a robust ecosystem that supports ETV. The active involvement of specific target groups helped to strengthen the solidity of the ecosystem and lay the foundations for the sustainability of the project results. In fact, the promotion campaign conducted so far has involved and informed institutional bodies, local authorities involved in GPP and policy makers who are actively involved in the implementation of policies aimed at supporting the transition to a circular economy. The ecosystem described is designed to improve collaboration and verification processes, particularly in the context of ETV.

By fostering a favourable environment for innovative technologies, these ecosystems not only facilitate the adoption of ETV, but also promote the dissemination and replication of project results. This model can serve as a valuable reference for other countries or sectors seeking to exploit the benefits of ETV, enabling them to capitalise on the opportunities it presents for sustainable development and technological progress.

By showcasing use cases and success stories, these ecosystems can inspire confidence and encourage broader engagement in ETV practices, ultimately fostering progress towards environmental sustainability and innovation.

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MONITORING, ACCEPTANCE LIMITS AND HEALTH CONSEQUENCE OF ODOUR NUISANCE: A SHORT LITERATURE REVIEW ON THE STATUS OF THE ART

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Abstract

The increased sensibility of people occurred in the last years towards the environment and the quality of life have led to the classification of odours as harmful atmospheric pollutants. Exposure to odours, pleasant as unpleasant, can generate different effects in peoples ranging from emotional reactions annoyance up to indirect health consequences due to stress reactions, affecting to a not negligible extent the overall quality of life. The monitoring of odour nuisance is then one of the growing environmental issues affecting the human life in industrial, rural, and residential areas. The main methodological approaches for odour detection developed in the past decades includes odour impact criteria and field inspections. The former typically relies on the use of physic-mathematical models for odour dispersion, able to consider both meteorological and topographical features of the area, returning the odour concentration (ouE/m^3) in the surrounding area. The latter involves the use of panels of assessors for detecting the effective exposure to odour and the extent of odour plumes. By the way significant differences exist in the worldwide regulations concerning the odour limit concentration, the percentiles of yearly hours, the averaging time and the peck-to-mean factor. Additionally, epidemiologic study concerning health consequences on people exposed to odour, resulted affected by several methodological biases. This makes the finding reported in the different studies not always fully reliable and meaningful.

Key words: exposure assessment, health effects, impact criteria, international regulations

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1. Introduction

According to Hangartner et al. (1989), odorants are defined as those substances able to stimulate the human olfactory system leading to the perception of an odour. Meanwhile, an odour can be defined as an organoleptic attribute of certain volatile substances once these are sniffed and hence perceived by olfactory organs (ISO, 2008).

The perceiving of odorous phenomena represents a matter of potential harassing for local population that requires adequate responses by local

authorities. Inhalable volatile chemical species able to generate the olfactory perception are well known (e.g. sulfides, mercaptans, ammonia, amines, ketones, alcohols) (UNI, 2022). Based on the current level of knowledge, it is not possible to establish absolute odorants concentrations thresholds able to prevent the risk of annoyance. The reason of this difficulty is addressable to many factors among which: the high level of subjectivity of each individual in being sensible to the specific odorants; the habituation of individual to given odours (Conti et al., 2020); the synergic effect that the mixture of the different volatile

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chemical species can have in emphasizing the odour perception; the specific urban, industrial and/or rural context (DEPA, 2002, 2009; MASE, 2023; Pringer et al, 2015).

While EU legislation has clearly identified the best available techniques (BAT) for at-source odour emission control in each industrial sector (EU, 2010), odour annoyance continues to be reported by local populations in several areas. The determination of the effective exposure of the local population to odorous phenomena and whether these phenomena can be effectively deemed acceptable or unacceptable represent a complex issue. In general, the approaches exploited for this purpose are based on two main concepts: the assessment of the concentration of the odorous compounds; the duration and the frequency of the odorous perception (EN, 2016a, 2022).

The determination of the first aspect is based on the application of atmospheric dispersion models (UNI, 2000) able to consider both the meteo-climatic and orographic characteristic of the area under study, once the amount of odorous emission has been determined according to the EN (2016 a, b). The determination of the second aspect, *i.e.* the frequency and duration of the odorous perception represents a quite complex task. Many approaches have been proposed mainly based on simulation models and on the exploitation of human assessors panel, experts, able (*i.e.* appositely trained) to recognize the presence of odours and their features (EN, 2022).

Based on what discussed above, the present paper provides an overview of the main methodologies and acceptance limits related to odour detection and control currently employed worldwide. Additionally, due to its significance, an investigation into the health consequences associated with odour nuisance has been also conducted, and the detected finding discussed.

2. Odour impact criteria

Based on the current impact assessment techniques, the dispersion of odour at different distances from a specific source is determined through dispersion models, which can predict calculate the odour concentration at designated receptors by considering the effects of topographic and meteorological conditions. This result can be obtained once the odour emission rate (*i.e.* odour concentration multiplied by the whole flow rate) per each given source in the area under investigation was determined. Once calculated, the concentrations are compared against the odour impact criteria (OIC) which are represented by the legal limits and parameters imposed by local authorities. In general, OIC consists of the following main elements (Tables 1-2):

- 1) threshold of odour concentration;
- 2) level of percentile compliance;
- 3) averaging time adopted for the calculation of the concentrations returned by the atmospheric dispersion models;
- 4) peak phenomenon.

Level of percentile indicates, in general, the number of hours on yearly basis during which the average odour concentration has to be lower than the threshold limit. In other words, for the number of hours exceeding the percentile level, an odour concentration higher than the odour concentration limit is considered acceptable. In general, percentile level adopted is $\geq 98\%$.

Time periods considered for averaging the concentration include usually the single hour of the year, *i.e.* 8760 h/year, but also lower periods like second and/or minute are exploited.

Finally, another important aspect associated with the odour concentration is the peak phenomenon that can occur in periods < 1 h causing the momentary sensation of odour. For considering this phenomenon, a multiplicative factor named peak-to-mean ratio is generally used for correcting the hourly average odour concentration (Conti et al., 2020).

2.1. Odour concentration

Odour concentration, together with its detectability, is a widely exploited approach in regulations for indicating the acceptance limits for local populations also based on the destination of the area and on the distance of the receptors from the emission source. Due to the large variability in the sensibility of individuals against olfactometric stimulations, the concept of detectability is associated to a given percentage of individuals able to perceive the presence of odour.

The determination of odour concentration at the emission source is performed in specific laboratories by the dynamic olfactometry procedure exploiting as sensor human noses of appositely trained panel of assessor. This because there are no other instruments or analytic procedure able to return satisfactory information about the olfactometric responses of humans. The procedure for the dynamic olfactometry has been standardized in different countries by apposite technical notes: for the EU UNI EN 17325:2022 (UNI, 2022); for Australia and New Zealand AS/NZS 5323.3:2001 (AS/NZS, 2001); for US ASTM E679-04 (ASTM, 2011).

In the EU 1 odour unit (ou_E) is defined as the specific concentration of a reference odorant, n-butanol. The concentration of n-butanol corresponding to 1 ou_E/m^3 is 0.04 mol/mol of n-butanol in neutral gas. This definition is then used to trace the odour unit of any odorant or mixture of odorants in ambient air. Briefly, the analytic procedure of the dynamic olfactometry consists of determining the odour perception threshold (OPT) by submitting to the assessor's panel diluted mixture of a given volume of the sample of odour air. The dilution is performed by given volumes of pure (*i.e.* deodorized) air, expressed in m^3 , and at the sample is conventionally assigned the values of 1 ou_E . This mixture is submitted to the assessor's panel at decreasing dilution ratios.

The OPT is then defined as the dilution ratio (ou_E/m^3) that is perceived by 50% of the exposed

populations and, for the dynamic olfactometry procedure, by 50% of the assessors (UNI, 2022). For a given sample of odorant or mixture of odorants, the higher is the ou_E/m^3 the stronger is its olfactive impact. The standardized procedure also indicates procedure for the odour air sampling as the other conditions and criteria to be respected during the dynamic olfactometry test. Additionally, as largely describe in the study of Hayes et al. (2023), the composition of the odours mixtures can also be determined by the exploitation of gas chromatography coupled with mass spectrometry or similar techniques. However, in practice, this is primarily useful for assessing the potential presence of harmful compounds even if this approach results of particular interest also for a deeper investigation of odour nuisance. In fact, as also reported in the study of Barczak et al. (2017), humans have a very different response and perception when exposed to the same volatile substances. The so-called hedonic tone of the odour can be perceived as pleasant by someone and unpleasant by others or the odour cannot be perceived at all. Furthermore, the different substances can have also very different effects on individuals depending on the concentration and on mixtures compositions. In other words, the hedonic tone can be classified as pleasant even if the substances generating this perception can be harmful and *vice versa*.

2.2. Atmospheric dispersion modelling

The presence of several technical and analytical obstacles for direct experimental determination of the odour concentration in the ambient air, requires the exploitation of dispersion modelling able to return the values of the ou_E/m^3 in the selected receptors once the odour emission at the source have been defined.

Qualitatively, dispersion modelling of airborne particles and/or aerosols aims to describe and predict the fate of chemicals in the atmosphere from a given source to given points (receptors). These last can also be represented by lines characterized by the same concentration of chemicals, isolines. From the quantitative point of view these models can return the concentration of chemicals and, in the specific case, of

odours (ou_E/m^3), once the meteorological parameters, sources feature, and topography of the analysed area were defined.

Simulation of atmospheric dispersion of pollutants can be performed by the following main approaches:

- Lagrangian approach, in which the variation of concentration is related to the fluid in movement;
- Eulerian approach, in which the concentration is related to a fixed system of coordinates;
- Gaussian approach, in which the distribution of pollutants and hence their concentration is determined by using a normal probabilistic distribution of fluctuation of wind vectors;
- Semi-empirical approach, mainly based on empirical parametrization;
- Stochastic approach, in which semi-empirical and statistic models are used to analyse periodicities, trends and interrelationships of air quality measurements and to forecast episodes of air pollution;
- Receptor approach, in which starting from the concentration observed at the receptor the contribution of different emission sources is estimated.

More recently, also computational fluid dynamics (CFD) was proposed (Yeo et al., 2020) as an approach for analysing the odour dispersion from pig house with complex terrain.

In general, the most widely used models for the determination of odours are represented by the AERMOD and CALPUFF for the Gaussian approach and AUSTAL2000 for the Lagrangian approach (Capelli et al., 2013). In any case, in Table 1 are reported the most diffused software available on the market with brief indications about the features, advantages and disadvantages.

2.3. Peak-to-mean factor

As already described in the previous paragraphs, calculation models are largely indicated by regulations as the most suitable method for the determination of the dispersion of odour emitted in the surrounding area of a given source.

Table 1. Software, approaches used and mean features

Software	Approach	Features	References
AERMOD (U.S.)	Gaussian	Enable to point, volume and areal sources	Baawain et al. (2017); Schulte et al. (2007),
LODM (Canda)	Gaussian	Oriented for livestock facilities	Yu et al. (2013a), Yu et al. (2013b)
STINK (Australia)	Gaussian	Specific for ground level areal sources	Galvin et al. (2004); , Smith (1995)
OdiGauss (Italy)	Gaussian	Enabled for odour from multiple sources	Danuso et al. (2015)
INPUFF-2 (U.S.)	Gaussian	Emission from semi-instantaneous and continuous points sources	Asadollahfardi et al. (2015); Zhu et al. (2000),
CALPUFF (U.S.)	Lagrangian	Multi-layer, multi-species, non-steady-state, evaluating the effect of time- and space-varying meteo conditions	de Melo et al. (2012);, Ranzato et al. (2012)
CALGRID (U.S.)	Eulerian	Photochemical, transport and dispersion model, three dimension temperature and air velocity	Scire et al. (2000);, Yamartino et al. (1992)

By the way, the hourly average odour concentrations in the air (ou_E/m^3) returned by these models resulted not adequate to account for the fluctuations of the odour concentration that is usually characterized by a sequence of peaks and null ou_E/m^3 values (Conti et al., 2020). The shorter is the averaging period considered, the higher are the concentration (ou_E/m^3) fluctuations. Considering that a typical human inhalation occurs every 1.6 seconds, the exploitation of the hourly average can result in inadequate method for assessing the presence of olfactometric nuisance. In other words, olfactometric nuisance could be masked by the hourly average.

To account for this phenomenon, the peak-to-mean approach has been proposed, aiming to calculate the short-term odours concentration starting from the hourly average (Drew et al., 2007). According to Smith (1968), Schauburger et al. (2012) and Piringer et al. (2015), the calculation of short-term concentration can be calculated starting from the long-term ones by using the correlation reported in Eq. (1).

$$C_p = C_m * (t_m/t_p)^n \quad (1)$$

In Eq. (1) C_p and C_m represents the short-term and the long-term concentrations, respectively, t_p and t_m the shorter and the longer averaging time, respectively, to whom the previous concentrations are referred whereas n is an empirical dimensionless exponent ranging from 0.2 to 0.5.

Starting from Eq. (1) a peak to mean factor F has hence been introduced, Eq. (2), also at regulatory level, for accounting C_p based on the C_m generally calculated on hourly average. The longer is t_m the higher is F .

$$F = C_p / C_m \quad (2)$$

2.4. A glance to main world regulatory frameworks

Table 2 summarizes the values reported by the main regulations in some countries around the world for the parameters associated with the odour impact criteria described in the previous paragraphs. These data have been retrieved both from the specific regulations of each country and area and from the work of Bokowa et al. (2021) and Conti et al. (2020). As it is possible to note there are some relevant differences among the countries but also among the different jurisdictions of the same country. For example, Australia showed a wide range of both odour concentrations limit but also of percentile and average time values, ranging from 1s up to 1h, based on the jurisdiction (e.g. Queensland, Tasmania, Victoria) and on other factors as the destination of the urban/industrial/rural areas considered. Similar considerations are also valid for other countries as Canada, Belgium and Netherlands.

3. Field inspection

Before the implementation of more relevant activities for preventing and decreasing the odours annoyance reported by the local population, the adoption of preliminary activities aimed to understand the importance and the extension of the problem is usually recommended. According to the current legislation (Table 2), the initial approach to be adopted in case of odours annoyance reported by the local populations, is represented by field inspections. In fact, the only presence of odour is not enough for being classified as a nuisance, but it is also necessary to establish its frequency.

For this reason, the objective of field visits is not to determine if the acceptance threshold has been exceeded but primarily to assess whether the frequency at which local populations are exposed to an odour result in a nuisance.

The methodologies adopted for this purpose are the grid and the plume methods (EN, 2016a, b). Both methods used a panelist of variable number of assessors depending on the number of measures to be performed. The grid one is more oriented to characterize odour exposure in a given assessment area whereas the plume one is more oriented to assess the extension of the odour impact (i.e. where the odour is perceived and where not) under specific emission and meteorological conditions (e.g. wind direction, rain, humidity, temperature, pressure). In the grid method the area under investigation is divided in a grid of a given number of squares with minimum side of 250m. The vertex of each square represents the point in which the panel of expert assessors makes the measurement. One measurement consists of 10 measurements performed over 10 minutes. Every 60 seconds the members of the panel are requested to sniff for 10s and note on a specific form, according to a preordered scale of values, the intensity and the quality of the odour perceived. For considering the different meteorological conditions and the odour fluctuation, the whole duration of the field inspection is generally established in 1 year. Grid methodology can be applied also in presence of several odours emission sources.

In the plume method two different approaches can be adopted: static and dynamic. For the static approach, the panel of experts moves upwind the source along parallel lines that are perpendicular to the plume extent. The goal is to determine the presence and the absence of odour along these lines. For the dynamic approach the panel of expert moves according to a zig-zag pathway with the same aim of determining the extension of the plume.

The plume method is applicable only when there is a given source of emission. In both methods the panel of expert have to be appositely trained according to what reported in the UNI (2022).

Table 2. Averaging time, limits for odour concentration (ouE/m³), associated percentile level (%) and peak to mean factor (F) for some countries

Country	Aver. Time	ouE/m ³ / Percentile (%)	F	References
Austria	5s	1/92 and 5/97	Variable	OAW (1994)
Australia	-	5/98 (Queensland)	10 (stack)	EHP (2013), DAFF (2012); DEC (2006), DEP (2002), SAEPA (2007), EPA Victoria (2001), EPA Tasmania (2004)
		5/99.5 (Queensland)	5 (ground)	
	3s	-(logD-4.5)*0.6 ⁻¹ /99 (New South Wales)	1.9-2.5	
	3min	2/99.5 (West) 4/99.9 (West) 4/99.9 (Victoria)	-	
	-	2.5/99.5 ((Queensland)	2	
	3min	-(logD-4.5)*0.6 ⁻¹ /99.9 (South)	-	
Belgium	1h	6/98 (pigs) 10/98 (poultry)	1	Government Walloon (2009) LNE (2008) VITO (2012)
Canada (Ontario)	10min.	0/95	1	MDDEP (2011, 2012)
Denmark	1min.-1h	5-10/99 (residential) 10-30/99 (industrial, rural)	7.8	DEPA (2002,2 009)
France	1h	5/98 (Composting plant) 5/99.5 (at 3km new animal by-product processing plant) 5/98 (at 3km existing animal by-product processing plant) <5 (at 500m for populated areas for other activities)	1	JORF (2003, 2008)
Germany	1s	0.02-0.1-0.15	-	GOAA (2008) , TA-Luft (2002)
	1h	0.5 (pleasant odours) 1.5 (poultry) 0.75 (fattening ptig) 0.5 (milking cow, fattening bulls, horses)	-	
Hong Kong	5s	5	N.A.	EPD (2016)
Italy	1h	1/98 (residential, hospital) 2/98 (market, offices, turistic) 3/98 (rural, leisure, sport) 4/98 (industrial, farm) 5/98 (country, unpopulated)	2.3	ARPAP (2014); MASE (2023); Provincia di Trento (2016); Regione Lombardia (2012)
Israel	5s-10min	1.5-10	N.A.	IMEP (2013)
Netherlands	1h	5/98 (existing situations) 1.5/98 (new situations) 0.5/98 (new sources)	1	InfoMil (2014, 2016); VROM (2006, 2007)
Spain	1h	3-7/98	1	DMAV (2005)
UK	1h	1.5 (most offensive odours) 3 (moderately offensive odours) 6 (Less offensive odours)	1	Bokowa et al. (2021); EA (2011)
New Zeland	1h	1/99.5 (high sens./unstable) 2/99.5 (semi unstable) 3/99.5 (high sens./stable)	1	NZMOE (2003)

Another preliminary approach available consists in the direct engagement of the same individuals, populations, reporting on the presence of odor. These are requested to note on a specific preset form record each odour nuisance episodes perceived, its intensity together with a description of the type of odour. This represents a less expensive approach, useful for the determination of the annoyance, but lacking scientific stability of the data due to psychological effect of citizens.

4. Odour pollution and human health

The toxic effect that specific odorant compounds, as H₂S, NH₃, COS can have if inhaled above the chronic reference dose (RdF) (mg/m³) (Barczak et al., 2022) is a well know aspect. Les clear appears what effects can indirectly generate on human health the short and the long-term exposure of odours due to stress reactions. In fact, odour nuisance can also be generated by synergic effect of mixtures of

different compounds even if the single concentration of each compound is largely below the odour perceiving and toxic thresholds. According to Guadalupe-Fernandez et al. (2021), this is due to a lack of standardized methods usable for environmental epidemiology studies able to include also specific psychological aspects and life quality of each single individual.

Several sources of bias have influenced the overall quality and reliability of the studies reported in the literature. Key biases include the identification of exposed and non-exposed populations, confounding factors such as lifestyle and exposure to other sources, the completeness of outcome data, and the application of appropriate statistical methods. Many of these studies are based on self-compiled questionnaires on which the engaged subjects report both the perceiving of odour annoyance and their symptoms.

Based on the discussion above, the already mentioned study of Guadalupe-Fernandez et al. (2021) identified five high-quality epidemiological studies, four of which consider the exposure of populations to odour emissions from animal feeding and one from a waste landfill. Table 3 summarizes the main health outcomes self-reported by the subjects exposed/engaged along with the main biases detected for each study.

The lack of other objective or clinical methods other than questionnaires' (Wroniszewska and Zwodziak, 2020) reported by Guadalupe-Fernandez et al. (2021) as a limiting factor for the assessment of odour annoyance, appears suitable of improvement by its integration with new promising methods combining questionnaire with electronic nose and specific trained staff for on-site surveys (Valli and Immovilli, 2008).

5. Discussions

The information reported above highlight large

differences at world level concerning the criteria exploited for setting the dispersion model (e.g. OPT, F, percentile, averaging time) (Bokowa et al., 2021). Such differences were justified by several factors as the destination of the area, e.g. residential, industrial, rural, and of the activities performed, e.g. livestock, chemical industries, waste management, water management, and other similar activities.

Other differences can also be found on the minimum distances from the odour sources. In general, in Asia and China the odour issue resulted more focused on specific sectors like the waste management one whereas other country showed a particular attention to livestock (Conti et al., 2020). Another difference noted was related to the models and approaches exploited for odour dispersion and concentration. For this aspect, a combination of different techniques including questionnaire, dispersion models and chemical analysis appears the one indicate as the most suitable by several authors (Baczak et al., 2017; Bokowa et al., 2021; Conti et al., 2020). A particular critical situation was detected for the EU area. In fact, EU aims to homogenize the legislation of the different member states (MS) in many sectors by the adoption of EU directives. The environmental sector is one of the most relevant in which the EU is more active since the late 50' and in which there is continuous implementation of new and advanced legislation. In the case of odour, several aspects require further understanding and regulation to prevent significant disparities among Member States.

Similar considerations can also be done for the field inspection approaches. In fact, despite the presence of specific technical notes able to standardize and to homogenize the methodologies and procedures to be adopted for the inspections, there is a lack of indications on threshold values (EN, 2016a). Such lack represents a matter of possible biases causes some bias for in assessing whether or not a population resulted exposed.

Table 3. Emission source, methodology used for the exposure assessment, health outcome assessment and main biases for the five epidemiologic studies classified of higher quality by Guadalupe-Fernandez et al. (2021)

<i>Author</i>	<i>Emission source</i>	<i>Exposure assessment</i>	<i>Outcome assessment</i>	<i>Main bias</i>
Avery et al. (2004)	Animal feeding	Self-reported questionnaire	Clinical measurements: Immune functions and allergy	Exposure assessment Confounding factor (other exposure)
Heaney et al. (2011)	Waste landfill	Self-reported questionnaire	Self-reported questionnaire: Gastrointestinal Mucus irritation Respiratory Skin	Exposure assessment Outcome assessment
Horton et al. (2009)	Animal feeding	Self-reported questionnaire	Self-reported questionnaire: Mood states Odour nuisance	Exposure assessment Outcome assessment
Schinasi et al. (2011)	Animal feeding	Self-reported questionnaire	Self-reported questionnaire: Gastrointestinal Mucus Respiratory Skin	Exposure assessment Outcome assessment
Wing et al. (2013)	Animal feeding	Self-reported questionnaire	Cardiovascular	Exposure assessment Confounding factor (other exposures)

Furthermore, this can also represent a matter of weakness of the odour exposure assessments that can generate criticism by the different actors involved in such investigations.

Finally, more research appears necessary for a definitive assessment of the impact of odour on human health. In fact, excluding the toxic effect of some well-known chemical compounds already regulated by specific legislations, the epidemiologic studies performed on the health consequences of individuals exposed to odour, in general, are characterized by the presence of several biases as: not proper consideration of confounding factors; absence of analytical results (large use of self-questionnaire); lack of adequate description of the source of the emissions; absence of adequate procedure able to give a clear and more deterministic indication about the effective exposure of population to odour.

6. Conclusions

The review performed in the present study highlighted that more research activity resulted necessary for a definitive solution of the different aspects involved in odour nuisance. Despite the quite extensive availability and reliability of physico-mathematical models and procedures, the assessment of the effective level of exposition and of the acceptable limits of odour concentration still remains a matter of discussion.

Many social, cultural and individual factors influence these aspects justifying the large differences of the values of the main odour impact criteria adopted worldwide. A decisive contribution to the definition of appropriate values for the above-mentioned criteria can be represented by scientific results concerning epidemiologic studies on population exposed to such nuisance.

Based on the state of the art, these studies are often influenced by numerous biases and methodological flaws, rendering the associated findings less reliable and challenging to utilize for this purpose. Among these drawbacks, the lack of clinical methodologies, other than questionnaires, able to definitively assess the exposure of the population to odor, represents one of the main limitations even if new integrated approaches seems able to give adequate response to this aspect.

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METHODOLOGY AND INFORMATION AND COMMUNICATION TECHNOLOGIES TOOL TO MEASURE AND COMMUNICATE PRODUCT CIRCULARITY

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Abstract

A methodology and an Information and Communication Technologies tool tailored to companies, in particular Small and Medium Enterprises, were developed in the framework of RECiProCo project, for measuring product circularity and water use, through a simplified and easy to be applied set of indicators based on a life cycle approach, considering all the phases of product life cycle. The indicators were integrated on a free of charge web Platform to be easily managed by the company, with the possibility to make available data and information to the public, i.e. final consumers, and other companies, to stimulate the transition to circular economy goals and environmental conscious purchases. A special focus was given to the indicators that could be useful to the paper, textile and construction sectors. These sectors, very important for the Italian industry and consumer's purchases were chosen for their relevance in terms of opportunity to improve the circularity of products and efficiency in water use. The circularity and water use indicators were tested in a sample of companies that found them easy to collect and calculate. The pilot companies participated with great interest and had the opportunity to increase their awareness about the environmental data related to their products and production processes and to identify actions to improve the circularity aspects of the product analysed. The RECiProCO web Platform can be used by companies to measure and monitor product circularity, to target improvement actions and to communicate to other stakeholders, in particular consumers, but also their customers or suppliers.

Key words: circularity indicators, consumers, water use, web platform

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1. Introduction

The transition to circular economy and sustainable development are major challenges for society in general and for organisations in particular. The European Commission has defined the frameworks for this paradigm shift with the adoption of the European Green Deal (EC Communication, 2019) for achieving the goal of climate neutrality by 2050, and the New Circular Economy Action Plan (EC Communication, 2020). The final objectives are the

conservation of resources and materials value as well as the reduction of waste and residues. In order to achieve these goals, both parameters, methodologies and indicators, especially addressed to companies and territories, must be defined to measure the distance to the targets and to assess the progress resulting from the application of specific circularity strategies.

The measurement of circularity for both products and services is a crucial step in the evaluation of resource efficiency, the definition of material and energy flows, and the identification of value and

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competitiveness components in processes. Furthermore, it is an essential tool for both monitoring progress against sustainability targets and providing companies with valuable insights to implement targeted strategies with the final aim to enhance sustainability. It is only through the measurement of circularity that the achieved results, or those towards which companies and society are striving, can be communicated in a reliable and transparent manner.

Nevertheless, the assessment of circularity is a complex issue that extends beyond the mere measurement of advancements in material recycling and reuse initiatives. It also encompasses the evaluation and communication of the advantages associated with the adoption of novel business models that are geared towards the transformation from linear to circular production and consumption models. In order to facilitate this transition, new measurement metrics have to be developed. Numerous initiatives have been carried out by organisations with different methodologies and approaches, all aimed at measuring the degree of transition to circular models (Circle economy and PACE, 2020).

The landscape of available tools appears to be very heterogeneous, but main features common to the different approaches can be identified. One of the key feature is the level at which circularity should be measured: the macro level (country system), where public policy attention is focused on circularity in relation to sustainable production and consumption models applied to urban and production systems (Yuan et al., 2006); the meso level (sector/industry), such as industrial eco-parks that take advantage of shared infrastructure and exchange of materials, waste, services and energy to improve circular performance; the micro level (organisation/company), i.e., the level of companies and productions committed to improve their environmental performance by adopting sustainable production models to save raw materials, use recycled materials and apply sustainable product design; the micro level (product/asset/service/process) proposed by Saidani et al. (2017), which focuses on the circularity of products, components and materials along the entire value chain and throughout their life cycle.

Other relevant features are the measurement and evaluation methodologies, which can be qualitative or quantitative; the types of resources to be included in the evaluation method; their classification as inputs or outputs; the main objective of the measurement exercise; the validation method in which the measurement is realised (certification, written-declaration or self-declaration) (Alliance for the Circular Economy, 2021).

Numerous literature studies have proposed an analysis of tools for measuring circularity based on several factors, such as the purpose and type of tools (assessment tools or guidelines to guide strategic choices), the level of accuracy, the type of data considered for measurement and the nature of indicators (qualitative or quantitative), the scope of

application (macro, meso, micro, nano) and the reports generated (Valls-Val et al. 2022).

At the European level, the document supporting circular economy policies is "A new Circular Economy Action Plan" adopted in 2020 (European Commission, 2020), which also presents a monitoring framework based on a set of indicators which can capture key aspects of circularity. New indicators on circular economy have been added to the four dimensions that identify the priority sectors and areas (production and consumption; waste management; secondary raw materials; competitiveness and innovation) towards which the plan's actions are directed, following the 2023 review (Eurostat, 2023). Specifically, new indicators to monitor material efficiency (material footprint and resource productivity) and new indicators to monitor whether EU consumption is within planetary boundaries (consumption footprint) have been implemented. Therefore, while the framework for measuring progress towards circular economy is defined at EU level (macro level), the micro level lacks an organic and standardized framework of measurement indicators (Valls-Val et al., 2023).

Various experiences to measure circularity have been developed at European and national level. An important standardisation tool applied at meso, micro and macro level is the one proposed by the Technical Committee International Standard Organisation (ISO) 323 "Circular Economy" (abbreviated as ISO/TC 323), which has been working since 2018 on the definition of a package of standards (ISO 59000 series on Circular Economy) consisting of a total of seven standards which define requirements, guidelines and supporting tools for the implementation of activities by all organizations involved, in order to maximize their contribution to sustainable development.

Moreover, the Global Reporting Initiative (GRI) through the development of GRI 306: Waste 2020 (GRI Standards, 2024) also provides a certification for companies of all sizes, sectors and geographical locations to report on waste-related impacts.

When considering the tools applicable at the company level (micro level), the most tested is the one launched by the Ellen MacArthur Foundation, a private foundation established in 2009 which researches and invests in the dissemination of circular economy issues. Launched in 2020, Circulytics (Ellen MacArthur Foundation, 2020) allows companies to understand their position towards circularity. This tool evaluates quantitative and qualitative parameters by a system which considers 'enabling factors', i.e. indicators to assess the conditions that enable a company to innovate and move forward, and 'outcomes', activity-specific indicators which measure inputs and outputs of a production process. Companies will receive an overall score based on customised factors, information and comments provided by Ellen MacArthur Foundation, which can be used in investor

and customer relations, while the Foundation commits to creating "inspiring" case studies for high-scoring companies. Currently, the Foundation is moving away from data collection and individual performance assessments based on Circulytics by encouraging organisations to disclose their circular economy performance as outlined in the European Sustainability Reporting Standards (ESRS 5) (EC Delegated Regulation, 2023).

Moreover, at the micro level, it is worth mentioning for Italy the Circular Economy (CE) Client Report tool of ENEL X (<https://corporate.enelx.com/en/our-commitment/sustainability/circular-economy/client-report#works>), where ENEL X offers a consultancy service for companies and public administrations to design a roadmap to increase their level of circularity. The analysis is based on qualitative and quantitative data and returns a report with a detailed view of the initial level and achievable targets in terms of circular economy, with the value of a self-declaration validated by a third-party certification body. Moreover, at the Italian level, the ICMQ (Istituto di certificazione e marchio qualità per prodotti e servizi per le costruzioni) has developed a product circularity index (NCI), which provides some indicators to measure the circularity of materials, energy, water and waste of the organization (<https://www.icmq.it/materiali-prodotti/certificazione-nci.php>).

Nevertheless, the analysis of the already available Italian tools and indicators for circular economy measurement and monitoring, although of primary importance to guide industries towards circularity and water efficiency as well as to improve the environmental performance of their products, shows that they can be difficult to apply, they are not always available free of charge but sometimes should be purchased by companies, and could require the support of external consultants for their application and for calculation procedures.

In order to overcome this problem, and to provide an easy tool to monitor and evaluate product circularity and water use, a methodology and an Information and Communication Technologies (ICT) tool tailored to companies, in particular Small and Medium Enterprises (SMEs), for measuring product circularity and efficiency in water use, were developed by ENEA in the framework of RECiProCO project (<https://www.reciproco.enea.it/>), funded through an agreement between ENEA and the Italian Ministry of Economic Development for the "Development of circular economy tools and initiatives for consumers".

The term 'product circularity' is used to describe a set of practices designed to optimise the use of resources and minimise waste throughout the production and consumption cycle of a product. This approach emphasises two key goals: sustainability and economic efficiency. RECiProCO was a wide project, which, in broad terms, aimed to support more sustainable and conscious consumers' choices and to

increase consumers, companies and citizens awareness about circular economy principles and approaches.

In particular, one of the project purposes was to carry out a feasibility study for the development of a voluntary environmental communication system for non-food and non-energy products, based on two types of indicators: a set of circularity indicators and a set of water use indicators, following a life cycle approach.

The sets of circularity and water use indicators, usable by all types of companies and products, except food and energy ones, as required by the project, are easy to be applied and referred to the product. Moreover, they were tailored to companies, in particular Small and Medium Enterprises (SMEs), to increase their awareness about circular economy principles and to support them towards the application of circular practices in their production processes, since companies, especially SMEs, could still have little knowledge or expertise about circular economy practices, methods and tools.

In addition, an ICT tool called "RECiProCO web Platform" was developed on a web platform; this tool contains the set of indicators and can be used directly and easily by companies, with the help of ENEA, if needed, to measure and monitor product circularity (self-evaluation), to target improvement actions and to communicate to other stakeholders, in particular final consumers but also their customers or suppliers. Moreover, the web platform allows companies to make available data and information to the public. Finally, a restricted number of indicators was identified for the communication to consumers through a QR code, for a more sustainable and immediate Business to Consumer communication, thus helping consumers in their purchase choices, with the final aim to boost the production of circular and water use efficient products.

The Agreement between ENEA and the Ministry of Economic Development prescribed that the set of indicators must be particularly suitable for the paper, textile and construction sectors. These sectors were chosen for their relevance in terms of opportunity to improve the circularity of products and efficiency in water use. Moreover, following the Agreement prescriptions, the indicators were tested in a sample of companies of the above-mentioned sectors, to develop the final version of the ICT tool, which was implemented on a free of charge web platform. ENEA helped companies, where necessary, to apply the set of indicators and verified the correctness and accuracy of both collected data and calculations, thus acting as a third-party reviewer.

This paper describes the methodological approach that led to the development of the set of indicators and the ICT tool; the testing phase in companies and the main results achieved; the main characteristics of the RECiProCO web platform and the description of its use by companies as well as final considerations and future developments.

2. Materials and methods

For the purpose of project activities, a preliminary survey was made on the main circularity and sustainability measurement tools, circularity indicators and environmental certifications which mainly affect the micro and nano level, i.e., applicable at the company level and at the product level, also identifying the potential that these tools, indicators and certifications have for the communication to the final consumer or for supporting companies to reduce the environmental impacts of products from an eco-design and circular economy perspective.

In the next paragraphs the methodological approach for the definition of the final sets of circularity and water use indicators and the design of the main features of the RECiProCO web Platform are described.

2.1. Development of the circularity and water use indicators

The circularity and water use indicators were developed to be used at the product level and are usable for all types of products, except for food and energy products, as required by the project; furthermore, they were identified following a life cycle approach, i.e. taking into account all phases of product life cycle, with a holistic approach. The Agreement with the Ministry of Economic Development prescribed that focus, in particular for the pilot phase, must be given to three production sectors: textiles, paper and construction.

