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Grau de Química

Treball Final de Grau

Characterization of plastic waste found in high mountain tourism areas of the Catalan Pyrenees. Citizen awareness- raising activities.

Caracterització dels residus plàstics trobats en zones de turisme d'alta muntanya dels pirineus catalans. Activitats de sensibilització ciutadana.

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What you do makes a difference, and you have to decide what kind of difference you want to make.

Dr. Jane Goodall

I would like to thank my tutor Dr. Mònica Martínez for her valuable guidance and tireless support throughout the entire project. I am very grateful for my laboratory partner, Joaquim Serra, for helping me whenever I needed it and for giving me insightful feedback.

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REPORT

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1. SUMMARY

Most of the plastics that are found in the sea forming the well-known plastic islands, have been transported from the population centers through the rivers. For this reason, one of the first tasks to be carried out is to prevent plastics from reaching the rivers, and for this purpose is necessary that citizens become aware of the need to make an appropriate management of plastic waste at the end of its useful life. Over the last few years, several campaigns have been carried out to raise awareness about proper waste management in homes, but this awareness seems to be forgotten when we are away from home and especially in leisure areas or other activities such as industry or agriculture. These wastes, that are not managed correctly, often reach the rivers or riverbeds and finally become part of the plastic islands. In order to provide scientific evidence of this fact and promote citizen awareness, the aim of this work is to separate, characterize and quantify plastic waste found in areas with a high number of tourists in the Catalan Pyrenees and to share this information with citizens through some awareness-raising activities. For this plastic waste, commonly known to be plastic bags and water bottles, alternatives are proposed based on an eco-audit. This work is part of actions 3 and 4 of the Plastic0pyr project of the Interreg POCTEFA program.

Keywords: Plastic waste, Mountain tourism, FTIR-ATR, Eco-Audit, Citizen awareness-raising

2. RESUM

La majoria dels plàstics que es troben al mar formant les conegudes illes de plàstic han estat transportats des dels nuclis de població a través dels rius. Per aquest motiu, una de les primeres tasques a dur a terme és evitar que els plàstics arribin als rius i, per això, és necessari que els ciutadans tinguin consciència de la necessitat de fer una gestió adequada dels residus plàstics al final de la seva vida útil. Durant els darrers anys, s'han dut a terme diverses campanyes per conscienciar sobre una gestió adequada dels residus a les llars, però aquesta consciència sembla oblidar-se quan ens trobem fora de casa i sobretot a les zones d'oci o altres activitats com la indústria o l'agricultura. Aquests residus, que no es gestionen correctament, sovint arriben als rius o lleres i finalment passen a formar part de les illes de plàstic. Per tal d'aportar proves científiques d'aquest fet i promoure la conscienciació ciutadana, l'objectiu d'aquest treball és separar, caracteritzar i quantificar els residus plàstics que es troben a les zones amb un elevat nombre de turistes als pirineus catalans i compartir aquesta informació amb els ciutadans mitjançant una activitat de conscienciació ciutadana. Per a aquests residus plàstics, coneguts habitualment per ser bosses de plàstic i ampolles d'aigua, es proposen alternatives basades en una eco-auditoria. Aquest treball forma part de les accions 3 i 4 del projecte Plastic0pyr del programa Interreg POCTEFA.

Paraules clau: Residus plàstics, Turisme de muntanya, FTIR-ATR, Eco-Auditoria, Conscienciació ciutadana

3. INTRODUCTION

Since the 1950's, plastic production has increased consistently [1] because of its ideal characteristics (inexpensive, durable, lightweight, abundant, and can be produced in any desired shape) [2]. But for these same properties, a growing portion of plastic is used for single-use purposes [2]. Most single-use plastics are disposed of in landfill sites; however, plastic persists and if not properly stored, may later surface to become "debris" [3]. This results in an accumulation of plastic debris not only in terrestrial environments but also in open ocean, shorelines, remote islands, and deep sea [3]. Due to the chemical persistence of plastics and their mechanical fragmentation to small fragments and particles [4], plastic pollution imposes threats on aquatic life, ecosystems and human health [2]. These issues are further complicated because our current knowledge on environmental behavior and ecological impacts of microplastics is limited [5].

Most of the research on quantification of plastic pollution and its effects has been focused on marine environment [2]. The sources of this pollutant include loss from waste management streams, fishing operations, illegal dumping and natural disasters [6], but a significant amount of marine plastic debris comes from land-based sources and rivers are possibly their major transport pathway [4]. Riverine plastic transport remains relatively understudied in comparison to marine plastic litter [2] and it is important to increase the studies on plastic pollution in freshwater ecosystems to achieve a global knowledge and therefore, better solutions.

If the problem is approached at the root rather than at the outcome, the prevention of plastic reaching the rivers is essential. Therefore, citizen-awareness campaigns are necessary to inform people of the appropriate management of plastic waste at the end of its useful life.

3.1. PLASTIC0PYR PROJECT

PLASTIC0PYR: Strategies to reduce plastic pollution in mountain ecosystems (Project nº EFA340/19) is a project co-funded by the European Regional Development Fund (ERDF) which aims to sustainably prevent the accumulation of plastics in mountain ecosystems to make tourism compatible with the conservation of these ecosystems. The project is carried out in natural sites located in the Pyrenees of Catalonia, Andorra, and France [7].

To accomplish its goals, the project is divided in 5 complementary and interdisciplinary actions that are led by one of the partner organizations, as well as in close collaboration with the other organizations. Actions 1 and 2 focus on project management and communication activities respectively. Action 5 studies the circular economy for plastics. This research work is part of actions 3 and 4:

- Action 3: plastic's life cycle. Based on citizen surveys and data from an app developed by the project and hosted on the Marine Debris application, a qualitative study of plastics in nature and a life-cycle assessment (LCA) is conducted. Alternative materials are also studied and proposed.
- Action 4: plastics in mountain rivers. Plastic debris of various sizes on mountain rivers is collected, characterized, and quantified. Citizenship will also be part of this study by means of a citizen-science activity.

3.2. PLASTIC DEFINITION

Plastics are a long chain class of synthetic or semi-synthetic polymers that have high molecular weights, generally of about 10^4-10^6 g/mol [8]. The synthesis of the large molecular weight polymers is called polymerization, and it is the process by which monomer units (smallest unit in a polymer) are joined over and over, to generate each of the constituent giant molecules. Plastics are largely derived from coal and petroleum products [9] but can also be derived from substances such as cellulose, corn, cotton and natural rubbers, or can be produced by living organisms [10].

Based on their behavior under applied heat, they can be classified as thermoplastics, thermosets and elastomers. Elastomers are lightly cross-linked and at room temperatures, have elastic properties, but when heated, often undergo irreversible chemical changes. Because thermosets are highly cross-linked, when they are melted and then solidified, they stay solid,

and if reheated, they decompose rather than melt. Thermoplastics, on the other hand, have some cross-linking which allows them to not change their chemical composition when heated and thus can be molded repeatedly. For this reason, most single use plastics are thermoplastics and they can be melted and remelted allowing them to be recycled [11].

