CHARACTERIZATION OF PLASTIC MATERIALS PRESENT IN MUNICIPAL SOLID WASTE: PRELIMINARY STUDY FOR THEIR MECHANICAL RECYCLING

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ABSTRACT

In the EU, 25.8 million tons of plastic wastes are generated each year and more than 30% end up in landfills. In Spain, this percentage rises up to 50%. Mechanical recycling is currently one of the best alternatives to reduce problems associated with poor management of plastic waste. In this paper, an analytical laboratory study of several samples of municipal plastic waste from Granada (Spain) was presented. The samples were supplied by the Waste Treatment Plant (Ecocentral). The study was based on the measurement of the moisture and dirt content of the selected plastic waste. Those parameters were determined by washing and drying the waste and analyzing the washing wastewater; in order to determine/justify the need of a washing step and a post-treatment of the washing water. The results showed that the differences in moisture and dirt content were significant between the different types of polymers, which could influence in the economic profitability of mechanical recycling. Polystyrene (PS) is the material that loosed the most weight while polypropylene (PP) loosed least weight. Moreover, the washing wastewater shows parameters that comply with the discharge regulations of Granada (Spain), except for the case of polyethylene film, whose wastewater would require pre-treatment prior to discharge. Overall, the results were satisfactory, as they show that most of the ordinary plastic waste can be recycled without high cost.

1. INTRODUCTION

Since the Second World War, plastic has been established as one of the essential materials in many areas of everyday life, gaining strength in sectors such as automotive industry, clothing and decoration. From the 1960s to the present day, the demand for plastic products has grown continously. The annual production of plastic has increased twenty-fold in the last fifty years. Among the most commonly used polymers today, different typologies can be cited (Table 1).

One of the major environmental problems is the large amount of plastic waste generated. Both, the production of this material and the incorrect waste management cause several environmental problems. One of those problems is the amount of oil needed to manufacture virgin polymers (up to 6% of world oil production). Other problems are greenhouse gas emissions during manufacture (more than 1% of the world total), low recycling rates of waste and dumping of this waste at sea (World Economic Forum

et al., 2016). It is estimated that 80% of the waste present in seas are plastics which come from land (Rojo-Nieto and Montoto, 2017). The problem is that it takes between 100 and 1000 years to degrade plastics, so they suppose a real threat for sea flora and fauna.

In 2015, 322 million tons of plastic were produced worldwide. Europe is the second largest producer behind China. Its production reached 58 million tons that same year. Of this amount, 25.8 million tons were introduced annually into the municipal waste stream, which supposed a 12.4% of the total municipal solid waste (MSW). A 30.8% of this plastic waste was deposited in landfills, 39.5% was utilized for energy recovery and 29.5% was recycled (PlasticsEurope, 2016). In Spain the situation is even worse than in the EU. In fact, the amount of plastic waste that ends up in landfills is over 50% in Spain (PlasticsEurope, 2015).

The statistics show that the production of plastics and the plastic waste recycling do not grow in the same way. Increasing the percentages of mechanical recycling would be

TABLE 1: Most commonly used polymers.

Society of the Plastics Industry (SPI) Code	Polymer	Applications
♻ 1 PET	Polyethylene terephtalate	Food packaging, carbonated soft drink bottles, water bottles, oil bottles, etc.
♻ 2 PEAD	High density polyethylene	Bags, detergent bottles, dairy bottles, etc.
♻ 3 PVC	Polyvinyl chloride	Pipes, cards, sanitary fittings, etc.
♻ 4 PEBD	Low density polyethylene	Bags, film, packaging, etc.
♻ 5 PP	Polypropylene	Food packaging, lids, reusable cups, etc.
♻ 6 PS	Polystyrene	Single-use plates and cutlery, yogurts, butter packaging, etc.
♻ 7 Otros	Other plastics	Multiple applications

a good alternative for reducing the amount of oil used and greenhouse gas emissions, as well as reducing landfill waste.