The above-mentioned sectors were chosen for the following reasons:

- interest for the final consumer and interest in a Business to Business perspective;
- importance in relation to circularity and water use (e.g., recycled content in textile, paper and construction products; potential product recyclability at the end-of-life; high water use in the production process or during the whole life cycle; content of by-product in the main products; product made of reused materials);
- relevant regulations at national Italian level: e.g., 110% bonus for construction (IG, 2020a), new legislation for mandatory separate collection of the textile fraction in municipal waste in accordance to the Legislative Decree 116/2020 (IG, 2020b), End of Waste 188/2020 regulation for paper and cardboard (MATTM, 2020), Legislative Decree 116/2020 on packaging labelling for collection/reuse/recovery/recycling and nature of materials used (IG, 2020b).
- availability of environmental labels and certification schemes based on life cycle approach, e.g. Ecolabel, Environmental Product Declarations, other sector labels, Minimum Environmental Criteria (Criteri Ambientali Minimi – CAM) for the Italian Green Public Procurement (GPP).

The following procedure was then followed to develop the methodological framework and to define the set of circularity and water use indicators, to be included in the ICT tool (Fig. 1):

- Literature analysis of some directives, regulations and product certifications schemes to identify and analyse the circularity and water use indicators already available at national and European level, with a specific focus on the three selected sectors. The main documents examined are the following:

- Public documents examined in the preparatory work of UNI CT (Italian Standard Body-Technical Committee) 057 "Circular Economy" related to the publication of the Italian Technical Specification UNI/TS 11820:2022 "Measuring circularity - Methods and indicators for measuring circular processes in organizations" (UNI, 2022); this document defines a set of indicators intended to evaluate, through a rating system, the level of circularity of an organisation or group of organisations.

- Circularity criteria available in the Italian Green Public Procurement (GPP) Criteria (Criteri Ambientali Minimi – CAM) for paper (MATTM, 2013), textile (MASE, 2023) and buildings (MiTE, 2022);

- Circularity criteria present in environmental product labels (both national and European/international), such as European Ecolabel, Environmental Product Declarations (EPD), other type I or type III environmental labels according to ISO 14020 (2022);

- Available methodologies and tools for estimating water stress and indexes for measuring the impact of water withdrawals and consumption, related to anthropogenic activities (Vanham et al., 2018), as well as various data platforms (e.g. Aqueduct 3.0 tool, developed by Hofste et al., 2022).

- Identification of a specific number of indicators that, at first analysis, were considered significant and useful for measuring product circularity and water use. These indicators were extracted from the aforementioned standards, regulations and certification schemes, with particular reference to the public documents examined in the preparatory work of UNI CT 057 "Circular Economy" and were suitably adapted and/or modified in order to choose a set of about 30 indicators, encompassing all phases of a product's life cycle, i.e. design, procurement, production, distribution/sales, use/consumption, end-of-use. These indicators have been considered representative of circularity and water use, are suitable for all types of products, except for food and energy ones, as required by the project, and are easy to be calculated by companies on a voluntary basis (Table 1).

- Once defined the set of indicators, dedicated meetings were organised with the main national consumers association, with the support of the

Ministry of Economic Development; during these meetings the methodology and type of indicators were shown to the consumers association in order to collect feedbacks and opinions, which were very positive, and plan any possible modification or change, with the final aim to bolster a more easy application by companies and improve the understanding of each indicator and its usability by consumers.

- Elaboration of a spreadsheet addressed to companies to support them in the calculation of circularity and water use indicators during the subsequent testing phase (see also par. 2.2). This spreadsheet aims to help companies to collect and retrieve data for the calculation of the indicators and contains appropriate columns: unit of measurement, calculation procedure, value obtained.

In order to identify indicators that are relatively simple to be calculated and that require a reasonable amount of time to obtain the necessary data for calculation, the spreadsheet also demands the

following information: difficulty in both finding data and in the calculation; time needed to find data for the calculation.

- Testing of the above-mentioned 30 indicators with selected companies of paper, textile and construction sectors, by means of calculation tests, carried out with the support of ENEA researchers (see par. 2.2).

- Validation of the final set of indicators: Selection of a small number of indicators for the development of a QR Code (Table 1, in bold) to be included in the ICT tool, to be affixed on products for easy understanding by final consumers, to whom this tool is mainly addressed. Similarly to the selection of the whole set of indicators, in this phase consumer's associations were consulted and their opinion were collected, in order to select the most understandable, effective and relevant indicators to be included in the QR code, which could easily use by consumers to support them during their purchase choices.

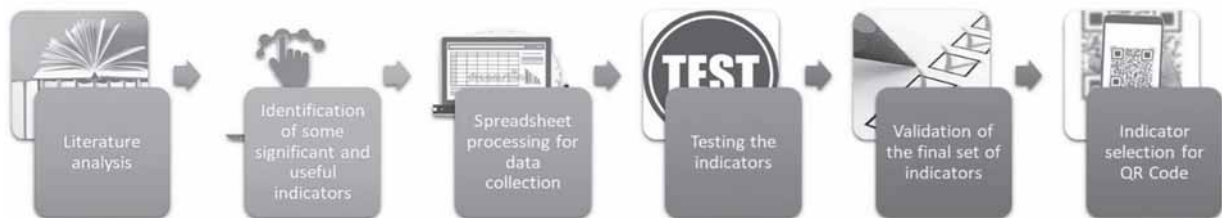


Fig. 1. Procedure adopted for the selection of indicators.

Table 1. Indicators of circularity and water use selected for the RECiProCO ICT tool. The indicators in bold are those selected for the communication to consumers (QR code)

Phase of the Life Cycle	Indicator	Type of indicator
Design	Is the product referable to circular design models?	Yes/No
Procurement	Percentage of by-products and/or secondary raw materials and/or recycled material used in relation to total material resources used	Quantitative
	Percentage of renewable raw materials used in relation to total material resources used	Quantitative
	Percentage of raw materials and secondary raw materials purchased and/or acquired from local suppliers (< 100 km) compared to total raw materials purchased and/or acquired	Quantitative
Production	Percentage of climate-changing emissions released in year n-1 compared to climate-changing emissions released in year n	Quantitative
	Percentage of renewable electricity consumed in relation to total energy consumption	Quantitative
	Percentage of renewable thermal energy consumed in relation to total energy consumption	Quantitative
	Percentage of self-produced electrical energy from renewable sources and/or recovery processes compared to total electrical energy consumed	Quantitative
	Percentage of self-generated thermal energy from renewable sources and/or recovery processes compared to total thermal energy consumed	Quantitative
	Percentage of waste sent for material recovery and/or recycling compared to total waste	Quantitative
	Percentage of by-products generated compared to total production residues	Quantitative
	Percentage of waste produced in year n-1 compared to total waste produced in year n	Quantitative
	Product-related water consumption	Quantitative
	Organisation-related water consumption (annual basis)	Quantitative
	Organisation-related water consumption (monthly basis)	Quantitative
	Percentage of water from recovery and/or recycling compared to total water consumed	Quantitative
Quantity of wastewater discharged (annual basis)	Quantitative	
Quantity of wastewater discharged (monthly basis)	Quantitative	

	Total amount of organic pollutants (total COD) in effluent before treatment (year n)/Total amount of COD in effluent before treatment (year n-1)	Quantitative
	Total amount of Total Suspended Solids (TSS) in effluent before wastewater treatment (year n)/Total amount of TSS in effluent before wastewater treatment (year n-1)	Quantitative
	Is wastewater treatment carried out at the farm or at centralised level (e.g. consortium purification plant, discharge to sewer)?	Qualitative
Distribution and sale	Percentage of primary packaging used per unit of product from renewable and/or recycled sources compared to total primary packaging used	Quantitative
Use/consumption	Is it possible to repair the product (Y/N)?	Yes/No
	Is it possible to replace its components? (Y/N)?	Yes/No
End of use	Is it possible to regenerate the product?	Yes/No
	Is it possible to reuse the product?	Yes/No
	Is the product disassemblable?	Yes/No
	Does the product have a suitable recycling chain?	Yes/No

2.2. The testing phase with companies

The activities of the testing phase were divided into three consecutive and closely connected phases:

- identification of companies from the three sectors by means of ENEA’s contacts;
- involvement of companies by e-mails and online meetings;
- data collection and calculation of indicators.

The first step was the identification of the companies to be involved. Thanks to the communication channels established through previous experiences, such as the Italian Stakeholder Platform on the Circular Economy (ICESP), the Symbiosis Users Network (SUN) and participation in national research projects, ENEA established contacts both with the trade associations of construction, paper and textile sectors and with individual companies in order to verify their interest and willingness to share company data aimed at calculating circularity and water use indicators of their products. Eight companies, representative of the Italian production from the three sectors, participated in the testing phase: two from the paper sector, four from the textile sector and two from the construction sector.

Once the companies were identified, the ENEA working group contacted organisations representatives by e-mail and organised first operational meetings with each company, aimed at both explaining the objectives of the project, and the methodology to be adopted. During the meetings, the first step was to identify, together with the company team, the product chosen for the testing phase. Then the set of indicators to be used (organised in the above-mentioned spreadsheet, described in par. 2.1) was explained to the companies and, at the same time, there were in-depth discussions on how to carry out the data collection.

The following products were chosen by companies:

- different types of paper for the paper sector;
- jeans trousers, coated fabric made with cereals, vegetable fabric obtained from pineapple peel and fair carpet for the textile sector;
- thermal insulation material made of 100% cereals and PVC flooring for the construction sector.

In order to calculate the indicators, company

data of different natures (e.g. data relating to consumption, products, waste and by-products leaving the company site etc.) and of different levels of detail were required.

Therefore, the importance of indicating the sources of the data used, as well as the procedure and any calculations made to provide the requested information, were highlighted during the discussion with the companies. In this phase companies were also asked to qualitatively evaluate the degree of difficulty encountered to obtain the data and the time needed to get the information. This phase aimed to evaluate the relevance, as well as the difficulty of application/calculation, of the complete set of indicators for the companies and their products.

According to such approach, a specific spreadsheet was produced by ENEA showing, for each indicator, the unit of measurement required, the method of calculation, calculation procedures and the final numerical values for each indicator. In addition to the value assumed by the single indicator, the following information was requested for each indicator and included in the spreadsheet:

- Value calculated/measured. The possible answer, selectable via drop-down menu was yes or no;
- Reason for non-calculation or non-measurement;
- Degree of difficulty encountered in finding the data. The possible answer, selectable via drop-down menu was: 'No difficulty', 'Medium difficulty' and 'Large difficulty';
- Time needed to find the data. The possible answer, selectable via drop-down menu was: "less than 30 minutes", "between 30 minutes and 1 hour", "more than 1 hour";
- Degree of difficulty encountered in the calculation. The possible answer, selectable via drop-down menu was: "No difficulty", "Medium difficulty" and "Large difficulty";
- Notes and sources of the data.

In order to provide a comprehensive understanding of the data collection process and the indicators calculation, some specific criteria and metrics were developed to assess the company difficulties encountered during these phases, with the final aim to increase the rigor in the testing phase (Table 2).

Table 2. Criteria and metrics for the definition of the different degrees of difficulty during data collection and indicators calculation

<i>Type of difficulty</i>	<i>Degree of difficulty</i>	<i>Definitions</i>
Data collection	None	Data are fully available in company management/accounting systems and can be retrieved easily and without any specific internal/external expertise
	Medium	Data are partially available in company management/accounting systems and can be retrieved with some specific operations /internal expertise/external expertise
	Large	Data are not available in company management/accounting systems and have to be searched by hand in bills/invoices/registers or only with the help of specific internal/external expertise
Calculation	None	All indicators are calculated by companies on their own in relation to the chosen product, without the need of any external help/expertise
	Medium	Some indicators are calculated directly by companies on their own in relation to the chosen product, whereas external help/expertise is needed to calculate the remaining indicators
	Large	Indicators are calculated only with the help of external expertise, which is needed to relate the collected data to the product (e.g. data are referred only to the whole company and the company needs help to relate them to the product)

Following this preliminary activity, which was carried out in the same way for each company ("formal" involvement via email and subsequent remote preliminary meeting), the work proceeded with each company in different ways, with different timing and critical issues, which were solved step by step by a joint cooperation between ENEA and the involved companies. Table 3 shows some of the problems encountered and the solutions proposed and implemented to overcome them.

2.3. Design of the ICT tool: the RECiProCO Platform

The RECiProCO Platform, developed by ENEA with the support of LASER ROMAE S.r.l, is an ICT tool which allows companies to publish, retrieve, measure and evaluate the circularity and water use indicators of their products, together with their relevant characteristics. The set of indicators contained in the Platform is that identified in the previous phases of the project. The Platform is a web application aimed at the acquisition, normalization and visualization of data relating to the circularity of products, to support companies in identifying areas of environmental improvement. The Platform is accessible through any web browser at the following link: <https://simulatore.reciproco.enea.it/#/home>; it is available in Italian, but a translation in English language will be evaluated in the next future.

The Platform architecture foresees two types of roles, supported by specific profiling mechanisms for The customisation of specific functions. The

predefined roles are: company employees/managers and Platform administrator (ENEA).

The employees/managers of a company, upon first login, can register and associate the user with their company. Moreover, they can insert products and circularity indicators by filling in guided questionnaires. ENEA, as the administrator, verifies and validates the data entered by companies, checking for any errors and requesting companies to correct them and enter any missing information.

The administrators have access to the lists of companies, products and circularity indicators included in the Platform and can search and export data. Moreover, the Platform implements a customized dashboard to display aggregate data in terms of products and indicators.

The Platform architecture is multi-layer, the various functions are logically separated, and are therefore divided into layers or engines. Each engine plays a specific role within the Platform architecture.

With reference to the architecture model, the engines are defined as follows:

- AuthEngine: Engine in charge of the authentication, logout and information retrieval functions of the current user;
- CryptoEngine: Engine that carries out entity encryption / decryption operations. It allows the encryption/decryption of a generic object or a file via the application's secret key or via a key passed as input. Its purpose is to encrypt the reporting entity and the files uploaded as "to be encrypted". Only one active CryptoEngine can be used at a time;

Table 3. Data collection critical issues

<i>Critical issues emerged during the test phase</i>	<i>Identified solution</i>
Difficulty in obtaining data for foreign suppliers of raw materials	Data collection focused on national suppliers
Difficulty in definition of the "upstream" system boundaries	Boundaries set starting from the supply of a secondary raw material input to company processes
Procurement of raw materials information from national retailers who refer to foreign producers	Procurement referred to the dealer present in the national territory
Inability to provide complete data on multiple years	Indicators based on data relating to several years not calculated
Presence on the national territory of sales offices, but production processes located abroad	Use of environmental labelling (for example EPD) for the (partial) evaluation of indicators

- **LoggingEngine:** Engine which carries out “logging” operations of the application. There are 5 “classic” log levels (DEBUG, INFO, WARN, ERROR and CRITICAL) and in its basic version it saves logs on the filesystem. There must be at least one active LoggingEngine; the Platform can manage multiple LoggingEngines (e.g. N on filesystem and remote M);
- **MetricsEngine:** Engine which calculates Platform statistics and supports different types of extraction (.XLS, .CSV, etc.). In its basic version it saves statistics on the application’s internal database and allows export in .XLS and .CSV format. Only one MetricsEngine is active at a time;
- **StorageEngine:** Engine that saves information relating to files, questionnaires and notification templates. In its basic version it saves this information on the filesystem. There must be at least one active StorageEngine; the Platform can manage multiple StorageEngines (e.g. 1 on filesystem, 1 on MongoDB). There is a background job that synchronizes between all active StorageEngines. The data format used is JSON;
- **WorkflowEngine:** Engine which perform the workflow of the phase transaction states. It allows the creation or removal of associations between two states, the start of a new process or its finalisation. In its basic version the workflow is saved on a database external to the application.

The Platform implements the entire treatment process, which involves, through complete automation of the workflow, the acquisition of data useful for calculating the indicators, the normalisation, processing, storage and visualisation.

Once the products and data useful for calculating the indicators have been entered, the Platform guides users through the transition of states, up to validation and publication. A selection of data relating to products and indicators can be inserted into a QR-Code. Finally, the Platform allows companies to calculate the level of circularity of the products inserted, for each life cycle stage, according to an algorithm which calculates an average value, expressed as a percentage, obtained from the ratio between the sum of all indicators in each stage and the total number of indicators available in that stage.

3. Results and discussion

3.1. Results of the testing phase with companies

The testing phase of circularity and water use indicators at company’s product level proceeded with a constant and close cooperation with ENEA, which provided continuous support to the organisations in order to collect the data and to calculate the indicators in the most appropriate way, to guarantee their completeness and correctness, to verify the data sources robustness and finally to ensure an adequate understanding of the calculation or estimation procedures adopted.

The companies joined the project with great interest and good cooperation with ENEA and most of them found few or modest difficulties in compiling the file containing the indicators; the average time required for finding the data and for calculation of indicators was 1 hour maximum. All data collected by the companies and included in the indicator tables (one table for each company/product) were reviewed and approved by ENEA, which acted as third-party verifier; in case of errors or lack of detailed information, companies were asked to review the data and calculations and correct the errors. In particular, ENEA applied specific verification procedures to verify if the data used were corrected, with the aim to ensure data reliability and accuracy. More in detail, ENEA checked data sources, how they have been searched and retrieved within company’s administrative or technical departments (e.g. from excel spreadsheets); moreover, ENEA verified the bills and invoices used, when needed, for each indicator, as well as the calculation procedures, checking if allocation procedures were needed to relate the data to the chosen product and finally if the calculation was correct or if it needed revision or adjustments. Figure 2 collects the feedback obtained from the eight companies that participated in the testing phase. All the eight companies completed the spreadsheet with their data, their calculations and the final indicators values. The elaboration of the data collected in the spreadsheets showed that most companies (81%-91%) had very low difficulties in collecting data and calculating the indicators, while some of them (7-18%) had medium difficulties (Fig. 2). Only 2% of companies had high difficulties (see Table 2 for difficulties criteria). Examples of difficulties, defined by Table 2, include the need to search data in bills and invoices because the company did not have a specific accounting system for the type of environmental data needed to calculate the indicators, or the help provided by ENEA to the company in order to perform allocation procedures needed to calculate, for instance, the amount of energy consumption of the chosen product, in case the company had only the energy consumption of the whole factory (i.e. not related to the product).

As regards the time necessary to collect the data for the calculation of the indicators, it turned out that a large part of the information was collected in less than 30 minutes (87%), a small part (12%) between 30 minutes and 1 hour and only 1% of the data took more than an hour.

3.2. The final set of circularity and water use indicators and the RECiProCO web platform

The results of the testing phase of the set of indicators and the spreadsheet for data collection and indicators calculation confirmed the validity and feasibility of the set of indicators previously identified (Table 1) which also met the requirements of:

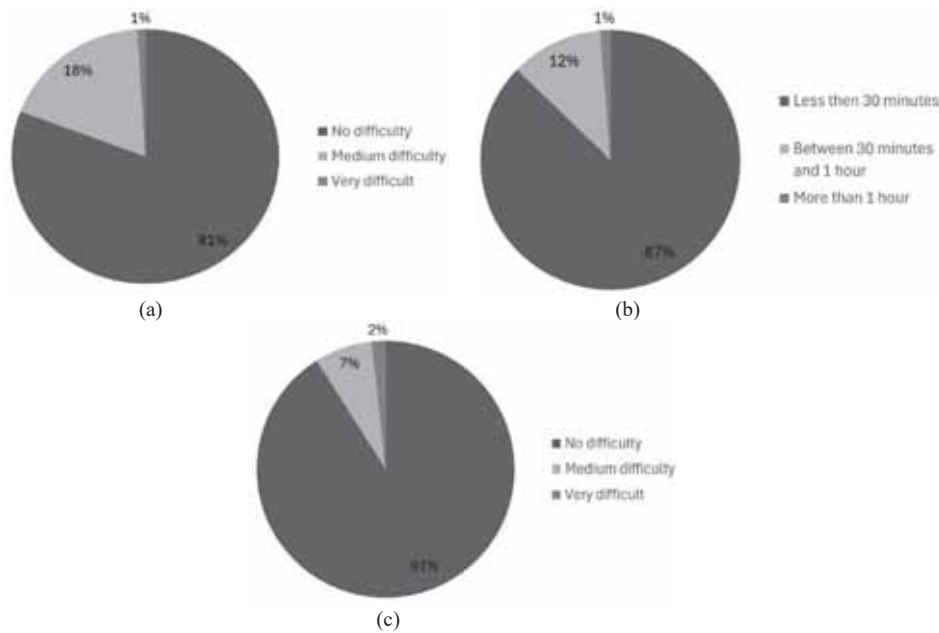


Fig. 2. Results from the testing phase regarding the data collection and indicators calculation: (a) difficulty in data collection, (b) data collection time, (c) difficulty in indicators calculation

- significance from the point of view of circularity and water use;
- suitability for measuring the circularity and water use of a product and for their use in the RECiProCo web Platform (which contains the whole set of indicators);
- simplicity and rapidity of retrieval and calculation by organisations.

The testing phase led to the definition of the final set of indicators (28) reported in Table 1, which are related to all the life cycle phases: design, procurement production, distribution and sales, use/consumption and end of use.

Within the set of indicators previously defined, a restricted number (11) were identified as clearly understandable to the consumer, to be used for consultation through the QR code available in the RECiProCO web Platform (Table 1, in bold) (see also par. 3.3).

The RECiProCO Platform was finalized as well, according to the final sets of indicators reported in Table 1 and made it available on line and free of charge for companies.

The Platform was presented and explained during some dissemination events, which included dedicated workshops with the Ministry of Economic Development and the project final event, with the participation of a wide public, such as companies, consumer's associations, research institutions and citizens.

3.3. Description of RECiProCO Platform

In order to register to the Platform, a company must connect to <https://simulatore.reciproco.enea.it/#/home> and proceed with the registration, creating a username and a password, by selecting "Sign in", on the top right

corner of the page.

After having registered, the company can enter the Platform with their username and password. At this point, the company dashboard is divided in two main parts (Fig. 3):

- a navigator at the top
- a consultation section at the bottom

In the left part of the top navigator the company can create/consult their contents and private pages. In the lower part of the page, the company can view the selected section.

Upon first login, the company must fill in the fields of the contact data (of the company and of the person fulfilling the questionnaire), the ATECO code, the name of the product they want to analyse.

ENEA will then proceed with the validation of the request and will authorize the company to access the Platform's functions.

At this point the company can create a new product sheet: to create a new Content, the company must click on the "Contents" button in the navigation bar at the top left of the homepage, then click "Dynamic Contents" and then on the "Add Content" button (Fig. 4). In the New content section (Fig. 5), the company has to click on "Form nuovo prodotto" and fill in the "Content title" with the name of the product.

After that, the company has to fill in the data about each circularity indicator with quantitative information (either a percentage or an absolute value) or with qualitative information; the indicators are divided in the following sections, representing the stages of product life cycle:

- product description;
- design;
- procurement;
- production;
- distribution/sales;

- use/consumption;
- end-of-use.

Finally, the company has to decide whether authorize the public access to their indicators or not. In fact, a company could decide to compare the results of its indicators for a certain product with those of a similar product produced by another company, thus boosting the improvement of company’s circularity performance.

ENEA will then proceed with the validation of the data provided by the company through the Platform and will authorise the publication of the questionnaire on the Platform. At the end of this process, the company can see on the Private Pages

menu, the level of circularity of its product (Fig. 6) and the one of other companies products available in the Platform, according to an algorithm which calculates an average value obtained from the ratio between the sum of all indicators in each life cycle stage and the total number of indicators available in that stage.

In the Private Pages menu the company can also see the QR code associated with its product (Fig. 7) and can use its for dissemination purposes towards other companies and also final consumers. A user guideline and training material will be soon available to support the correct fulfilment of all sections, with the goal to make the company autonomous in the quantification of the indicators.



Fig. 3. Dashboard of the Platform for the user: contents and private pages (on the top) and the selected section



Fig. 4. Page of the Platform where a company can create and add a new content

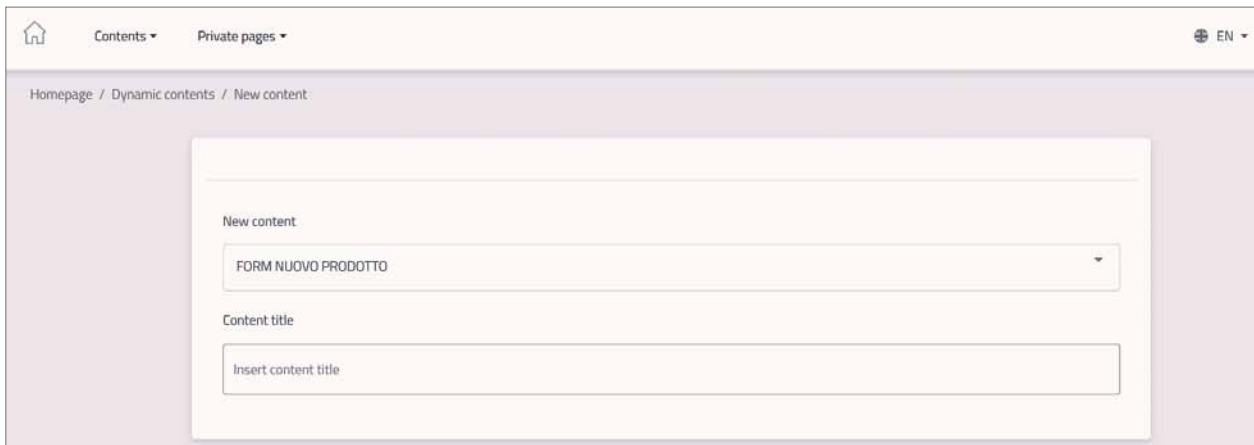


Fig. 5. Page of the Platform where a company can add a new product and its circularity and water use indicators

PRODUCT	AGENCY	LEVEL OF CIRCULARITY	DESIGN	PROVISIONING	PRODUCTION	DISTRIBUTION/ SALE	USE/ CONSUMPTION	END OF USE
testiera nera di plastica dura	-	78.41%	100%	80%	64.29%	60%	100%	75%
product 1	-	23.53%	100%	0%	0%	100%	0%	50%
product 2	-	25.88%	100%	46.67%	14.29%	0%	0%	25%
product 3	-	17.65%	100%	33.33%	0%	0%	0%	25%

Fig. 6. Level of circularity for company’s product and for any other product available in the Platform (also from other companies)

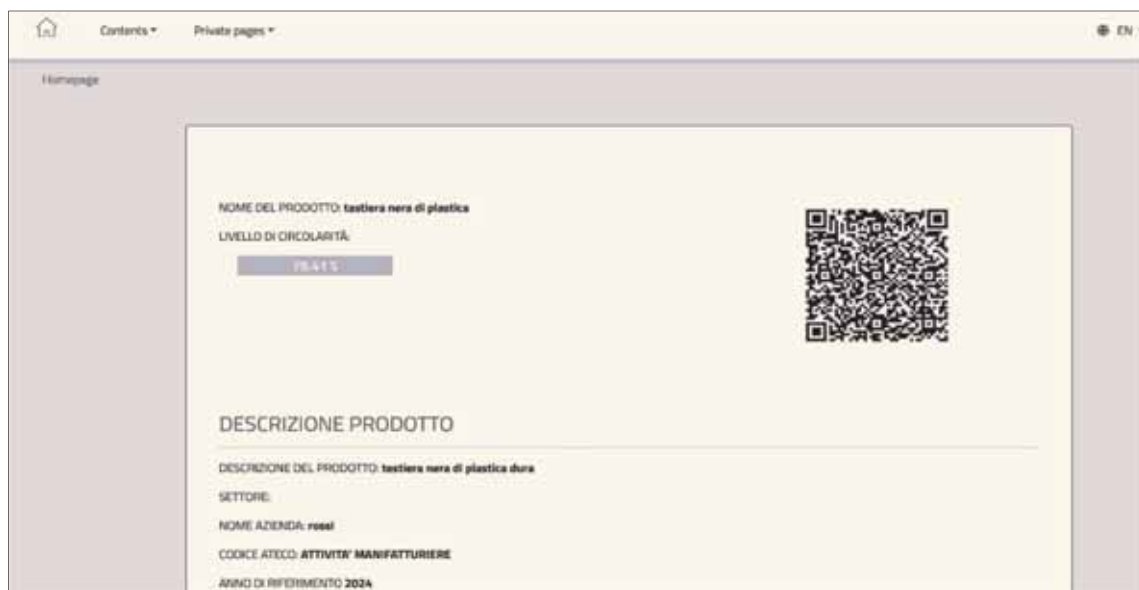


Fig. 7. Example of QR code of a company’s product

4. Conclusions

The activities carried out in the framework of RECiProCo project led to the final identification, on the basis of a life cycle approach, of a set of 28 indicators of circularity and water use, which are significant, easy and quick to be calculated. The set of indicators can be used by organisations, also without the need of external support, to measure and monitor over time the circularity and water use of their products and to communicate them to other companies (for example customers and/or suppliers) in a Business to Business approach. Furthermore, 11 of these indicators were chosen for communication to final consumers, because they were considered to be clearly understandable by non-expert’s buyers, who can use the QR code information as support and guidance for responsible and environmentally conscious purchases.

Moreover, a free of charge web Platform was developed, addressed to companies, especially to SMEs, aimed at the acquisition, normalization and visualization of data relating to the circularity and

water use of products. The Platform contains the whole set of indicators developed in the project and can be used directly by organisations to calculate the level of circularity of their products, to identify areas of potential environmental improvement and to communicate their environmental commitment towards circular economy goals to final consumers and to other companies.

The set of indicators was developed by means of open consultations with several national consumer’s association, which provided useful feedbacks, and suggested modifications, in order to boost a more easy application by companies and improve the clarity of each indicator and its usability and effectiveness by consumers.

The companies involved in the testing phase were grateful to ENEA for having been involved in the project and participated with great interest; the calculation of the indicators allowed them to become more conscious about the environmental data related to their products and production processes, and was a first step towards the management and monitoring of

these type of data, especially for companies, mainly SMEs, which did not have any previous expertise or little knowledge in the field of circular economy.

The Platform is now in its launching phase and a supporting guideline for users will be available and published soon on the Platform, with the possibility to develop further training materials, such for example videos or power point presentation on circular economy, environmental sustainability and efficiency in water use concepts.

The Platform was presented and explained during some dissemination events, such as the project final workshop, with companies, consumer's associations and research institutions.

At the moment, some new companies have already registered to the Platform, tried its functions and are evaluating how to use it within other projects in collaboration with ENEA. Moreover, in order to boost a broader use and knowledge, the Platform will be explained and disseminated in further communication events with the cooperation of consumer's associations and different type of companies.

In particular, specific dissemination action plans and capitalisation activities of the Platform, including its possible improvements, could be planned by ENEA with the collaboration of either national consumer's association or the Italian Circular Economy Stakeholder Platform (ICESP), addressed to both industries, consumers associations and citizens could be organised. These dissemination activities will enable ENEA to gather new important feedbacks from consumers about their understanding of the set of indicators, their effectiveness to support sustainable and circular purchasing choices and finally to obtain any suggestions for possible improvements or modifications to the set of indicators.

Moreover, further tests of the Platform will be performed in other companies, also in the framework of national or European projects about circular economy indicators and sustainability assessment to which ENEA participates. The outcomes from this larger sample of companies will lead to more comprehensive and robust considerations about the actual effectiveness of the set of indicators and the Platform.

In addition, possible improvements and extension of both the methodology and the Platform to other types of products and industrial sectors could be designed and implemented, by the adaptation and integration of the indicators already available, also on the basis of sector specific characteristics and peculiarities as well as on the basis of the results of the new testing phases.

Finally, within this further developments and tests of the Platform, a specific verification procedure will be developed, with dedicated guideline and rules, which could be applied also by external reviewers.

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“GREEN” EXTRACTION OF BIOACTIVE MOLECULES FROM VEGETABLES AND FISH INDUSTRY BY-PRODUCTS

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Abstract

Food companies are increasingly interested in the effective application of sustainable and innovative techniques able of making by-products management less expensive if not even a source of additional incomes. Since several by-products coming from the food processing industry are rich in high-power bioactive molecules, the recovery of such substances is currently the most promising means, either for reusing them as ingredients in functional foods or for applications in many sectors such as the packaging one. Indeed, combining bioactive molecules extracted from vegetables and fishery by-products can contribute to make biofilm with antimicrobial and antioxidant effects. Moreover, from the point of view of sustainability, it is necessary to replace chemical extraction techniques, characterized by heavy environmental side effects. In this paper, the chitin extraction from deep-water shrimp (*Aristeus antennatus*) by-products was performed by using enzymes and organic acids in alternative of harsh chemicals. As for the vegetable products, the ultrasound-assisted extraction (UAE) was applied for a more sustainable extraction of the bioactive molecules from tomato pomace by-products (seeds and peels). Preliminary results showed that the enzymes reduced significantly the protein content in the shrimp by-products, while formic acid was able to remove 100% of the minerals, being as effective as hydrochloric acid. The UAE, performed using water as the “green” solvent, for 20 min at temperature below 40°C, was effective in the complete extraction of polyphenols from tomato pomace by-products. Moreover, an amount of β -carotene of 8.64 mg/100g DW, equal to 50% of what reported in the literature was extracted.

Key words: bioactive molecules, green extraction technologies, shrimp by-products, tomato by-products

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1. Introduction

The seafood production sector has been increasing over the years in order to meet the growing demand for seafood (FAO, 2022). As described in the report FAO (2022), the global production of crustaceans exceeded 10 million tons in 2020, most of which produced from the aquaculture sector. This overwhelming amount of crustaceans has generated a considerable amount of by-products coming from the processing sector, mainly represented by heads, tails, shells, which can account up to 70% of the raw material total weight (Yan and Chen, 2015; Zhang et

al., 2023). In developing countries, crustacean by-products are often just dumped in landfill or the sea, while in developed countries, disposal can be very expensive, representing up to US\$150 per ton in Australia, for example (Yan and Chen, 2015). Therefore, companies are increasingly interested in research and development of technological solutions capable of making waste management less costly if not even a source of additional income. Indeed, a lot of valuable compounds can be recovered from these by-products such as chitin, astaxanthin, hydrolysates, that could be reused in many sectors, e.g. pharmaceutical, cosmetic, food, feed, wastewater

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treatment sectors and so on, increasing the prospective of sustainability, profitability and competitiveness of food companies (FAO, 2022; Zhang et al., 2023).

Chitin, the world's second most abundant polymer in nature after cellulose, is present in large quantity in crustacean shells (ranging from 15 to 40%), depending on the species, organism, nutrition status, processing methods and other factors (Yan and Chen, 2015). Considering the high amount of chitin, as well as other valuable compounds, as calcium carbonate, proteins and pigments, in crustacean shell, an important challenge in the view of sustainability, is for sure the extraction of these compounds in such a way that does not adversely affect the environment (Yan and Chen, 2015). In most cases, the chitin extraction is carried out by using chemical solvents as sodium hydroxide and hydrochloric acid to remove proteins and minerals respectively (Pakizeh et al., 2021), resulting unfriendly for the environment and making the obtained minerals and proteins unsuitable as human and animal nutrients (Pakizeh et al., 2021; Yan and Chen, 2015). To avoid the use of hazardous chemicals, it is very intriguing and advantageous to explore the feasibility of using enzymes to degrade seafood waste, taking into account their power of producing bioactive peptides and good quality chitin (Mathew et al., 2021). Proteolytic organisms or commercial enzymes, like alcalase, trypsin, delvolase, papain, pancreatin, etc. have been used to achieve enzymatic deproteinization (Islam et al., 2023). Moreover, organic acids, like lactic and formic acid, were used to remove minerals from crustaceans shells, in order to substitute harsh chemicals like HCl (Islam et al., 2023).