Thermoplastics are synthesized as spherical pellets or nurdles typically about 0.5–5 mm in size and then they are heated, extruded, or blow molded in the shape of its purpose. Additionally, additives are added depending on the purpose, such as flame retardants for electronic plastics or flexibility enhancers for packaging [2]. The most produced polymers in terms of market shares are high- and low-density polyethylene (HDPE and LDPE), polypropylene (PP), polyvinyl chloride (PVC), polyethylene terephthalate (PET), polyurethanes (PU), polystyrene (PS) and polyamides (PA). [12].

It has been seen that the fate of plastics in the environment varies with the plastic's properties. Travel distances, likelihood of accumulation, and degradation rate may vary considerably between plastic polymer type, shape, and size [2].

3.2.1. Plastic types

The density of the polymer affects the extend of transportation in aquatic environments, as well as its propensity for degradation [2]. The density values of the most produced polymers are shown in Table 1.

Table 1. Density values for the most produced polymers [12]

Polymer	Density [g/cm^3] [13] [14]	
	Min. value	Max. value
HDPE	0.940	0.970
LDPE	0.917	0.940
PP	0.900	0.910
PVC	1.150	1.700
PET	1.300	1.400
PUR (generic polyurethane)	0.871	1.42
PS	1.040	1.250
Nylon 6,6 (polyamide)[(a)]	1.140	

(a) From all the polyamides, the value of Nylon 6,6 is displayed because it will be used in the experimental section.

3.2.2. Plastic shapes

The shape of plastics affects exposed surface area, which can be important for transportation processes and chemical leakage. Shape groups observed in former studies are the hard plastics (solid pieces), pellets (preproduction), films (thin layered), and fibers (elongated lines) [2].

3.2.3. Plastic sizes

The size of the plastic is useful to determine the source and assess the final environmental impact. However, there is a lack of a cohesive terminology for plastic sizes. The most used terms are nanoplastics (<0.1 μm), microplastics (0.1 μm – 5 mm), mesoplastics (5mm – 5 cm), macroplastics (>5 cm) and megaplastics [2].

3.3 PLASTIC POLLUTION IN MOUNTAIN AREAS

Based on the results of the 1st campaign of the PLASTIC0PYR project, an abundance of $6.2x10^{-4}$ kg/m^2 macroplastics, and the same abundance for mesoplastics were found in the Catalan Pyrenees areas. Most of the plastics found were PE bags, either from mountain tourists or agriculture.

Microplastic debris can be classified as primary microplastics or secondary microplastics. Primary microplastics are directly released in the environment as small particles and secondary microplastics originate from the degradation of larger plastic objects [8]. Most of the microplastics found in the mountains are secondary microplastics from the degradation of the macroplastics and mesoplastics evidenced before, and others can be airborne [15].

3.3.1. Extraction of microplastics in inorganic and organic matrices

For the separation of microplastics from an inorganic and organic matrix, frequent in mountain samples, a few methods are being used but have not been optimized nor unified. Some of the methods carried out consist of a separation of the inorganic matrix by difference of densities and the removal of the organic matter via digestion or extraction with organic solvents [12] [16].

The separation by densities consists of the preparation of a solution with a desired density in which once poured over the sample, the denser particles sink and the less dense particles float. For microplastics in inorganic and organic matrices, many saline solutions with different

densities have been tested: NaCl (1.2 g/cm^3), $BaCl_2$ (1.3 g/cm^3), NaBr (1.4-1.6 g/cm^3), $ZnCl_2$ (1.5-1.7 g/cm^3), NaI (1.6-1.8 g/cm^3) and others [12]. The solutions of $ZnCl_2$ and NaI have higher densities therefore can also separate denser polymers like PVC and PET, but they are both classified as environmentally harmful [12].

The most common digestion for this type of samples is done with H_2O_2 30% but depending on the complexity of the organic matrix (seeds, stems) it is not entirely successful. The digestion with HNO_3 60% has been tested [16], and had a good performance.

3.4. CHARACTERIZATION OF PLASTICS

To study the impact of plastics on the environment, it is necessary to have an accurate identification and characterization of these. Fourier-transform infrared (FTIR) spectroscopy is very useful in the characterization of plastics as it gives information of the chemical functional groups of polymeric samples. Every polymer has its unique set of spectroscopic bands and that allows the differentiation of the polymer type. The vast database of spectra makes polymer identification an easy task. If a sample is a microplastic, the micro FTIR option may be used. FTIR has the advantage that it is a nondestructive method. For this analysis, operational modes such as attenuated total internal reflectance (ATR), Transmission, and reflectance can be used. ATR and reflectance modes do not need an extra step in sample preparation, but the Transmission mode does. Moreover, operational mode ATR gives a stable and reliable line data even for samples with uneven surfaces [8].

3.5. LIFE-CYCLE ASSESSMENT

Life cycle assessment (LCA) is a tool for measuring the environmental impacts of a product over different life cycle stages. LCA evaluates all the resources inputs, including energy, water and materials, and environmental loading including CO_2 emissions, solid wastes and liquid wastes of a product [17]. LCA's have four essential phases:

- Goal and scope definition, where the purpose, objectives, functional and system boundaries are defined.

- Inventory analysis, which consists of collecting all data relating to inputs, processes, emissions, etc. of the whole life cycle.
- Impact assessment phase quantifies environmental impacts and input resources based on the inventory analysis.
- Interpretation of the results calculated from the impact assessment phase and to recommend improvement measures.

Depending on the goal and scope of the LCA, as well as the database used for the inventory analysis, LCA's can vary for a same subject. Thus, the definition of this phases is crucial for the study of the environmental impacts of a product.

There are several methods for doing and LCA, but in this study we will be using the Eco-audit.

3.5.1. End of life potential

In the context of manufacturing and product lifecycles, the end of life refers to the final stages of a product's existence. Some end of life options can be landfill, combust (for energy recovery), downcycle, recycle, re-manufacture and reuse.

The end of life potential of a product is the difference between the obtention of the material from a virgin source and the obtention of the material from a recovered source. For example, if looking at the energy consumption for a product with recycling as its end of life, the end of life potential would be the difference between the energy consumption for the obtention of the material from a virgin source and the obtention of the material from a recycled source, taking into account that the process of recycling also consumes a certain amount of energy. The energy balance is usually negative, as less energy is needed to obtain the material from already existing sources than to obtain it from virgin sources. The CO_2 emission balance tend to also be negative except for when the end of life is energy recovery, which as a result of the combustion, emits CO_2.

4. OBJECTIVES

The main aim of this work is determining the typology of plastic waste found in high mountain areas with high human activity to carry out citizen awareness- raising activities, with the intention of reducing the amount of plastic waste.

To achieve this goal, some specific objectives are considered:

- Quantify and characterize mountain plastic waste to better understand the plastic pollution problematic.
- Develop a method for the separation of microplastics in complex matrices (inorganic and organic).
- Propose the most sustainable alternative for a plastic water bottle and a plastic bag based on the results of an eco-audit.
- Propose a citizen awareness-raising activity.