The aim of this study is the characterization of the various plastic materials existing in mixed municipal solid waste and to assess the need of the washing/drying steps and their potential impact as preliminary preparation steps on the global recycling process line.

2. MATERIALS AND METHODS

2.1 Materials

The raw material used in this study came from the municipal solid waste collected and treated at the Waste Treatment Plant (Ecocentral) in Granada (Spain), and corresponded to the fraction that had not been collected

selectively. Municipal solid waste in Granada is made up of different fractions (Figure 1), among which the organic matter (34.4%) stands out, while plastic represents 12.6% of the total. This information comes from periodical characterizations carried out in the plant.

Figure 2 shows the different fractions of the plastic waste. Among them, polyethylene film is the most important, representing approximately 43% of the total. Plastic waste from all fractions except for rigid high-density-polyethylene, HDPE (mainly bottles) and plastics belonging to the category "Others" were analysed in the laboratory. The category "Others" includes many multilayer plastics, fibres and other polymers which are not the focus of this study.

At the Waste Treatment Plant of Granada, plastic waste is mechanically pre-treated to separate one type from
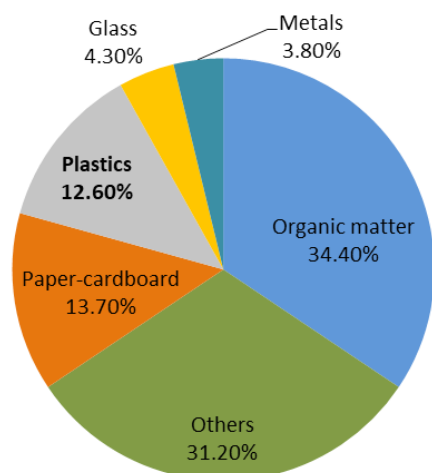
FIGURE 1: Composition of municipal solid waste in Granada (Spain).
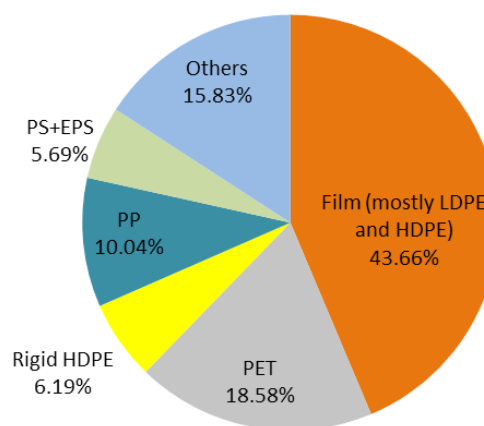
FIGURE 2: Composition of plastic fraction present in municipal solid waste of Granada (Spain).

another to facilitate the subsequent recycling and baling. The samples analysed in the laboratory came from the bunker corresponding to each type of plastic separated inside the plant (Figure 3). The plastic types analysed are:

- Polyethylene (PE) film (mostly bags and packaging film);
- Polyethylene terephthalate (PET) packaging;
- Polypropylene (PP) packaging;
- Polyestirene (PS) and Expanded Polystyrene (EPS) packaging.

2.2 Methods

The first step was the characterization of the plastic waste in the Waste Treatment Plant of Granada (Spain). The plastic waste was then transferred to the laboratory, where a series of analyses were carried out in the following order (Figure 4): 1) measurement of moisture content, 2) grinding, 3) washing and 4) measurement of dirt content. In parallel, the wastewater from the washing of each type of plastic material was analyzed, measuring the dissolved solids content, the total solids content and the chemical oxygen demand (COD).

2.2.1 Characterization of raw material in the treatment plant and in the laboratory

The purpose of this characterization was to determine the type of polymer that make up the waste material in order to separate and classify it. Several techniques were used for this purpose. At the municipal waste treatment plant, the plastic waste obtained from the bunkers was analyzed directly by Near Infrared Spectroscopy (NIR) using a portable Panatec Thermo Scientific microPhazir AG, with a wavelength range of 1600-2400 nm. The analysis was performed on the materials while they were in the waste stream, i.e. dirty and wet. This portable NIR spectrometer gives a reference spectrum with a correlation coefficient that indicates the similarity between the two spectra, appart from the measured spectrum.