As well as seafood production sector, another fundamental sector to be better investigated is the tomato processing chain, because of the huge amount of the global production, the waste generated from the industrial processing, and the valuable molecules that could be recovered from their by-products.

Italy is the third producer of tomatoes for industrial use in the world, behind USA and China. The Italian tomato processing industry is the leader in Europe, followed by Spain, Turkey and Portugal. Indeed, in the Mediterranean area, Italy plays a major role for the tomato derivatives production, with 13% of the total world production and almost 50% of the European one, with a turnover of more than 4 billion €. The tomato processing by-products are unsuitable tomatoes for industrial processing, MOT (Material Other than Tomatoes), cleaning and transport water used in the line of production and, finally, by-products produced in the processing lines (tomato peels, seeds and pomace). In a "circular economy concept", and considering that the waste produced during the industrial processing operations represents generally the 2.3-2.5% of the total fresh matter, the recovery of these by-products and their reuse to valorise them have to be considered in the future (Sandeï et al., 2015). A valid alternative is the possibility of using Tomato Processing By-products (TPB) as a potential source of bioactive substances, including carotenoids,

polyphenols, which have antioxidant, anti-inflammatory and chemo preventive properties, and prevent cardiovascular diseases (Coelho et al., 2023). With this regard, it is important to use "green" extraction techniques in order to replace the conventional extraction methods with high environmental impact. One of the emerging "Green Technologies" considered in this work is the Ultrasound-Assisted Extraction (UAE) that can increase the extraction yield of the bioactive molecules naturally present in the TPB produced during the common industrial operations (tomato pomace – peels and seeds) by using eco-friendly solvents (water and or ethanol). UAE is an high-potential technology that can accelerate heat and mass transfer and has been successfully used in the extraction of several classes of food components such as aromas, pigments, antioxidants, and other organic and mineral compounds (Chemat et al., 2011). This technology, with affinity to hydrophilic molecules, fails to recover all the lipophilic molecules that are even present in the waste and therefore has to be combined with other techniques such as supercritical CO₂ to recover all bioactive compounds (Riera et al., 2004).

The need of the hour, it is not only to valorise agri-food wastes and by-products, by extracting from them bioactive molecules in a green and sustainable way, but even to find solution that prolong seafood shelf life, and of all food items, characterized by a very high perishability, preferably employing natural compounds, more and more demanded by consumers. In that point of view, a very sustainable attempt could be represented by biofilms that are made of molecules extracted from both seafood and vegetables, which with their combined power can act as natural preservatives. Towards this direction, the first step and the main objective of the present work was to verify the goodness and effectiveness of "green" techniques (not using chemicals) for the extraction of some bioactive molecules from vegetables and fish industry by-products, in order to contribute to valorise such by-products in a sustainable, efficient and economically profitable way.

In particular, the potentiality of the UAE of extracting bioactive molecules from TPB was investigated, as well as the use of enzymes and organic acids for the deproteinization and demineralization of chitin from deep-water shrimp (*Aristeus antennatus*), to replace harsh chemicals normally used in the extraction of chitin.

2. Materials and methods

2.1. Sample supplying and preparation

Deep-water shrimp (*Aristeus antennatus*) by-products (heads, shells and tails) were supplied by local restaurants and fish markets in Campania (Italy), immediately transported to the laboratory under refrigerated conditions and frozen at -18 °C until use. For the extraction of chitin from shrimp by-products,

the samples, after thawing, were washed several times with distilled water to remove impurities, dried in an oven at 55 °C for 12h, and then crushed into powder by using a mixer for solid samples (Bosch VitaPower Serie 4).

The peel and seed (35 kg) used for the “green” extraction of bioactive molecules were obtained during the tomato processing operations carried out at the SSICA technology laboratory during summer 2023 (Fig. 1).

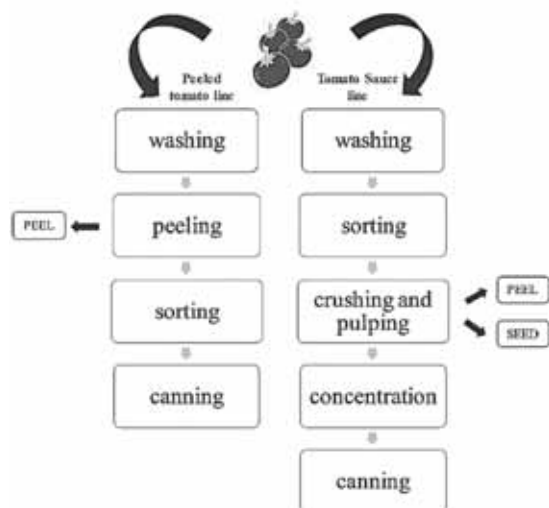


Fig. 1. Processing plant with peel and seed sampling points

2.2. Deproteinization of shrimp by-products

The enzymatic hydrolysis was performed in triplicate in an enzymatic digestion unit (GDE, VELP Scientifica) on 10 g of dried by-products at which 100 mL of distilled water was added (1:10 g/mL). The mixture was heated at 90°C for 15 minutes to inactivate the endogenous enzymes, then temperature was set at 55°C, pH adjusted at 8, and two proteases (Protamex® from *Bacillus* sp. or Alcalase® from *Bacillus licheniformis*, Sigma-Aldrich) were added to the mixture (5% w/w of by-products powder). The reaction was performed for 180 minutes under constant agitation (80 rpm) and continuous pH monitoring and addition of NaOH 2M to maintain the pH of 8. At the end of the reaction, temperature was set at 90°C for 15 minutes to inactivate the enzymes.

For chemical deproteinization, the dried powder was treated with 0.75 M NaOH at a ratio of 1:6 (g/mL) for 24h at room temperature under constant agitation (80 rpm). After reactions, the samples were drained, washed several times with distilled water until neutral pH and oven-dried at 55 °C. The upper layer, containing the hydrolysates, was stored at -18°C.

2.3. Demineralization of shrimp by-products

Demineralization was performed on the deproteinized powder using 5% solution of three organic acids (citric, or lactic or formic) (Carlo Erba)

at a ratio of 1:10 g/mL for 1h at room temperature and under constant agitation (200 rpm).

As for the chemical demineralization, the dried samples were treated with 1.25 M HCl at a ratio of 1:10 (g/mL) for 1h at room temperature under constant stirring (200 rpm). After reactions, the samples were drained, washed several times with distilled water until neutral pH and oven-dried at 55°C.

2.4. Ultrasound-assisted extraction (UAE) of bioactive molecules from tomato by-products

The by-products from tomato processing lines were collected from SSICA pilot plants and stored in under vacuum bags, then immediately blast chilled and stored at -18°C, to reduce oxidative phenomena affecting the bioactive molecules content. In a second moment, the tomato by-products were thawed and treated in a crushing process directly with the UAE equipment. The UAE used for this extraction process was “Ecotecne ES15” equipped with a crusher with ECO 10 generator settled directly by the company (Fig. 2).



Fig. 2. Ultrasound extractor ES15

Extraction tests were performed mixing the tomato by-products with water at a ratio of 1:10. The extraction process was carried out as follows: maximum power and frequency of 25.3 KHz ($I_{out} = 2.3$ A, $V_{out} = 310$ V, $P_{out} = 710$ VA), temperature variation between the start and end of the process of about 10 °C, without exceeding 40 °C (Sengkhamparn and Phonkerd, 2020). At the same time, in order to have a reference sample, a similar extraction was performed using the same ES15 UAE system, but using only the stirring mode, without the ultrasound generator.

At the end of the process, each extract was separated from the coarse material through a 0.4-mm fine-mesh sieve and subsequently filtered with a TNT EC1223 cloth, then stored in aluminised vacuum bags at -18°C.

At the end of the extraction process, a total volume of 350 litres of aqueous extract was produced and then 15-fold concentrated by means of evaporation and concentration steps using a vacuum

concentrator, a semi-industrial pilot plant, at the Experimental Station for Food Processing Industry.

2.5. Chemical analyses

The shrimp by-products and chitin extracted were analysed in order to determine total nitrogen, chitin content, total lipids, proteins, ash and moisture contents. The analyses were carried out in triplicate by the following methods: moisture by the AOAC 90.15:1993, lipid content by the Soxhlet method (AOAC 920.39), total nitrogen by AOAC 954.01/988.05 and the ash content by AOAC 938.08. Chitin and protein contents were evaluated by determining total nitrogen (Nt) and non-nitrogen compounds (i. e. ash, moisture and lipids) as described above and applying the Eqs. (1-2) (Díaz-Rojas et al., 2006):

$$\text{Chitin \%} = \frac{(Nt \cdot Cp + K - 100) \cdot Cq}{(Cp - Cq)} \quad (1)$$

$$\text{Protein \%} = \frac{(Nt \cdot Cq + K - 100) \cdot Cp}{(Cq - Cp)} \quad (2)$$

where: *K* is equal to the sum of the non-nitrogen compounds, while *Cp* (6.25) and *Cq* (14.5) are conversion coefficients that relate the mass fraction of nitrogen with protein and chitin, respectively (Díaz-Rojas et al., 2006).

The demineralization and deproteinization degrees (DDM% and DDP%, respectively) were calculated by using Eqs. (3-4):

$$\text{DDM \%} = \frac{\{[(MO \cdot O) - (MR \cdot R)] / MO \cdot O\} \cdot 100}{100} \quad (3)$$

$$\text{DDP \%} = \frac{\{[(PO \cdot O) - (PR \cdot R)] / PO \cdot O\} \cdot 100}{100} \quad (4)$$

where: *MO* and *MR* are the ash contents (%) before and after demineralization; while, *PO* and *PR* are the protein concentrations (%) before and after deproteinization. *O* is the mass (g) of original sample and *R* represents the demineralized residue (Eq. 3) or hydrolysed residue (Eq. 4), in dry weight basis.

On the aqueous extracts obtained from tomato by-products, total polyphenols, were determined by means of Folin-Ciocalteu reagent (Sigma-Aldrich), and using gallic acid as a standard (Sigma-Aldrich) (Hudz et al., 2019). On the concentrated aqueous extract, dry mass, degree brix, organic acid, sugars, total polyphenols and carotenoids were determined.

Dry mass and degree brix were determined following methods described in the Italian Ministerial Decree (1989), while citric acid and malic acid by enzymatic kits (Citric acid and D-malic acid kits, R-Biopharm). The speciation of sugars was performed by HPLC-ELSD (Ma et al., 2014). D-(+)-glucose, D-(-)-fructose and sucrose were obtained from Sigma-Aldrich. Lycopene and beta-carotene were determined by HPLC-UV (De Sio et al., 2001). Standards of

lycopene and b-carotene were purchased from Sigma-Aldrich.

2.6. Statistical analysis

Experiments and analyses were carried out in triplicate. One-way analysis of variance (ANOVA) and Tukey HSD multiple comparisons were performed using RStudio software (2023.12.1+402) to analyse significant differences ($p < 0.05$).

3. Results and discussion

3.1. Composition of shrimp by-products

By-products composition of *A. antennatus* is shown in Table 1. These findings were similar to those obtained by Liu et al. (2021), who analysed the meat and by-products composition of five species of shrimps. Composition of *A. antennatus* by-products indicated they can be used as a rich source of bioactive compounds to develop natural nutraceuticals, or to be employed in other sectors, such as food packaging.

Table 1. Proximate composition of *A. antennatus* by-products (g/100g, mean ± SD)

Proximate composition	g/100 g of by-products
Protein	6.00 ± 0.32
Fat	1.44 ± 0.03
Ash	15.30 ± 0.27
Chitin	12.56 ± 0.05
Moisture	64.70 ± 0.78

3.2. Deproteinization and demineralization steps

Commercial proteases can be useful for the extraction of chitin, because, unlike crude proteases extracted from microorganisms, they can give a more reliable product and can be easily scaled up for industrial extraction of chitin/chitosan (Mathew et al., 2021). Usually, after enzymatic deproteinization, 5-10% of proteins remains adhered to chitin, and are not completely removed, unlike what done by chemical deproteinization (Mathew et al., 2021). However, the latter method could damage proteins extracted and negatively affect the safety of the hydrolysates preventing their use as animal feeding or human nutraceuticals.

In terms of deproteinization, the proteases used in the present study at the optimal conditions of pH and temperature previously defined, were able to reach a DDP of 95%, very similar to that obtained by using 0.75 M NaOH (DDP = 96%), as shown in Fig. 3, without statistically significant differences observed between treatments ($p > 0.05$). In terms of optimization of the deproteinization step, in the future other techniques will be used after enzymatic extraction, like ultrasound, which could be useful to remove the protein residue remained attached to the chitin. The degree of deproteinization obtained in the present work was slightly higher than that obtained in previous studies by using enzymes (Islam et al., 2023).

However, present results confirmed what obtained in terms of high power of alcalase and protease from *Bacillus* sp. to remove proteins from seafood by-products (Dey and Dora, 2014; Dumay et al., 2006; Valdez-Peña et al., 2010). Alcalase® is a bacterial serine endopeptidase prepared from a strain of *Bacillus licheniformis*. Alkaline alcalase was proven to be efficient in obtaining the highest protein recovery and DDP% among the proteases tested from shrimps by-products (De Holanda and Netto, 2006; Dey and Dora, 2014; Valdez-Peña et al., 2010).

Protamex® is a *Bacillus* sp. protease complex, which was used to recover proteins/lipids by *Sardina pilchardus* viscera (Dumay et al., 2006), giving slightly higher protein recovery (61%) than Alcalase (60%), with degrees of hydrolysis quite similar (3.1 and 3.3%, by using Protamex and Alcalase respectively). However, Messina et al. (2021), who performed enzymatic extraction of bioactive peptides from *Parapenaeus longirostris* by-products at a pilot scale, found a higher hydrolysis degree by using Protamex® on *P. longirostris* by-products (10-16%).

Concerning the demineralization step, it was observed that the use of the three organic acids allowed reducing drastically the mineral content from the raw by-products, with statistically significant differences between DDM% obtained from the three organic acids (Fig. 4). In particular, the highest percentage of demineralization was reached by using formic acid, which was able to remove completely the minerals (DDM% = 100%), followed by lactic acid that contribute to reach a DDM% of 60%, and by citric acid with a DDM% = 44%. The controls, i.e. the samples demineralized with HCl, showed a DDM% equal to 100% (Fig. 4). The results of the present study agree with those obtained by Baron et al. (2017), who observed that formic acid was the only acid able to reach a demineralization degree of 99% with a pH of 3.5. Another study (Hu et al., 2020) obtained a slightly lower (90%) demineralization degree of shrimp by-products using a 5% solution of formic acid. As regards the other organic acids, it was found that lactic

acid achieved a demineralization degree of 60%, lower than that (90%) obtained by Mahmoud et al. (2007), by using lactic acid (75.6 g L⁻¹) on *Pandalus borealis* deproteinized shells, at a ratio of 1:10 (g shell/mL acid) and room temperature, but considering a reaction time of 2 hours, instead of 1 h considered in the present work. Retention time is one of the most important parameters in the demineralization process, because could affect the quality of the purified chitin. Citric acid was also used as a promising alternative to HCl to demineralize shrimp shells, but as it was shown by Pohling et al. (2022), to have a complete removal of minerals from shrimp shells, it could require much more concentrated citric acid solution and two-steps of demineralization unlike formic acid, for which a single step was enough.

3.3. Total phenolic content

The values of total polyphenols obtained from the analysis of samples taken at regular intervals of 5 min in an extraction test lasting 30 min are shown in Fig. 5. Statistically significant differences ($p < 0.05$) in total polyphenol content were observed between the extraction performed with ultrasound and that carried out by agitation, because the ultrasound improved solvent penetration into plant cells and cell wall disruption, facilitating the release of bioactive molecules.

As it can be seen in Fig. 5, an extraction time of 20 minutes allowed a complete extraction of the polyphenols contained in the processed by-products, because any significant variation was observed after that time. Subsequent trials were carried out on the tomato waste for a time of 20 min. The extracts obtained were combined and 15-fold concentrated from 0.41 to 6.20°Bx.

3.4. Characterization of the concentrated extract

The results of the characterization of the concentrated extract are shown in Table 2.

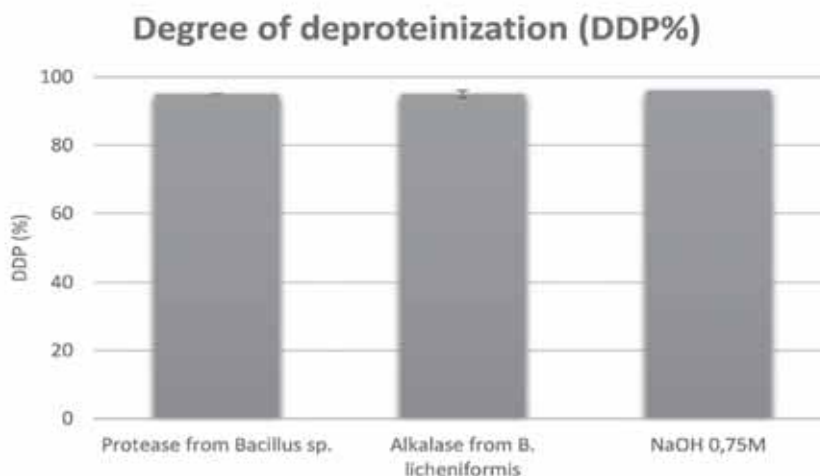


Fig. 3. The DDP% reached by the proteases and sodium hydroxide used for the deproteinization step

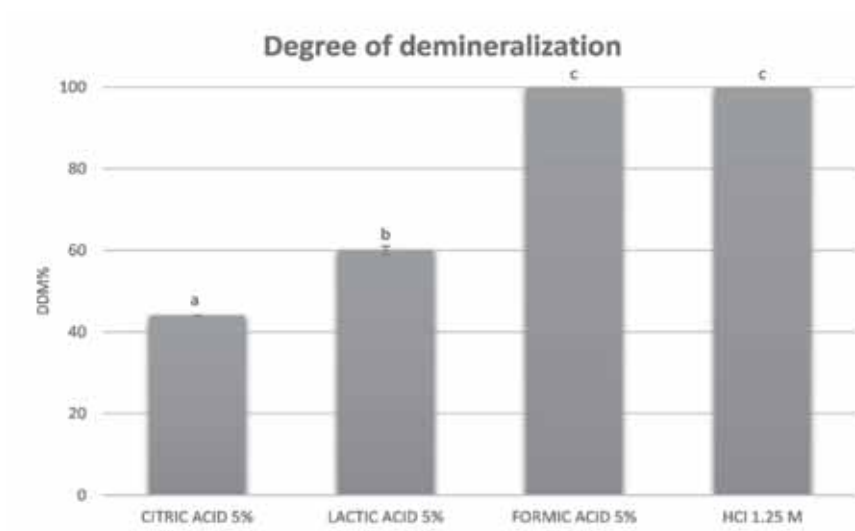


Fig. 4. The DDM% reached by the four acids used for the demineralization step. Different lowercase letters indicate statistically significant differences (p<0.05)

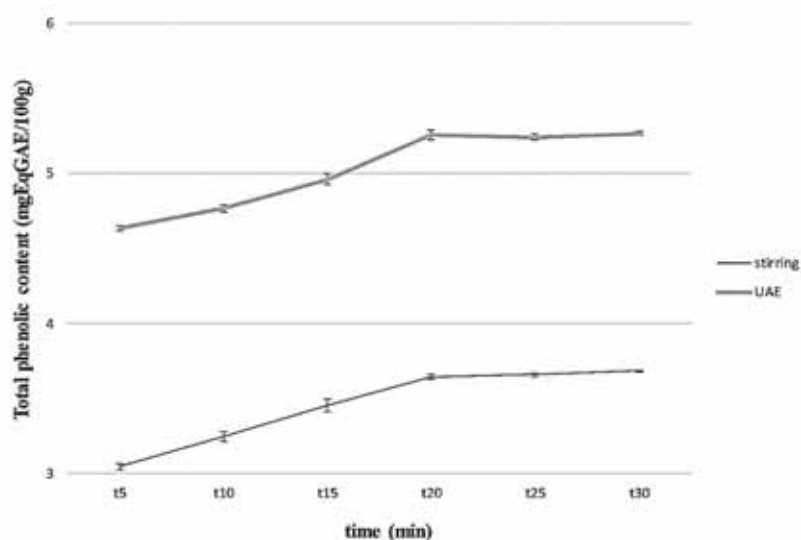


Fig. 5. Total phenolic content at different extraction times with UAE and under agitation.

Table 2. Sugars, organic acids and carotenoids in concentrated aqueous extract (mean ± SD)

Sugars		Organic acids		Carotenoids	
Glucose (g/100g dw)	Fructose (g/100g dw)	Citric acid (g/100g dw)	Malic acid (g/100g dw)	Licopene (mg/100g dw)	β-carotene (mg/100g dw)
4.86 ± 0.37	9.96 ± 0.12	0.36 ± 0.01	0.21 ± 0.02	3.24 ± 0.25	8.64 ± 0.63

A direct comparison with data reported in the literature is not possible given the variability of the raw material due to the different percentages of peel, seed and pulp in the by-products, which depends on tomato processing lines (tomato purees and paste, or peeled or diced tomato for example), type of crop, variety and ripeness of the tomato. It is also difficult to find solid data on organic acids and sugars content; only an average of 25% of carbohydrates has been reported (Coelho et al., 2023).

It should be highlighted that although the ultrasonic extraction technique is more suitable to extract hydrophilic substances, the cell-wall rupture also brought into solution a certain amount of

carotenoids; the content found was low for lycopene but for β-carotene it was about 50% of the amount reported in the literature (Coelho et al., 2023). In order to obtain a higher amount of carotenoids, the process could be optimized by using a more homogeneous raw material in terms of peel size; this operating condition will be evaluated in the future.

4. Conclusions

As far as the chitin extraction was concerned, the “green” extraction method, carried out by considering enzymes and formic acid, allowed to obtain chitin with less than 1% of protein and mineral

contents, which are recommended both as food ingredient and for chitosan applications in food packaging. Further analyses will be carried out on the extracted chitin in order to characterize it in terms of solubility, degree of acetylation, molecular weight, and finally converted in chitosan.

In terms of scalability, this field has not been much explored so far, but the studies have increased in the last years, with studies that scale up from a laboratory to a pilot scale, providing important results on the feasibility of this kind of shrimp shell processing on an industrial scale. In terms of costs, using enzymes for extracting chitin could be slightly more expensive than using hazardous chemicals but much greener. Indeed, the use of enzymes and organic acids is in accordance to the 12 principles of the green analytical methods (GAC) that focuses on reducing or possibly eliminating hazardous substances used in some processes or analyses to minimize their negative effects on human health and the environment. However, the higher costs can be balanced by the possibility to obtain hydrolysates rich in essential amino acids that, in turn, can be integrated in formulated diets for aquaculture as sources of biologically active peptides.

One of the main goals of this work was to valorise and develop a “green and sustainable process” protocol able to valorise the huge amount of Italian tomato processing by-products, by the application of novel “green” extraction technologies. Preliminary tests of UAE showed that a treatment of tomato by-products for 20 minutes was enough to obtain a complete extraction of the polyphenols as well as a high β -carotene level. Next step of this research will be to apply in a “cascade process model” this technique with others “green extraction systems (i.e. Supercritical CO₂ extraction)” in order to add and exploit the extraction of the lipophilic fraction of the tomato by-products biomolecules.

It is conceivable that the green technique based on the use of ultrasound for extracting bioactive molecules from tomato pomace by-products could be scaled up for industrial application being affordable for the food companies. Indeed, pilot plants of ultrasound are not so costly, how a market research can show, moreover, the use of water as solvent is more sustainable and avoids the necessity of more expensive chemicals.

Moreover, the bioactive molecules successfully extracted from shrimp and tomato pomace by-products in eco-friendly processes, might be thought to be used together in the future to make packaging solutions that can help prolong food shelf life, giving even more support to food companies to make their production more sustainable and profitable.

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ELLIPSE: EFFICIENT AND NOVEL WASTE STREAMS CO-PROCESSING TO OBTAIN BIO-BASED SOLUTIONS FOR PERSONAL CARE AND AGRICULTURAL SECTORS

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Abstract

Sustainability is a pillar to develop and maintain global strategies such as the EU Bioeconomy Strategy, the EU Green Deal and the SDGs. The transition from a linear to a circular economy including resource recovery, reuse and recycling is essential. However, meeting the requirements for converting bio-based waste streams into renewable raw materials, like bioplastics, with strict purity and high performance to ensure both proper processing and meet product request, demand inter-disciplinary cooperation among high skilled experts from EU. ELLIPSE project will address the valorisation of two heterogeneous waste streams: slaughterhouse waste and paper & pulp sludge, to produce cost-efficient polyhydroxyalkanoates for agricultural and personal care applications, by the coprocessing with other organic waste such as sludge from the dairy industry and glycerol from the biodiesel industry, as well as recovering nutrients to produce bio-based fertilizers. This will be achieved by applying the cascade biorefinery approach using acidogenic fermentation where, by one hand a VFA enrich stream will be generated from the selected feedstocks and coupled to PHB fermentation production system, and by other hand, the solid fraction produced after the acidogenic fermentation will be used to recover nutrients such as N and P. The integration of these waste streams as biorefinery feedstocks will allow reducing the volumes of landfilled waste, opening new avenues for platform chemicals and bioplastics production while creating additional revenue for the related industries generating them, with added advantages of water recycling, decreased soil degradation, groundwater pollution and methane emissions. ELLIPSE approach will be based on lab-scale optimization of organic waste mixtures and operation conditions, then these conditions will be up-scaling to treat at least 100 tonnes of slaughterhouse waste and 20 tons of wastewater sludge derived from pulp and paper industry.

Key words: bioeconomy, waste management, bioplastic, bio-based fertilizers

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1. Introduction

Heterogeneous organic wastes are challenging waste streams due to the presence of impurities that can compromise valorisation and are therefore usually sent to landfill. By one hand, slaughterhouses are one of the parts of the meat production chain where most waste is produced. In these places large volumes of solid waste and wastewater rich in organic contaminants and nutrients (particularly phosphorus) are produced. However, several problems are associated with the end of life of slaughterhouse waste such as waste segregation, high moisture content, presence of infectious pathogens (bovine spongiform encephalopathy) or heavy metal contaminations (Bhunia et al., 2022).

Among them digestive tract content (belly grass) is a major waste produced at cattle slaughterhouses and is comprised of partially digested cattle feed, mainly containing grass and grain. It has been reported that approximately 92 kg of wet belly grass are produced per animal (Smyth et al., 2011). Such a high volume of production makes it a waste that is difficult to manage and whose disposal, most of the time entails economical losses.

By the other hand, a global review of manufacturing sectors divulged that 17% of the global waste comes from paper industries (Camberato et al., 2006). The waste produced from pulp and paper industry is categorized in rejects such as pulping, bleaching, washing, primary sludge, and secondary sludge. Pulp and paper sludge is produced from the treatment of wastewater. The industrial sludge composition has been reported as highly variable with carbohydrate contents varying between 20% and 75% but also it contains heavy metals and toxic chemicals, which leads to toxicity of water bodies and impurities as filler, stickies, dye or ink which makes a waste difficult to manage (Gupta and Shukla, 2020).

At the same time, the environmental damage caused by petroleum-based plastic and the diminution of fossil reserves is translated into an increase in general public awareness of environmental issues and a necessity of a transition to a circular economy, where resources be renewable. This growing awareness in the population has increased industry's interest in the production of plastics from materials that can be eliminated from our biosphere in an "environmentally friendly" way (Leal Filho et al., 2021).

Bioplastics offers many benefits over traditional plastics including reduced greenhouse gas emissions, biodegradation and usage of sustainable materials. In addition, they can be produced from residues which allows to achieve two objectives simultaneously: produce a more sustainable polymer and reduce the huge quantities of waste generated at urban and industrial level. Despite the many advantages of bioplastics, they are not yet as well integrated into the market as fossil-based plastics, mainly because of their price, but also because their properties may not be adequate in certain cases (high fragility, low

thermal stability and low nucleation density) (Atiwesh et al., 2021).

Among the most widely produced bioplastic worldwide is polyhydroxybutyrate (PHB), the most common bioplastic from polyhydroxyalkanoates (PHAs) family, due to its biodegradability in all environments. In addition, PHB can be obtained from different heterogeneous wastes as belly grass, sludges from different industries or glycerol (Brojanigo et al., 2021; Thirumala et al., 2010; Wendy et al., 2022). In this process, a first step is carried out where the organic waste is subjected to a pretreatment to avoid presence of impurities and an anaerobic digestion to produce volatile fatty acids (VFAs) and a digestate. In a second step, the VFAs streams is converted into PHB by bacteria present in mixed microbial cultures or pure cultures (Sirohi et al., 2020).

2. Why ELLIPSE's solutions?

The production of PHB a biobased polymer with versatile biodegradability properties in most environments is addressed in the framework of ELLIPSE project. PHB represent a promising material for a wide range of applications: PHB is biodegradable in soil which makes it ideal for agricultural applications (mulching films or fertilizer coatings) where products are disposed of in the soil and recycling system cannot be implemented (Chen et al., 2021). PHB is also a polymer that can be used in the manufacture of certain types of packaging and can be easily recycled by chemical recycling or enzymatic recycling, producing the monomer 3-hydroxybutyric acid that can be reincorporated into the fermentation as fermentation substrate (Parodi et al., 2021).

At the same time, in the process of PHB/PHBV production from sludge VFAs, a large amount of nitrogen (N) and phosphorous (P) remains in the digestate. Non-renewable mineral fertilizers, formed by N, P, K are the base of the current agricultural system, and the European Union agriculture is highly dependent of imports for fertilizing purposes. According to Fertilizers Europe (<https://www.fertilizerseurope.com/wp-content/uploads/2022/09/Industry-Facts-and-Figures-2022.pdf>) the total fertilizer consumption of European Union was about 17 million tons. Among them 32 % of nitrogen had to be imported as well as 65 % of P₂O₅ (Fertilizer Europe, 2022). Some of them such as P or Mg have been qualified as Critical Raw Materials by the EU COM(2017)490 and these are crucial for EU growth, competitiveness and especially for a sustainable food industry. In a context of migration to production models based on the postulates of the Circular Economy, it is essential to close the cycle by making use of all by-products generated during the industrial processes (Könjnger et al., 2021). For this reason, a good strategy that leads to a complete valorisation of the waste produced at industrial level is essential to reduce dependence on fossil fuels and foreign imports.

ELLIPSE project emerges as a solution for the coprocessing of heterogeneous waste streams generated in significant amounts in Europe (slaughterhouse waste, paper and pulp sludge, dairy sludge and glycerol) to produce cost-efficient PHAs for agricultural and personal care applications, as well as recovering nutrients to produce bio-based fertilizers (BBFs). The integration of these waste streams as biorefinery feedstocks will allow reducing the volumes of landfilled waste, opening new avenues for platform chemical and bioplastics production while creating additional revenue for the related industries, with advantages of water recycling, decreased soil degradation, groundwater pollution and methane emissions.

2. Case studies

2.1. Current state of the art

2.1.1. Acidogenic fermentation (AF) to produce VFAs

Acidogenic fermentation is considered as an efficient method for VFA production using a variety of organic waste as a substrate (sludge, food waste or organic fraction of municipal waste among others). Various methods to promote VFAs yield in waste acidification step have been developed, e.g., using pre-treatments, through in-situ processing control such as the regulation of pH, temperature and additives (Chen et al., 2021; Karki et al., 2021). pH control is widely used strategies to inhibit methanogenesis and enhance VFA production through anaerobic digestion of sludge or other organic matters, and it is found that pH was the main factor affecting the various indicators, especially alkaline pH regulation facilitated solubilisation, hydrolysis, and acidogenesis while simultaneously inhibiting methanogenesis. In recent years, zero-valent iron, Fe(0), has also been reported to enhance the biological processes by stimulating microbial activity and creating favourable reducing conditions (Luo et al., 2014). The presence of Fe(0) can, for example, serve as a buffering agent to react with and compensate for the acidity, and then relieve the low pH inhibition on microbial activities for the generation of VFA (Cao et al., 2019). On the other hand, anaerobic membrane bioreactors (AnMBR) have proven to be effective in treating waste streams from different sources, even food and pulp and paper industry, improving yield and quality of the VFAs produced.

The combination of advanced technologies in previous studies, such as the use of Fe(0) and AnMBR, has allowed to improve the production of VFAs and to validate the industrial viability of these technologies. These trials have provided experimental evidence that the process is scalable and cost-effective, offering a practical solution for the circular bioeconomy (Mahmood et al., 2022). The advantages include a stable and high yield of VFAs, improved product purification due to continuous separation, and more effective control of organic loading by regulating the waste stream. For the ELLIPSE project, this

technology could be critical in the valorisation of heterogeneous waste, increasing the efficiency of nutrient recovery and maximising the production of specific VFAs. Unlike other anaerobic systems, AnMBRs can process waste with high concentrations of solids, such as complex industrial waste. This makes it possible to work with more challenging waste streams while maintaining high efficiency in the production of VFAs. These innovations in AnMBRs represent a significant improvement over traditional anaerobic fermentation methods, allowing for a more efficient and pure production of VFAs. However, membrane fouling can emerge as a serious problem in the AnMBR especially when the system is operated at acidic or alkaline conditions, those suitable to produce VFAs (Parchami et al., 2020). A variation in operating pH directly affects the cell morphology along with a major alteration in adhesion and flocculation phenomena.

Considering the selected feedstocks to be treated in ELLIPSE project (slaughterhouse waste and pulp and paper sludge), there are not many reports on VFAs production in spite of their potential carbohydrate content. But reports on VFA yields during biogas generation confirm the feasibility of VFAs production from this waste. Optimizing a process for VFAs production from primary sludge alone or in combination with secondary sludge under favourable acidogenic conditions, will open new avenues for platform chemicals and bioplastics production from paper and pulp mills waste. In this context, ELLIPSE plan to valorise 100 tonnes of slaughterhouse waste, 10 tonnes of sludge derived from pulp and paper industry, 10 tonnes of glycerine, and 20 tonnes of waste reject from deinking process. Nowadays, these waste streams are derived to landfill, so ELLIPSE will provide solutions to empower the circular economy and valorisation of this wastes.

2.1.2. Production of PHAs (PHBV) from VFAs

Bacterial fermentation processes using VFAs as carbon source have emerged as a sustainable approach for PHA production due to their abundance in waste streams from organic sources, such as agricultural residues, food processing, and wastewater treatment (Montiel-Jarillo et al., 2021). Thus, harnessing the bacterial metabolism to convert VFAs, such as acetic acid, propionic acid, butyric acid, valeric acid and caproic acid into PHA, offers a greener alternative to traditional petroleum-derived plastics (Yin et al., 2016). Further, by using waste-derived streams, the cost of carbon is lower than using refined materials and the competition for raw materials used in food and feed applications, such as plant-derived sugars and oils, is avoided (Vu et al., 2021).

The process starts by screening and selecting strains known to efficiently use VFAs and convert them into PHA. Strains from genera such as *Cupriavidus*, *Bacillus* and *Pseudomonas* have been extensively studied and optimised for this purpose (Pradhan et al., 2018; Sabarinathan et al., 2018).

Optimizing fermentation conditions is critical for maximizing PHA production efficiency and reducing production costs, in addition to tailoring of the polymer composition, which results in the production of a polymer with specific mechanical properties. Parameters such as VFA concentration and composition, pH, temperature, aeration, agitation, and nutrient supplementation are carefully controlled to maintain optimal microbial growth and PHA accumulation (Mengmeng et al., 2009). The main challenge of the ELLIPSE project is the production of a PHB with the appropriate characteristics for agricultural and packaging applications. PHB usually has quite poor mechanical properties for use in applications such as blow molding (weak elongation break and melt strength and high brittleness), which are widely used in the plastic industry (Di Lorenzo and Righetti, 2013). By optimizing fermentation parameters, the type of PHB obtained can be modulated, thus varying its mechanical properties.

Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) is a type of PHA formed by chains of hydroxybutyrate and hydroxyvalerate. The presence of valerate (a larger carbon chain) in PHBV disrupts the crystalline structure that is characteristic of PHB (Grousseau et al., 2014; Policastro et al., 2021). This disruption increases the amorphous regions in the polymer, leading to enhanced flexibility (Avella et al., 2000). Odd-chain fatty acids (valeric acid and propionic acid) are precursors of the 3-HV fraction during PHBV accumulation (Policastro et al., 2021). Therefore, in the ELLIPSE project, by modulating and increasing the proportion of valeric and propionic acids during fermentation, we will obtain PHBV with a higher hydroxyvalerate content, making it suitable for applications where PHB is not feasible due to its poor mechanical properties (such as packaging or mulching films).

After fermentation, the bacterial biomass with PHBV granules is harvested from the culture medium followed by PHBV recovery and purification. Several recovery approaches include two main strategies: PHA recovery with solvents (aqueous solvents, halogenated solvents, alkanes, alcohols, esters, carbonates and ketones) and PHA recovery by cellular lysis (with mechanical treatments, oxidants, acid and alkaline compounds, surfactants and enzymes or combinations thereof) (Koller et al., 2013). One of the main challenges for the green production of PHA is the development of solvent-free recovery processes. ELLIPSE uses aqueous-based methods for the digestion with environmentally friendly re-agents of non-PHA cellular material, more concretely, specific enzymatic methods for extraction are attractive alternatives for purification of PHA.

2.1.3. Transformation of PHBV into valuable products

More than 150 different PHAs building blocks constitute the PHAs family. However, the commercial portfolio is mainly restricted to five types of PHAs, including PHBV. The current commercially available PHAs are mainly produced from sugars and/or

vegetable oils. Besides, the PHBV building block contains only between 1-2% of valerate content, which is limited for further processability.

Several technical difficulties arise when processing current PHA compounds into different end applications. PHB has a narrow processing window, during processing (particularly at temperatures of 160 degrees and higher, and with high shear and/or long residence time), thermal degradation and chain scissions occur. This results in an undesirably large decrease in molecular weight (Mw) and viscosity. PHB has also low nucleation density making a crystalline polymer at room temperature, with around 60-70 % of crystallinity and fast crystallization rate between 80 and 100 °C, but slow below 60°C or above 130 °C, so that the material remains amorphous and sticky (dos Santos et al., 2017).

Apart from that secondary crystallization can occur, which results in brittle and stiff material and slow processing cycles. The material properties then change over the next few days, so you do not immediately have the final properties. To overcome these technical challenges, it is possible to combine the different commercial PHA building blocks and transform the polymer into suitable material that can be processable into injection moulding, extrusion blow moulding, coating and film blowing technologies to end applications.

Within ELLIPSE, the customized produced PHBV with different valerate contents (5-25%) and different Mw (150-500 KDa) is investigated, formulated and transformed into end-applications, including bottles for cosmetic personal care, coating & encapsulates fertilizers and mulch films for agricultural sector.

2.1.4. Nutrients recovery and coated fertilizers

The unsustainability of the fossil-based fertilizer production, the depletion of the conventional P-based fertilizer reserves, combined with the increasing of the geopolitical scenario, highlight the urgent need for new sources of bio-based alternatives for fertilizer production.

The intensive agriculture and livestock concentration across Europe could offer a valid source of nutrients and an alternative to classic synthetic fertilizers. Typically, the related residues and livestock effluent also represent the feedstock to feed more than 1600 anaerobic digestion (AD) plant around Europe. In the AD process, the organic matter is degraded while N and P are released in the mixed liquor which leads to high concentration of nutrients in the effluent. The latter is called anaerobic digestate which includes the stabilized organic matter, N and P compounds, potentially exploitable to produce bio-based fertilizers (Lorick et al., 2020).

The available technology developed and exploited at industrial level, for nutrient recovery from anaerobic digestate, comprise on chemical/physical steps aiming firstly at the solid/liquid separation of the organic and the mineral and soluble forms (Selvaraj et al., 2022). This step is accomplished mainly by screw-

press, centrifuge and belt filter, which lead to the production of a solid fraction characterized by a total solid concentration between 20-25% and high amount of organic N and P (Munasinghe-Arachchige et al., 2020). The liquid fraction is much more pure on suspended solids but is rich in ammoniacal nitrogen and orthophosphate.

Among the other technologies, the ELLIPSE project will explore and develop a nutrient recovery technique based on membrane separation. Specifically, the system comprises on a preliminary solid/liquid separation aiming the removal of all the fibers and particles and an ultrafiltration unit (UF) with a consequent obtaining of a concentrate fraction rich in suspended solids, microorganisms and organic nitrogen. The free ammonia gases permeate the wall of the shell side of the membrane contactor and dissolved from the counter current flow of sulphuric acid recirculated in the lumen side. The final product will be a solution of ammonium sulphate with a final nitrogen up to 7%.

Another promising option are photosynthesis-based technologies that incorporate the use of microalgae to recover nutrients while simultaneously sequestering carbon dioxide (CO₂) and producing beneficial biomass. Cultivating microalgae on wastewater has gained popularity in recent years as a novel method to recover nutrients before they are released into the environment (Acien Fernandez et al., 2018). Algae has strong potential for use in large-scale systems for upcycling N and P into biomass due to their rapid growth rates (Arumugam et al., 2018). A sustainable farming system promoting the circular N and P-bioeconomy concept could involve growing these aquatic species on either diluted manure or biogas digester effluents and harvesting them for use as a mineral fertilizer substitute (Königer et al., 2021; Rajagopal et al., 2021). Currently, the scale-up of autotrophic cultures is a time-consuming process occupying a significant area of the industrial production plants. Several studies had proposed a two-stage cultivation strategy involving heterotrophic growth in the first stage for high-density inoculum preparation and to achieve a decrease in the photobioreactors (PBRs) volume required to start (Wang et al., 2017). Even so, this type of cultivation in PBRs still represents an infrastructure and land space greater than the heterotrophic cultivation which can take place in any type of closed fermenter. The technology consists of three main stages, (i) microalgae cultivation in two stages, which consume the organic matter and nutrients contained in the digestate; (ii) harvesting or separation phase to recover the solid fraction of microalgae as high value by product and the liquid fraction as reusable water; (iii) a drying stage by spray dryer to recover the dry microalgae (Barros et al., 2019).

The European Union produces an estimated 180 million tons of digestate annually, containing high concentration of nitrogen (2-5 kg/m³) and phosphorous (0.5-1.5 kg/m³) compounds. By recovering these nutrients, the project can reduce

reliance on synthetic fertilizers, thereby decreasing environmental pollution and promoting soil health. Utilizing these waste streams as biorefinery feedstocks for the recovery of N and P, the ELLIPSE project not only addresses waste management challenges but also contributes to a circular economy. This approach reduces the volumes of landfilled waste, opens new avenues for a platform chemical and bioplastic production, and creates additional revenue for related industries.

The escalating demands of a growing global population coupled with diminishing arable land per capita necessitate continual advancements in fertilizer application practices. However, conventional nutrient application strategies have faltered in optimizing the use efficiency of key elements like N and P. To address this challenge, the adoption of innovative fertilizer products capable of enabling a controlled release of salts and micronutrients, the controlled release fertilizers (CRFs) has emerged as a promising tailored nutrient management solution to curtail environmental risks while preserving crop productivity.

This granulated fertilizers still represent niche and high-valuable products with applications in horticulture, lawn maintenance (including sport fields), landscaping (including green area in municipalities) and agriculture (Shaviv et al., 2000). These fertilizer products are based on different technologies which create a barrier around the granules, protecting the salts and nutrients from immediate washing and controlling to some extent the release of nutrients into the soil up to 24 months. This strategy of “doing more with less” has several advantages: (a) avoiding an excessive use of fertilizer salts; (b) Reduced nutrient losses (N, P, K and micronutrients) to the environment (‘run-offs’) and the associated impacts (e.g. accelerated eutrophication in aquatic environments from P) (c) Maintained /increased crop yield rates at a lower nutrient application rate (d) Improved quality of plants that need a continuous supply of nutrients at a low rate (e) Cost saving and reduction of labour associated to a reduce number of applications on field. On the other hand, these fertilizer products are obviously more technological challenging (spray coating from organic solvents, fluidized beds), they are produced at higher cost, and even small defects hinder functionality (high quality needed).

In this context, the ELLIPSE project will scale-up well-known technologies for the preparation of biodegradable in soil granulated products in which a biodegradable matrix will protect and act as a barrier to control the release of nutrient in soil and soil. ELLIPSE partners has been improving a technology that combine biopolymeric materials and mineral fertilizer in a one-step extrusion process to obtain a granule in which the salts are finely dispersed in the matrix. The PHBV biodegradable biopolymeric matrix will protect the minerals from run-off and will enable a gradual intake of nutrient to the plant. After the nutrients have been released and in-taken by the

plant, the remaining empty biopolymeric matrix will biodegrade in the substrate or soil without bioaccumulation and without the generation of microplastics, satisfying the requirements by the EU fertilizer products regulation. As the same time, the ELLIPSE project will foresee the upscale of a technology that enable the coating of pristine mineral granules by spraying solutions of biodegradable materials in rolling drums.

2.2. Challenges and innovations

ELLIPSE addresses different challenges that involve different topics such as organic waste valorization into VFA, PHBV production and purification or biofertilizers, among others. The main challenges and innovations associated are depicted in Table 1.

2.3. Relevance and significance.

The European Green Deal is the European Union Strategy to transform the economy and society to achieve net-zero GHG emissions by 2050. It

emphasizes the transition from linear to a circular economy through the Circular Economy Action Plan. ELLIPSE is fully aligned with this approach, where waste streams are bioconverted into high-value products closing the loop of these organic wastes and providing sustainable materials such as biobased and biodegradable bioplastics and new sustainable fertilizers.

Besides, the Green Deal also aims to decarbonize the industry and foster the adoption of clean and sustainable technologies. ELLIPSE promotes the innovation in clean technologies providing alternatives to waste streams that are directly landfilled or incinerated.

Considering the fertilizers, the new European regulation on controlled released fertilizing products address different aspects as the soil health and microplastic pollution, as currently the main controlled released fertilizing products are based on non-biodegradable products, the new fertilizer produced in ELLIPSE based on biodegradable polymers suitable in soil conditions will be a big breakthrough in this field promoting the sustainability avoiding the microplastic pollution.

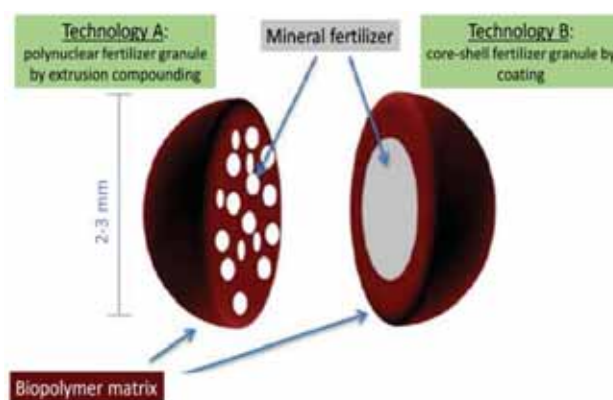


Fig. 1. Technology for bio-coated fertilizers

Table 1. Main challenges of ELLIPSE project and innovations related

Challenge	Innovation
Maximize VFA production to improve PHAs production efficiency.	A combined strategy of the following approaches will be implemented: <ul style="list-style-type: none"> • Use of AnMBRs • Addition of zero-valent iron nanoparticles to enhance the microbial metabolic activity • Optimization of the ratios between the main waste streams and other OW (glycerol or dairy sludge).
PHBV purification through more efficient and sustainable processes	Organic solvent-free methodology to purify PHBV including: <ul style="list-style-type: none"> • Use of aqueous-based methods for the digestion with environmentally friendly reagents of non-PHA cellular material. • Fine-tuning the processing conditions to tailor the final average molecular weight of the PHA to the envisaged specification. • Use of specific enzymatic methods for extraction and purification of PHA, adapting a previously developed method to the specificities of the organisms to be used.
Maximize the production of VFA of interest (propionic and valeric) to produce PHBV copolymers with tailored content in valerate.	<ul style="list-style-type: none"> • Optimization of the global operational, including waste and inoculum pretreatment needs, co-digestion ratios and operational conditions to produce PHBV grades with valerate content between 5-25 %. • Use of pre-selected microbial strains known to use VFAs for PHA accumulation and selection of those strains that more efficiently metabolise the specific VFA mixes produced and yield PHA with the required monomeric compositions.

<p>PHBV fulfilling the requirements for target applications (rigid packaging, mulching film, paper coating and fertilizers' encapsulation)</p>	<ul style="list-style-type: none"> • Formulate the obtained tailored PHBV copolymers in specific new compounds and wet compounds according to the processing technologies to produce the different prototypes and final applications' requirements.
<p>Maxime nutrient recovery from the digestate through innovative microalgae hybrid cultivation technology</p>	<ul style="list-style-type: none"> • Optimize the hybrid cultivation system at pilot scale by obtaining in the autotrophic phase a quality inoculum with this type of digestate, capable of improving and completing the recovery of nutrients in the heterotrophic phase, resulting in an algal biomass with fertilising properties. • The combination of these two types of microalgae cultivation modes (PBR+HBR) presents a synergistic effect that will minimize the inherent setbacks of PBRs and HBRs, while harnessing the advantages such as overcoming the inhibition effect with the sufficient sunlight and nutrients supply in the PBR and saving surface area and time.
<p>Zero-waste strategy: biofertilizers algae/ biofertilizers</p>	<ul style="list-style-type: none"> • Improved recycling/recovering of nutrients from OW by assimilating them into algae biomass with the later collection by spray drying of a product of commercial interest as BBFs. • By supplying recycled nutrients acquired from OW streams to plants and offsetting the use of synthetic fertilisers, algae BBF grown in OW will reduce the environmental impacts. Due to the slow-release, microalgae can be used in organic, low input and conventional farmings with a frequency 3-4 consecutive seasons in the same field and can be combined with compost as a carrier.

3. Results and discussion

3.1. Objectives

Despite all the useful and unique characteristics of PHB, several of which are superior to corresponding polymers, there are still several limitations when PHB is produced at industrial scale capacities.

At the present, the main bottleneck for the large-scale production and incorporation of PHA in the market is their elevated production cost, ranging from 2.2 to 5.0 € per kilogram. This cost is at least three times higher than that of the predominant fossil-based polymers, which are priced at less than 1.0 € per kilogram (Gholami et al., 2016). Another aspect that can hinder the possibility of incorporating PHB into the market is their properties are not very suitable for packaging applications. PHB is characterized by a high crystallinity, usually between 55 and 70 % which is an important cause of PHB brittleness and by a melt strength weaker than other non-biodegradable synthetic polymers used in packaging. In order to improve flexibility, the introduction of comonomers such as 3-hydroxyvalerate (3HV), to form the copolymer PHBV, can be carried out. Compared to PHB, PHBV with higher contents (> 2%) is less crystalline and more flexible with improved impact resistance and toughness which makes PHBV more practical for packaging application (Abbasi et al., 2022). To overcome these limitations ELLIPSE project attempts to: (1) reduce the costs of production and achieve economic viability in competition with the current low petroleum manufacturing costs by using free-charge organic wastes as substrate for PHB production and by recovering, (2) enhance physical and chemical characteristics, such as better mechanical flexibility or lower toughness by producing a more versatile polymer as PHBV and (3) provide sustainable performances in the production of PHBV but also considering the end-of-life of the polymer. To achieve this the following objectives are

addressed:

1. To apply effective pretreatments for heterogeneous wastes and improve the yield of VFA production.

ELLIPSE project will set an effective pretreatment system to minimize the presence of the non-biobased impurities on the selected heterogeneous waste according to the waste requirements for subsequent acidogenic fermentation process. The project will also implement a methodology that enhances VFAs accumulation in a liquid stream, especially the accumulation of valeric and propionic acid which facilitates PHBV production by microorganisms in further steps.

2. To obtain PHBV copolymers through efficient and sustainable processes.

The VFAs rich stream from acidogenic fermentation of the processed organic wastes will be directly used as a carbon source for PHAs production, specifically PHBV copolymers, using in-house bacterial strains. Novel green enzymatic method will be employed for PHBV separation and purification to achieve the most efficient and sustainable process of PHBV production.

3. To obtain different PHBV grades and compounds according to the requirements of the final application.

In the ELLIPSE project PHBV copolymers with different contents of valerate will be produced due to the possibility of changing flexible properties and melt strength depending on 3HV content. Copolymers with high valerate content (up to 15%) will be produced for mulch film and rigid packaging applications. Copolymers with lower valerate content (up to 5%) will be produced for coating application and encapsulated fertilizers.

4. To validate new and innovative End-of-life forms to recover monomers than will be reincorporated into the production process.

Both enzymatic and chemical recycling will provide derived 3HB and 3HV monomers that will be additional monomers in the fermentation for PHBV

production, besides VFAs from acidogenic fermentation, increasing the yield of bioplastic production and producing the original biopolymer with the same properties.

5. To recover nutrients (nitrogen and phosphorous) to be used as bio-based fertilizers.

To maximize the valorisation of the organic waste, digestate stream will be treated with cutting-edge technologies (microalgae treatment and membrane filtration) to recover nutrients, in particular nitrogen and phosphorus. Recycled nutrients will be used in form of powder or struvite as fertilizer for agriculture and horticulture use, closing the loop.

6. To demonstrate environmental and socio-economic sustainability and apply mature/novel digital technologies in the PHBV production process.

The ELLIPSE project will implement methods and tools for the economic and environmental assessment of the impact from cradle to grave of the compounds/products to guarantee the safety and sustainable production of PHAs for personal care and agricultural applications. This includes the application of life cycle assessment (LCA) and life cycle cost (LCC) analyses and assessment methods of the contribution to a local circular economy. The project will also implement process simulation software and mathematical models to determine and improve the economic and social impacts and reduce the environmental risks associated with the technology process.

3.2. General concept

To achieve the previous objectives ELLIPSE project will apply a 4 phases methodology (Fig. 1) to expand the opportunities for the valorisation of bio-waste in all stages and across all sectors and meet the market requirements for the proposed application in the personal care and agriculture sectors.

3.2.1. Phase 1: Waste stream selection, analysis, pretreatment and VFA production

In ELLIPSE an efficient solution for the excess waste generated in meat production industries and paper and pulp sludges will be implemented. A pretreatment of the waste streams will be required because slaughterhouse waste normally contains about 5% of different impurities and solids such as plastics that could interfere in the acidogenic fermentation (AF).

Residues from the paper industry, consist of organic matter (e.g. cellulose fibres) but also other impurities such as filler (kaolin, calcium carbonate), ink, binder/stickers and other impurities (e.g. plastics). Before using it as a substrate for VFA production, it is necessary to separate problematic inorganic materials and impurities using a pretreatment step. This separation step will be carried out using existing technologies such as pulper, de-inking processing unit, cavitation unit, among others, and other steps such as sedimentation, sieving will be evaluated if

necessary. Then physical and chemical treatments will be implemented to first obtain homogeneous waste stream and adequate particle size and promote the hydrolysis through basic and/or acidic pretreatments. Then, combination of pretreatments and organic wastes will be studied and VFA yield and VFA profile will be evaluated to optimise their production for its use in next phases (Phase 2).

The slaughterhouse bellygrass and paper and pulp sludge will be used as feedstocks to produce VFAs by AF. AF will be optimized through a design of experiments targeting an improvement in the propionic and valeric content. During this phase strategy as mixture of wastes, controlling pH to inhibit methanogenesis and dose iron nanoparticles will be used. ELLIPSE develops AnMBR technology and acidogenic fermentation under pilot-scale conditions to demonstrate the efficiency of membrane bioreactors in the separation of products such as VFAs or biomass and how this technology overcomes traditional limitations by significantly improving organic matter degradation and selectivity of VFA production.

AnMBRs allow for greater retention of microbial biomass due to the physical separation offered by the membranes, which maximises the efficiency of substrate conversion to VFAs. Once optimized operational conditions at lab scale in the 2L reactors for the acidogenic fermentation, an AnMBR will be used to validate the parameters selection and to improve the yield and quality of the VFA produced: (i) check that the operating pH selected does not affect the durability and performance of the AnMBR membranes by previous test at an existing 30L-scale AnMBR plant before scaling the process; (ii) assess degree of retention of Fe(0) nanoparticles in the reactor, favouring the economy of the process; (iii) check the possible effect of undissolved solids in the AnMBR operation, and if required, include a phase separation stage before the fermenter to remove part of the solids entering the system.

A relevant example of AF at pilot and industrial scale is the use of poultry waste in an AnMBR (Yin et al., 2022), which has proven to be very effective for the production of VFAs that achieved a high yield of 0.90 g-VFA/g-VS under weakly alkaline pH conditions and an organic loading rate of 2 g-VS/(L-d), which is in line with the parameters tested at ELLIPSE. In addition, the system was able to maintain stable productivity for more than 50 days under high solids concentration and high flow rate conditions. The best conditions will be upscaled in two pilot plants: in BEST (Austria) the approach is to simulate a direct implementation into pulp and paper production process to produce lower chain VFAs aimed for coating materials for paper/cardboard. In Green Generation (Ireland) the approach is on slaughterhouse waste targeting longer-chain VFAs intended for PHBV for agricultural applications (Placido and Zhang, 2018).

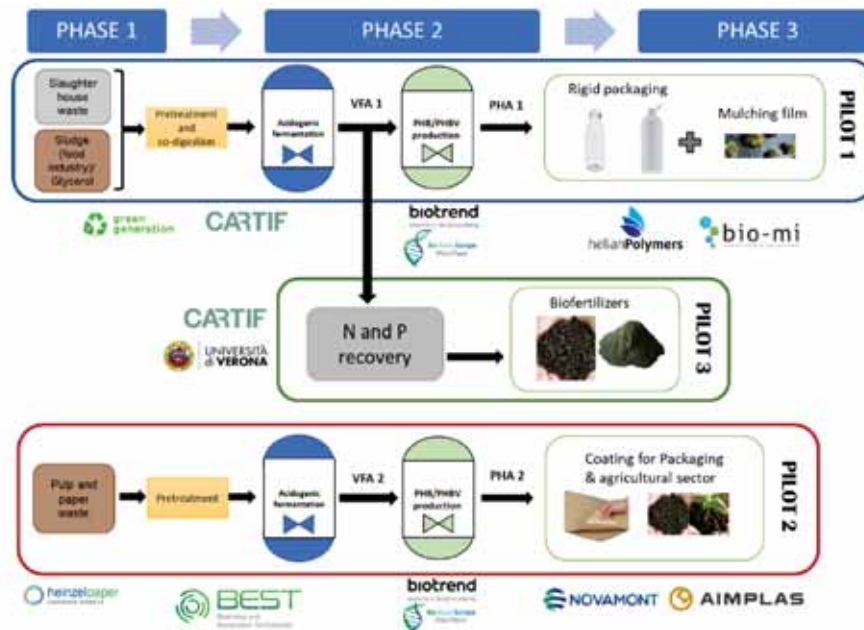


Fig. 2. ELLIPSE methodology

3.2.2. Phase 2: Intermediary products

In this phase, two approaches will be tackled: VFAs produced in the phase 1 will be used to produce PHAs by biotechnological processes. During this phase strains able to growth on carboxylic acid in different concentrations coming from Phase 1 will be selected. The bacterial producers selected will be grown in fed-batch cultures with the VFA mixes obtained from the AF. The cultivation strategy will be optimized to attain high polymer productivities by changing parameters such as carbon source feeding regime to avoid substrate inhibition, the optimum dissolved oxygen profile and the best trigger of polymer accumulation (nitrogen vs phosphate depletion). Different strategies such as pH stat and cell recycling systems, in case a diluted substrate stream will be tested. The most optimal fermentation process on the VFA mixture selected substrates will be scaled in 1500 L bioreactors in BBEP facilities (Belgium).

The digestate will be treated by two different technologies (microalgae bioreactor and different types of membranes bioreactors) to recover the nutrients, mainly N and P. These nutrients are the base for future formulations of biobased fertilizers (BBFs.)

By one hand, using microalgae bioreactors, the digestate generated in phase 1 will be used as feedstock for microalgae cultivation. Once the algae biomass is harvested it will be separated from the solution by centrifugation and spray-dried to estimate the quantitative potential of nutrient recovery per unit volume of feed. Besides, supernatant will be removed, and remaining biomass heated until completely dried. Algae control will be evaluated with spectrophotometry and optical microscopy. The best operating conditions to maximize the quality and quantity of the recovered nutrients will be selected.

By other hand, nutrients recovery from fermentation digestate will be performed through

sequential unit operations including solid/liquid separation of the digestate through a screw-press, ultrafiltration of the permeate, reverse osmosis and ammonia recovery as ammonium sulphate by membrane contactor. The process will be implemented by treating of 100-200 L of digestate per day. At the same time a membrane contactor for ammonia recovery from digestate will be used where NH_3 from the origin solution, passing through the gas-filled porous membranes and recovered by sulfuric acid to obtain ammonium sulphate.

3.2.3. Phase 3: Applications

There are two big sectors in which the focus is set to validate both PHA and nutrients: personal care and agriculture. By one hand, personal care will use the PHBV with less content of valerate for rigid packaging and the PHBV more flexible for paper coated flexible packaging. Agriculture sector will include the use of the PHBV for mulching film production, together with the combination of the recovered nutrients and PHA into a control-release BBF.

3.2.4. Phase 4: End-of-life alternatives

Finally, end of life alternatives of the validated products will be studied to confirm their recyclability and biodegradability respectively: Mechanical recycling route for the PHBV based rigid packaging and the mulch films; chemical recycling for rigid packaging, to check the feasibility of recovering monomers that could be further introduced in the PHB/PHBV production process and enzymatic recycling for PHBV coating in paper. Mulch films and slow-release fertilizers will also be tested for biodegradability in soil according to ISO 23517:2021.

In addition, a full life cycle assessment (at technical, economic and social level) will be carried

out to evaluate the whole ELLIPSE value chain. These three phases are complementary in three different pilots, where the starting organic waste and applications are different:

3.3. Pilot phases

3.3.1. Pilot 1

In pilot 1 a co-digestion of feedstocks will be carried out with the aim of ensuring the most optimal conditions to produce VFAs. Slaughterhouse waste will be the main residue used coupled to either sludge from food industry or glycerol. In this pilot 1, the co-digestion of the residues will be optimized to assure the highest odd-chain VFA concentration (valerate and propionate). This is because in the next step when bacterial strains are accumulating PHBV, the presence of odd-chains VFAs trigger the accumulation of a higher fraction of 3-HV in the PHBV polymer making it more flexible and with lower melt strength, which makes it more suitable for application in the personal care and agricultural sectors.

Rigid packaging for the personal care sector and mulching film for agriculture are produced. In this way, valerate high-content polymer will be used to produce compounds to produce bottles by EBM and flexible films (mulch films) obtained by blown film extrusion.

3.3.2. Pilot 2

In pilot 2, different residues produced during the paper production are addressed. This pre-sorted wastepaper industry waste will be used in the production of VFA. For this pilot the requirements of a VFA stream rich in valerate is not so high, as the final applications achieved do not require the same processing conditions than in pilot 1.

In this context, A higher total concentration of VFAs will be sought rather than a higher partial concentration of propionate and valerate. As the valerate fraction is lower in this pilot the valerate-low content PHBV with lower melting point will be used to produce wet compounds suitable for coating application for paper-based flexible packaging for liquid personal care products using conventional printing technologies.

3.3.3. Pilot 3

In pilot 3 a strategy to recover N and P will be implemented with the aim of producing bio-fertilizers. Two different technologies will be validated.

- The technical feasibility of nutrient recovery via hybrid microalgae cultivation process (photoautotrophic and heterotrophic) while treating digestate from acidogenic fermentation will be demonstrated.

- Nutrient recovery will also be performed through physical methods optimizing two different technologies to recover ammonia, as ammonium sulphate: i) pressure drive membrane technologies (ultrafiltration and reverse osmosis) and ii) membrane contactor.

The products will be converted into pellets will be further processed to produce two types of innovative fertilizers with tunable kinetics of nutrient release, applying PHA from pilot 2 in the formulation of polynuclear fertilizer granules by extrusion compounding and core-shell fertilizer granules by coating. These final products will be tested for biodegradability in soil according to ISO 23517:2021.

3.4. Consortium members

The aforementioned issues are being tackled through a collaborative initiative represented by a project consortium, consisting of 1 academic team (University of Verona), 3 technological centers (AIMPLAS, CARTIF and BEST) and 10 industry partners (BBEPP, BIOTREND, NOVAMONT, BIOMI, ZERO-E ENGINEERING, HELIAN POLYMERS, ENCO, GREEN GENERATION and LARKIRCHEN PAPIER) from 8 different countries. The role and the background are the following (Table 2).

3.5. Preliminary results

During the first period of the project (20 months), the project consortium worked mainly on tasks related to the production of VFAs from different wastes and its optimization. A brief summary of the results is presented below.

3.5.1. Characterization and selection of wastes

The waste streams samples were provided by:

1. Pulp & paper processing industry provided by Heintzelpaper. Residues from pulp and paper industry (rejects from deinking process), OCC (old corrugated containers) process and chemical pulp production. The samples were specifically taken from four different processes (Fig. 3). Two secondary fibres processes: i) De-Inking Process (DIP; used to regenerate raw material from waste paper for the production of white paper), ii) Recycled Fibre (RCF) process (RCF; first step of recycling brown paper, e.g. cardboard), iii) the production of pulp from primary fibres/wood (POELS) and iv) wastewater treatment (ARA).

The characterization provided analytical data for the following parameters of the waste streams: total solids (TS), volatile solids (VS), pH, chemical oxygen demand (COD), soluble chemical oxygen demand (sCOD), total Kjeldahl nitrogen (TKN) and biomethane potential (Table 3) to evaluate the suitability of the samples for the acidification process. Samples from the processes DIP and ARA show a low methane yield. Sample RCF10-04 achieved the third highest methane yield, however most of the sample is inorganic sand which can be very problematic when put inside a reactor. The samples with the most promising results are RCF10-03, RCF10-05, RCF10-06 and POELS-03. These samples are regarded as most interesting for the acidification process and were therefore selected for further analysis and pre-treatment trials.

2. Organic waste provided from the company Green Generation, such as slaughterhouse waste (belly grass or rumen content), wastewater treatment sludge from dairy industry (Glanbia Ballitore WWTP Sludge,

Rosderra Edenderry WWTP Sludge) and waste from bioprocess (Glycerine from biodiesel production). The characterisation of glycerine, belly grass and sludge are shown in Table 3.



Fig. 3. List of wastes from Heinzelpaper production process, including ID, description and picture. The abbreviations mark the processes they come from: DIP = deinking process; RCF = recycled fiber; POELS = pulp from primary fibres; ARA = wastewater treatment

Table 2. List of participants in ELLIPSE project

<i>Participant</i>	<i>State</i>	<i>Country</i>	<i>Role</i>
AIMPLAS	Technological center	Spain	Mechanical, chemical and enzymatic recycling of personal care and agricultural products
Bio Base Europe Pilot Plant (BBEPP)	Industry partner	Belgium	Scale up of the PHA fermentation process in 1500 L reactor and scale up of PHA recovery and purification
University of Verona (UV)	Academic team	Italy	Green nutrient recovery through physical methods and ammonia recovery from liquid phase of digestate by membrane contactor
Biotrend (BIOT)	Industry partner	Portugal	Technical scale implementation of the PHA fermentation process from VFA-rich streams.
Novamont S.p.A (NVMT)	Industry partner	Italy	Compounding, coating, prototyping and validation of PHA based bioplastic into innovative fertilizers with tunable kinetics of nutrient release
Fundación CARTIF	Technological center	Spain	Pre-treatments of waste streams. Acidogenic fermentation to produce VFAs as substrate for PHA fermentation. Nutrient recovery from hybrid microalgae cultivation
Zero-Emissions Engineering (Zer0-E)	Industry partner	Netherlands	Full life cycle assessment at the technical, economic, and social levels to evaluate the value chain feasibility and scalability.
Helian Polymers (HP)	Industry partner	Netherlands	Compounding, prototyping and validation of PHA based bioplastic into rigid packaging for personal care products applications
ENCO srl	Industry partner	Italy	Dissemination, exploitation, awareness and market uptake
Bioenergy and Sustainable Technologies (BEST)	Technological center	Austria	Pre-treatment of pulp and paper waste for the acidification process. Acidogenic fermentation in a pilot reactor to produce VFAs
Green Generation Ireland Ltd (GG)	Industry partner	Ireland	VFAs production at pilot scale from co-digested sludge
Laakirchen Papier AG (HEPA)	Industry partner	Austria	Supply of substrates directly from the pulp and paper production process
BIO-MI	Industry partner	Croatia	Compounding, prototyping and validation of PHA based bioplastic into mulch film application

Table 3. Characterization and analysis results of samples from Heinzelpaper production process

ID	pH [-]	TS [%]	VS [%]	COD [g/kg]*)	sCOD [g/L]	TKN [g/kg]	Biomethane potential [Nm ³ /t FM**)]
RCF10-03	7.0	3.2	2.7	20.59 ± 4.54	4.7	0.2	6.7
RCF10-04	6.8	26.0	4.7	82.78 ± 9.21	7.0	0.3	5.6
RCF10-05	6.1	3.3	2.9	36.81 ± 0.79	5.2	0.1	7.1
RCF10-06	6.6	1.4	1.1	13.43 ± 3.09	4.1	0.0	3.3
DIP11-01	6.8	6.2	1.6	23.81 ± 18.02	3.7	0.0	2.8
DIP11-02	6.7	1.3	1.0	11.78 ± 12.73	2.9	0.2	2.2
ARA-A-01	7.2	1.2	0.5	4.07 ± 3.45	0.3	0.5	0.8
ARA-A-02	6.8	4.9	3.9	25.38 ± 2.41	0.7	1.1	2.4
ARA-A-03	7.1	0.5	0.2	1.87 ± 1.21	0.4	0.0	0.4
POELS-02	5.8	1.1	1.1	9.61 ± 0.11	0.2	0.0	2.4
POELS-03	6.3	0.8	0.8	5.93 ± 5.41	0.0	0.0	3.0

*) Samples were inhomogeneous which made sampling difficult. Therefore, the COD values (duplicate analysis) show a high deviation. **) FM = fresh matter

3.5.2. Impurity removal and pre-treatments

Samples from the pulp and paper industry were inspected to find strategies to remove potential impurities. Common impurities are plastic pieces, polystyrene, stickies, and metal pieces. Those impurities are problematic for microbial processes and should be removed before the acidification. In the RCF process for cardboard recycling, impurity removal is already performed during the process in the production plant. Samples RCF10-03, RCF10-05 and RCF10-06 are almost free from impurities, only small styrofoam pieces are left in RCF10-03. Therefore, it was decided that no additional impurity removal for the RCF-samples is necessary. No impurities were detected in samples from the production of pulp from primary raw material (wood). Thus, no impurity removal is necessary for the sample POELS-03. ARA and DIP samples were not selected for acidification which is why impurity removal of those samples was not considered.

With the chosen samples (RCF10-03, RCF10-05, RCF10-06 and POELS-03, see above), pre-treatment trials were performed to enhance the potential yield of VFAs in a subsequent acidification step. The sCOD was used as an indicator for easily available organic matter; an increase in sCOD was considered a positive effect for acidification. Four methods were investigated: two physical treatments to obtain a homogeneous stream (i) heat (125 °C), (ii) high-shear mixing (blender), and two chemical treatments to promote hydrolysis and diminish the possibilities of methanogenesis (iii) acid addition (H₂SO₄, pH 5 or 4), (iv) base addition (NaOH, pH 9-11) (Ortner et al., 2015).