5. EXPERIMENTAL SECTION

The experimental part of this work consists of three main sections. The first section consists of the separation and characterization of plastics found in the Catalan Pyrenees. The second section analyzes a few common plastic objects that citizens bring to the mountains and some of their alternatives through an Eco-Audit. In the third section, a citizen awareness-raising activity is carried out.

5.1. SEPARATION AND CHARACTERIZATION OF PLASTICS SAMPLED IN MOUNTAIN AREAS

The PLASTIC0PYR team have done two sampling campaigns so far in the Pyrenees. The 1st sampling campaign was done in September and October of 2020, and 2nd sampling campaign in March and April of 2021. In each campaign, the sampling was done in some Pyrenees sites by CEAB and Universitat de Girona teams, and different types of samples were sampled: riverside samples, manta net samples, grab sampling, biofilm, sediment, and fish samples.

The manta net samples and the sediment samples can consist of a polymeric fraction in an inorganic and organic matrix. If the polymeric fragments have a visible size, like macroplastic and mesoplastics, they can be easily located and separated from the matrix. But in the case of microplastics, the separation from the matrix cannot be done simply by visual selection. In this study, various separation methods for microplastics in an inorganic and organic matrix are tested.

In addition to that, the quantification and characterization of riverside samples for the 2nd campaign is carried out. They include macroplastic and mesoplastic samples, but in this study only the macroplastic samples were analyzed.

5.1.1. Separation of microplastics (from 5 mm – 0.25 mm) in an inorganic and organic matrix

For developing a method of separation, it is important to understand the characteristics of the sample. In this case, the sample consisted of a mixture of organic matter (OM), which includes microplastics (MP) and inorganic matter (IM), mostly sand and gravel.

Based on the current methods found to separate microplastics in soils, two different methods were tested. Method 1 follows some of the steps explained in reference [12] and Method 2 is derived from reference [16]. For Method 2, the reference works with HNO_3 60%. Because of the availability, HNO_3 70% is used instead. For each method, various experiments were carried out tweaking the some of the conditions. To test the methods, a mix was made with sand as the IM, leaves as OM and microplastics cut in the laboratory (< 1 mm).

For weighing the samples, the electronic balance scale Gram AH-600 was used and for centrifuging the sample, the MSE Super Minor Centrifuge was used. As for chemicals, sepiolite from Sigma Aldrich (St. Louis, MO, USA) was used for the IM, in the digestions, H_2O_2 30% from PanReac AppliChem (Darmstadtand, Germany) HNO_3 70% from Fisher Chemical (Loughborought, UK) were used. For the saline saturated solutions, NaCl and $BaCl_2$ from Scharlab (Sentmenat, Spain) were used.

5.1.1.1. Method 1. H_2O_2 30% digestion and $BaCl_2$ saturated solution

For method 1, two experiments were done (Table 2):

Table 2. Conditions for the experiments 1 and 2 of method 1

Exp.	IM	OM	Nº of MP	Digestion conditions	Salt used	Reference article
1	Sepiolite	2 grounded leaves	1 of each polymer type[(a)]	H_2O_2 30%, 24h at 60ºC	$BaCl_2$	[12]
2	Park sand, sieved		3 of each polymer type[(a)]			

(a) PE, PP, PET, PS, PVC and NYLON.

The first experiment consisted of three replicates of 12 g of plastic-free sepiolite and two dry grounded leaves spiked each with 6 microplastic particles, 1 of each of some of the most common polymers: PE, PP, PET, PS, PVC and NYLON [9]. These polymers were photographed, and their spectra was recorded by FTIR-ATR for further inspection. 50 mL of H_2O_2 30% were added. The solution was left 24h with stirring and heating at 60ºC to remove all the OM except the microplastics. For separating the microplastics from the IM, the separation by densities technique was used. The sample was transferred to a beaker and 50 mL of a saturated solution of $BaCl_2$ (1.296 g/cm^3) was poured over. The solution was stirred for 5 minutes. It was not possible to see any microplastic floating because of the opacity of the solution and the presence of OM, therefore the solution was discarded and no microplastics were extracted.

For the second experiment, three replicates of 12 g of sieved (5mm – 0.25mm) park sand and two dry ground leaves were spiked with 18 microplastic particles, 3 of each polymer listed before. These polymers were also tracked as explained in the first experiment. 50 mL of H_2O_2 30% were added to each replicate and a standard sample with only the microplastics and hydrogen peroxide was also done to study the possible degradation of the polymers during the digestion. The digestion was done in the same conditions as experiment one. After 24h, the OM was partially digested. The solution was then filtrated, and the solid fraction was dried at 60ºC. Once the solid fraction was dry, it was transferred to a beaker and 50 mL of the saturated solution of $BaCl_2$ was poured over. The solution was stirred for 5 minutes. After collecting the floating microplastics, it was decided to filtrate separately the floating fraction of the solution and the sedimented fraction, and then dry it for 24h at 60ºC. For the floating fraction, the OM was manually separated for better identifying the microplastics. Then, each fraction was inspected through a magnifying glass and the detected microplastics were collected. The salt solution was saved for future separations in order to decrease the amount of liquid waste during the separation process.

5.1.1.2. Method 2. HNO_3 70% digestion and NaCl saturated solution

For method 2 (Table 3), the order of the steps of the method 1 was changed; first, the IM was separated, and then the sample was digested to remove the OM.

Table 3. Conditions for the experiments 1 and 2 of method 2

Exp.	IM	OM	Nº of MP	Digestion conditions	Salt used	Reference article
1	Park sand, sieved	2 grounded leaves	6 of each polymer type(a)	HNO_3 70%, 24h at 60ºC	NaCl	[16]
2			4 of each polymer type(a)			

(a) PE, PP and PS

In the first experiment for method 2, three replicates of 12 g of park sand and two dry grounded leaves were spiked with 18 microplastic particles, 6 for each of the 3 most common polymers found in the Pyrenees mountain areas based on this study: PE, PP and PS (section 6.2). The polymers were photographed, and their spectra was recorded by FTIR-ATR for further inspection. 70 mL of a saturated solution of NaCl (1.18 g/cm^3) were added to the sample and the solution was mixed for 10 minutes. Once mixed, the solution was placed in a centrifugation tube and was centrifuged at 2000 rpm for 30 minutes. Then, the solution was poured into a beaker and 50 mL of HNO_3 70% were added. This was left for 24h with stirring at 60ºC. The OM was degraded and the microplastics were filtrated and collected for analysis. 50 mL of HNO_3 70% were added to a standard sample with only the microplastics to study the possible degradation of the polymers during the digestion.

The second experiment for method 2, consisted of the same procedure as the first experiment but 4 microplastics of each polymer were put instead of 6, two replicates instead of three and controlling that the temperature never went above 65ºC.