Only the measurements with coefficients higher than 0.90 were considered. This value indicates that the material of the polymer can be considered to be the same as the reference material. However, this equipment does not provide the numerical values of the absorption peaks. In addition, moisture and dirt present in the material can lead to erroneous absorption peaks, which do not correspond to the polymer.

Thus, in the laboratory, this technique was complemented with Fourier Transform Infrared spectroscopy (FTIR), once the materials were washed and dried, to avoid disturbances caused by moisture and dirt. In addition, this equipment provides the values of the main absorption peaks, which allows comparing them with those in the literature (Bozaci et al., 2012; Rodríguez-Bruceta et al., 2014; Smith, 1999; Vahur et al., 2016; Zieba-Palus, 2017) and confirming the type of polymer that compose the waste.
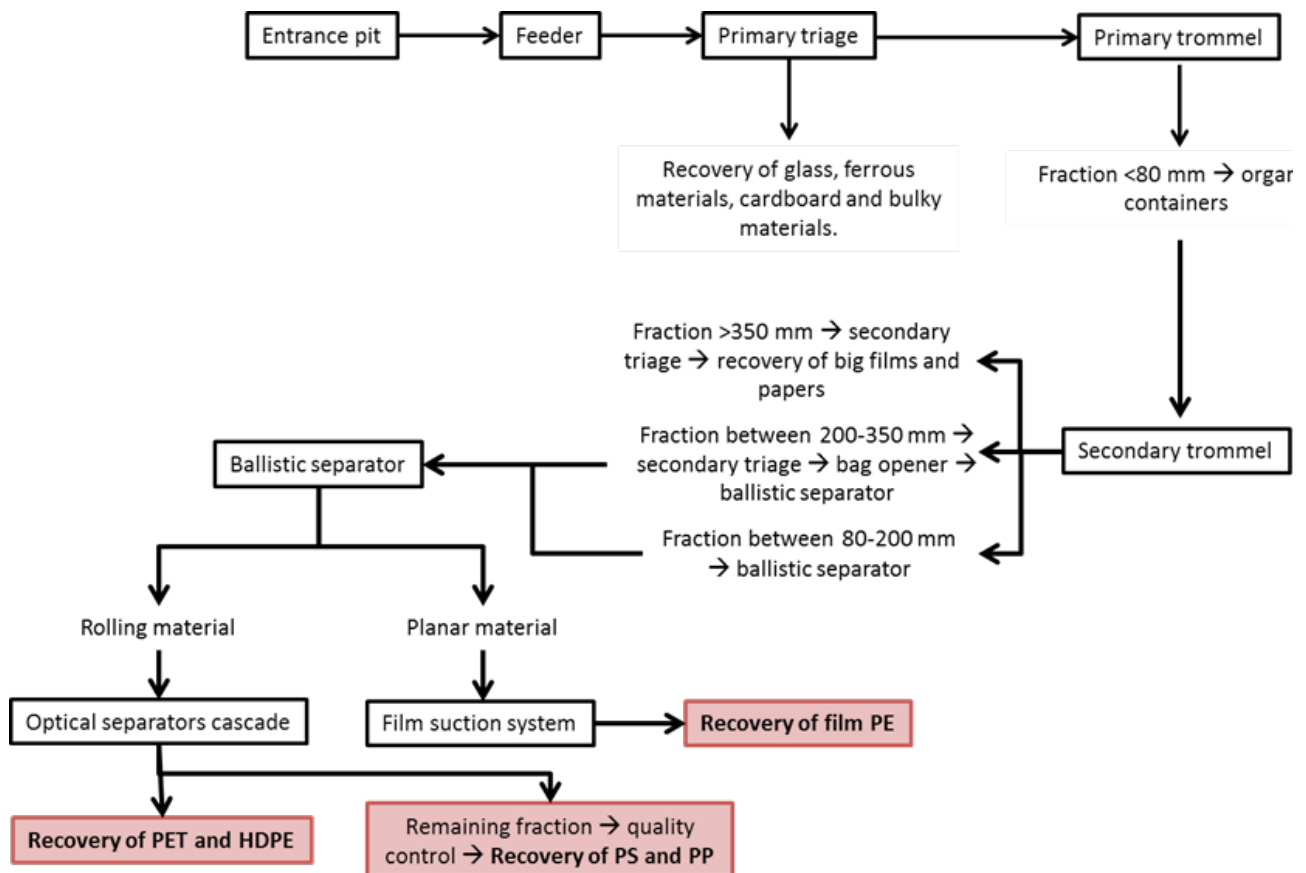