(i) Heat treatment was performed in a 12 L bench autoclave. A volume of 300 mL homogenized sample were transferred to 500 mL bottles and then put in the autoclave. The autoclave was heated up to 125 °C and the temperature was held for 15 minutes. Afterwards it cooled down to room temperature.

(ii) The samples were mixed to break down particles and with that increase the surface available for (bio-) chemical reactions. A blender was used for this and the samples were mixed until no further change in fibre or liquid texture was observed (approximately 30 sec).

(iii) The pH value was adjusted to 5 using H₂SO₄. After the adjustment of the pH, the samples were stored at 4°C for 24 hours, and analysis (sCOD, VFA) was performed afterwards. The samples were stirred during the pH adjustment using either a magnetic mixer or a spoon if the sample was too viscous.

(iv) The pH was adjusted to 9-11 using NaOH. After the pH adjustment, the samples were stored at 4°C for 24 hours, and analysis (sCOD, VFA) was performed afterwards. The samples were stirred during the pH adjustment using either a magnetic stirrer or a spoon if the sample was too viscous and base was added.

At first, the different treatments were performed separately, and original samples were treated using either heat, high-shear mixing, addition of acid or base. In Fig. 4, the sCOD content in the original samples compared to after the treatments is shown.

Each individual pre-treatment method increased the sCOD content, except for high-shear mixing of sample RCF10-03 which had no effect. Heat treatment had the highest impact and increased the sCOD by 15-40 %. The sCOD of the sample POELS-03 was very low in the beginning (0.03 g/L) and was only increased to around 0.1 g/L with each treatment. Considering a subsequent microbial acidification process, those results indicate that the used pre-treatments solubilize solid organic matter and thus make it more available for microbes. The planned conversion of organic matter to fatty acids could be increased if a pre-treatment is performed.

The content and composition of VFA in the samples after the separate pre-treatments is shown in Fig. 5. The VFA content in the original samples was between 1.9 g/L and 2.8 g/L in the RCF-samples and were not considerably increased by the pre-treatments. However, the composition changed in the sample RCF10-05, where lactic acid was formed during heat treatment and acid and base addition. No VFAs were detected in the sample POELS-03. After all treatments were tested separately, a combination of treatments was used. The pre-treatment cascade consisted of high-shear mixing, pH adjustment to pH 4 followed by heat treatment.

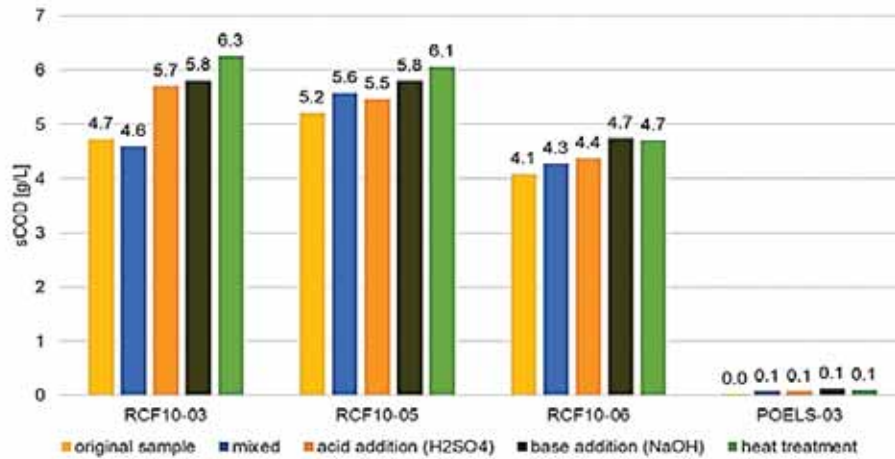


Fig. 4. sCOD content in original samples compared to after separately performed pre-treatments

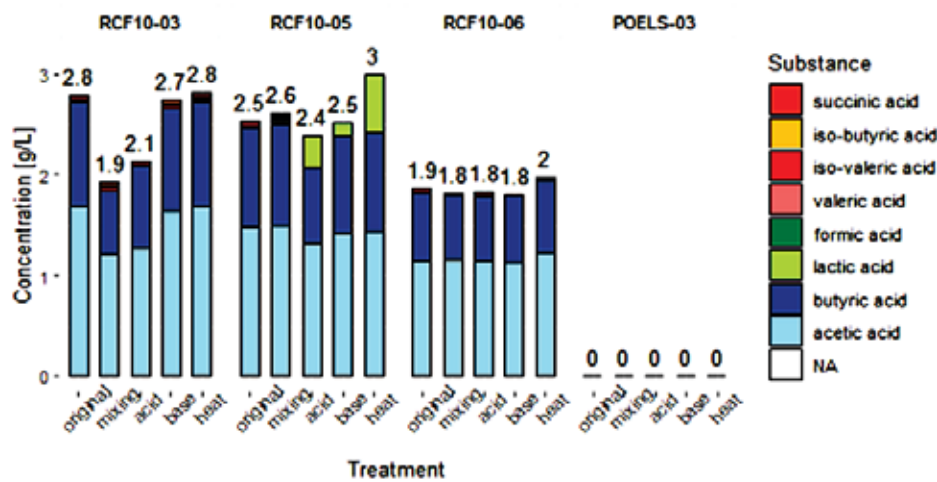


Fig. 5. Volatile fatty acid (VFA) content and composition of original samples compared to after separately performed pre-treatments

After each individual step, the sCOD and VFA were determined (results not shown). Combining the treatments did not increase sCOD or VFA content. In the case of the organic waste samples from Green Generation, physical treatment (blending and chopping) was performed for the homogenisation, for the most solid samples (bellygrass). The goal was to test the samples in the most natural and easy/feasible way possible.

For the chemical treatment, H₂SO₄ and NaOH was applied to the samples adjusting the pH to values 5.5 and 10 respectively. The VFA content was influenced by the modification of the pH, increasing the concentration of acetic and propionic acids mainly at a basic pH of 10 and has reached a yield of 0.5 g VFA/g VS.

3.5.3. Acidogenic fermentation tests

A series of acidogenic fermentation tests were performed to evaluate the feasibility of producing VFAs for the development of marketable bioplastics. The trials were carried out in glass bioreactors with a working volume of 1 and 2 L filled with different

volumes between 700 mL and 1100 mL. The total volume depended on the amount of waste (substrate) added to the mixture according to the substrate-to-inoculum S/I ratio, with value of 2 g VS/g VS used as positive control (inoculum from Valladolid’s Wastewater Treatment Plant in Spain), and in order to inhibit methanogens, it was chosen to allow O₂ to be present in the reactor (Wainaina et al., 2019). A correction of the methane production from the inoculum was done using a blank test containing only inoculum.

For each waste stream and pH condition, replicates were added to ensure that the results obtained were similar under these conditions. With the characterization of the waste streams, the amount of waste and inoculum was calculated based on the VSs. The trials focused on the effects of acidic (pH 5.5 adjustment with H₂SO₄) and alkali pH 10 with NaOH addition) on VFA production efficiency and VFA composition. To maintain a homogeneous mixture, the reactors were placed on an orbital shaker (shaking table) which was located in a temperature-controlled room where the mesophilic reaction temperature was set to 38°C (Fig. 6).

The retention time was 10-13 days, ensuring acidogenic fermentation and avoiding methanogenesis. A minimum of 15 mL samples were daily withdrawn to analyze the evolution of VFA concentration and profile. Samples were centrifuged and stored at 4 °C prior to their analysis. Phosphoric acid (150 µL H₃PO₄/15 mL sample) was added to the samples to avoid volatilization of the fatty acids to ensure their preservation. Gas chromatography-flame ionization detection (GC-FID) was the analytical technique used to separate and analyse mixtures

consisting of volatile components to quantify VFA in samples. By examining the pH values for the same samples, better results are generally obtained under basic conditions than under acidic conditions. The pH was monitored and adjusted during the tests. The samples of glycerine, sludge and bellygrass with pH 10 has the highest concentration of total VFAs with 1927.64 mg/L, 1657 mg/L and 1652.38 mg/L respectively (Fig. 7). For glycerine the maximum was reached at day 7 and for the bellygrass and sludge was at day 12.



Fig. 6. Anaerobic digestion reactors on the orbital shaker

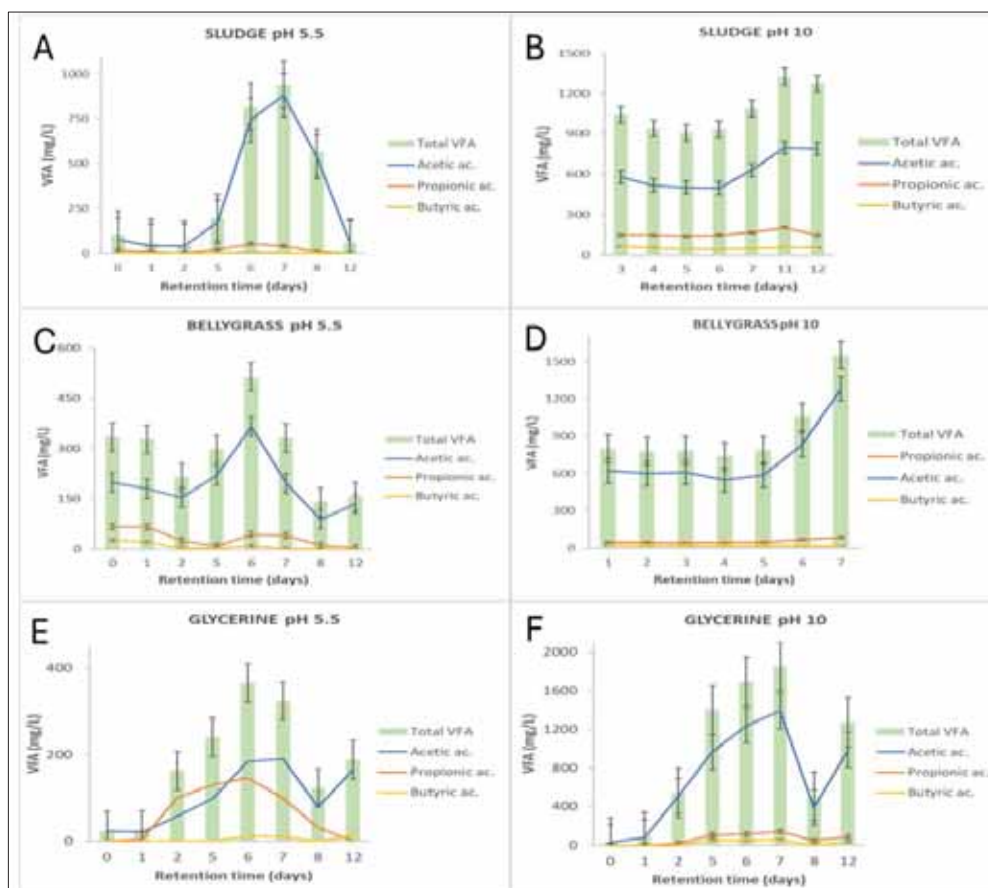


Fig. 7. VFA concentration (mg/L) of bellygrass (A, B), sludge (C, D) and glycerine samples (E, F) for 12 days pretreated at pH 5.5 and pH 10

The soluble COD (sCOD) COD was measured with quick tests using HACH DR 1900 Portable Spectrophotometer and Cuvette Test System with the Dry Thermostat Reactor LT200 (Hach) obtaining values of 2690, 1800 and 4080 (mg/ L) for glycerine, sludge and bellygrass respectively for that residence time. The VFA yield reached a value of 0.33 g VFAs /g VS glycerine, 0.47 g VFAs /g VS sludge and 0.27 g VFAs /g VS bellygrass. Under both pH conditions, VFAs represented a predominance of acetic, propionic and butyric acids. Figure 7 shows the concentration of these acids (mg/ L) over the residence time (days) of two replicates for each organic waste under the basic conditions. Similar concentration around 2 g/L of total VFAs has been founded in other acidogenic fermentation where the substrate was sludge coming from pulp and paper mills. In this case also acetic acid and propionic acid were predominant, but higher concentrations of valeric acid were founded (Li et al., 2022).

4. Conclusions

In general terms, impurity removal is already performed during the production process and the selected samples do not need further removal steps. VFA content depended mainly on the nature of the samples and the pH, as physical pre-treatments in most samples hardly affected the content of VFA in the samples. VFA content is not influenced significantly by the treatments. A combination of more than one method was not beneficial. Mixing the samples do not increase VFA content. As expected, samples in which pH was adjusted to 10 resulted in improved production of VFAs compared to pH 5.5 because the alkaline condition enhances the hydrolysis.

Green Generation's samples (bellygrass, glycerine and sludge) generated more VFAs than Heinzelpaper samples. This is caused by the higher COD content in the Green Generation's samples. Most of the VFAs produced are acetic acid though it would be interesting to have higher contents of propionic and valeric acids, which would allow to produce PHBV, a more interesting polymer than homopolymer PHB.

Experimental results in the preliminary phase of the first laboratory scale trials showed that adjusting the pH to 10 during fermentation allows higher production of PHBVs, reaching concentrations of up to 2 g/L in glycerine samples, with high yields also in bellygrass and sludge. These advances are crucial for the production of biopolymers such as PHBV and need to be improved in terms of co-digestion of the different organic wastes and further operational parameters and the addition of Fe(0) nanoparticles will be tested. As initial trials have shown promising results, further tests will be conducted in future stages by adjusting different operational parameters, such as pH, temperature, waste concentration and substrate ratio, in order to maximise efficiency and improve the production of VFAs. The results obtained confirmed the feasibility of obtaining VFA from these different

organic and pulp and paper wastes but, in order to obtain PHB from VFA, it is necessary to reach a higher concentration, so it is necessary to further investigate parameters to optimise the acidogenic fermentation and to study possible downstream concentration techniques. Different alternatives also can be applied to increment the concentration of VFAs. For example, in similar experiments, ultrasonic pretreatments have been used to increase the concentration of VFAs from pulp and paper sludges. These type of pretreatments can increase the VFAs production in the acidogenic fermentation by at least double.

These additional tests will be key to optimise the process and ensure its industrial scalability.

In addition, the use of membrane bioreactors will lead to better biomass retention and more efficient product recovery, overcoming the limitations observed in traditional technologies. The combination of these factors positions ELLIPSE as an innovative project in the circular bioeconomy, contributing to sustainability and efficiency in industrial waste management.

At the same time, one of the main objectives in ELLIPSE project is to obtain a PHBV biopolymer with more suitable properties and processability than brittle PHB polymers. To achieve this a strategy focused on the increment of valeric and propionic acids, which are precursors of the 3HV monomers, has been implemented. Thus, valerate and propionic acids concentration in the culture medium strongly affects the 3HV fractions of the PHBV polymer. In experiments with slaughterhouse wastes, as for example animal blood, the production of valeric acid is higher, representing in some cases around 20% of the total VFAs. However, these substrates normally contain higher organic matter, measured both as volatile solids (VS) of 80-100 gVS/kg or as COD of 180-260 gCOD/kg, while the organic matter of the samples used during ELLIPSE experiments was considerably lower. For example, bellygrass samples contains only 15.33 gVS/kg. These findings make it necessary to look for pretreatments that lead to an increase in the organic matter available for the acidogenic fermentation processes, which will be translated into an improvement of fermentation yields and consequently a higher concentration of propionic and valeric acid. Similar results in the content of valeric acid (less than 5% of total VFAs) when glycerol was fermented as the main substrate, but higher concentration of propionic acids was founded, being ever more abundant than acetic acid (45% of propionic acid and 40% of acetic acid of the total VFAs produced). One possibility to improve these results could be the implementation of anaerobic co-digestion which provides an opportunity to overcome the drawbacks of mono-digestion by simultaneously digesting two or more feedstocks. Among the principal benefits of co-digestion, promoting a more diverse microbial community, better nutrient balance or increasing the bioavailability of nutrients could help

to achieve a better VFA concentration during acidogenic fermentation.

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CIRCULAR ECONOMY INITIATIVES IN THE MARCHE REGION: IMPLEMENTING OF INDUSTRIAL SYMBIOSIS WITHIN THE MARLIC PROJECT AND MAPPING REGIONAL SKILLS AND GOVERNANCE

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Abstract

To reduce material use in production, we should embrace circular economy (CE) principles. Industrial symbiosis (IS) enables material, expertise, and service exchanges, though legislative and technical limits restrict local efforts. The MARLIC project was initiated in response to the necessity of establishing a central point of reference in the Marche region in Italy, to develop new materials and promoting circular use. This study aims to present the findings of two activities conducted by ENEA. The first activity involves the application of a methodology for the identification of roles and skills within the CE sector in the region, along with the collection of evidence useful for the proposal of an organisational framework at the regional level. In this regard, according to the evidence obtained, it can be supposed that the best organizational model for CE skills systematization can be achieved by creating a specific regional structure for CE and resource management. The second activity is a pilot IS application with local enterprises, following ENEA's methodology. In a workshop, 30 companies identified 85 matches and over 300 shared resources, including paper, leather, and plastic. The strong participation and positive feedback from organizations at the IS workshop show their commitment to enhancing process circularity and applying IS for improved environmental sustainability. Furthermore, the parallel activity of identifying a regional governance model for CE has provided a more concrete definition of the path towards transitioning from linear to circular production models.

Key words: circular economy, governance, industrial symbiosis, marches, resources circularity, skills on circular economy

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1. Introduction

The concept of a "circular" economy entails the production of goods and services through the reintroduction of materials into industrial ecosystems or their economic revaluation through reuse, recycling, and recovery, thus avoiding the waste that typically occurs in a "linear" economy. This approach

intervenes across all stages of the product lifecycle, from design to end-of-life management. In a circular economy (CE), the value of products and materials is preserved for as long as possible, shifting away from the traditional linear model of production and consumption. This shift represents a departure from the notion of waste minimization towards a holistic perspective that reconciles economic and

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environmental interests, recognizing them as interconnected rather than conflicting. The CE embraces a broader concept of well-being that encompasses both economic prosperity and environmental sustainability. Moreover, the CE fosters the development of sustainable business models capable of closing production cycles efficiently and making more effective use of resources within a given area. One notable strategy within this framework is Industrial Symbiosis (IS), which involves the exchange and sharing of resources (such as raw materials, water, energy, waste, services, skills, etc.) among companies and other stakeholders in the region. This approach enhances the competitiveness of industrial activities and enriches the local territory by maximising the utilisation of resources within the community rather than dispersing or outsourcing them. The IS was defined as “*the economic exchange of material flow, resources and residual waste in a multiple, collaborative network facilitated by trust, available information and existing stimuli*” (Agudo et al., 2022). The transition to a CE and sustainable development represents a significant challenge for society at large and for organizations. The European Commission has established the framework for this paradigm shift with the adoption of the European Green Deal (European Commission, 2019) to achieve climate neutrality by 2050 and the new Circular Economy Action Plan (European Commission, 2020). These initiatives aim to conserve resources and material value while reducing waste and residues.

In Italy, the National Strategy for the Circular Economy (NSCE) (MASE, 2013) was approved alongside the National Waste Management Program through Ministerial Decree No. 259, dated June 24, 2022 (MASE, 2022). The NSCE serves as a programmatic document outlining actions, measures, and objectives to advance institutional policies towards a CE. It identifies IS as one of the nine key areas of intervention necessary to achieve this transition in Italy. Furthermore, the NSCE supports the development of projects in this area through specific regulatory and financial instruments, demonstrating the country's commitment to advancing CE principles. However, while the benefits and potential of IS are clear, there are numerous political, regulatory and fiscal barriers, not to mention those related to innovation and business culture, which tend to hinder the spread and adoption of this practice. The barriers are often cultural, linked to a lack of knowledge about the benefits of collaboration and exchange between companies, a reluctance to invest in innovation because it is considered 'risky', or a general lack of awareness of the existence of different options for managing waste resources. In addition, the low price of raw materials makes the cost savings from adopting IS-based business models unattractive. The lack of legislation to support the management of many types of waste contributes to the lack of viability of material exchange between companies. This article illustrates the activity carried out by ENEA (Italian Agency for New Technologies, Energy and

Sustainable Economic Development) within the MARLIC (Marche Applied Research Laboratory for Innovative Composite Materials) project on IS. The Marche region, nestled in central-eastern Italy between the Apennine mountains and the Adriatic Sea, spans roughly 9.500 square kilometres. With six provinces comprising its territory, the Marche boasts a population of approximately one and a half million residents from a demographic standpoint (Assocalzaturifici, 2024).

The MARLIC project (2023) aims to establish a regional focal point for the advancement of new materials, with a specific focus on biomaterials and the adoption of circularity principles in the utilization and reutilization of raw and secondary materials within the Marche region. The project consortium comprises 21 local companies and 5 university and research centres. The project's activities are organized into two distinct, sequential actions:

- the first project concerns the development of biomaterials and mixed advanced materials studying the utilization of new raw and/or secondary materials;
- the second project follows a circular approach through a “De-manufacturing with a view to CE according to the rules of the 4Rs (Reduce, Reuse, Recycle, Recover).

Moreover, the project represents the first structured attempt to enhance the CE and implement IS in the Marche region. Among the project activities, ENEA oversaw those related to the following topics:

- identification of roles and competencies within the CE sector in the region, along with the collection of evidence to support the proposal of an organizational framework;
- establishing an IS pilot through the facilitation of IS workshops with local businesses and stakeholders based on ENEA's methodology.

Although IS is recognized, even by the SNEC as one of the most effective strategies for transitioning production systems towards a CE, there are typically no dedicated regional structures to support this approach. Regional offices, for example, are traditionally organized by specific competencies, separating waste management from industrial policy functions. For this reason, the two activities outlined above aim to identify the competencies and roles associated with the CE and particularly with IS, while also implementing a pilot initiative to guide businesses in the practical application of this strategy.

The project activities progress along two concurrent development paths. This paper endeavours to accurately depict the project and its activity advancements on CE. Therefore, the document structure is arranged into two sections corresponding to each sub-activity. The first activity is focused on the identification of actors and skills within the reference territory. This is crucial to implement CE actions inside it and to ensure an effective ecological transition. Therefore, to support the proposal of a specific circular organizational model tailored for the Marche region, a specific methodology was applied to identify the institutional actors and technical skills in

the field of CE at a regional level, basically through: a) mapping the state of the art and creating a database of skills and roles on the CE key areas; b) consulting the identified relevant/skilled actors; c) collecting evidence/recommendations useful to define an organizational model to structure CE skills. The second activity is instead dedicated to the establishment of an IS pilot in the targeted region.

The aim of this pilot activity is to provide the companies participating in the workshop with a concrete opportunity to compare and exchange information on possible and available resources. A methodology validated by ENEA will facilitate the exchange of information and the subsequent processing of the data collected during the workshop. Once the potential synergies have been identified, the aim is also to help the companies overcome the obstacles that prevent the implementation of a symbiosis path. This is done by providing them with the regulatory and technical information that is functional for the effective realisation of the synergy, which can be further replicated in the future by other companies sharing the same type of resources.

According to a validated methodology due to decades of experience on this issue. In 2010 ENEA started the development and implementation of an IS network model thanks to several projects in Italian regions (Cutaia et al., 2014, 2015, 2016a, b; La Monica, 2016; Luciano et al., 2016). ENEA's sustainability laboratory has effectively applied its methodology to assist companies in achieving IS matches within the MARLIC project. As part of the project, an IS workshop was convened and took place at the University of Camerino. The creation of an IS pilot in the Marche region passes through different steps of the well-established ENEA methodology: (a) stakeholder individuation in the designed area (b) preliminary activities for the IS workshop (c) development of the IS workshop (d) identification of the significant flow resources and data analysis (e) identification of the synergies and in-depth analysis from a technical and economic point of view. The initial phase involves identifying stakeholders in the Marche region. An analysis conducted in the targeted area reveals a significant presence of firms in the manufacturing sector, accounting for approximately 76.7% of the total participation in the IS workshop. Notably, the area has a longstanding history in the footwear sector. Furthermore, the industrial sector linked to footwear includes a diverse array of companies engaged in the processing and manufacturing of plastic/rubber materials, as well as the tanning and leather industries.

In Italy, the tanning sector comprises 1.154 companies as of 2022, with a total turnover of 4.2 billion euros (UNIC, 2021). A significant portion of the leather produced is primarily destined for the footwear industry, accounting for approximately 32.8% of its usage. This evolution is attributed to both the progress of materials utilized in footwear production, such as those for internal and external soles and the necessary co-evolution of key

industries within the footwear production chain.

2. Material and methods

ENEA approaches for the two project activities are outlined below. Firstly, the identification of CE competencies in the targeted territory involved combining surveys to gather field data. Secondly, leveraging ENEA's methodology for IS workshop organization and management, the effort to engage stakeholders within the region proved challenging yet significant.

2.1. Identification of roles and competencies in the Marche region

Combining surveys with other search methods is generally recognized as a valid opportunity to gather data. Researchers commonly adopt different approaches to gather and collect data for a specific purpose (Taherdoost, 2021) and different sources such as experiments, surveys, interviews, and questionnaires are normally used to achieve primary data, also in qualitative research (Kabir, 2016, Taherdoost, 2021). Moreover, specific "practical" approaches that also rely on a structured engagement with the target actors are adopted when the object of the (re)search (such as CE competencies intended to be mapped as reported in the literature (Ferreira and Matias, 2021), was not extensively examined by the literature or identified case studies.

The methodology developed by ENEA to identify roles and competencies related to the CE key areas in the Marche region, therefore followed a combined practical approach consisting of three working steps, , summarised in the methodological approach diagram in Fig. 1. The steps are as follows:

- Desk mapping, for preliminary identification of institutional actors and skills;
- Consultation of relevant/competent actors, to deepen the comprehension of roles and competencies;
- Summary of evidence/recommendations useful to support the proposal of an organizational model to systematize CE skills, based on the outcomes of the previous steps.

The methodological approach is summarized in Fig. 1. The desk mapping was aimed at defining the state-of-the-art skills and roles related to the CE key areas at the regional level and, therefore, it was aimed at providing the main information regarding the various relevant actors. An appropriate data collection sheet was created to properly store information characterizing the various subjects in terms of internal organization; institutional contacts (registered office, operational offices, telephone, email, website etc.); type of subject (e.g. organisation, agency, research centre etc.); relevant structures in the territory (offices, laboratories etc.); relevant skills for CE topics. A web search was therefore carried out, focussing on relevant public bodies (e.g. Region, University etc.) and using specific keywords related to CE topics, to define a list of reference "macro-structures" (MS).

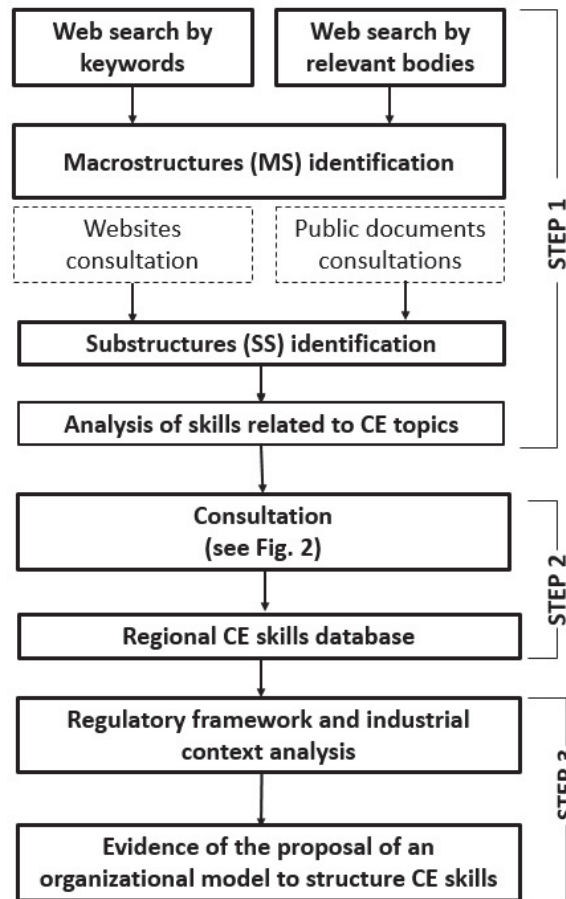


Fig 1. Methodological approach adopted by ENEA (source ENEA)

Subsequently, through the consultation of the websites (and their specific sections) and the freely available documents on the web regarding these MS (such as declaratory statements, organizational charts, etc.), a list of “sub-structures” (SS) was defined and classified according to the key CE topics (e.g. Production, Consumption, Waste Management, Secondary Raw Materials, ...). At the end of this phase, the first version of the CE skills database was developed, and the consultation of the relevant identified actors was carried out to detect in detail the skills of each one of them, thus integrating and deepening the first identification of CE skills developed during the desk mapping phase. The consultation was implemented through a survey tool specifically developed by ENEA following the process formulated by Peterson (Peterson, 2000), also adopted by other authors for CE skills identification (Sumter et al., 2021).

Such an approach was followed to realize an effective survey and, therefore, to carefully determine the types of questions to be asked, their specific wording and order based on the study’s objective, and using as much as possible a non-academic phrasing. When a research project requires information from an individual’s questionnaire construction is one of the most delicate and critical activities and it is fundamental to ask the “right questions”, i.e. the

questions able to provide valid and reliable information at the aim of the project itself.

For the sake of brevity, it is not possible to list all the questions of the survey, that were organized in specific sections to collect general information regarding the reference body, its structures in the territory and its peculiar skills regarding CE topics. Regarding CE skills detection, questions aimed to categorize them by CE key area (Design, Production, Consumption, Waste Management, Secondary Raw Materials, Innovation, Distribution/logistics, Financing/investments), also asking for information useful to characterize skills by type (political / programmatic, administrative, technical/ technological skills etc.). Other sections of the questionnaire were instead aimed at detecting synergies and collaborations between the different actors, to have a more in-depth understanding of the subjects themselves and their relationships with the territory and to identify any overlaps or gaps in skills.

The survey contained both open and closed-ended questions and it was developed in Google Forms to easily share it with the respondents and simply collect answers. To check the proper operation of the online form and the clarity/comprehensibility of the included questions, the survey was preliminarily tested through an internal small-scale pilot involving ENEA researchers. The sample of participants for the

consultation consisted of the relevant actors identified during the desk mapping phase, that were involved following the methodological approach illustrated in Fig. 2. In this working phase, the role of the ENEA office at the University of Camerino was fundamental to identify the contact person to be actively involved in the survey for each identified MS or SS. The responses to close-ended questions were analysed to obtain quantitative statistical data regarding the sample, while the answers to open-ended questions were coded and categorized to obtain clustered evidence from them. At the end of this step, the first version of the CE skills database was completed with detailed CE skills.

To support the proposal of an organizational model for structuring CE skills and supporting companies in undertaking CE actions and initiatives, moreover, two other relevant aspects have been explored, i.e. the regulatory framework, in terms of laws and regulatory measures relating to the CE, including regional guidelines for Ecologically Equipped Industrial Areas (EEIA), and the industrial context, in terms of willingness to collaborate through circular approaches and IS.

2.2. Establishing an IS pilot and organization of workshop

Since 2010, ENEA has developed an ecosystem of integrated tools to support companies in facilitating IS, which is a cooperative systemic approach for sharing and transferring resources (by-products, energy waste, services, skills and expertise) so that one company's waste can be used by another as a raw material. ENEA started the development and the implementation of an IS network model thanks to three projects in different Italian regions: the "Eco-Innovation Sicily" project (Cutaia et al., 2014,

2016a, b; La Monica, 2016; Luciano et al., 2016), the "Green Project - Industrial Symbiosis" in Emilia-Romagna (Cutaia et al., 2014, 2016a), and the "Industrial Park of Rieti-Cittaducale" project in Lazio (La Monica, 2016).

These experiences provided the basis for the design and implementation of the first Italian IS platform, the SYMBIOSIS® platform, aimed at companies and other operators in the territory to match demand and supply of resources and to activate transfers also through an expert connection system (Cutaia et al., 2015). The methodology developed by ENEA is based on three basic pillars:

- The language of symbiosis i.e., the identification of a shared language articulated in the Format Sheets (FS) for the collection of information, both company master data and input and output resources;

- Communication with companies, which aims to establish mutual, collaborative and continuous communication throughout the symbiosis implementation;

- Knowledge and experience, i.e., all the knowledge that allows ENEA to intercept synergies, not only those identified during the workshops but also those that to be realized need to include intermediate processes that allow the resources coming out of a production process to become suitable for reuse in a third production process. ENEA's IS platform is also part of this pillar.

The objective is to initiate a structured action of the Marche region on IS through the methodology. ENEA has managed the above activity by applying its methodology for the implementation of IS, which is based on a "horizontal" "network" approach and whose objective is to create synergies between supply and demand with respect to the resources made available or requested during the IS workshop.

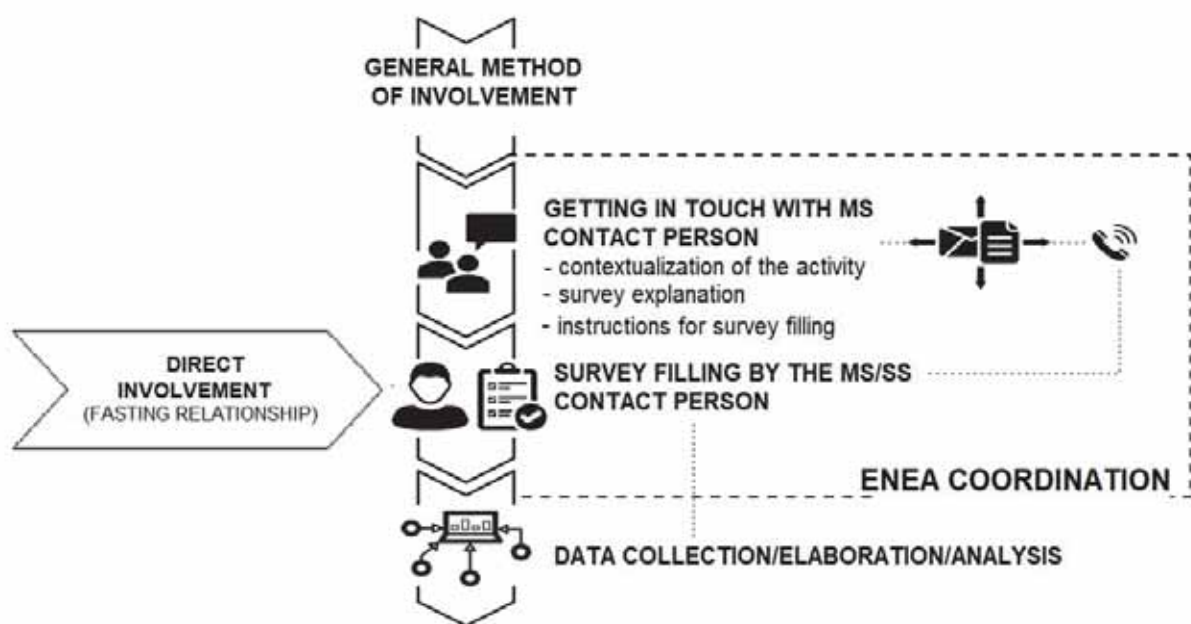


Fig. 2. Methodological approach for the consultation of relevant actors (source ENEA)

These activities include:

- *Involvement of actors on the ground:* The involvement of companies from different production sectors is one of the basic requirements to intercept potential synergies, even unseen ones;
- *Preparatory activity for IS workshop:* The organization of the workshop among the companies involved as fundamental moments of comparison, knowledge and exchange of information and data.
- *Workshop with collection of resources and identification of initial synergies:* A phase of elaboration and systematization of the collected data in which potential exchanges are identified;
- *Ex-post data processing,* uploading data into the SYMBIOSIS® platform to identify other potential exchanges;
- *Workshops with companies* for in-depth study of certain aspects and issues related to resources and processes;
- *Preparation of Operational Manuals* for one or more groups of synergies.