5.1.2. Quantification and characterization of macroplastics sampled in mountain areas

The samples analyzed were sampled by CSIC-CEAB and Universitat de Girona teams in four sites of the Catalan Pyrenees: Catllar (CAT), Setcases (SET), Vallter (VAL) and Concròs (CON) during the 2nd sample collection campaign of the PLASTICOPYR project. The samples were divided by macroplastics and mesoplastics and their dimensions, shape and weight were recorded by the CSIC-CEAB team.

Once the macroplastic samples were received by the UB laboratory, a small fraction of each sample was cut, cleaned, and characterized by FTIR-ATR (PerkinElmer FT-IR Spectrometer Spectrum Two) with the program PerkinElmer Spectrum™ 10. If this database was not able to find a coincidence, it was checked with the program KnowItAll U Edition. Metallic samples were not characterized because FTIR-ATR cannot record their spectra and are not the subject of the study. The mesoplastic samples were not characterized in this study due to lack of time. The characterization of each sample was inputted in a spreadsheet for every site.

5.2. ECO-AUDIT

Based on the results of previous citizen surveys and plastic waste found in PLASTIC0PYR's first campaign, the technical committee of the project proposed two objects to analyze their life cycle by an eco-audit and propose some alternatives. The selected objects are a 100% virgin PET bottle and a 50-70% r-PE bag. The proposed alternatives for each of them are shown in Table 4.

Table 4. Selected objects for the Eco-audit study and their proposed alternatives.

Object	Alternatives
100% virgin PET bottle (500 mL)	25% r-PET bottle (500 mL)
	50% r-PET bottle (500 mL)
	Tetra Pak carton (500 mL)
	Aluminium can (500 mL)
50-70% r-PE bag	Woven PP bag
	Paper bag
	Cotton bag

The purpose of these analysis is to study the environmental impact of the production of these objects and their alternatives (in Spain) and determine which option is more sustainable. This eco-audit is carried out via the program GRANTA EduPack 2021R1 with the Level 3 database which include the full materials and process database, as used in industry.

The eco-audit software consists of the following steps to fill in with the information of the product:

- **Material, manufacture and end of life:** all the components of the product are added and the material of each of them selected. It has the option to indicate a percentage of recycled content, the mass, the primary process and the end of life. For every product the corresponding information was filled in. The information was extracted from reports, articles and in some cases, weighing and analyzing the products. See specifications for each product in Appendix 1.

  For the Tetrapak carton, two different recycling percentages data were extracted from the reference [18]. As shown in Table 17 in Appendix 1, the two are referred as entry 4 and entry 5 respectively.

  In reference to the water packaging items, the secondary or tertiary packaging is not considered. This is because often, when a water bottle is bought in mountain areas, they are bought in units.

- **Transport:** for all the product variations (of the water packaging and bag) it was narrowed down to once the product is made in Barcelona, it is transported to Setcases (one of PLASTIC0PYR's sampling points) where it could be theoretically sold in a market. The transport type is a 14 tonne truck with a total distance of 140km. The transport for the previous steps (production to logistics center, logistics center to distribution) was not considered because the information was not available.

- **Use:** this step is not necessary in this study. It has been assumed that the water bottle will not be refrigerated and the bag does not require additional energy for its use.

With this information, using its materials and process database, the program calculates the equivalent environmental burden of energy (MJ) and CO_2 footprint (kg). Based on the end of life selected, it calculated the end of life potential.

5.3. DEVELOPMENT OF A CITIZEN AWARENESS-RAISING ACTIVITY

As stated before, action 4 of PLASTIC0PYR project consists of the sampling of plastic waste in mountain areas along with citizen-science activities, operating as complimentary actions. The protocols that are being developed for both sample collection and separation of plastics are being thought of so that they can be carried out by citizens, therefore raising-awareness as well as increasing the data for a better knowledge on plastic waste in mountains.

Based on the scientific articles referenced before, a method for extracting microplastics from an inorganic and organic matrix has been developed (section 6.1). In this section, a prototype for this separation is tested with materials and reagents easily accessible to citizens and simplified and adapted the vocabulary, for it to be understood by everyone. This separation is thought to be for soil samples, which have inorganic and OM.

The activity was tested on one teenager and two adults. For a substitute of an actual mountain soil, plant soil was used. Table salt and tap water was used for the saline solution. For the material, two glasses were used per person, a pair of tweezers and a spoon.

In a glass, four teaspoons of plant soil were placed and spiked with 4 PP microplastics cut from a packaging container. In another glass, four finger-sized volume of water was poured, and a spoonful of table salt was added and then stirred. This process was repeated until a sediment of salt remained in the bottom of the glass even if stirring. At this point, the mixture was poured over the glass with the soil. The mixture was again stirred for 1 minute and then left for 30 minutes to set. Afterwards, the OM floated and the microplastics were visible. The microplastics were extracted with a pair of tweezers.

6. RESULTS AND DISCUSSION

6.1. DEVELOPMENT OF A METHOD FOR SEPARATING MICROPLASTICS FROM AN INORGANIC AND ORGANIC MATRIX

Two different methods were tested and the microplastics were recovered and characterized.

6.1.1. Method 1

In experiment 1 it was seen that the use of sepiolite as the IM did not work well because of its absorbent nature. The solution became a paste, and it was not possible to filtrate, so it was left to dry at 60ºC. Once dry, it was broken apart with a spatula to create a sandy sample that could be manipulated, but because of that and that its grain size very small, the solution became opaque and it was not possible to see any microplastics floating when the $BaCl_2$ saturated solution was added. No microplastics were extracted.

For experiment 2, it was decided to replace the sepiolite as the IM with sand from a park. Even though the sand might not be plastic-free, because the microplastics added were thoroughly tracked, the error from other surfacing microplastics was dismissed. 50 mL of H_2O_2 30% was added to the sand to see how it reacted and if it was possible to filtrate once the digestion was done. It was seen that the solution became dense and it filtrated very slow. It was then decided to sieve the sand through 1 mm and 0.25 mm grain sieves and keep the middle fraction to eliminate the smaller size grain sand which stayed in suspension and made the solution very opaque. The number of microplastics added was increased from 1 to 3 of each type of polymer because it was seen that 1 microplastic was not representative enough. With this changes, when adding the salt, the solution was clearer, but it was still hard to see the floating microplastics because of the remaining organic particles and the slight opacity of the solution. The microplastics recovered and characterized are shown in Table 5.

Table 5. Results of the microplastics extracted with method 1 in experiment 2 for the three replicates.

	R1	R2	R3
Total MP in sample	18[(a)]	18[(a)]	18[(a)]
MP extracted	2 (PS) 2 (PE) 2 (PVC) 2 (PET)	3 (PS) 2 (PE) 1 (PVC) 3 (PET)	3 (PS) 3 (PVC)
Total MP extracted	8	9	6
Recovery percentage	44.4%	50%	33.3%
Mean recovery percentage	42.6%		

(a) 3 PE, 3 PP, 3 PET, 3 PS, 3 PVC and 3 Nylon.