FIGURE 3: Scheme of mechanical pre-treatment carried out inside the Waste Treatment Plant of Granada (Spain).
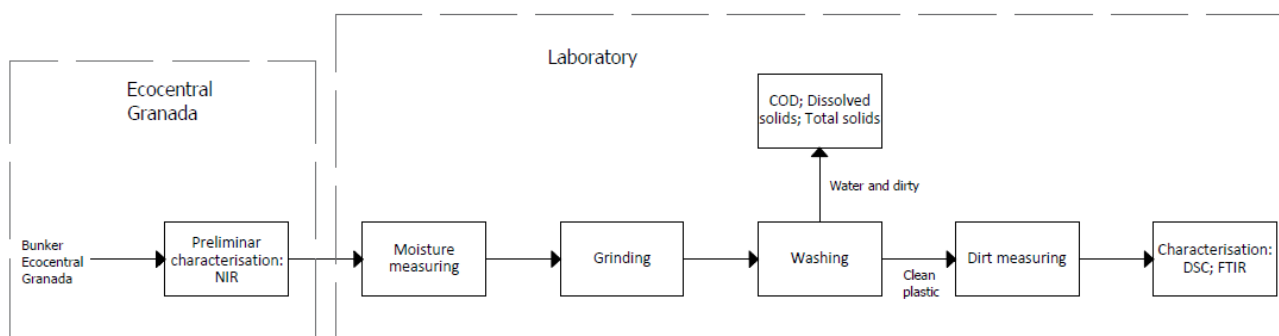
FIGURE 4: Experimental sequence of the study.

The analysis was performed with a Perkin-Elmer FT-IR Spectrometer model Spectrum 65, which has a wavelength range of 4000-400 cm^{-1} and a resolution of 2 cm^{-1}.

In the laboratory, tests were also carried out using Differential Scanning Calorimetry (DSC) on the samples in order to obtain the melting points of the different polymers that compose the plastic waste, a technique that is also widely used to complement the previous ones. The analysis was performed on Perkin-Elmer Thermobalance model STA 6000, in accordance with the standard ISO 11357-3:2018.

2.2.2 Determination of moisture

Moisture content was determined according to standard UNE-EN ISO 18134-3:2016 using a drying oven at a temperature of 105 ± 2 °C during 24 hours.

2.2.3 Washing process

Three samples of each of the plastic materials described in section 2.1 were taken and washed. In order to determine the amount of water used in the washing process, several companies expert in the design of washing machines for the mechanical recycling of plastic were consulted. The minimum quantity acceptable for a correct washing is 1 L per 100 g of plastic. This quantity was complied for all the polymers except for EPS. A solid:liquid ratio of 1:40 was used for this material, because it is very bulky and it has a very low density, so it takes large amounts of water to submerge it completely and wash it properly. The duration of each wash was 30 minutes. Similar to our methodology, different water quantities were reported for diverse polymers. For example, after performing a cradle-to-gate life-cycle inventory (LCI) (EU data) for polymer recycling from post-consumer sources, Hopewell et al., (2009) found that the amount of consumed water was 32 kL/tonne for high-density polyethylene (HDPE), 43 kL/tonne for polypropylene (PP), 66 kL/tonne for (PET) and 140 kL/tonne for polystyrene (PS).