Following the IS workshop, ENEA catalogued, processed and standardized the data collected during the event. The first activity was to digitize the important amount of data collected and to catalogue them based on the model developed by ENEA during previous experiences. The resources, shared by the companies that took part in the event, were then indexed through the creation of a code that uniquely identified them. This code is made of a) a company code (exclusively determined), b) resources typology (INPUT/OUTPUT), and c) its sequential number (Eq. 1).

$$M000^{(a)} / YYYPUT^{(b)} / 000^{(c)} \quad (1)$$

3. Result and discussions

The outcomes of the project endeavours are highlighted below. The process of identifying roles and competencies has resulted in the identification of numerous pertinent stakeholders encompassing regional public administration, university entities, and business associations. ENEA spearheaded the IS workshop with support from local associations, held in Camerino. Thirty (30) local companies spanning diverse production sectors actively participated in the event. Distinct criteria were established for delineating various macro-categories of resource flows. Consequently, eight macro-categories were identified: plastic, paper, tissue, services, wood, skins, organic, and miscellaneous.

The activities carried out reflect the Marche Region's commitment to CE themes, also demonstrated through specific regulatory initiatives. Findings from the assessment, as well as discussions with companies and stakeholders during the IS pilot program, reveal that the business community is highly attentive to CE issues and is interested in the IS

mechanism, seeing it as an opportunity to improve resource efficiency.

Thus, there are clear opportunities for system-wide eco-innovation: regional authorities could advance these by promoting actions that facilitate IS, while other public or private actors might also capitalize on businesses' openness to adopting new business models, such as IS. The pilot program not only identified potential synergies among participating companies but also highlighted their strong inclination for collaboration - a crucial foundation for the effective application of CE strategies in general and IS in particular.

3.1. Identification of roles and competencies in the Marche region

The desk mapping phase allowed to identification of a total of 26 relevant/competent actors including MS (12) and SS (14), belonging to regional public administration and regional bodies (9), universities and research centres (6) and also to business associations (11), as shown in detail in Table 1. The consultation phase involved 18 actors among the 26 identified and, in particular 3 MS and/or SS belonging to regional public administration and regional bodies, 6 MS and/or SS belonging to universities and research centres and 9 MS and/or SS belonging to business associations, and a total of 12 survey responses were collected.

Table 1. Number and characteristics of the identified relevant actors (source ENEA)

<i>Type of subject</i>	<i>MS</i>	<i>SS</i>	<i>Total</i>
Regional PA	2		2
Regional Bodies	2	5	7
Universities	4		4
Research Centres	2		2
Business Associations	2	9	11
TOTAL	12	14	26

Figures 3 and 4 show some of the main general results obtained from the analysis of the collected responses. The strategic role of these key CE areas of expertise was evident, with strong external collaborations beyond the MS/SS. Ten out of the 12 involved actors responded to a question regarding awareness of similar structures/expertise on key CE topics, identifying Design, Waste Management, Innovation, and Financing/Investments as the primary areas of focus. The gathered information also highlighted that these collaborations led to significant positive results, including participation in international and national projects, clusters, and platforms, all of which are considered very valuable by the involved actors.

Furthermore, analysing the given suggestions on how collaborations between structures could be improved, it can be asserted that the identification and collection of the real needs of the various stakeholders

(regarding enterprises), as well as the sharing/coordination/aggregation of CE expertise, are considered key elements by the involved actors. On the other hand, it emerged a lack of skills regarding other CE key areas of expertise, such as Secondary raw materials and Training. Moreover, detailing the analysis of results, it is noteworthy that skills regarding Design, Consumption, and Waste Management are declared by the actors belonging to regional PA and regional bodies, while universities and research centres, in addition to these, also declared skills on Production, Consumption, Innovation and Financing/investments. Skills regarding Secondary raw materials are instead declared only by the actors belonging to business associations.

These results show a context that seems to be characterized by a lack of relevant skills on the key

aspect of resource efficiency (in particular, for institutional actors), in which broadening and improving such expertise appears particularly strategic to support the implementation of specific CE projects/activities. It is also worth noting that this evidence indirectly confirmed the soundness of the other project activity related to establishing an IS pilot/organizing specific workshop.

The review of the regulatory framework showed that the Marche Region legislated on CE consistently and coherently in the last five years, with 9 published regional laws having some specific reference to the CE scope. It is therefore quite clear the Region's intent to create a systematic skills model focused on CE and the regulatory field can be considered a strong driver to develop knowledge and skills in this priority area.

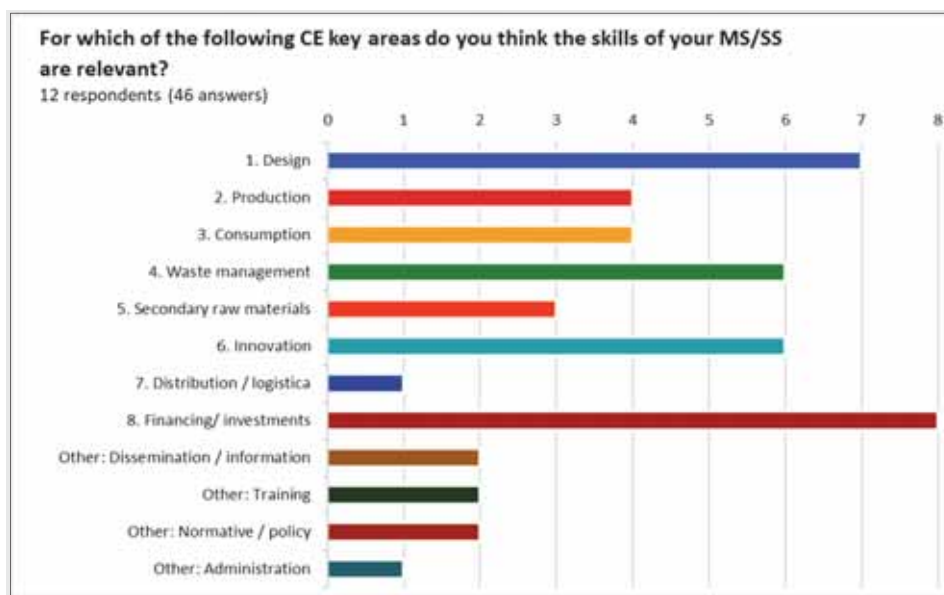


Fig. 3. Outcomes from returned questionaries’: CE skills classification(MS= macro-structures; SS=sub-structures) (source ENEA)

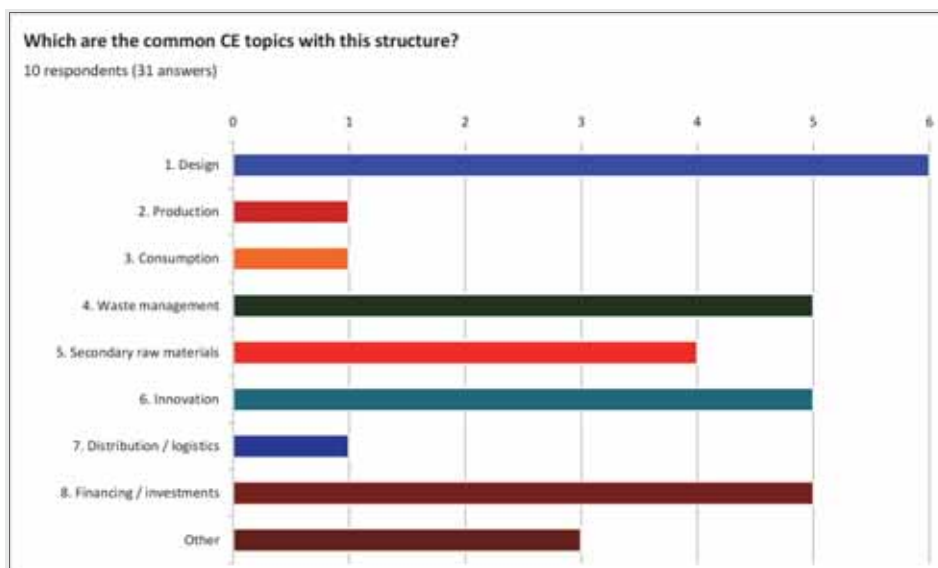


Fig. 4. Outcomes from returned questionaries: structures common CE skills (source ENEA)

In detail, the EEIA regional guidelines already mentioned and strongly requested an industrial model in line with the principles of CE, which should facilitate small and medium-sized enterprises (SMEs) in achieving an improvement in their environmental performances, through the provision of infrastructures and high-quality common services and allowing the control and reduction of the cumulative impacts generated by all SMEs.

The analysis of the regional industrial context identified four industrial clusters focused on the CE - Manufacturing, E-living, Agrifood and Furniture. These clusters play a strategic role in advancing projects and promoting clean processes and technologies within the regional economy. At the regional level, cultural barriers to CE seem partially overcome by companies' willingness to collaborate on various CE-related projects, particularly in IS.

3.2. Establishing an IS pilot and organization of workshop

The workshop was coordinated by ENEA, aided by local associations, and took place in Camerino. Thirty (30) local companies from various production sectors were actively involved in the event. Figure 5 illustrates the distribution of various production sectors, categorized by the ATECO code of the companies. Additionally, the macro-sectors of the companies were identified: companies in the "manufacturing sectors" are depicted in blue, those in the "production and distribution of water, sewerage, and waste management" category are represented in yellow, companies in the "services of communication" and "agriculture, hunting, and forestry" sectors are shown in red and green respectively, and companies in the "legal activities and consultancy" sector are highlighted in orange. Figure 5 highlights a predominant presence of companies in the manufacturing sector (76.7%), with a notable concentration in the "manufacture of rubber/plastic material articles" (6 companies, accounting for 20%). Furthermore, an analysis of the size distribution of participating companies was conducted based on current reference legislation (MISE, 2005), revealing a relatively even distribution across size categories, with a slight majority of large companies (30%), followed by micro (26.7%) and small firms (23.3%).

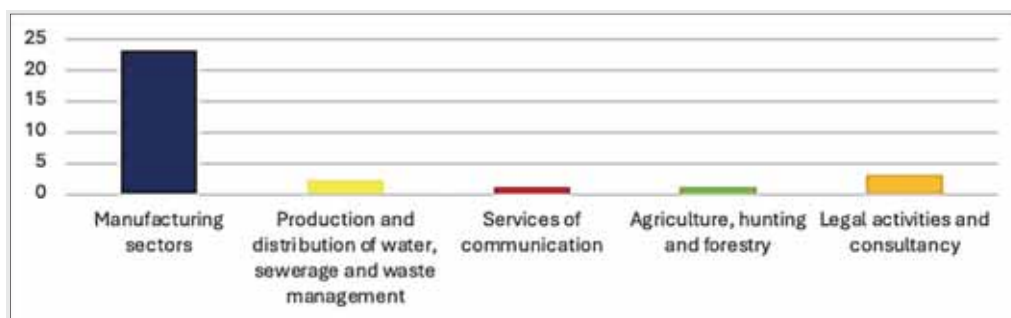


Fig. 5. Production sectors of the thirty companies that participated in the IS workshop (source ENEA)

Figure 6 shows the geographic distribution of the companies attending the IS workshop, according to the ATECO classification of economic activities. The data obtained from the workshop underwent post-processing, and, with more than 300 shared resources (as outlined in the next table) the outcomes show a total of 85 matches identified. Companies shared mainly material resources (88% in number), then services & skills (11%) and lastly energy (1%).

Specific criteria were defined for the creation of different macro-categories of resource flows. The following 8 macro-categories were therefore identified: plastic, paper, tissue, services, wood, skins, organic, and other. Based on this classification into macro-categories, those with the highest number of shared resources and synergies were pinpointed. Specifically, the "leather," "paper/cardboard," and "plastic/rubber" sectors garnered the most attention from both an economic and quantitative perspective. Consequently, a detailed analysis was conducted on these sectors, resulting in the identification of three distinct flow diagrams (Figures 7-9). The continuous line represents synergies from the IS workshop, while the dash-dot line depicts those from ENEA's data post-processing. These diagrams contain information about the companies engaged in potential synergies.

The IS workshop focused on potential synergies involving Ethylene-vinyl acetate (EVA) polymer. EVA, a thermoplastic commonly used in the footwear industry, is essential for crafting outsoles and midsoles known for their excellent shock absorption. During manufacturing, EVA is injected into heated molds to shape these components. In Fig. 7, the EVA waste resource is denoted by the unique identification code M003/OUTPUT/001 within the flow diagram about plastic/rubber materials. The annual quantity of this material amounts to approximately 94,000 kilograms. Such quantities underscore the potential for reusing or recycling the material, suggesting a potentially beneficial pathway due to the substantial volume involved. To guarantee adequate polymer flow within the mould, gates and runners (channels through which the plastic flows from the injection moulding machine into the part cavity) are essential. Their dimensions may vary based on the size and shape of the parts to be produced, but they can represent up to 20% of the total weight of the midsole (Bianchi et al., 2023).

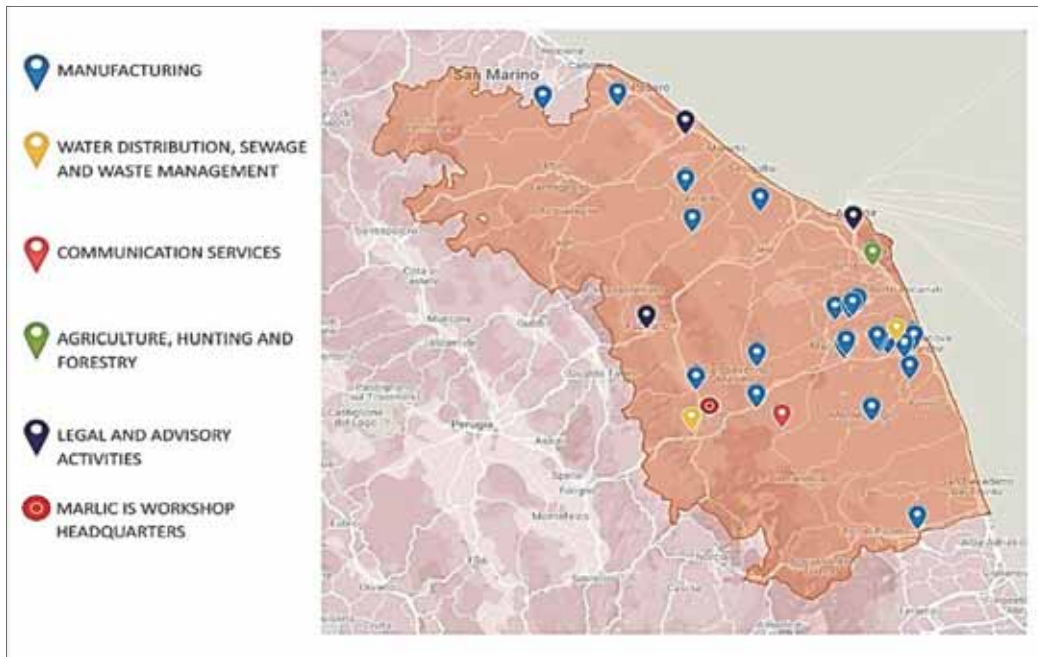


Fig. 6. Location and type of companies participating in the MARLIC IS workshop (Source ENEA)

Table 2. List of resources emerged during the IS workshop (source ENEA)

Competence/Service	34
Competence/Service	34
Materials	280
water	7
carbon	2
paper and cardboard	29
chemicals	19
composites	3
sludge	5
rubber	7
inert	13
wood	24
metals	17
mixed	16
oil	6
organic	21
plastics	67
RAEE	7
textile	33
toner	1
glass	3
Energy by-product	3
energy	3
Total	317

Currently, there is no concrete evidence that a real industrial process can recover and reutilize EVA scraps/waste. The footwear annual production is, in 2022, equal to 23.9 billion pairs, most of which contain EVA soles (World Footwear Yearbook, 2023). It is hence evident the importance of the recycling and reuse of materials from footwear production (Muthu and Li, 2021). In scientific literature, numerous studies have been conducted to assess the viability of alternative recovery systems for EVA waste. For instance, Lopes et al. (2015) utilized EVA waste as a filler for natural rubber, styrene-

butadiene rubber, and acrylonitrile-butadiene rubber in compression while Pavia Junior et al. (2021) evaluated the recovery of EVA micronized and added to the virgin material. The outcomes that emerged from the workshop in the Marche region show evidence of the possible synergistic integration of EVA scraps/waste in a virtuous loop of recycling. The evaluation with industry experts has underscored the potential of technologies already capable of facilitating the reuse of this material within the existing production cycle, requiring only minimal technical adjustments to the process.

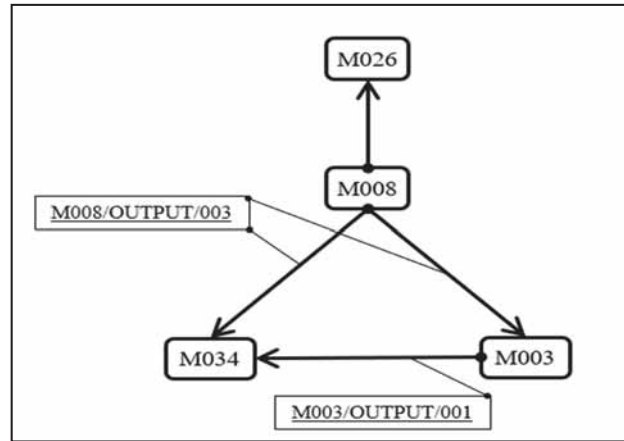


Fig. 7. Flow diagrams related to plastic/rubber flow material (source ENEA)

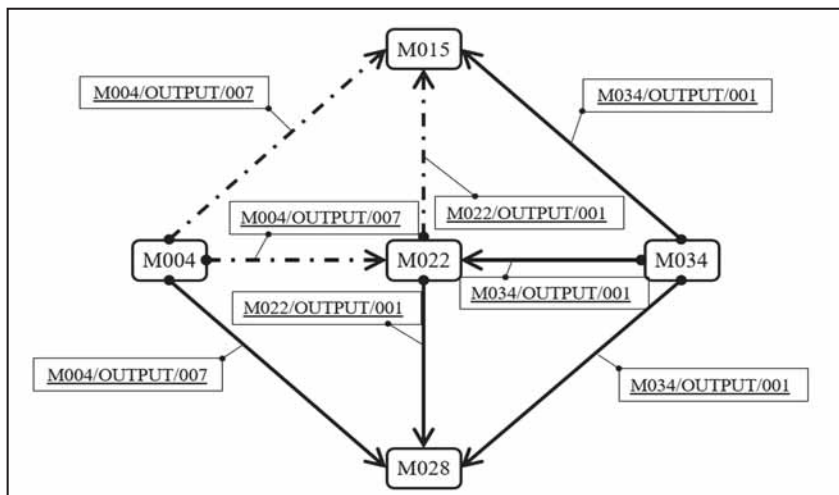


Fig. 8. Flow diagrams related to leather flow material (source ENEA)

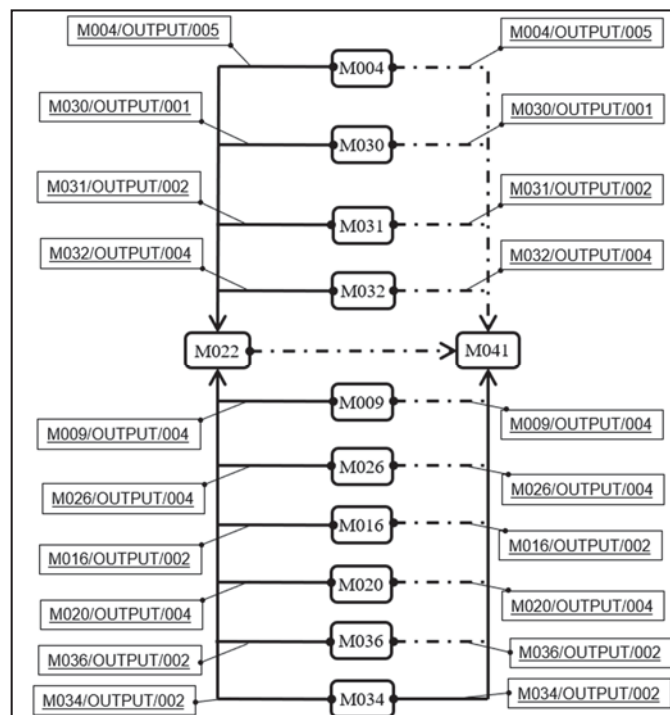


Fig. 9. Flow diagrams related to paper/cardboard flow material (source ENEA)

This material is currently disposed of in large quantities due to high production rates. However, ENEA's consultations with local stakeholders and companies reveal a tangible opportunity for recycling under specific technical conditions. Furthermore, a feasible recycling pathway already exists, even though market demands often favour the use of virgin materials due to technical constraints and brand motivations. Midsoles made from partially recycled EVA material may exhibit slightly lower performance compared to those made from entirely virgin material. However, cited studies have confirmed the possibility of effectively utilizing this waste to manufacture footwear components with mechanical performance comparable to virgin ones (Lopes et al, 2015, Paiva Junior et al., 2021).

The identified symbiosis scenarios offer promising economic benefits and enhanced resource efficiency for participating companies. They also provide opportunities for the region to leverage economies of scale, reducing both resource consumption and waste disposal.

The interest of companies and stakeholders in this initiative is demonstrated by the feedback received from IS experience. Most of the participants, whose opinions on the activity were collected through an evaluation questionnaire, rated the workshop as a good opportunity to establish a network of cooperation and information exchange between companies in the region, as well as an opportunity to get in touch with other companies in the area and their different needs.

4. Conclusions

The MARLIC project involved two parallel activities that dealt with the implementation of the CE in the Marche region. In particular, the first concern was to recognize the skills and related governance that exist in the region. The second aim was to demonstrate the effectiveness of the IS approach and the interest of the production system in its implementation through the implementation of a pilot activity.

The first activity focused on mapping skills and governance related to the CE in the Marche Region, indicating that the subject is addressed from various angles. Moreover, it is dispersed across different areas of governance and lacks integration within a dedicated structure. Conversely, discussions during events such as the IS meetings and engagements with local stakeholders underscore a significant interest in embracing CE solutions, including symbiosis.

Therefore, to promote and simplify the transition toward a "circular region" paradigm, it would be advantageous to establish a governance model (the specifics of which, in terms of functions, stakeholders involved, funding etc. represent a future development of the research here presented) that consolidates and, if possible, harmonizes the diverse functions and expertise of the CE. This model, aligned with the principles outlined in earlier regulatory documents like the EEIA regional guidelines, should

also streamline the procedures for launching businesses in the production sector. Additionally, it should engage companies in an ongoing enhancement of environmental performance and encourage a trajectory toward environmental responsibility for both individual companies and the broader economic system.

The results from the first phase of activities have been discussed and made available to interested public and private stakeholders. These findings could serve as a basis for reorganizing and refining skills related to the CE, to enhance their organizational effectiveness and address any gaps in integration. As previously noted, at the time of the analysis, there was no clear reference to IS, either in terms of competencies or in the presence of bodies to facilitate and support it at the regional level. However, the IS workshop revealed significant interest from local companies in this approach, despite the absence of specific regional policies and organizational actions. It is therefore desirable that public authorities and relevant stakeholders build on this analysis to encourage a broad and systematic adoption of IS across the region.

In the IS workshop, organised on October 2022 by ENEA with the collaboration of University of Camerino, Camera di Commercio delle Marche, and Confindustria Macerata, about 30 companies, coming from several industrial sectors, shared 220 output and 97 input (88% as materic flows, 11% as skills and services, and 1% energy). During the IS workshop, over 85 potential synergies were identified. Eight macro-categories (namely plastic, paper, tissue, services, wood, skins, organic, and others) were identified for materic flows. Specific focus, with flow diagrams, was then realised for matches regarding scraps of leather, paper/cardboard, and plastic/rubber.

It should be emphasized that the identified synergies are potential; however, all involved companies have been provided with the necessary information to mutually activate and implement the operational, organizational, and commercial agreements needed to bring these synergies to life. The decision to proceed rests with the independent initiative and confidentiality of each company involved, in consideration of the boundaries of authority held by the research organization that conducted the workshop.

However, given the importance of the footwear industry in the Marche Region, which includes 1099 companies (representing 30.8% of the total on a national basis) and 16961 employees (23 % of the total), with a business export volume of 12.7 billion euros, the opportunity was taken to explore further a specific synergy that emerged. This synergy would enable significant regional-scale advantages, as the EVA flow under review can facilitate multiple collaborations among the companies involved. Recovering production waste and EVA from unsold footwear can create a circular system within the same production chain.

Thus, particular attention was for the last, given the potential of extensive use of EVA polymer in footwear production processes, particularly due to the significant presence of this industrial sector in the Marche region, as well as an estimated huge amount of such a kind of waste materials, seems to be an interesting CE solution (enabling the closing of the cycle in the same sector). The synergy identified, in particular, involved 4 companies, resulting in an EVA flow of 94000 kilograms per year. Despite the strong interest in this strategic supply chain for the region, to date, even if technologies for reusing that waste are potentially available, the related market, and thus the potential of IS, is un-exploited, showing, therefore, a significant potential that can be harnessed, both in terms of efficiency improvement and in system-wide eco-innovation.

The literature analysis confirms the potential for reusing EVA scraps, although current technologies do not guarantee that recycled products will match the technical characteristics of new ones. Discussions with stakeholders at the IS workshop identified key bottlenecks hindering the practical development of an EVA scrap recycling pathway. The strategy must be multifaceted - technical, regulatory, and market-driven, focusing on consumer engagement campaigns around products made from recycled EVA material, resolving outstanding technological gaps, and addressing legislative inconsistencies concerning recycled EVA use.

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STRUCTURAL AND ENVIRONMENTAL ASSESSMENT OF HIGH-PERFORMANCE ASPHALT MIXES WITH A HIGH RECLAIMED ASPHALT PAVEMENT CONTENT: A CASE STUDY

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Abstract

Nowadays, attention in the construction sector is increasingly turning towards the concept of sustainability, efficiency and durability to reduce atmosphere emissions. The A4 Turin-Milan motorway is pushing in this direction. In particular, from the pavement point of view, one of the most important innovations introduced by the road manager is the study of bituminous mix with about 70% of reclaimed asphalt pavement (RAP); this methodology is today possible thanks to the use of rejuvenating agents, the implementation of next-generation asphalt plants, and the introduction of a graphene-enhanced polymeric compound (GPC), through which it is possible to obtain high-performance asphalt concrete (up to +75% of service life) with a consequent reduction in future maintenance if compared to the existing pavement. The new technology was experimented first in the construction of a 1 km long trial section at the end of 2022. With the aim of validating the Life Cycle Assessment (LCA) and Life Cycle Cost (LCC) analyses prior to the production of the mixtures, laboratory and field tests were carried out specifically aimed at assessing the structural characteristics of the mixtures. Mixtures used were previously tested in the laboratory and showed an increase in the average stiffness and strength values at 20 °C and 25 °C up to 50% compared to average values of traditional mixes. From the time of construction, the trial section is being monitored visually as well as by Falling Weight Deflectometer (FWD) tests. The results obtained in the first 500 days of life seem to be promising compared to the assumptions made for the LCA and LCC analyses (10% less deflection when compared to materials traditionally used by the road operator). In fact, a very stable and effective behaviour of the new pavement under relevant traffic loads could be recognized.

Key words: asphalt concrete, graphene-enhanced polymeric compound, service life, sustainability, rejuvenator

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1. Introduction

The transport sector has grown strongly in recent decades, and this has led to a significant impact on the environment (Yang et al., 2015). Several countries around the world are pushing for policies that urge the replacement of the linear economy model with a circular economy model (Mantalovas and Di Mino, 2020). These directives have also been implemented in the field of road construction. In order to drastically reduce atmospheric emissions and

achieve climate neutrality by 2050, the two strategies promoted by the European Union are the use of recycled material and the extension of the service life of pavements (MIMS, 2022). Specifically, materials must be carefully selected to take into account both performance and economic, social and environmental aspects (Aurangzeb et al., 2014).

Nowadays, to assess the environmental and economic impact of a technology, or simply to compare two different solutions with each other, there are two different tools, namely Life Cycle Assessment

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(LCA) and Life Cycle Cost Costing (LCC) (Salehi et al., 2021).

The use of by-products and/or waste has the dual benefit in reducing environmental impact, in terms of reduced space in the landfill, and limiting the use of raw materials from both renewable and non-renewable resources (Andersen, 2007; Yaro et al., 2023). An example of circular economy application is the use of RAP for the construction of new asphalt concrete mixes (da Costa et al., 2024). However, in some countries around the world, RAP is not seen as a valuable resource and therefore national regulations severely restrict their use in road pavements (Jaawani et al., 2021). RAP consists of aggregates coated with aged bitumen (which has undergone oxidative processes over time), which when used in high quantities, if not properly treated with regenerating products, leads to a drastic increase in the stiffness of the final bituminous mix, resulting in premature cracking (Prosperi and Bocci, 2021). There are various types of products on the market for using RAP, however it is important to emphasise that not all of them are capable of restoring the chemical properties of RAP aged bitumen (Abe et al., 2023). Among them, we can distinguish between softening agents, which simply reduce the viscosity and stiffness of aged bitumen (Ismael and Khaled, 2018), and regenerating agents, that restore the physical-chemical properties of aged bitumen (Loise et al., 2021). Thanks to the latter and the advent of increasingly advanced asphalt plants, it is possible to recycle large quantities of RAP up to 60-70% (Lee et al., 2015; Lu et al., 2019; Pasetto et al., 2021; Porot et al., 2017).

Another possibility for sustainability is to increase the service life of pavements using high-performance materials. The reduced environmental impact is due to the minimised use of materials and energy during the life cycles (PIARC, 2019). There are numerous studies by the scientific community on high-performance materials (Huang et al., 2023; Li et al., 2017; Ma et al., 2016; Mi et al., 2022; Moreno-Navarro et al., 2016; Ranieri and Celauro, 2018). Noteworthy among them are asphalt mixes modified with graphene-based compounds (Asim et al., 2022). These special compounds improve asphalt performance by increasing the rutting resistance (Hafeez et al., 2019), the fatigue resistance (Adnan et al., 2021), the fracture toughness and fracture energy (Wang et al., 2018), and reducing the temperature sensitivity (Hafeez et al., 2019) and the ageing susceptibility (Li et al., 2021a; Li et al., 2021b; Wu et al., 2017). In the last years, a special compound of graphene and selected recycled plastics has been introduced. These compounds allow both to improve the performance of the asphalt mix, but at the same time to reduce the environmental impact thanks to the reuse of plastics normally destined for waste-to-energy plant (Russo et al., 2022). Being a dry modification method, they are added directly into the mix and not through the bitumen (wet modification method), so that highly modified mixes can be obtained even when high quantities of RAP and

consequently low quantities of raw bitumen are used (Bruno et al., 2024). They have already been used in several important applications such as bridges, high traffic roads, motorways and airports showing excellent potential (Meroni et al., 2022; Moretti et al., 2021; Russo et al., 2022; Venturini et al., 2022).

In the last decade, the increase in vehicle traffic, technological development and the introduction of self-driving vehicles have led to roads being thought of not only as a means of carrying loads, but rather as a means of communication and information exchange, if not even as a source of energy (Smart Roads) (Trubia et al., 2020). Among the systems that can be used in a smart road are those that allow real-time monitoring of vehicular traffic, analysis of user behaviour, electronic payment, charging of electric vehicles, monitoring of pavement condition through Building Information Modelling (BIM) models, self-driving vehicle movement, etc (Bosurgi et al., 2019; Finogeev et al., 2019; Pompigna and Mauro, 2022; Trubia et al., 2020).

This paper presents the results of LCA and LCC analysis of asphalt mixes modified with GPC and high percentages of RAP (approximately 70%) suitably designed to realize the asphalt concrete (AC) of the A4 Turin-Milan motorway (one of the busiest roads in the northern Italy), at the end of the service life of the existing pavements. The investigation was conducted by comparing the proposed technology (identified by the code GPC) with those traditionally used on motorways, i.e. a conventional non-modified AC (marked with the code NM) and a polymer modified bitumen (with Styrene Butadiene Styrene, SBS)AC (code: PMB). The estimation of the service life of the different technologies was carried out using the rational design method, employing the results of laboratory tests obtained with respect to previous applications on other reference test sites. The design hypotheses were then verified in full scale with both laboratory tests and in situ road pavement surface monitoring, with the aim of validating the assumptions made for the LCA and LCC analyses.

2. Site description

The trial section was 1 km long. The work was carried out in the month of October, 2022, and the new technology (GPC) was laid only on the slow lane of the carriageway in the direction of Milan, which is the most trafficked by heavy vehicles (Fig. 1). In detail, the new pavement was spread out over the existing 20 cm thick subbase layer, which was made of reclaimed asphalt and at the moment of the work appeared in very good conditions (Fig. 2), and was composed by the following AC layers:

- wearing course (4 cm) in SBS-modified porous AC;
- binder course (6 cm) in AC modified with GPC and 66.6% of RAP;
- base course (10 cm) in AC modified with GPC and 67.0% of RAP.

Figure 3 shows the paving stage of the proposed asphalt concrete.

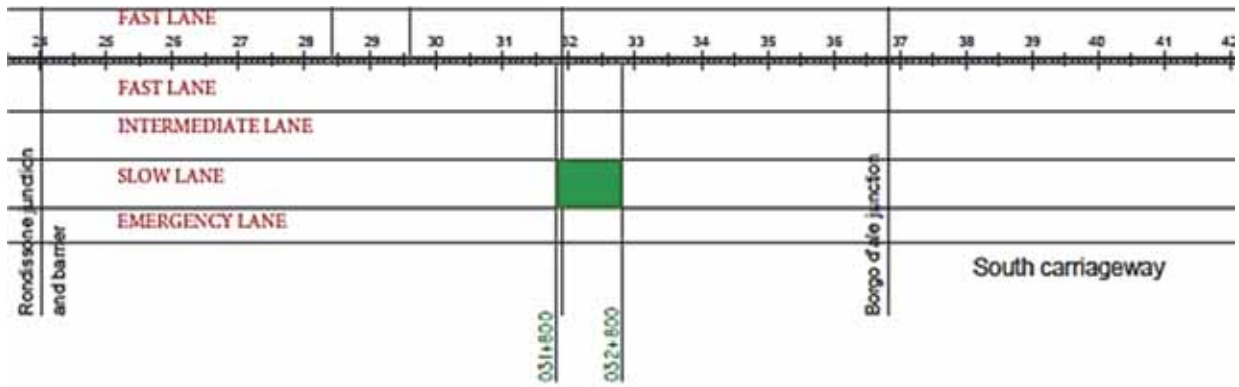


Fig. 1. Trial section layout



Fig. 2. Exiting pavement detail after the milling operation



Fig. 3. Paving stage of GPC modified AC

3. Materials

RAP aggregates were sampled at the mix plant and characterized in terms of gradation (EN 933-1) and bitumen content (EN 12697-1) in accordance to the European standard. Raw and RAP aggregates were then adequately proportioned in order to fit the grading envelope in accordance with the standards

provided by one of the most important road management authorities in Italy (ANAS, 2021).

Two different types of RAP aggregates were used, with a maximum size of 12 mm and 20 mm, and a bitumen content of 3.68% and 2.45% respectively. Due to the large amount of RAP used, it was necessary to incorporate a rejuvenating agent capable of restoring the chemical properties of the RAP aged

bitumen. In particular, an additive consisting of a mixture of vegetable derivatives was used at a quantity of 0.1% by weight of RAP. The characteristics of the additive are shown in the Table 1. This type of additive was selected based on the investigation on the preliminary given properties of the additives available in the market and the potential of being used in future project. Instead, no RAP material was included for the wearing layer. It is worth noting that a new generation asphalt plant was used for the production of the mixes, which is capable of handling very high percentages of RAP and at the same time allows high hourly production rate (Santolini et al., 2024).