The standard sample with only the microplastic fragments and 50 mL of H_2O_2 30% showed no changes during the digestion in most microplastics but PVC, that became slightly opaque; therefore, it can be stated that H_2O_2 30% at 60ºC for 24h does not degrade the microplastics significatively and that if microplastics are not found after the procedure, is because of its inherent error.

Almost all the PS microplastics were found, and this can be attributed to the fact that the polymer selected was white, thus making the detection much easier. This also happened with PVC, as H_2O_2 changed its opacity make it more noticeable. For a better extraction, polymers could be tinted.

PP and Nylon microplastics were not found. The PP selected was transparent and very thin, so it was more prone to sticking to the walls of the beaker and going unnoticed. Nylon microplastics were fibers from tights and were very fine and got easily attached to the OM. As the OM was not entirely digested, probably the fibers got stuck to it and got removed together when filtrating.

6.1.2. Method 2

For method 2, some changes were done taking into consideration the previous results in method 1:

In the separation by densities, for selecting the proper salt three factors where determined: density, toxicity and usage. The density of the solution is associated with the type of polymer

that will float and ultimately, will be separated. The toxicity of the solution is very important in this kind of work where its aim is to understand and reduce contamination, so the method itself should be the most eco-friendly possible. The usage refers to how it performs in terms of facility of filtration and reaction with the sample.

As seen in the results of the macroplastic characterization (section 6.2), the most common macroplastics found in the Pyrenes mountain areas are PE, PS and PP. Because microplastics are mainly degraded macroplastics, it was assumed that the most common microplastics in the Pyrenes are PE, PS and PP.

Considering this assumption, more toxic and dense salts as $ZnCl_2$ (1.5–1.7 g/cm^3) or NaI (1.6–1.8 g/cm^3) were discarded because PET and PVC could be omitted. The most appropriate salt was decided to be NaCl, because it is environmentally friendly, has a density of around 1.18 g/mL which that allows the extraction of PE, PS and PP and is very easy to filtrate.

In method 1, the fact that the solution became opaque because of the IM, did not allow a good detection. For tackling this problem, the order of the steps of method 1 were inverted; a first separation of the IM was done centrifugating the solution, and then the OM was digested. This change made a very big difference and the solution was much clearer and the microplastics could be detected. Also, the solutions filtrated faster.

The other thing that did not allow a good detection was the remaining OM. For this method, the digestion was done with a stronger acid, HNO_3 70% and the OM was fully degraded. It was thought that the concentrated acid would degrade the filtrating paper when filtrating, but it was not an issue.

For the first experiment, two of the replicates were in a hotter setting (75ºC) and the microplastics got burnt. They looked black and some of them were attached to others. The burnt plastics were the most recovered because they were black and easier to identify.

All burnt microplastics, even de PS and PP, had a PE spectrum. When looked at it with a microscope, it was clear that some of them were attached together and others had tiny specs of something white. For this reason, a proper characterization could not be done and in Table 6 the polymer name it is not displayed.

Table 6. Results of the microplastics extracted in experiment 1 of method 2 for the three replicates

	R1	R2	R3
Total MP in sample	18[(a)]	18[(a)]	18[(a)]
Total MP extracted	15	10	14
Recovery percentage	83.3%	55.5%	77.7%
Mean recovery percentage	72.2%		

(a) 6 PE, 6 PP and 6 PS

The first experiment of method 2 recovered more microplastics than the method 1, but because some of the microplastics got burned, the method was repeated in experiment 2. This time 4 microplastics of each polymer were put in the sample instead of 6, only two replicates were done and the temperature controlled at 65ºC. The results are shown in Table 7.

Table 7. Results of the microplastics extracted in experiment 2 of method 2 for the two replicates

	R1	R2
Total MP in sample	12[(a)]	12[(a)]
MP extracted	4 PP 4 PE 4 PS	4 PP 3 PE 4 PS
Total MP extracted	12	11
Recovery percentage	100%	91.6%
Mean recovery percentage	95.8%	

(a) 4 PE, 4 PP and 4 PS

In experiment 2, all the polymers were extracted but 1 PE, and this is because the PE polymer was very fine and got stuck to the walls of the beaker. Table 8 shows the mean recovery percentage for each experiment performed.

Table 8. Mean recovery percentage of each method and experiment

	METHOD 1	METHOD 2	
	Experiment 1	Experiment 1	Experiment 2
Mean recovery percentage	42.6%	72.16%	95.8%

Method 1 had the worst performance of recovering the microplastics, and between the experiments of method 2, experiment 2 recovered almost all of the microplastics in the sample.

6.2. MACROPLASTIC CHARACTERIZATION RESULTS

For all the locations, the quantity of each polymer and its corresponding area, weight and abundance were calculated. For a global study of the Catalan Pyrenees, all the results have been added up together.

The samples classified as "x" or "unknown" represent the samples that their spectrum was not identifiable with the database used. Some samples were a combination of various polymers, and because of the complexity of separating them and calculating the proportionate area and weight, and the small amount found, these items are expressed as PE+PE, PE+PE-C, PET+PE, PET+PE+PP, PE+PP, PET+x, PE+PS. To simplify the graphics, the label "others" includes all the polymers with a <1% quantity of the total. This includes: PMMA, PE+PE, PE+PE-C, PET+PE, PET+PE+PP, PE+PP, PET+x, PE+PS, CELLULOSE and NYLON.

A total of 217 samples were characterized with a total surface area of 7.42 m^2 and 2.17 kg. The total area of sampling consisted of 4800 m^2 (1200 m^2 x 4 sites). Therefore, 0.15% of the total surface area sampled had macroplastics and with an abundance of $1.8x10^{-3}$ kg/m^2. In Table 9, the information for each site is displayed.

Table 9.Total nº of items, area (m^2) and weight for each site and for the total surface area

	CAT	SET	VAL	CON
Nº of items	48	53	115	1
MacP area [m^2]	3.17	2.80	1.32	0.13
Weight [kg]	0.77	0.45	0.85	0.10
Items/total area [items/m^2]	0.0400	0.0441	0.0958	0.0008
MacP area/total area [%]	0.26	0.23	0.11	0.01
MacP weight/total area [kg/m^2]	$6.4x10^{-4}$	$3.7x10^{-4}$	$7.0x10^{-4}$	$8.3x10^{-5}$

In the CON site, only 1 item was sampled. In CAT and SET, a similar number of items were sampled and in VAL, it doubled the items sampled. However, excluding the CON item, the VAL items have the least total area in comparison with the other sites, because the samples were much smaller. The site with the most abundance of macroplastics was VAL ($7.0x10^{-4}$ kg/m^2), followed by CAT ($6.4x10^{-4}$ kg/m^2) and SET ($3.7x10^{-4}$ kg/m^2). The site with the least abundance of macroplastics was CON ($8.3x10^{-5}$ kg/m^2).

6.2.1. Most common polymers by number of items

Based on number of items, the most common polymers were PE with 64% (140 items), PP with 10% (21 items), and 7% of PS (15 items) polymers. For every site, most of the samples found were PE, the majority of them being plastic bags. The percentage results are represented in Figure 1.