Washing was carried out using water from the urban network, which has a total dissolved solids (TSD) of 74.4 mg/l.

With regard to temperature, the plastic was washed at room temperature (23-25°C) and using hot water (60°C) following the recommendations given by other authors in the literature (Al-Sabagh et al.,, 2016; Awaja and Pavel, 2005; Kratofil et al., 2014; Luijsterburg, 2014; Rodríguez-Bruceta et al., 2014) (Table 2).

2.2.4 Determination of dissolved solids, total solids and COD in wastewater

After the washing, the wastewater was analyzed and the following parameters were measured: dissolved solids, total solids and chemical oxygen demand (COD). These parameters were measured because they are the ones that determine the quality of the wastewater before it is discharged into the network. The methodology used to determine each of these parameters is described below:

- Dissolved solids: They were determined gravimetrically by vacuum filtration, according to UNE 77031:2015, using a Filter-Lab 1240 filter, with a pore size of 14-18 μm. The filtered water was then allowed to dry in a 105°C oven and the remaining solid residue was weighed. The result is expressed in g/L.
- Total solids: They were determined by the difference in weight between dirty plastic and clean plastic. The result is expressed in g/L.
- COD: This parameter has been determined in accordance with ISO 6060:1989, using the dichromate method. The chemical oxidizer was added to the wastewater and boiled. It remained in this state for a time, after which it was reduced through a reducing agent. Finally, it was evaluated in order to measure the amount of chemical oxidant consumed, expressed in mg/L of equivalent oxygen.

2.2.5 Determination of dirt loss

Dirt loss was determined considering the weight loss of the plastic after the washing process. This parameter corresponds to the total solids parameter, as both measure the same solid fraction, but expressed in different units.

TABLE 2: Washing conditions.

Polymer	Temperature	Ratio (plastic:water)
PE film	Room; 60°C	1:10
PET packaging	Room; 60°C	1:10
PP packaging	Room; 60°C	1:10
PS packaging	Room	1:10
EPS packaging	Room	1:40

This is why the same values are presented in Table 3 for dirt loss as in Tables 4 and 5 for total solids.

The loss of dirt is expressed in percentage.

3. RESULTS AND DISCUSSION

3.1 Identification of polymers existing in raw material

The infrared spectra obtained by the NIR technique in the plant revealed that the majority of plastic materials analysed were composed of PE, PET, PP, PS, EPS and PA. This result coincides with the characterization presented in Figure 2, so it can be stated that the majority of plastic wastes contained in MSW are composed of PE, PET, PP, PS, EPS and PA (Figure 5).

However, due to the heterogeneity and characteristics of the sample in the field analysis with the NIR equipment, in some cases FTIR technique in the laboratory was performed as well (Figure 6). The spectra obtained were compared with spectra from the literature of pure polymers (Bozaci et al., 2012; Rodríguez-Bruceta et al., 2014; Smith, 1999; Vahur et al., 2016; Zieba-Palus, 2017). It was found that the absorption peaks coincided with those described for PE, PET, PP, PS and PA by these authors. In the case of PE, in the characterization with FTIR, it was possible to differentiate between high density polyethylene (HDPE) and low density polyethylene (LDPE), since there is at least one different absorption peak between both, according to other authors (Kochetov et al., 2017; Rodríguez-Bruceta et al., 2014; Smith, 1999).

Another method used to verify the type of polymer, as described in section 2.2.1, was the DSC. This method is considered as the most decisive in polymer characterization, since it provides the melting temperature. All the polymers analyzed gave consistent results except PA. In the characterization phase, this material was detected only in multilayer products, containing PET or PE in addition to PA, and the layers could not be separated. This resulted in numerous overlapping and difficulties to identify fusion peaks in the DSC. For that reason, the DSC of these materials has not been included in the results.