Table 1. Properties of the rejuvenator used in this research

Typical properties	Unit	Value
Appearance	-	liquid
Colour	-	dark brown - purple
Density at 25°C	g/cm ³	0.85-0.95
Viscosity at 25°C	mPa·s	50-150
Flash point	°C	≥ 200
Pour point	°C	≤ 0

The virgin binders used in the present study were a 50/70 bitumen for binder and base layer (with a content of 4.65% and 4.01% respectively) and an SBS-polymer modified bitumen for the wearing layer (5.0%). Lastly, a GPC composed by graphene nanoplatelets and selected recycled hard plastic (Fig. 4) was used as AC modifying agent of the binder and base layers (6% by weight of bitumen). The physical properties of the GPC additive are shown in Table 2. Further information on the chemical properties of this product is available in the experimental study by D'Angelo et al. (2022).



Fig. 4. Graphene-enhanced polymeric compound

Table 2. Properties of the GPC additive

Typical properties	Unit	Value
Appearance	-	granules
Colour	-	black
Apparent density at 25°C	g/cm ³	0,4-0,6
Softening point	°C	160-180
Melt Flow Index at 190°C/5kg	g/10'	4-10

4. Test methods

4.1. LCA and LCC analyses

The LCA analysis was conducted according to a “cradle-to-grave” approach in compliance with the European regulations (UNI EN ISO 14040, 2021; UNI EN ISO 14044, 2021). The methodology consists in four interconnected phases: goal and scope, inventory analysis, impact assessment and interpretation. The LCC analysis has also been carried out in parallel to assess the economical sustainability of the two solutions.

In particular, the proposed solution (see Chapter 2) was compared with a solution normally used on motorways, and consisting of open-graded surface course, binder course and base course in SBS-polymer modified asphalt concrete (with no RAP content for surface course and 30% for binder and base courses).

Figure 5 shows the system boundary considered to perform the analysis. In particular, in this system, recycled material (RAP) was considered as an external product that would otherwise be landfilled in its original life cycle (“avoided waste”). The analysis was carried out considering a reference service life of 35 years and a number of resurfacings for each individual layer shown in Fig. 5.

Moreover, the two different solutions were also investigated and compared to highlight the effective environmental advantages of the use of high reclaimed asphalt percentages and high-performance technologies capable of increasing the service life of the pavement. The environmental analysis was conducted using the OpenLCA software and the Ecoinvent v3.7.1 database. Table 3 shows primary data on consumption and costs of raw materials and production processes taken from surveys carried out by the road operator and asphalt production plants, as well as from the OpenLCA software database. The analysis were performed considering the production of SBS-modify asphalt concrete (with 30% of RAP) and GPC asphalt concrete (with about 70% of RAP) in two different production plants since the incorporation of about 70% of RAP requires the use of a new generation asphalt plant that is technologically advanced. The production of traditional hot mix with 30% of RAP can be produced in a traditional asphalt plant (Nicolini et al., 2023). Furthermore, it is important to note that the contribution of the chemical additives in the life cycle of the pavement, even if these are used in small quantities, has been evaluated as an increase, albeit minimal, in the amount of virgin bitumen entering the system (hypothesis). It has not been possible to consider the exact contribution of the products constituting the regenerating agent because the exact chemical nature of these products is the intellectual property of the manufacturer.

A section of road 1 km in length, 4 metres in width and 20 cm in thickness was considered to perform the analysis, taking a reference period of 35 years: this corresponds to the best theoretical

estimation of the service life for the GPC-based pavement technology, under the traffic loads expected by the Road Manager, following the implementation of Miner’s law on the base of the stress-strain calculations that were performed with the help of a computational software (Fig. 6). This service life was estimated during the design phase using an empirical-rational method, based on a number of variables whose knowledge was possible thanks to the contribute of the Road Manager and to the availability of historical information in Sina’s databases. The

mechanical properties of the subbase, foundation and subgrade layers were derived from in-situ investigations having been realized from the birth of the motorway, while the expected performance of the asphalt mixes was derived from laboratory mix pre-qualification tests and the outcomes documented with respect to previous applications on other reference test sites. Results were reported in terms of kilograms of carbon dioxide equivalent (CO_{2equ}), energy consumption (kW/h), raw material consumption (kg), and reuse of recycled plastics (t).

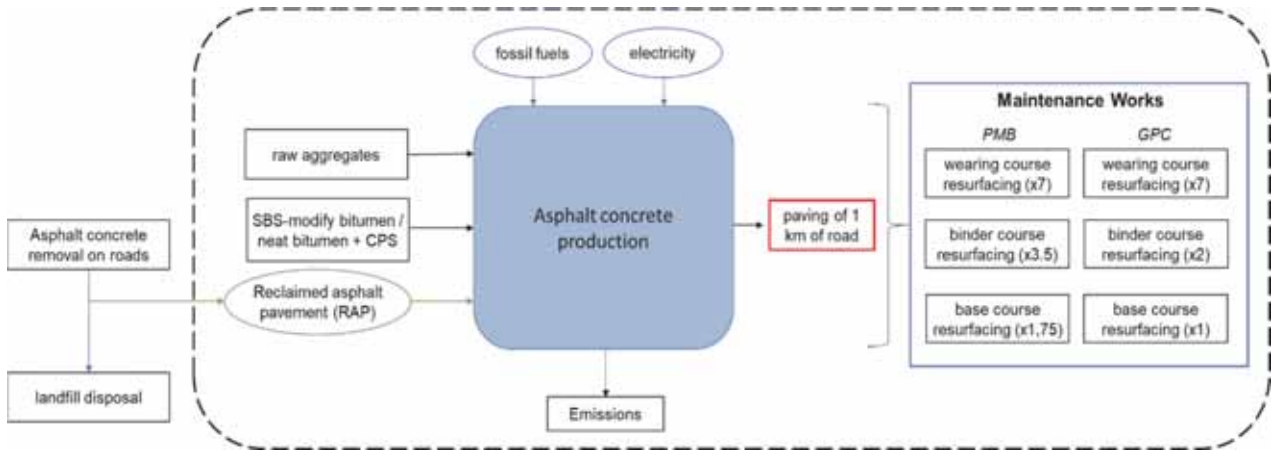


Fig. 5. System boundary considered for the analysis

Table 3. Input data for LCA

Input	Type of asphalt plant	
	Discontinuous	New generation
Type of fuel	Methane*	methane
Fossil fuel consumption [m3/t]	3.5*	8.0
Production [t/h]	120*	240*
Power consumption [kWh/t]	1.7*	1.7
Virgin aggregate transport distance [km]	100*	100*
Bitumen transport distance [km]	100*	100*
RAP transport distance [km]	65*	65*
Aggregate cost [€/t]		11*
Modified bitumen cost [€/t]		550
Methane cost [€/kW h]		0.106
Electricity cost [€/kW h]		0.0624
Transport costs [€/t km]		0.425
Asphalt concrete disposal cost [€/t]		10*

*data provided by the road operator

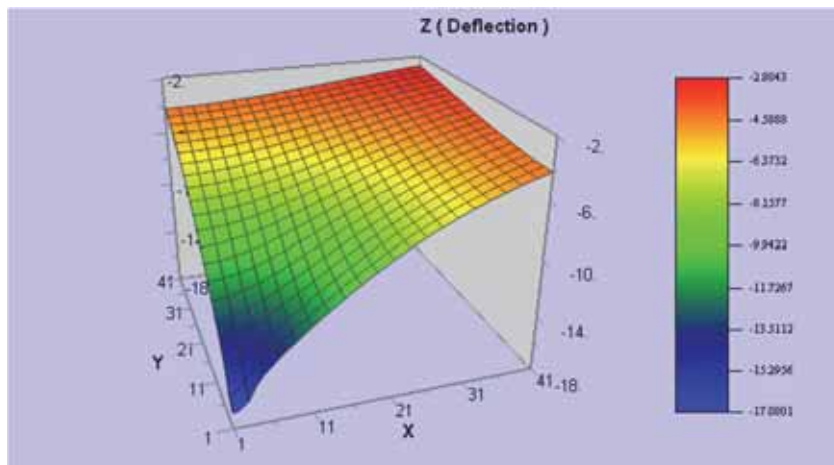


Fig. 6. ALIZE-LCPC software output

4.2. Laboratory tests

In order to check the quality of the mixes laid in the test field and to confirm the assumptions at the basis of the pavement design, material samples were taken in situ. The results were then used to verify LCA and LCC analyses. Subsequently, specimens were produced using a gyratory compactor equipment with up to 200 rotations (EN 12697-31) and through roller compaction (EN 12697-33). The following parameters have been determined on the AC specimens: volumetric properties, stiffness, indirect tensile strength, water sensitivity, resistance to permanent deformation, tendency to crack propagation at low temperatures and fatigue resistance.

4.2.1. Indirect Tensile Strength Modulus (ITSM) test

The Indirect Tensile Strength Modulus is used to evaluate the elastic properties of ACs in different climatic condition (Iwański, 2020). Moreover, it is considered a very important performance parameter in pavement design (Bocci et al., 2020). The tests were carried out in according to the standard (EN 12697-26). In particular, the standard defines the ITSM parameter (in MPa) as follow (Eq.1):

$$ITSM = \frac{F \cdot (v+0.27)}{(z+h)} \quad (1)$$

where F (N) is the applied vertical load, z (mm) is the amplitude of the horizontal deformation obtained during the load cycle, h (mm) is the mean thickness of the test sample, and v is the Poisson's ratio.

The test was performed at three different temperatures (10 °C, 20 °C, and 30 °C) in control-horizontal displacement configuration. A different horizontal deformation was applied during the test as the temperature changed to avoid damage to the material. Specifically, 5 µm was chosen at 10 °C, 7 µm at 20 °C, and 9 µm at 30 °C.

4.2.2. Indirect Tensile Strength (ITS) test

The mechanical resistance was determined in terms of indirect tensile strength (ITS), according to the EN 12697-23 (2017). The test was conducted at 25 °C with a constant deformation rate of 50±2 mm/min until the load, after failure, reaches 30% of the maximum load. The specimens produced for this test were compacted at 120 cycles.

4.2.3. Water sensitivity of bituminous specimens

The water sensitivity of bituminous specimens (A method) is defined through the indirect tensile strength ratio (ITSR), which as defined by the EN 12697-12 (2018) is calculated by the following equation (Eq. 2):

$$ITSR = 100x \frac{ITS_w}{ITS_d} \quad (2)$$

where: ITSR is the indirect tensile strength ratio, in percentage (%); ITS_w is the average indirect tensile

strength of the wet specimens group, in kPa (at 15°C); ITS_d is the average indirect tensile strength of the dry specimens group, in kPa (at 15°C). The specimens produced for this test were compacted at 120 cycles.

4.2.4. Resistance to permanent deformation

The resistance to permanent deformation was assessed by means of Wheel-Tracking Device (WTD). The test was performed following the B method (small size) indications of the EN 12697-22 (2020) standard. This device simulates the pavement distresses by rolling a wheel (10000 cycles) across a sample of AC under specific temperature condition (60°C). Specifically, the slabs were compacted through roller compactor (EN 12697-33). Two different test repetitions were performed (in dry condition). The wheel-tracking slope (WTS), in mm per 10³ load cycles, was calculated as (Eq. 3):

$$WTS = \frac{(d_{10000} - d_{5000})}{5} \quad (3)$$

where d_{5000} and d_{10000} are the rut depth after 5000 and 10000 load cycles respectively (in millimetres).

Moreover, the rut depth (RD) after 10000 cycles (in mm) and the proportional rut depth (PRD) after 10000 cycles (in percentage) were determined.

4.2.5 Semi-circular bending test (SCB)

Low temperature crack propagation tendency was assessed by means of the SCB test (EN 12697-44) at 0°C. By means of an elettro-mechanical press, a constant rate of deformation of 5.0±0.2 mm/min was applied on the samples until the crack propagated from the notch to the edge of the samples.

The fracture toughness (K) was calculated by the Eq. (4):

$$K \left[\frac{N}{mm^{1.5}} \right] = \sigma_{max} \cdot Y \cdot \sqrt{\pi \cdot a} \quad (4)$$

where: σ_{max} is the maximum failure stress of the specimen; Y is the effort intensification factor in normalized mode; a is the cutting depth of the specimen (in millimeters).

4.2.5 Indirect Tensile Fatigue (ITF) test

The determination of a material's fatigue resistance is fundamental to the evaluation of a pavement's durability in the field and to design the road structural package (Bruno et al., 2024; Wen and Bhusal, 2013). In this research, the resistance to fatigue was analysed in indirect tensile configuration (EN 12697-24). Different horizontal stress amplitudes (750, 1000 and 1250 kPa) were set and a temperature of 10°C was fixed to obtain an initial horizontal deformation ε_0 between 70 and 400 µm/m and a fatigue life between one hundred and one million cycles. The load frequency was 2 Hz. The fatigue failure of the different samples (N_f) was assumed in correspondence to the number of cycle when the specimen broke. Test results were plotted in a log-log

scale graphs, as function of the horizontal strain measured on the undamaged specimen at the beginning of the test.

4.3. In situ road pavement surface monitoring

Non-destructive in-situ testing of pavement condition is a very effective tool for checking the state of the road and for predicting the remaining service life without damaging the pavement or slowing down traffic (Liand Wang, 2018; Vyas et al., 2021).

The investigation of pavement structural conditions was based on the use of the FWD device (Fig. 7), whose first application on the interested site is dated many years before the realization of the new pavement. The FWD testing protocol provided for a loading plate with 300 mm diameter, an impact load of 120 kN and nine geophones positioned at 0, 200, 300, 450, 600, 900, 1200, 1500 and 1800 mm from the centre of the loading plate.



Fig. 7. FWD device

The assessment of the bearing capacity of the existing pavements (both as a whole and with respect to the subbase layer only) was propedeutical to the identification of the requirements of the new GPC-based technology to be applied, and to the design of the proposed solution. Its repetition during the work, instead, supported operators to recognize the suitability of the selected thicknesses and materials, while the continuation of FWD testing over time in the future, at last, has to be identified as the monitoring action that will help the Road Manager understanding the real performance of the applied solution, compared to the one that was considered in the design phase.

5. Design evaluations and results after almost a two years application under traffic

5.1. Life Cycle Assessment (LCA)

Based on the LCA methodology described previously (section 4.1) the potential environmental impacts were calculated. Fig. 8 presents the results for the two different technologies (GPC vs PMB). The LCA analysis highlighted the positive environmental contribution of a larger use of RAP aggregates and a

neat bitumen with an innovative GPC compound in the binder and base layers, rather than using the traditional mixes. The use of the GPC mixture allowed a reduction of 38.5% in CO_{2-eq}, 30% in energy consumption, 38% in raw bitumen, 40% in virgin aggregates, and the use of 4.9 tons of recycled plastics.

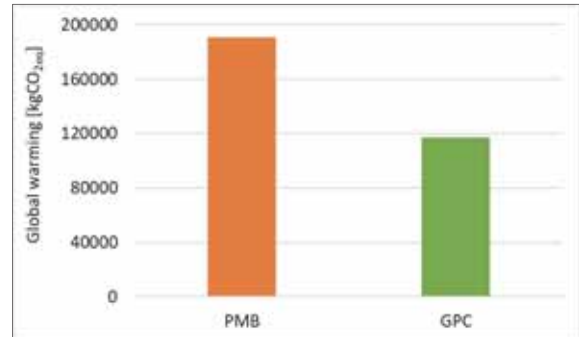


Fig. 8. Equivalent CO₂ consumption for the two solutions (PMB vs GPC)

5.2. Life Cycle Cost (LCC)

The LCC analysis has been performed based on the primary data collected from the company, without opening the boundary of the systems. As can be seen in the Fig. 9, the life cycle cost assessment related to the production of the different layers, also considering maintenance work over the service life (35 years), were 29.7%, 35.3% and 28.3% less for wearing, binder, and base layers respectively. There are also process-related costs to consider, which account for less than 5% of the total cost of the work (“other”) and were 102.1% more in the case of the GPC mixture. Overall, the cost of the GPC mixture was therefore 29.4% lower than the solution traditionally (PMB) used by motorway operators in Italy.

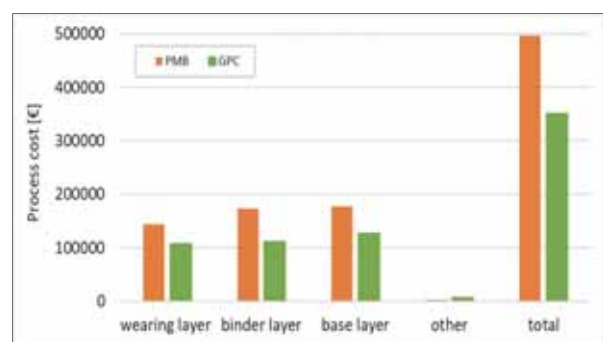


Fig. 9. Process cost for the two solutions (PMB vs GPC)

5.2. Laboratory tests

After the test field was realised, the different GPC mixes were tested (end of October 2022) to check whether they complied with the specifications. The laboratory results of the two innovative mixes (GPC mixtures for base and binder layers) were compared with the test results in the Sina database i.e. NM and PMB mixtures.

5.2.1. ITSM results

Table 4 shows the results of indirect tensile strength modulus at different test temperatures for GPC mixtures (GPC base and GPC binder). The two different mixtures showed no substantial differences in terms of stiffness at the same temperature (with the exception of the results at 10°C), despite a different aggregate grading curve and bitumen content. Furthermore, the table shows that GPC mixtures at a temperature of 20 °C had a stiffness that was about 45% higher than the NM mixture and 27% higher than the PMB mixture.

Finally, both mixtures met the requirements of the specifications (SATAP, 2022), which stipulate that the material stiffness must be greater than 11000 MPa at 10°C, 7000 MPa at 20°C, and 2000 MPa at 30°C for bituminous mixtures modified with polymeric compounds (dry method).

Table 4. Average values of ITSM

Test parameter	Lab results		Reference results	
	GPC Base	GPC Binder	NM	PMB
ITSM at 10°C (MPa)	17036	19200	-	-
ITSM at 20°C (MPa)	11007	10898	5000-7000	7000-9000
ITSM at 30°C (MPa)	4861	4620	-	-

5.2.2. ITS results

Table 5 shows the results in terms of indirect tensile strength. For the control mixtures NM and PMB, a reference range from the Sina group database was provided. The GPC mixtures had an average ITS of 1.69 MPa for the base layer and 1.98 MPa for the binder layer. These values were 70-100% higher than for NM mixtures and 10-25% higher than for PMB mixtures. These higher strengths were due both to the use of polymeric compounds, but also to the simultaneous use of very high quantities of RAP. In general, the use of very high RAP content can be deleterious in asphalt mixes, but thanks to the use of the correct dosage and type of rejuvenator product, it was possible to avoid a doubling of ITS values typical for mixes with very high RAP percentages and no rejuvenator agent.

Table 5. Results of indirect tensile strength at 10°C

Test parameter	GPC base	GPC binder	NM	PMB
ITS (MPa)	1.693	1.979	0.9-1.2	1.2-1.8

5.2.3. ITSR results

Table 6 showed that both GPC mixtures have no problems in terms of water susceptibility, ITSR correspond to 98%, well above the minimum acceptability limit of 90% (main Italian specifications for NM and PMB mixtures).

Table 6. Average values of indirect tensile strength ratio at 15°C

Test parameter	GPC base	GPC binder	NM	PMB
ITSR (%)	98	98	>90	>90

5.2.4. WTD results

Table 7 represents the results of Wheel tracking test. It is noticeable that the values of RD, PRD and WTS are well below the maximum regulatory limits for the acceptance of a mixture specified by the road operator (SATAP, 2022). These results showed that these high-modulus GPC mixtures did not exhibit any problems in terms of accumulation of permanent deformations at high temperatures.

Table 7. Wheel tracking test results at 60°C

Test parameter	GPC base	GPC binder	NM	PMB
RD (mm)	1.97	1.76	<2.5	<2.5
PRD (%)	3.28	2.93	<5.0	<5.0
WTS (mm/1000 load cycles)	0.05	0.05	<0.1	<0.1

5.2.5. SCB results

Table 8 shows the results from SCB tests carried out at 10°C in terms of fracture toughness (K). In this case, a significant difference in K value was evident between the two GPC mixtures (17.76 N/mm^{1.5} for the base and 30.36 N/mm^{1.5} for the binder). Both materials met the minimum specification requirements (>15 N/mm^{1.5}), with the average value in the case of the GPC binder that was double the minimum required, indicating a good resistance of the material to crack propagation.

Table 8. Semi-circular bending test results at 0°C

Test parameter	GPC Base	GPC Binder	NB	PMB
K (N/mm ^{1.5})	17.76	30.36	>15	>15

5.2.6. ITF test results

The ITF test results (number of cycles to failure) were plotted in two log-log graphs, as function of the initial horizontal deformation for GPC binder and base mixtures (Fig. 10). For each material, a power function (with a linear trend in the log-log plane) was superimposed to the experimental data to describe the fatigue behaviour. Furthermore, ϵ_0 values corresponding to 10⁶ load cycles were determined from the equation of the determined fatigue lines. The results were then compared with average values present in the Sina Group database, which refer to a NB mix (Table 9). The results showed that GPC mixtures perform better than NM mixtures commonly used by the motorway operator. Specifically, GPC base mixture had an ϵ_0 at 10⁶ cycles of 79 μ strain 20% higher than the NM mixture, while GPC binder mixture had a value of 86 μ strain 10% higher.

Table 9. Initial deformation of mixtures at 10^6 load cycles

Type of mixture	ϵ_0 (μ strain)
GPC (base layer)	79
NM (base layer)	63
GPC (binder layer)	86
NM (binder layer)	78

5.3. In situ road pavement surface monitoring

FWD testing and visual inspections started being performed after the construction of the new pavement to assess its behaviour under traffic. After more than 500 days during which the reference section was trafficked by almost 12 million total vehicles (3 of which were heavy vehicles), there is no evidence on the new pavement of any surface degradations nor deformations at all (Fig. 11).

Instead, having a look at the old pavements being adjacent to the 1 km long section in which the new GCP mixture was applied (Figs. 12-14), it is possible to recognize some typical effects produced by the end of their service life (it is notable to highlight that these pavements were built at the start of the millennium and that their design life was 20 years). After 500 days of traffic, results from FWD testing showed a highly stable behaviour of the new pavement under traffic. Tests performed in comparable climatic conditions and temperatures between 10°C and 20°C, in fact, showed that deflection basins recorded with the use of 9 geophones are very similar independently of the season (spring or autumn) and, most important,

of the time passed from the construction. According to this, constant trend rates were determined for some deflection basin indicators (DBIs) that were considered for comparison purposes.

Figures 15 and 16 show the results of FWD testing belonging to both the 1 km long test section and a longer section (2 km at all) which includes two adjacent old pavements together with the new one. The new pavement demonstrated trend rates around zero as regards the $AREA_{@25^\circ C}$ (representative of the whole pavement structural condition) and the $D_{1@25^\circ C}$ (representative of the AC layers) DBIs: this means that no structural modifications occurred from the time of the construction. Also, looking at the Table 10 and at the Fig. 17 representing the surface modulus in (MPa) vs the depth in (mm) it is possible to highlight that there was a strong decrease of the difference between deflections under the AC layers (see D_3 - D_5 column and the shift between the red curve – 1 km long section – and the blue one – 2 km long section): this confirms that the new AC structure is able to absorb more stress from the top if compared to that in the old pavements, thus preventing the subbase layer and the unbound bottom layers to receive extra solicitations.

Based on the recorded deflection basins, back-calculation operations were running out in the end. As a result, calculations led to understand that the current elastic moduli of the GCP new pavement layers could be placed between 6500 and 7500 MPa, as the elastic modulus of the whole pavement including the open graded wearing course was estimated in a range of 5200÷5700 MPa.

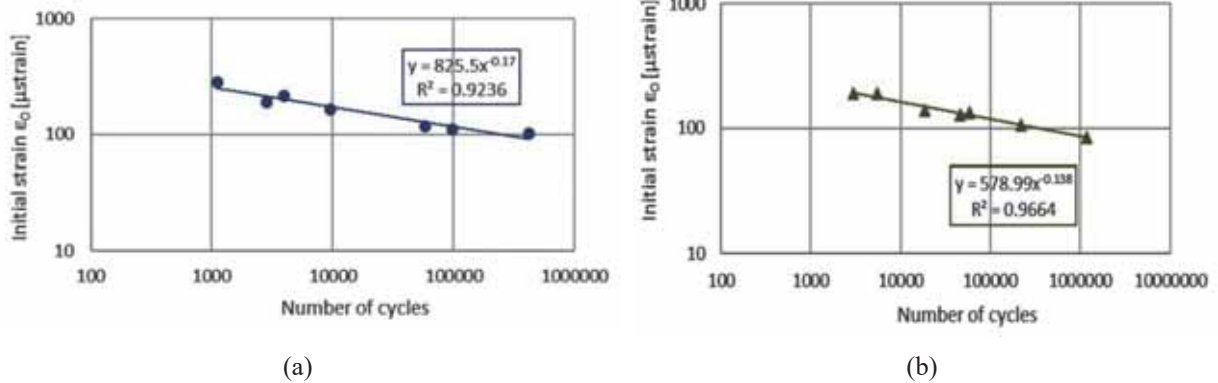


Fig. 10. Initial strain fatigue line of GPC mixture: (a) for base layer (67.0% of RAP) (b) for binder layer (66.6% of RAP)



Detail a



Detail b



Detail c



Detail d



Detail e

Fig. 11. Visual inspection 500 days after the construction



Fig. 12. Existing pavement monitoring (detail a)



Fig. 13. Existing pavement monitoring (detail b)



Fig. 14. Transverse regularity of the existing pavement (detail c)

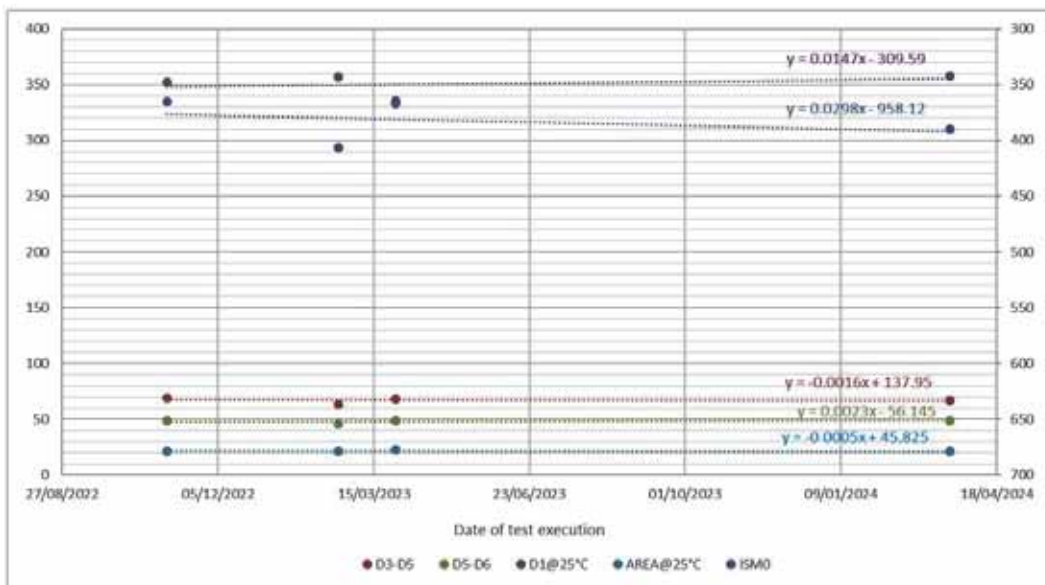


Fig. 15. FWD test result for trial section (1 km long)

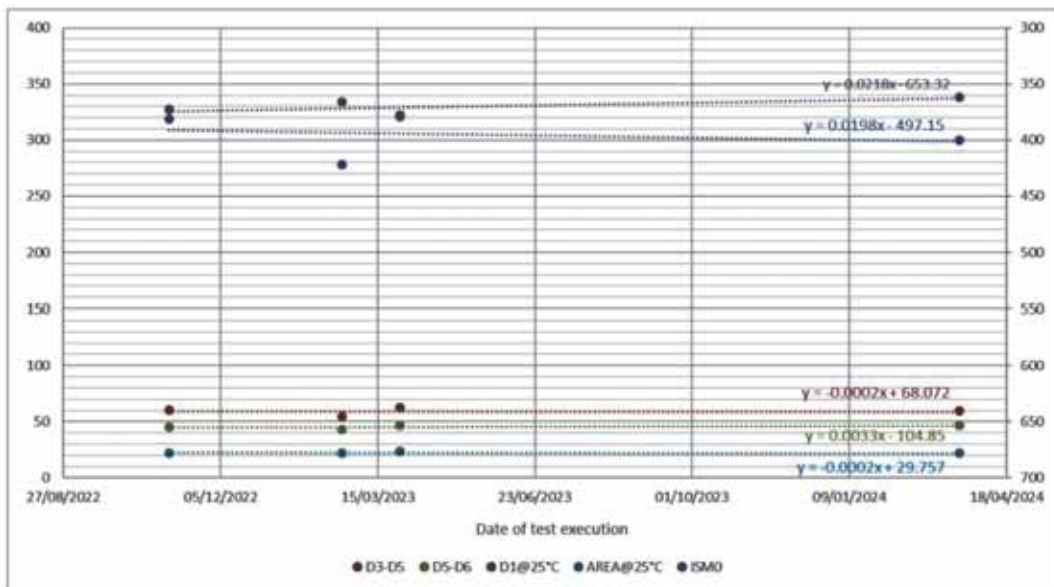
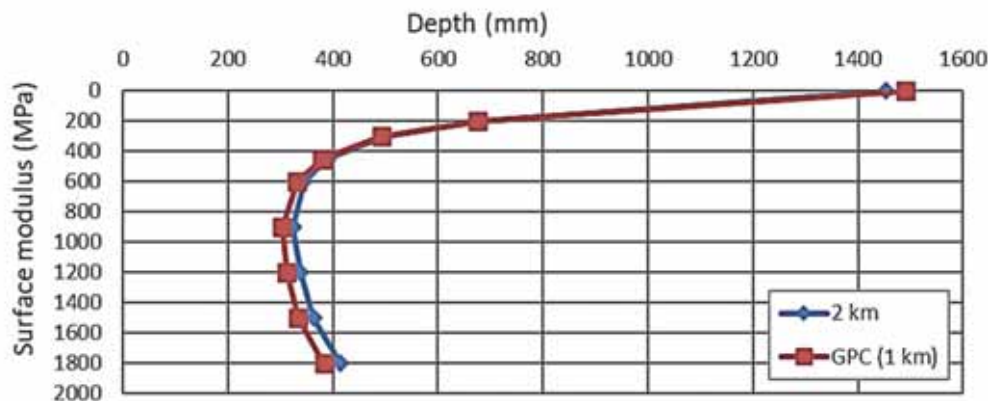


Fig. 16. FWD test result for trial section and adjacent old pavement (total length of 2 km)

Table 10. Falling weight deflectometer test results

Date	Progressive	Temperature (°C)	ISM ₀ (MPa)	D ₃ -D ₅ (MPa)	D ₅ -D ₆ (MPa)	D _{1@25°C} (MPa)	AREA@25°C
Nov/2022	31+300 – 33+300	20.32	366	69	49	352	22
	31+800 – 32+800	20.58	381	61	45	327	22
	Difference, %	1	4	-12	-7	-7	2
Feb/2023	31+300 – 33+300	13.58	407	63	46	357	21
	31+800 – 32+800	13.63	422	55	43	334	22
	Difference, %	0	4	-13	-7	-6	3
Mar/2023	31+300 – 33+300	20.38	367	68	49	336	22
	31+800 – 32+800	20.70	378	62	47	321	23
	Difference, %	2	3	-9	-4	-5	4
Mar/2024	31+300 – 33+300	17.82	390	67	49	358	21
	31+800 – 32+800	17.78	400	59	46	338	22
	Difference, %	0	3	-11	-6	-6	4

**Fig. 17.** Surface modulus vs depth

6. Conclusions

The objective of this research was to quantify the reduction in environmental impact achieved using high-performance AC modified with GPC compound and with a high RAP content (about 70%). An LCA analysis was carried out, which allowed us to estimate a reduction in environmental impact in terms of CO_{2eq} of almost 40% compared to the technologies normally used in motorways, considering a 35-year analysis time interval.

A subsequent LCC analysis, on the other hand, made it possible to estimate the economic savings brought out by these technologies, which is equal to almost 30%, mainly due to the lower maintenance operations that the road operator is expected to carry out during the pavement's life cycle. To refine the assumptions underlying the LCA and LCC analyses and to understand the behaviour of the designed mix under traffic at a full-scale level, a 1 km long test field was set up on the A4 Turin-Milan motorway. After the test field was set up, laboratory tests were carried out to verify the performance of the materials against the specification limits.

Laboratory tests and in situ road pavement assessments were carried out to verify the performance of the materials against the minimum requirements for the hypotheses based on the LCA and LCC analyses to be verified. The results showed that these materials performed well despite their very

high RAP content.

The results were then compared with the data in the motorway management group's database, which showed that the materials studied performed better than those normally used on motorways (the ITSM value at 20°C was 10-25% higher than that of PMB and the average PD of 1.86 mm was significantly below the typical values for NC and PMB mixtures). Finally, the pavement was monitored with FWD tests as well as visual surveys, which showed that almost two years after the construction of the test field, the pavement is in very good condition and no issue has been detected. On the basis of the FWD surveys, it was possible to recognise a significant improvement in the deflection basin indicators considered (around 10%).

These results would seem to convince that the material is on the right track to be able to aspire to a service life significantly higher than 20 years (between 30 and 35), thus letting the design assumptions be thought as reasonable. However, this section will need to be constantly monitored in the coming years to reinforce this statement.

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EXTENDED PRODUCER RESPONSABILITY (EPR) IN AVIATION SECTOR: A NEW RISK ANALYSIS APPROACH

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Abstract

The introduction of Extended Producer Responsibility (EPR) into European legislation will have a significant impact on EU industry. Obviously, in order to make the management of an EPR-based process efficient, it is necessary to define strategic approaches that help companies to be compliant with the European Legislation. Therefore, the objective of this study is to understand whether it is possible to propose a risk analysis model that can help company management to develop an efficient ERP process. A possible solution to the research question is to propose a new risk analysis approach in which two widely used methods in the field of risk analysis are combined: the Functional Resonance Analysis Method (FRAM) model of Prof. Hollnagel combined with the Analytic Hierarchy Process (AHP) method proposed by Saaty. The new approach has been applied to the aviation sector, which will be affected by Extended Producer Responsibility in the short term, due to the increase in the number of aircraft at the end of their life cycle and to the evolution of regulations in the aeronautical sector.