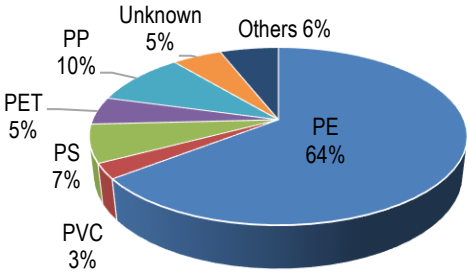

Figure 1. Percentages of each polymer by number of items

6.2.2. Most common polymers by surface area

For the surface area, the most common polymer was PE with 90% (6.65 m^2). The rest of the polymers comprised the other 10% of the total surface area. For every site, PE had the vastest surface area in comparison with the other polymers. The percentage results are represented in Figure 2.

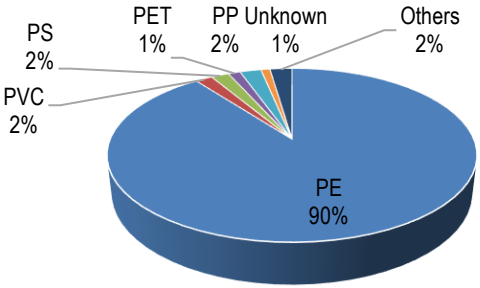

Figure 2. Percentages of each polymer by surface area (m^2)

6.2.3. Most common polymers by weight

Based on weight, the most common polymers were PE with 40% (0.87 kg), "others" with 32% (0.70 kg), which 24% (0.52 kg) was from a single item of PET-x, and 10% of "unknown" polymers. For the CAT and SET sites, PE was the most common item by weight (PE in CAT: 47%, PE in SET: 60%). In VAL, PE was the second most common item by weight (15%) topped by the single item of PET-x (62%), included in "others". The only item found in CON was a PE sample. The total percentage results in the 4 sites are represented in Figure 3.

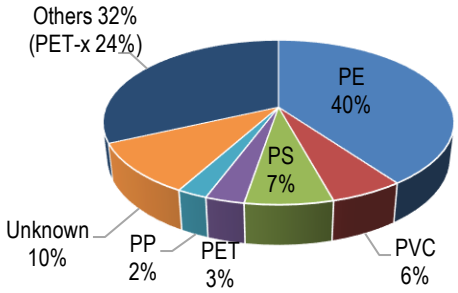

Figure 3. Percentages of each polymer by weight (kg)

6.3. ECO-AUDIT ANALYSIS

6.3.1. Water packaging results

All the alternatives studied are single use. For the Tetra Pak carton, two sources of end of life data have been found, entry 4 and entry 5 (section 5.2. and Appendix 1). The two of the variants are analyzed.

6.3.1.1. Energy

In this section, the environmental burden of energy (MJ) of each alternative is studied.

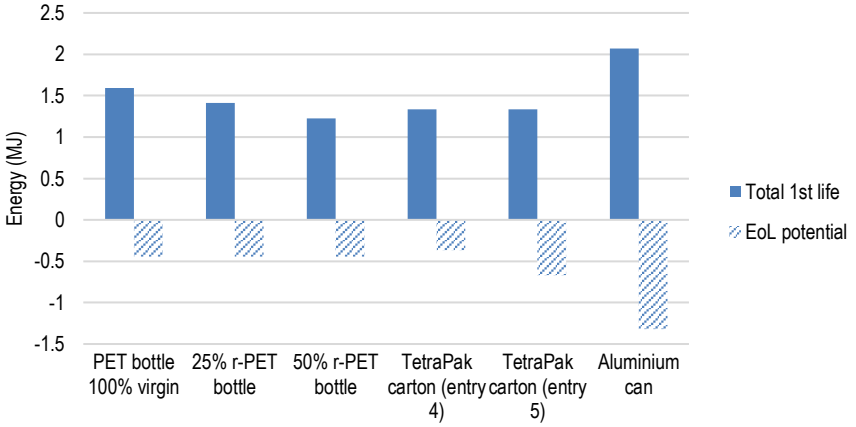

Figure 4. The environmental burden of energy is represented for each water packaging alternative.

As seen in Figure 4, the aluminium can is the alternative that consumes the most energy to produce (2.07 MJ), followed by the PET bottle 100% virgin (1.59 MJ) and the 25% r-PET bottle (1.41 MJ). However, because 86% of the aluminium cans in Spain are recycled, it is the alternative with the highest end of life potential (-1.32 MJ). This is followed by the Tetra Pak carton (entry 5) (-0.67 MJ) and all the PET bottles (-0.44 MJ).

6.3.1.2. CO_2 footprint

In this section, the environmental burden of CO_2 footprint (kg) of each alternative is studied.

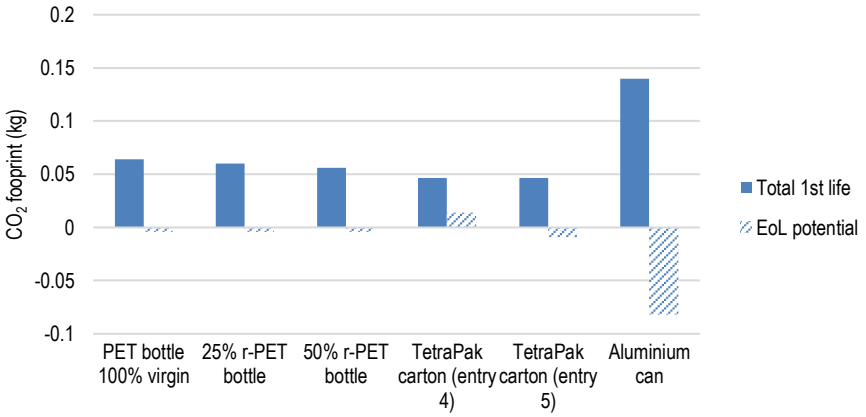

Figure 5. The environmental burden of CO_2 footprint is represented for each water packaging alternative.

In a similar pattern than the environmental burden of energy, in Figure 5 is seen that the aluminium can is the alternative that leaves a bigger CO_2 footprint (0.14 kg), also followed by the PET bottle 100% virgin (0.06 kg) and the 25% r-PET bottle (0.06 kg). Equally, the aluminum can is the alternative with the highest end of life potential (-0.08 kg), followed by the Tetra Pak carton (entry 5) (-0.01 kg) and all of the PET bottles (-0.004 kg).

The Tetra Pak carton (entry 4) in the process of end of life does not subtract the quantity of CO_2 footprint, it adds more (+0.01 kg). This is because 78% of the waste is incinerated and CO_2 is emitted from this combustion.

6.3.1.3. Total

In Table 10 it is shown the results for the environmental burden of energy and CO_2 footprint for each water packaging alternative for the 1st life and the total when the end of life potential is subtracted from the 1st life.

Table 10. Total environmental burden of energy and CO_2 footprint for each water packaging alternative.