Figure 6 shows the results obtained in the laboratory. In the case of PET, the melting temperatures reported by other authors (Awaja and Pavel, 2005; EAG Laboratories, 2018) were between 250-265°C, although slightly lower temperatures can be obtained,which is consistent with the results presented in this paper. For PP, the melting temperature is usually higher than 160°C (Hindle, 2018; Mofokeng et al., 2011), coinciding with the DSC shown in Figure

6. In the case of PS and EPS, the temperature obtained in the DSC is the glass transition temperature since they do not have melting temperature because they are amorphous polymers (Oliveira et al., 2013; Parres-García, 2005). This value is usually around 100°C, which is in accordance to the value obtained in the present investigation.

It should be noted that, within the PE, the DSC technique could be used to distinguish between HDPE and LDPE whose melting temperatures are different. HDPE may have melting temperatures between 120-130°C (Chianelli et al., 2013), but in most cases the values are closer to 130°C or even higher, such as those obtained by some authors (Araújo et al., 2008; Shnawa et al., 2015) and in the DSCs presented in this article. On the other hand, the melting temperature of low-density polyethylene is usually between 115-125°C (Batra, 2014), but it can fluctuate slightly above or below these values since other authors have obtained melting temperatures of 113°C or 127°C (Ashraf, 2014; Poley et al., 2004). These values are also in accordance with those shown in Figure 6 for the LDPE.

All the results obtained by these three techniques allowed to separate and quantify precisely the composition of the fraction of plastic material contained in the MSW, as well as to confirm the results of Figure 2.

3.2 Moisture and dirt content of plastic materials

Table 3 shows the data related to moisture and dirt obtained for all types of plastic materials separated from mixed municipal solid waste of the province of Granada (Spain). The samples with the highest moisture content were PS, EPS and PE film. Other authors such as Carranza et al.,, 2010 obtained moisture values of 10-20% for PE film waste from greenhouses.

With regard to the dirt present in plastics, it was found that most of it was made up of organic matter (soil, plant debris, etc.), paint and chemical residues, as well as labels and glue residues. Organic matter was more abundant in PE waste, paint and chemicals were present in PP waste, while labels and glue residues were more abundant in PET, PS and EPS. The labels were removed after the material had dried, as they were easily detached. After removal they were weighed. They represent between 10-14% of the plastic material and they are therefore an important parameter to take into account for mechanical recycling. Organic matter and paint residues were removed during washing.

The waste that contains the most dirt is PE film, followed by PS. PP had very low dirt loss values. This causes the viability of mechanical recycling and the quality of the final product can vary from one material to another. The

TABLE 3: Moisture and dirt content at different temperatures for each polymer.

	PE	PET	PP	PS	EPS
Moisture (%)	11.78	8.90	1.58	20.98	16.10
Dirt (%) at room temperature	13.79	8.50	2.65	10.20	7.91
Dirt (%) in hot water	13.17	6.45	1.53	--	--

TABLE 4: Characteristics of washing water at room temperature.

	PE	PET	PP	PS	EPS
Total dissolved solids (g/l)	5.64	3.52	0.59	5.71	1.00
Total solids (g/l)	13.79	8.50	2.65	10.20	7.91
COD (mgO$_2$/l)	1920	851.57	208.67	526	340.25

TABLE 5: Characteristics of hot washing water.

	PE	PET	PP	PS	EPS
Total dissolved solids (g/l)	5.68	2.04	0.40	-	-
Total solids (g/l)	13.17	6.45	1.53	-	-
COD (mgO$_2$/l)	1733	510.29	210	-	-

loss in weight of dirt is not necessarily determined by the temperature of the washing.

Washing with hot water did not imply a greater loss of dirt in terms of weight. In fact, the opposite effect was observed (Table 3). It was found no significant difference in the loss of dirt from the material when washing PE, PET and PP at room temperature or in hot water. Therefore, PS and EPS were washed only at room temperature, in order to save water and electricity. However, the use of hot water made it possible to better remove paint and glue residues and improved the loss of fat, giving the washed material a brighter appearance.