Key words: aircraft, end of life, extended producer responsibility, recovery, risk analysis

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1. Introduction

The aim of this research is to apply the principles of Extended Producer Responsibility to the end-of-life management of aircraft. The first step of this article is to define - Extended Producer Responsibility (EPR), placing it in today's industrial reality and taking into account the recent regulatory reforms that have affected this institution. The concept of EPR, introduced in 1990, has been defined by Lindhqvist (2002) as *"a strategy to reach an environmental objective of a decreased total environmental impact from a product, by making the manufacturer responsible for the entire life-cycle of the product and especially for the take-back, recycling and final disposal of the product"*. In other words, it is a particular strategy through which the producer of a

given good has complete responsibility for his product throughout its life cycle, therefore also during the so-called post-consumer stage (Lifset, 1993; Lindhqvist, 2000). These responsibilities will require the manufacturer to perform several tasks. First of all, the manufacturer must provide customers with all the necessary information for the correct management of potential environmental impacts associated with its products. In addition, those who produce the product have to take charge of the physical and logistical management of the product throughout its entire life cycle, in order to ensure its correct disposal/recovery when it becomes, economically or technically, obsolete. Finally, the manufacturer is responsible for any damage caused by the product to the environment during its life cycle. On closer inspection, this is a very broad responsibility, ranging from the regulatory

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aspect to the technical-o-informational aspect up to affecting the company's credibility towards stakeholders (Lindhqvist, 2000). In recent years, EPR has fully entered the Circular Economy System promoted by the European Union, as a strategic approach aimed at promoting the latter, (Bocken et al., 2016; Compagnoni, 2022; Jensen et al., 2021). From an operational point of view, this vision has translated into - practical application of the EPR concept in various areas of strategic interest. By way of example, a sector that immediately embraced this principle is the electronic and electrical sector (Directive 2002/96/EC), following numerous pressures from stakeholders who had to effectively manage a constantly growing flow of materials (Compagnoni, 2022; Gui et al., 2013; Huisman et al., 2008; Pérez-Belis et al., 2015; Wasserbaur et al., 2022). To be efficient, an EPR system must start from a system that is able to measure its impacts so as to demonstrate its goodness and technical-economic feasibility. In the aeronautical sector, the application of EPR is a very delicate issue, especially with regard to the correct management of aircraft that are at the end of their operational cycle. Aircraft recovery processes are now becoming increasingly important and this interest will increase in the years to come (Scheelhaase et al., 2022). This statement is confirmed by recent studies carried out in relevant literature (ICAO, 2019; Ribeiro and de Oliveira Gomes, 2015; Scholz, 2022), according to which about a thousand commercial aircraft a year will end their life cycle by 2030. However, determining the real obsolescence of an aircraft is very complex as the "technical life" of the aircraft itself does not always coincide with the economic obsolescence. In this sense, - ICAO (2019) estimated that compared to the twenty - six-year average duration of an aircraft from a technical point of view, there could be a significant reduction in its life span from an economic point of view as disassembly activities, if properly managed, could be more profitable than keeping certain categories of aircraft in operation.

In other words, the choice to keep an aircraft alive depends on numerous variables, which are also influenced by general macroeconomic trends and the competitive positions of the various stakeholders as they operate in the sector (De Brito et al., 2007; Keivanpour et al., 2013; Wong et al., 2020). wanting to deal with the issue of sustainable management regarding the recovery process of an aircraft at the end of its life cycle, the first aspect that must be evaluated is the extreme complexity that characterizes the process of decommissioning an aircraft. As demonstrated in a recent study by ICAO IN 2019, the recovery process of an aircraft tends to be distinguished into two major phases. The first phase concerns the processes apt to obtaining "reusable" parts for other aircraft. The second, on the other hand, concerns the pure dismantling and recycling of parts that cannot be used on other means. The distinction between the two phases is very important as the possibility of reusing parts obtained from other aircraft

is strictly regulated by the regulations that guarantee the safety of the sector. (Böckmann and Schmitt, 2012; Elsayed et al., 2019; Fera et al., 2020; Maaß, 2020; Scheelhaase et al., 2022). In principle, these components must maintain "airworthiness" status before being reused on other aircraft. For this reason, anything that cannot be considered efficient for safe use will have to undergo the recertification process and obtain the relevant authorizations before returning to service (ICAO, 2019). Taking into account this aspect, the pre-eminent objective of any company that intends to proceed with the recovery of one of its aircraft is to remove most of the valuable components, maintaining, given the technical condition, their status of "certifiability" so that they can be immediately placed on the market. Having dealt with the recovery process, these have a different value from a purely economic point of view. As highlighted by Scholz (2022), there are two different business models applicable to the sector under consideration. The first concerns the case in which the components obtained from the activities of disassembly remain the property of the owner of the aircraft. The second case, on the other hand, provides for the components obtained from the recovery process to become the property of the recycler. The proposed solutions have important repercussions in terms of determining the trade-off of the recovery process. In fact, if on the one hand the cost of the recovery activities varies according to purely technical aspects such as the class to which the aircraft belongs to or the number of engines present, the value obtained from the disassembly activities is closely linked to the resale of the most valuable components. Therefore, thanks to their correct recovery, more significant results will be obtainable from an economic point of view, both for the owner companies and for the resellers of the components (Zhao et al., 2020).

Taking into account the above, it is clear that there is a need to put in place procedures and reference standards that are able to comply with the characteristics of the economic and environmental sustainability of the disassembly processes provided for by current legislation. In this sense, on the basis of the study conducted by de Brito et al. (2007) several manufacturers have put in place initiatives aimed at ensuring the efficient management of the End of Life, in the light of current legislation. The two main initiatives, led respectively by Boeing and Airbus, have led to the creation of two consortia, AFRA and PAMELA. Without going into - specific merits, - it should be noted that the objective of AFRA (Carberry, 2008) is not to provide for - direct management of the recycling activities but to create standards that are a reference both for the industries in the sector and for the governments that will have to indicate the legislative action. In order to create an EPR compliant management system and to obtain a method of analysis suitable for the objectives of this work, it is therefore necessary to carry out a wide-ranging analysis, that analyzes not only the reference literature in the aeronautical field, but that also analyzes the

EOL methods used by industry leaders (i.e. Boeing, Airbus) for the correct management of the recovery activities associated with their aircraft. Taking into account the above, the present study aims to answer a specific research question: what are the main phases that characterize the dismantling of an aircraft at the end of its life cycle? Moreover, what are the critical steps to be taken into account in order for recovery procedures to be efficient from a technical and economic point of view? To answer these questions, a hybrid approach of risk analysis was chosen. This approach relates to a process based on the combination of two well-known evaluation methodologies - the Functional Resonance Analysis Method (FRAM) model, used in - risk analysis processes of organizational processes, and the Analytic Hierarchy Process (AHP) model useful to better characterize the subjective choices that emerge during the brainstorming phases which are carried out during the analysis of the abovementioned processes. The aim of this study is to answer a specific question: is it possible to set a new risk analysis framework that helps companies to meet the new EPR-related challenges?

2. Material and methods

The present study aims to identify a series of inputs useful for the preparation and updating of these standards. Given the context, it is therefore possible to say that the management of a sustainable recovery system is characterized by the simultaneous presence of heterogeneous elements (i.e. technical, physical, organizational) interconnected with each other. These, therefore, will have to be treated in such a way as to be able to immediately manage any failures, stemming any effects that may cause inefficient conditions to the system. For this reason, the purpose of this research is to create a resilient management process that can effectively respond to any negative event that may harm the technical-organizational efficiency of the recovery process. By embracing what has emerged in the reference literature on resilience engineering (Arcuri et al., 2022; Dinh et al. 2012; Haimes, 2009; Hulme et al., 2019; Patriarca et al., 2020; Sujun et al., 2022), a risk analysis model based on the joint use of FRAM: the Functional Resonance Analysis Method proposed by Hollnagel (2014) and the so-called Analytic Hierarchy Process (AHP) model of Prof. Thomas Saaty (2008), is proposed.

The Functional Resonance Analysis Method (FRAM) is a risk analysis method mainly used for the analysis of so-called socio-technical systems that aims to analyze how the individual activities of a given business process are carried out. Its objective is to understand the real efficiency of the process under analysis rather than the nature of failures that may affect the process itself. The FRAM is based on five steps. First, one must clearly define the purpose of the analysis to be performed, identifying the different functions that characterize the process. Then, for each function, one must understand what relationship exists between this function and the function immediately

downstream. Hollnagel (2012), for each function, has identified six elements Input (I), Output (O), Time (T), Preconditions (P), Resources (R), and Control (C). These elements are represented graphically by means of a hexagon. Each function is connected to another function so as to obtain an interconnected network that allows the analyst to immediately visualize the procedural flow related to a given activity. Although FRAM has undoubted advantages in terms of process visualization and adaptability to different organizations, the model under analysis nevertheless has several limitations.

The most important one is that the FRAM is a qualitative analysis model. For this reason, it is necessary to associate the FRAM with an additional method of analysis in order to make FRAM-related choices less 'subjective'. For this reason, the Analytic Hierarchy Process (AHP) has been associated with the FRAM. This is a multi-criteria decision-support technique used by organizations to select an alternative from those available and to rank a set of criteria according to an order of desirability based on a rational framework of quantitative comparisons (Saaty, 1980). The AHP model proposed in this study is that proposed by Saaty in its general form, constructing a pairwise comparison matrix using linguistic scales where the experts' opinions were used to rate intensity. Compared to the various methods in the literature, the decision to use the AHP is linked to its ease of use by the research team as well as its adaptability to other analysis criteria such as the FRAM.

The FRAM/AHP model, based on the methods and principles of ISO and NIST, aims to make the advantages that derive from the use of the proposed systems its own, while reducing the limits underlying their use. The Functional Resonance Analysis Method (FRAM) theorized by Hollnagel (2014) is a very useful method for managing contexts characterized by a clear predominance of the human/organizational aspect over the purely technical one (Hollnagel, 2017). This ability allows the organization to respond appropriately to adverse events, making it resilient to such situations (Aguilera et al., 2016; Bergström et al., 2015; Bjørnson et al., 2020; De Carvalho, 2011; Tian and Wasserbaur, R., Sakao, T., & Milios, L., (2022), Interactions of governmental policies and business models for a circular economy: A systematic literature review. , *Journal of Cleaner Production*, 337, 130329., 2020). However, the FRAM, if used as a model of independent analysis, has the limitation of being strongly influenced by the individual aspect due to the simultaneous presence of several experts, belonging to different professional areas, who, by participating in its drafting, can, with their many individual choices, negatively influence its construction. This, as evidenced by the reference literature, risks reducing its effectiveness as the barriers supporting risk mitigation would be identified exclusively in the face of personal choices and evaluations (Buikstra, et al., 2020; De Carvalho 2011; Rosa et al., 2015). To overcome this limitation, it is therefore necessary to introduce an

additional analytical tool to help process actors identify the risk factors associated with the functions identified in the process so that they can identify a priority scale for resolving intervening issues in those functions. To this end, it was decided to build a hybrid FRAM/AHP model, where the AHP method would be used as a semi-quantitative analysis tool capable of reducing the subjectivity underlying the construction of the FRAM model. The model, built on the basis of the methods contained in the International Standards ISO 31000:2018 and NIST Special Publication 800-300 Rev.1 of September 1, 2012, will lead to the creation of a flowgram in which the process under analysis will be divided into interrelated functions that will be evaluated according to two fundamental parameters: Time and Precision. Time and Precision are the subject of particular attention in Saaty's Analytic Hierarchy Process, a measurement method that takes place through pairwise comparisons based on judgments developed by experts in order to obtain priority scales. These scales, appropriately used, will be used to measure "subjectivity in a mathematically objective way" (Saaty, 2008). The comparison of the pairs will take place through a particular scale of values, shown below, which represents the intensity that each judgment has in the comparison between these pairs (Table 1).

Table 1. Comparison matrix between pairs (our elaboration taken from Saaty, 2008)

Intensity	Explanation
1	Given two activities, they contribute equally to the objective
3	Given two activities, experience and judgment show a slight preference for one over the other
5	Given two activities, experience and judgment demonstrate a moderate preference for one over the other
7	Given two activities, experience and judgment demonstrate a strong preference for one over the other
9	Given two activities, experience and judgment show that one activity is to be preferred without a shadow of a doubt to the other
2,4,6,8	Intermediate Values

From the values obtained from Table 1 we will construct a matrix based on the values contained in this table and their reciprocals. Therefore, when an element "i" (a decision) has one of the values in the table, it is compared with another element, called "j" (a decision), then "j" has a reciprocal value when this is compared with "i". All these elements will be represented in a square matrix "M", where each value of this matrix represents the result of the comparison between the decision criteria of row "i" with column "j" (Eq. 1). The element will be equal to 1 (= 1), if "i" is equal to "j", this means that the factor is equal with respect to itself. Therefore, the main diagonal of this square matrix, where the elements with $i = j$ are always

present, will always have numerical values of 1, since there is no priority or dominance relationship between closely equal elements.

$$M = \begin{pmatrix} 1 & \dots & a_{1n} \\ \vdots & \ddots & \vdots \\ \frac{1}{a_{1n}} & \dots & 1 \end{pmatrix} \tag{1}$$

In addition, the $a_{ij}a_{ji}$ will be the inverse of the element, i.e. = -1. This represents the inverse mathematical relationship that proves the so-called "inverse element opposition". The next step in the model proposed by Saaty (2008) is the determination of - comparison weights between the elements of the square matrix "M". These values are obtained by the partial results of the set "A" and $v_i(A_j)$ where $v_i(A_j)$ is the impact value of the alternative "j" with respect to the alternative "i". These data represent the numerical representation of the opinions expressed by experts with respect to the proposed alternatives. These results are then normalized through the Eq. (2):

$$\sum_{i=1}^n v_i(A_j) = 1 \text{ with } j = 1, \dots, n \tag{2}$$

where: n is the number of alternatives or items compared. Each element of this sum consists of (Eq. 3):

$$v_j(A_j) = 1 \text{ with } j = 1, \dots, n \tag{3}$$

This causes the priority vector of the alternative "i", in relation to the importance criterion, to be defined by the Eq. 4:

$$v_k(A_i) = \sum_{j=1}^n \frac{v_j(A_i)}{n} \text{ with } i = 1, \dots, n \tag{4}$$

that is, having a square matrix "M" to determine the importance weights of each factor in the matrix, it is necessary to add up all the factors and add them together in each column. Then you need to make another "MRW" matrix where each of its elements will be the relative weight of each of the elements in the left column, with respect to each of the items in the top row. To do this, divide each part of the "M" matrix by the sum of the parts in each column. In this "MRW" matrix, the simple weighted average of the elements in each row will give you the relative weight "RW" of each element. To validate and ensure the validity of these considerations and calculations, the AHP methodology also involves a consistency analysis of all the processed data. Since the matrix "M" is a reciprocal matrix, if all the experts' value decisions were good, it would be possible to verify that all expert value decisions were adequate in all comparisons (Eq. 5).

$$a_{ij} \times a_{jk} = a_{ik} \quad \forall i, j, k \tag{5}$$

Therefore, according to this model, the "M" matrix would be consistent. Let "n" be the number of elements the auto vector of "M" and "w" the priority vector. If the opinions expressed by the team are all coherent, then λ_{max} (Gomes et al., 2004) (Eq. 6):

$$\lambda_{max} = n \& a_{ij} = \frac{w_i}{w_j} \tag{6}$$

However, starting from the premise it is always possible to find inconsistencies, these can be evaluated by assuming that the closer the value λ_{max} is to "n", the greater the consistency of opinions. Thomas Saaty (2008) showed that given a matrix "M" one must identify a vector - that - satisfies Eq. (7):

$$A_w = \lambda_{max} \times w \tag{7}$$

and that to obtain the auto vector from this equation, it is necessary to calculate Eq. (8):

$$\lambda_{max} = \frac{1}{n} \sum_{i=1}^n v_j \frac{[A_w]_i}{w_j} \tag{8}$$

It is important to note that small variations - imply small variations -, where the deviation of the autovector with respect to n (order number of the matrix) is considered a measure of consistency. Therefore, it can be said that it allows us to evaluate the proximity of the scale developed by Thomas Saaty (2008) with the scale of ratios or quotients that would be used or quotients that would be used if the matrix "M" were totally coherent. This can be done by means of a consistency index (CI). According to Thomas Saaty's (2008) Theorem 1, "M" is consistent if, and only if $\geq n$.

That is, if the matrix "M" is consistent, then when calculating the magnitude of the perturbation of the matrix "M" using the ratio (Eq. 9)

$$CI = \frac{(\lambda_{max} - n)}{(n - 1)} \tag{9}$$

the CI will have a value of less than 0.1 (Saaty et al, 2012). Saaty proposes the calculation of a Consistency Ratio (CR) based on these issues related to the consistency of matrix data. The Consistency Ratio is defined in Eq. (10):

$$CR = \frac{CI}{RI} \tag{10}$$

where CI is the coherence index calculated from the

equation presented above. RI is the Random coherence Index. RI is obtained by square matrices of order "n" of Oak Ridge National Laboratory - USA. from Oak Ridge National Laboratory - USA, presented in Table 2 (Saaty, Thomas L., et al., 2012).

The larger the CR, the greater the inconsistency. When n = 1 or 2, the CR is zero; when n = 3, the CR must be less than 0.05; and when n = 4, the CR must be less than 0.08. In general, an inconsistency is considered acceptable, for n > 4, when CR is less than or equal to 0.10. If CR is less than 0.10, judgments will need to be revised. AHP provides an index of consistency for an entire hierarchy. An inconsistency of 10% or less implies that the adjustment is small compared to the actual values of the autovector items (Saaty, et al, 2012). The joint application of the AHP method with the FRAM model will give an objective foundation to the subjective assessments of the variability of the FRAM-obtained Outputs made in terms of time and accuracy.

5. Results and discussions

The case study of this work has been drawn up in compliance with the ISO 9001:2015 standard "Quality Management Systems", where it is provided that, for the executive execution of an End of Life project such as the one proposed, it is necessary to preliminarily frame:

1. Who are the subjects and which processes are involved in the EOL process of an aircraft with clear identification of the existing interrelationships between them;
2. Define the roles, tasks and responsibilities of the parties involved, in order to avoid any process inefficiencies
3. Build a risk analysis model capable of identifying the main project tasks and any issues related to this path

The three elements described will lead to the construction of a project plan, drawn up in accordance with the provisions of paragraph 8.3 of the ISO 9001:2015 standard which, once validated by the Management, will constitute the operational framework of the entire process (Fig. 1).

The first result that the team has obtained, through the application of the FRAM/AHP model, is a complete reformulation of the disassembly process of an aircraft at the end of its life cycle. It is demonstrable, in fact, that with an adequate brainstorming activity, the team was able to rework the flowgram proposed by ICAO (2019) (Fig. 2) INTO a "dynamic" scheme (Fig. 3), where, for each downstream function, a specific condition capable of characterizing the upstream function is defined.

Table 2: Random Consistency Index (R.I.) Source: Saaty and Vargas (2012)

N	1	2	3	4	5	6	7	8	9	10
Random consistency index (R.I.)	0	0	0.52	0.89	1.11	1.25	1.35	1.40	1.45	1.49

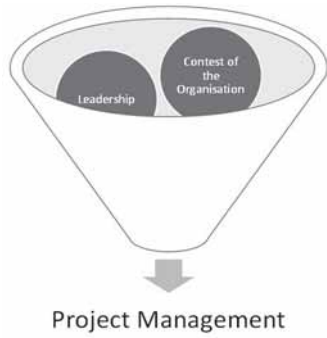


Fig. 1. Operational flow chart of the model (our processing)

Another very interesting result related to the application of the FRAM model derives from the fact that, through - analysis of the "Technological, Organizational, Human" characteristics of the identified functions, it is possible to obtain a classification, in terms of the two variables proposed by Erik Hollnagel in 2014: "Time" and "Precision". These two elements, according to the author, are fundamental in order to define the degree of criticality of the functions of the process in question, as their presence is able to characterize the degree of variability that these have and how this variability influences the interactions between the functions downstream and upstream of a given process. through the analysis of the "Time elapsed for the realization of the activity" and the "Precision used for the realization of the activity", the working group analyzed the different functions that characterize the disassembly process of an aircraft at the end of the life cycle, arriving at the identification, through an internal brainstorming process, of the following functions considered critical for the performance of recovery functions (Table 3).

The highlighted functions are considered critical by the research team because they have a high capacity to influence the efficiency of downstream functions. In particular, according to the team's analysis, the 'critical' functions affect downstream

activities for two reasons: the first is related to the fact that, given the accuracy required to carry out disassembly activities, the time that operators will have to devote to such activities could be prolonged beyond what is expected, creating bottlenecks all along the way. Secondly, possible misunderstandings between operators may generate errors in the identification of parts, leading to delays along the recovery line.

The other functions, although important in the economy of the process, are not considered critical by the team because, from the evaluations carried out, their inefficiency could be easily managed through the down streaming of the functions. The analysis process carried out with the FRAM method was therefore supported by the AHP method, through which the working group intended to "confirm its evaluations by purifying them, as much as possible, from the subjective assumptions that allowed the graphic realization of the FRAM model, Starting from the so-called Common Performance Condition (CPC) theorized by Woltjer Rogier and Erik Hollnagel (2008) and adapted to the recovery process, it is possible to establish that time and precision are respectively functions of the following CPCs Eq. (11) (Table 4 and Table 5).

$$f_{TIME} = (T_1; T_2; T_3; T_4; T_5)$$

$$f_{ACCURACY} = (P_1; P_2; P_3; P_4; P_5) \tag{11}$$

From the characterization of the c.d. Priority matrix of the variables under analysis, two different matrices have been created, one for the Time variable and the second for the Precision variable. Subsequently, the working group examined how the individual variables are able to influence the functions considered critical through FRAM analysis by constructing two matrices, one for priority and one for the determination of the score associated with the analyzed variable with respect to the examined function. The data obtained have been summarized in the following table and graph (Table 6) (Fig. 4).

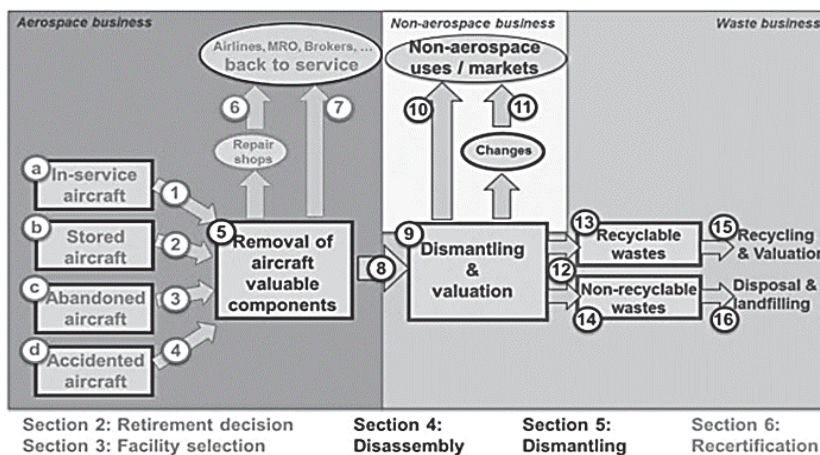


Fig. 2. Process of decommissioning an aircraft (ICAO, 2019)

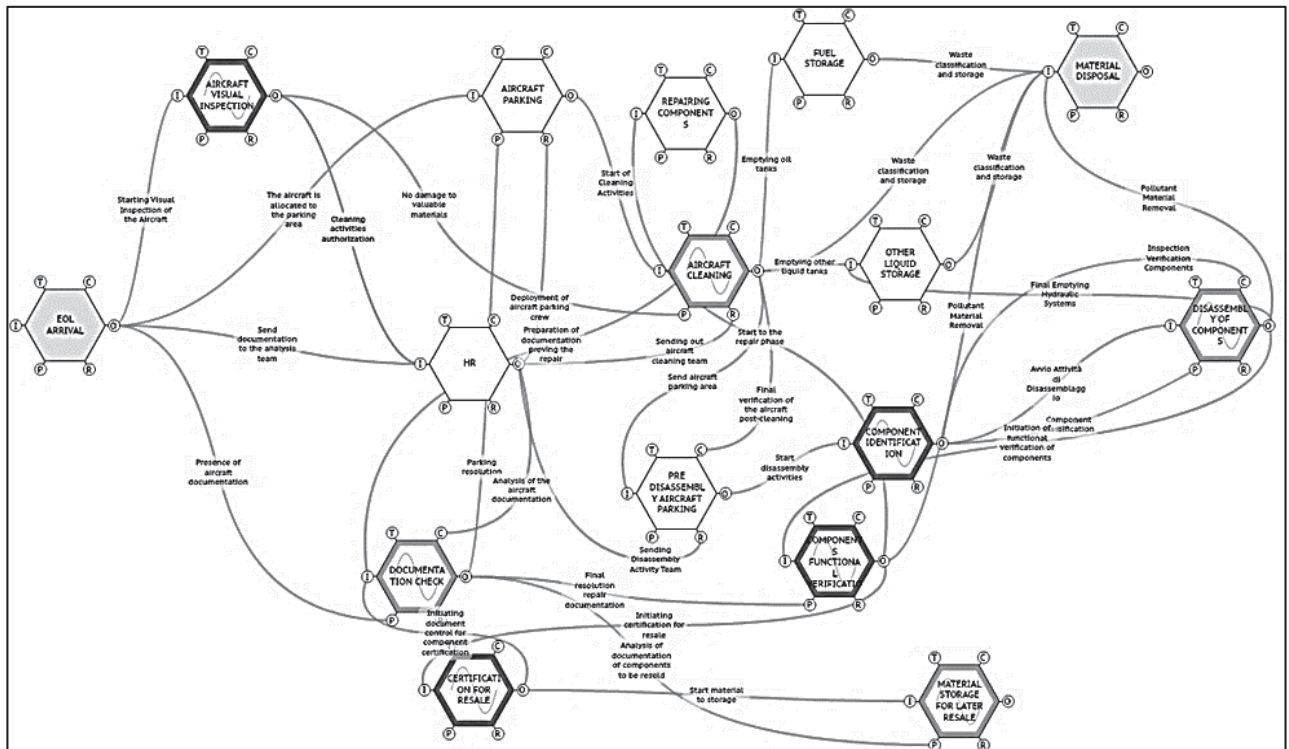


Fig. 3. FRAM representation of decommissioning of AN aircraft (our processing)

Table 3. Critical functions of the decommissioning process of an aircraft (our processing)

Function name	Description	Function type
AIRCRAFT VISUAL INSPECTION	The Work Team provides an initial inspection of the aircraft waves and retrieves the accompanying flight documentation	Human
CERTIFICATION FOR RESALE	The product to be resold undergoes re-certification by the relevant agencies to be remounted on the aircraft	Human
COMPONENTS FUNCTIONAL VERIFICATION	Components are tested for subsequent reuse	Technological
COMPONENT IDENTIFICATION	Components to be disassembled are first identified and classified	Technological
DISASSEMBLY OF COMPONENTS	Components are disassembled from the aircraft and destined for functional testing	Technological

Table 4. CPC considered as a function of time (our processing)

Time-assessed CPCs	
Resource availability	T1
Training and experience of the resources involved	T2
Type of fastner/connection	T3
Component manipulation	T4
Working conditions	T5

Table 5. CPC considered as a function of precision (our processing)

CPCs rated for accuracy	
Resource availability	P1
Training and experience of the resources involved	P2
Quality of internal/external communication	P3
Working conditions	P4
Quality of collaboration between team members	P5

Table 6. Summary table of the results obtained through AHP (our own processing)

<i>Critical Function</i>	<i>Time</i>	<i>Precision</i>
AIRCRAFT VISUAL INSPECTION	0.386	0.367
CERTIFICATION FOR RESALE	0.329	0.355
COMPONENTS FUNCTIONAL VERIFICATION	0.132	0.134
COMPONENT IDENTIFICATION	0.102	0.096
DISASSEMBLY OF COMPONENTS	0.061	0.046

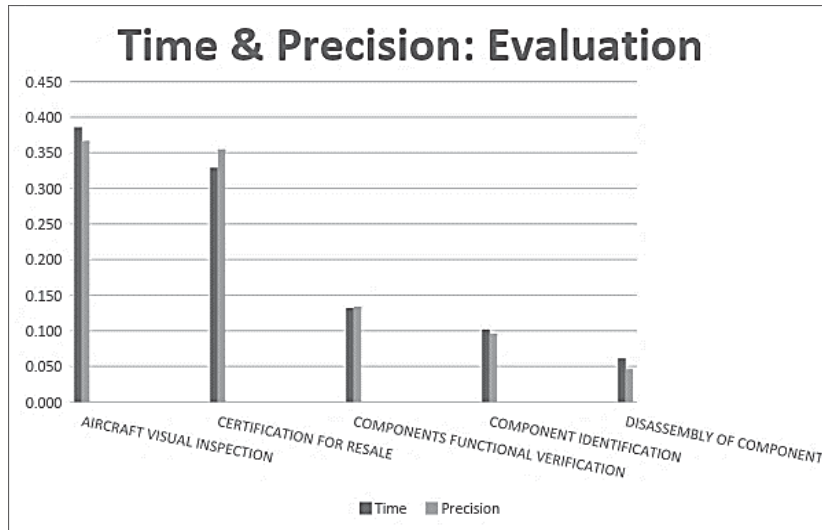


Fig. 4. Time and accuracies evaluated according to the AHP method (our own processing)

As can be seen from the data obtained (Fig. 4), the application of the AHP model to the case examined substantially confirmed what emerged during the application of the FRAM model, namely that the visual inspection function of the aircraft and the certification of the components recovered for subsequent resale/reuse on other aircraft requires particular attention from the workers. Going into the details of the results obtained, the visual inspection of the aircraft sees a prevalence of the "Time" factor (Fig. 5) compared to the "Precision" variable (Fig. 6).

This data is confirmed by the operational experience in the field, where the on-site verification of the condition of the aircraft requires adequate timing by the operators in charge who will have to identify any damage to the external elements of the aircraft that make the analyzed element unsuitable for immediate reuse. The second aspect of attention is that of certification for resale. Looking at the "Time" factor and the variables that most influence its performance (Fig. 7), the AHP method has shown how, for the work group, the availability of manpower connected to the technical capacity of the resources involved in the analyzed function are fundamental for the performance of efficient and effective performance.

Now let's look at how the "Precision" function is affected (Fig.8); according to the results collected and processed with the AHP method, we note a very important novelty compared to the case dealt with for the analysis of the "Time" function: For the "Precision" function, in fact, in addition to the skills

and availability of the workers, the work team gives high importance to the communication aspects both between the members of the group responsible for managing the certification of the components and that between the latter and external parties (i.e. deputies issuing of certifications, public authorities).

The evaluation proposed by the team finds, in this case as in others, its correspondence in operational practice. In fact, any element that is susceptible to reuse from a technological point of view must necessarily be accompanied by all the necessary documents in order to be authorized for use by the competent authorities.

By deepening the analyses in reference to these two functions "critical for the disassembly process", the AHP analysis has allowed us to understand how to improve the performance of the variables "time" and "precision" as a function of the variables being evaluated. With regard to the "time" function, the inspection of the aircraft requires, in order to be efficient, an adequate number of operators dedicated to the purpose.

As far as "precision" is concerned, here too the aspect related to the skills of the staff is highly relevant compared to the other variables. With regard to the other critical function, that of "certification for resale/reuse", the "time" and "precision" functions are also influenced by the presence of an adequate number of people appointed for the purpose and who are adequately trained to manage the operational activities related to the activity analyzed.

Aircraft Visual Inspection-"Time"

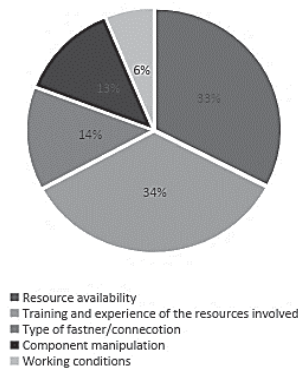


Fig. 5. Aircraft Visual Inspection - Evaluation of Weather Incident Variables (our processing)

Aircraft Visual Inspection-"Precision"

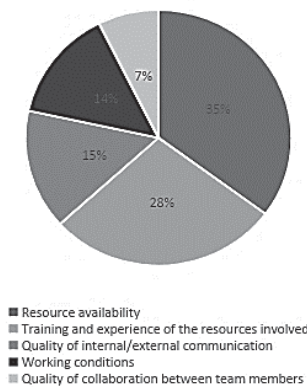


Fig. 6. Aircraft Visual Inspection - Evaluation of Accuracy Variables (our own processing)

Certification For Sale - "Time"

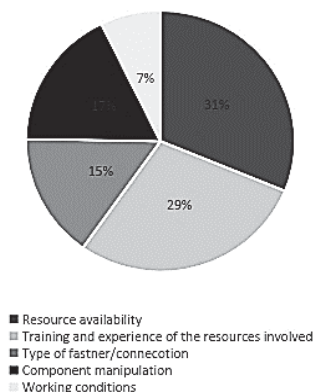


Fig. 7. Certification for Sale- Evaluation of Variables Affecting Time (our elaboration)

6. Conclusions

The aim of this study was to present a methodology designed to support companies involved in the disassembly of aircraft at the end of their life cycle, ensuring efficient recovery activities that align

with the principles of extended producer responsibility.

Certification For Sale - "Precision"

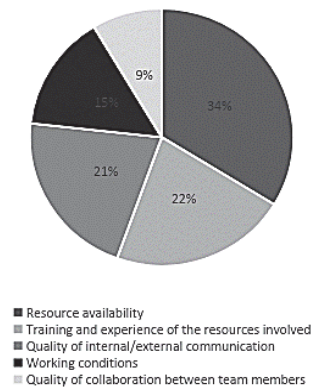


Fig. 8. Certification For Sale - Evaluation of variables affecting Accuracy (our elaboration)

Following a brief review of relevant literature, the study introduced a process analysis methodology combining FRAM and AHP models. This approach facilitated the identification and validation of key outcomes by the team responsible for managing the various phases of disassembly necessary for the recovery of aircraft components for reuse and recycling. The decision to develop this methodology stems from the observation in the literature that there are few models available for managing aircraft disassembly at the end of their life cycle. This highlights the novelty and developmental stage of this sector. Additionally, the evolving regulatory framework for extended producer responsibility adds complexity and opens avenues for further research.

The analysis emphasized the critical importance of "time" and "precision" factors, which, when defined through specific variables, must consider the human and organizational elements. These include the availability of resources and the requisite skills of the actors involved in the recovery process, both of which are essential for achieving outputs that are economically viable for companies. The economic evaluation of the presented process is proposed as a future area of research to provide comprehensive decision-support models that enhance the economic and environmental sustainability of the aviation sector. The proposed risk analysis model aligns with current evidence and studies.

However, ongoing regulatory and technological advancements in the aviation sector will necessitate further refinements. The authors anticipate that future work will confirm the validity of the proposed approach. Additionally, the methodology will need to be expanded to include cost-benefit analyses aimed at improving the efficiency of critical functions identified. This will involve the application of economic analysis methods, which are currently under review and will serve to further support the

decision-making processes in aircraft recovery at the end of their life cycle.

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Faber K., (2000), *Biotransformations in Organic Chemistry - A Textbook*, vol. VIII, 4th Edition, Springer, Berlin-Heidelberg-New York.

Handbook, (1951), *Handbook of Chemical Engineer*, vol. II, (in Romanian), Technical Press, Bucharest, Romania.

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Stanciulescu D., (2004), *Environmental impact assessment generated by C.E.T.1 Company, Iasi* (in Romanian), MSc Thesis, Gheorghe Asachi Technical University of Iasi, Romania.

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USEPA, (2007), Biomass Conversion: Emerging Technologies, Feedstocks, and Products, Sustainability Program, Office of Research and Development, EPA/600/R-07/144, U.S. Environmental Protection Agency, Washington, D.C., On line at: <http://www.epa.gov/Sustainability/pdfs/Biomass%20Conversion.pdf>

EC Directive, (2000), Directive 2000/76/EC of the European Parliament and of the Council of 4 December 2000, on the incineration of waste, Annex V, *Official Journal of the European Communities*, L 332/91, 28.12.2000, Brussels, Belgium.

GD, (2004), Governmental Decision no. 1076/2004 surnamed SEA Governmental Decision, regarding the procedure for strategic environmental impact assessment for plans or programs, *Romanian Official Monitor*, Part I, no. 707 from 5th of August, 2004, Bucharest, Romania.

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