	Total 1st life		Total (considering the EoL potential)	
	Energy [MJ]	**CO_2 footprint [kg]**	**Energy [MJ]**	**CO_2 footprint [kg]**
100% virgin PET bottle	1.59	0.0640	1.15	0.0598
25% r-PET bottle	1.41	0.0600	0.97	0.0558
50% r-PET bottle	1.23	0.0559	0.78	0.0517
TetraPak carton (entry 4)	1.33	0.0465	0.97	0.0604
TetraPak carton (entry 5)	1.33	0.0466	0.66	0.0375
Aluminium can	2.07	0.1398	0.75	0.0577

As it was seen in Figure 4 and Figure 5, the aluminium can alternative consumes the most energy and emits the most CO_2, but if the EoL is taken into account (energy: 0.75 MJ; CO_2 footprint: 0.057 kg), it is topped by the PET bottle 100% virgin in CO_2 emissions (0.059 kg) and in energy consumption (1.15 MJ).

The Tetra Pak carton (entry 5) has the lowest energy consumption (0.66 MJ) and CO_2 emissions (0.0375 kg).

6.3.2. Bag results

A first analysis has been done studying the 1st life for the plastic bag and its alternatives. The end of life potential is not studied nor compared between the alternatives because some of the alternatives are single use (plastic bag and paper bag) and others are multiple use (cotton bag and woven PP bag). In a second analysis, it has been calculated the number of uses the alternatives have to have to provide the environmental performance of the plastic bag in terms of energy consumption and CO_2 footprint.

6.3.2.1. Energy

In this section, the environmental burden of energy (MJ) for each bag alternative is studied (Figure 6).

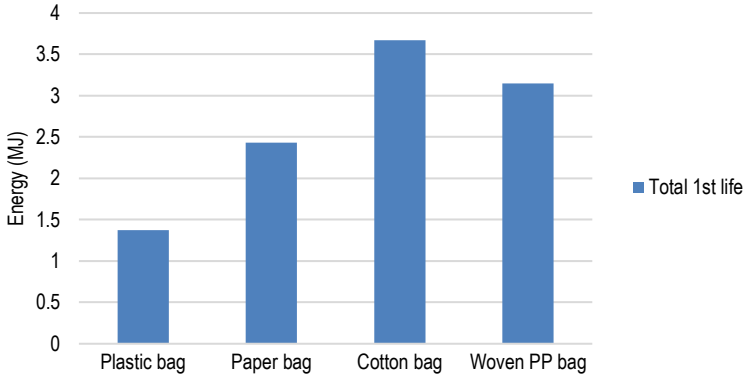

Figure 6. The environmental burden of energy is represented for each bag alternative.

The cotton bag is the alternative that consumes the most energy to produce (3.67 MJ), followed by the woven PP bag (3.15 MJ) and the paper bag (2.43 MJ). The option that consumes the less energy to produce is the plastic bag (1.37 MJ).

6.3.2.2. CO_2 footprint

In this section, the environmental burden of CO_2 footprint (kg) for each bag alternative is studied (Figure 7).

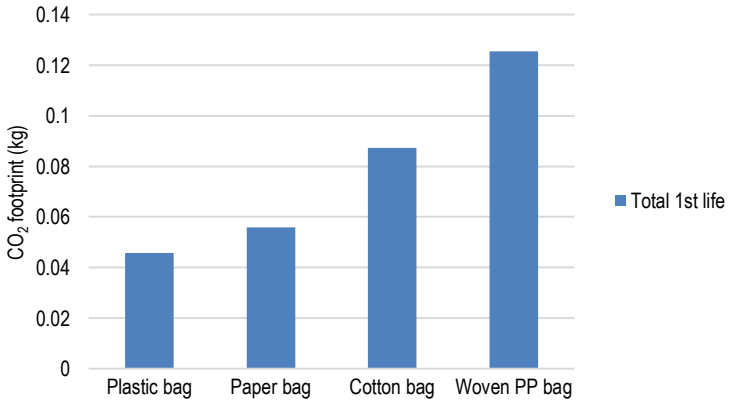

Figure 7. The environmental burden of CO_2 footprint is represented for each bag alternative.

The woven PP bag is the alternative that emits the most CO_2 when produced (0.12 kg), followed by the cotton bag (0.09 kg) and the paper bag (0.05 kg). The alternative that leaves the less CO_2 footprint when produced is the plastic bag (0.04 kg).

6.3.2.3. Total

Table 11. Results of the 1st life environmental burden of energy and CO_2 footprint for each bag alternative.

	Total 1st life	
	Energy [MJ]	**CO_2 footprint [kg]**
Plastic bag	1.38	0.0458
Paper bag	2.43	0.0558
Cotton bag	3.67	0.0872
Woven PP bag	3.15	0.1254

As seen in Table 11 and Figures 6 and 7, the plastic bag is the option between the alternatives that consumes the less energy and emits the lesser amounts of CO_2. For this reason, a study of the number of uses the alternatives need to provide the same environmental performance as the 50-70% r-PE bag follows.

Table 12. Result table that illustrates the number of times the different bag alternatives need to be used to provide the environmental performance of the 50-70% r-PE bag.

	50-70% r-PE bag		
	Energy [MJ]	**CO_2 footprint [kg]**	**Total**
Paper bag	5	4	5
Cotton bag	8	6	8
Woven PP bag	7	8	8

(a) if the use of the 50-70% r-PE bag is considered to be 3 times

In Table 12 it is seen that the alternative that will need the most uses to provide the same environmental performance in energy from the plastic bag is the cotton bag with 8 uses, followed by the woven PP bag with 7 uses and the paper bag with 5 uses. In terms of CO_2 footprint, the alternative with the most uses will have to be the woven PP bag with 8 uses,

followed by the cotton bag with 6 uses and the paper bag with 4 uses. In total, considering the energy and the CO_2 footprint, if a paper bag is used 5 times it will have the same environmental performance as the 50-70% r-PE bag, and the cotton bag and woven PP bag will need 8 uses.

If the durability of the alternative is taken into account, the paper bag is the most probable to get damaged before the 5 times of use and not reach the environmental performance of the plastic bag. Even though the cotton bag and the woven PP bag have to be used 3 more times than the paper bag to have an equivalent the environmental performance of the plastic bag, their properties make them more durable and the probabilities that they will get damaged before are much lower.

6.4. CITIZEN AWARENESS-RAISING ACTIVITY

The activity procedure was easy to follow and quickly understood by all the participants. All the materials were easily obtainable and unproblematic to manage.

The extraction of the microplastics itself imposed some issues; because the OM also floated with the microplastics, the floating fraction was very opaque. Despite that, some of the microplastics were still detected and extracted. The microplastics recovered from the prototype activity are displayed in Table 13.

Table 13. Results of the microplastics extracted from in the prototype activity

Participant	MP extracted
Teenager	3 out of 4
Adult 1	2 out of 4
Adult 2	0 out of 4

As show in Table 13, the teenager extracted the most microplastics. Adult 1 extracted half of them and adult 2 could not extract any as they did not see them because of vision issues. It is therefore stated that this activity is better for younger citizens with clearer vision.

Adult 2 found it hard to find the microplastics because they did not know what they were looking for.

Evidently not every size of microplastic can be found with this method because optical vision has its limitations. This procedure was done at home, but if applied in a school setting, a magnifying glass could be used to see the microplastics better or if available, a microscope.

7. CONCLUSIONS

The separation, quantification and characterization of mountain plastic waste and a citizen awareness-raising activity were successfully carried out and the following conclusions have been drawn:

- In the separation of microplastics from an inorganic and organic matrix, the ratio of the two fractions dictates the order in which the separation by densities (inorganic matter removal) and the digestion (organic matter removal) is carried out.
- For the matrix tested and the microplastics PE, PP and PS (from 5 mm – 0.25 mm), the method 2, which corresponded to a first separation by densities with a saturated solution of NaCl followed by a digestion with HNO_3 70%, extracted the most microplastics.
- Following the method 2, if the temperature goes above 65ºC, the plastics may get burned.
- The location of microplastics from visual input is best when the particles are colored. For future separations, the use of a tint is advised.
- The most common macroplastic found in the Catalan Pyrenees is PE, the majority of which is from plastic bags. Only 5% of the samples collected were PET macroplastics, thus the plastic waste generated from PET bottles is not the most predominant.
- Based on the CO_2 footprint and the energy consumption (with a certain scope), the most sustainable alternative from the 100% virgin PET bottle for single use is the TetraPak carton (if the end of life percentages are 80% recycled and 20% combusted, as in entry 5).
- For the bag options, all of the alternatives are more sustainable than the plastic bag if reused a certain amount of times to provide the same environmental performance than the production of the plastic bag.

- The citizen awareness-raising activity proposed has a high potential because a fraction of the microplastics can be extracted without specific laboratory material and at the same time educates on the problematic of plastic waste in mountains.
- As the activity worked best with younger people, it could be implemented in schools.
- As some of the participants could not find the microplastics because they did not know how they looked like, a field sheet must be done including photographs of real microplastics found in this type of matrices.

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9. ACRONYMS

ATR: attenuated total reflectance

CAT: Catllar

CON: Concròs

FTIR: Fourier-transform infrared

HDPE: high density polyethylene

IM: inorganic matter

LCA: life-cycle assessment

LDPE: low density polyethylene

MacP: macroplastic

MP: microplastic

SET: Setcases

r-PE: recycled polyethylene

r-PET: recycled polyethylene terephthalate

OM: organic matter

PA: polyamide

PE: polyethylene

PET: polyethylene terephthalate

PP: polypropylene

PS: polystyrene

PU: polyurethane

PVC: polyvinylchloride

VAL: Vallter

APPENDICES

APPENDIX 1: MATERIALS AND EOL SPECIFICATIONS

Water packaging

Material Specifications

Table 14. 100% virgin, 25% r-PET and 50% r-PET, 500 mL PET bottle specifications

Qty	Component name	Material	Recycled content [%]	Mass [kg][(c)]	Primary process [19]
1	Cap	PE-HD[(a)]	0	0.0013	Polymer molding
1	Ring	PE-HD[(a)]	0	0.0002	Polymer molding
1	Label	Cardboard	0	0.0009	Incl. in material value
1	Bottle	PET[(b)]	0[(d)] 25[(e)] 50[(f)]	0.0136	Polymer molding

(a) General purpose, molding & extrusion.
(b) Unfilled, amorphous.
(c) This data was obtained by separating the different components of a 500 mL PET bottle and weighing them. It is assumed that the three alternatives have the same weight.
(d) For the 100% virgin PET bottle.
(e) For the 25% r-PET PET bottle.
(f) For the 50% r-PET PET bottle.

Table 15. 500 mL Tetra Pak carton specifications [20]

Qty	Component name	Material	Recycled content [%]	Mass [kg]	Primary process
1	Polymer (sleeve)	PE-LD[(a)]	0	0.0041	Polymer extrusion
1	Paperboard (sleeve)	Cardboard	0	0.0027	Incl. in material value
1	Aluminium (sleeve)	Aluminium[(b)]	0	0.0007	Casting
1	Polymer (closure)	PE-HD[(c)]	0	0.0029	Polymer molding
1	Polymer (top)	PE-HD[(c)]	0	0.0039	Polymer molding

(a) Molding and extrusion.
(b) Commercial purity, S150.1:LM0-M, cast
(c) General purpose, molding & extrusion.

Table 16. 500 mL Aluminium can specifications [20]

Qty	Component name	Material	Recycled content [%]	Mass [kg]	Primary process
1	Aluminium can	Aluminium[(a)]	50%	0.0016	Casting

(a) Commercial purity, S150.1:LM0-M, cast

End of life specifications

Table 17. 500 mL End of life percentages for each water packaging alternative

Entry	Alternative	EoL	Percentage
1	100% virgin PET bottle[1]	Recycling	50.7%
		Energy recovery	15.5%
		Landfill	33.8%
2	25% r-PET PET bottle[1]	Recycling	50.7%
		Energy recovery	15.5%
		Landfill	33.8%
3	50% r-PET PET bottle[1]	Recycling	50.7%
		Energy recovery	15.5%
		Landfill	33.8%
4	Tetra Pak carton[18]	Recycling	21.4%
		Energy recovery	78.6%
5	Tetra Pak carton[18]	Recycling	80%
		Energy recovery	20%
6	Aluminium can[21]	Recycling	86%
		Landfill	14%

(a) Two different percentages for the Tetra Pak carton were found. In the table, both are displayed.

Bag

Material Specifications

All the bag alternatives have a similar volume. For the woven PP bag, the mass of the bag was divided by two to have a similar volume than the rest of the bag alternatives.

Table 18. 50-70% r-PE PE bag specifications

Qty	Component name	Material	Recycled content [%]	Mass [kg][(b)]	Primary process[22]
1	Plastic bag	PE-LD[(a)]	60	0.0245	Polymer extrusion

(a) Molding and extrusion.
(b) This data was obtained by weighing a 50-70% r-PE PE bag.

Table 19. Woven PP bag specifications [23]

Qty	Component name	Material	Recycled content [%]	Mass [kg][(b)]	Primary process
1	Woven PP bag	PP[(a)]	70	0.0685	Polymer extrusion

(a) Homopolymer, high flow
(b) This data was obtained by weighing a woven PP bag. Its weight was divided by two to have similar volumes in all alternatives.

Table 20. Paper bag specifications

Qty	Component name	Material	Recycled content [%]	Mass [kg]	Primary process
1	Paper bag	Cardboard	0	0.052	Incl. in material value

(a) This data was obtained by weighing a paper bag.

Table 21. Cotton bag specifications

Qty	Component name	Material	Recycled content [%]	Mass [kg][(a)]	Primary process
1	Cotton bag	Cotton fiber	0	0.077	Fabric production

(a) This data was obtained by weighing a cotton bag.