Differences in overall weight loss were significant between different types of waste: While PP had an overall weight loss of approximately 4%, the weight loss measured for PS was 30% of its weight after drying and cleaning by water. These results are of special interest from the point of view of the recycling process of these materials.

3.3 Determination of dissolved solids, total solids and COD on washing water

Tables 4 and 5 show the characteristics of the washing waters at room temperature and hot temperature. Dissolved solids, total solids and COD were determined for the different kinds of water. Washing in hot water did not imply a greater presence of dissolved solids in the water.

The wastewater will need to be pre-treated depending on the legislation concerning discharge to the sewerage networks of each city or country.

In the case of Granada, the Spanish province where this study was carried out, both the values of dissolved and total solids and the COD value comply with the limits established by the Municipal Ordinance regulating discharges to the sewage network of the Granada City Council, except in the case of polyethylene film. The COD values obtained for this material exceed the limits (1400 mgO$_2$/L), so its wastewater will need to be pre-treated prior to discharge. This pre-treatment would include several steps of decantation and aerobic degradation designed in order to achieve the discharging limits. The need of these steps will increase the initial cost of the recycling plant. In those cases, where a separate discharging network for industrial waste stream exists, purifying equipment will not be necessary.

4. CONCLUSIONS

The study carried out in this work is the first part of an investigation for the mechanical recycling of different types of plastic materials in order to promote this type of
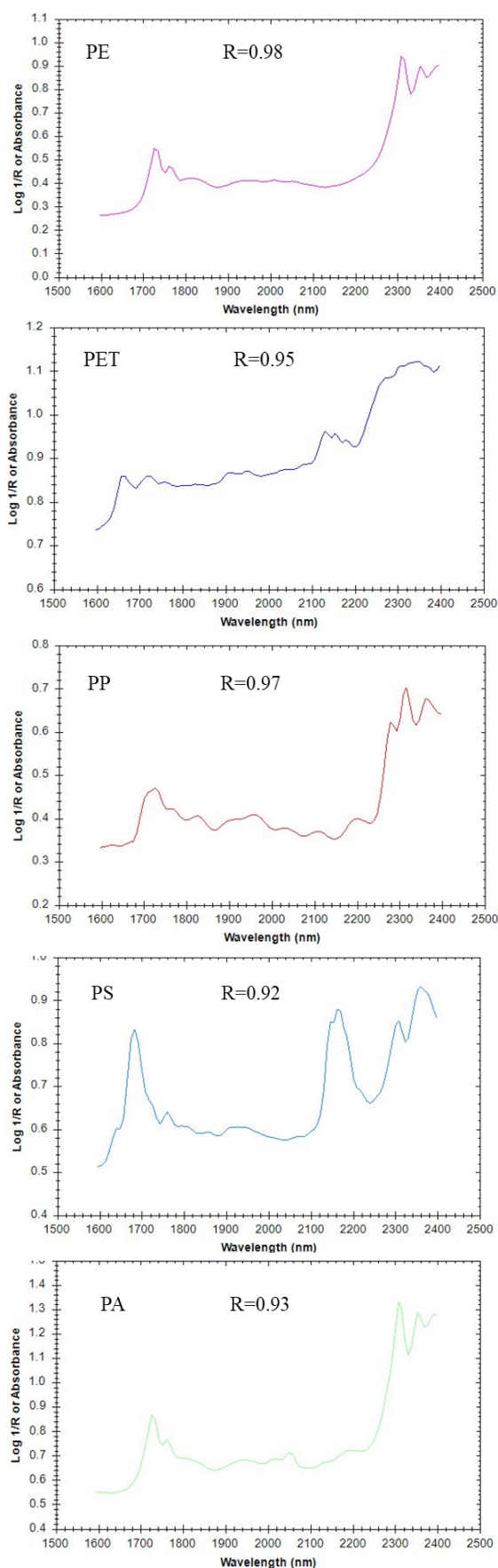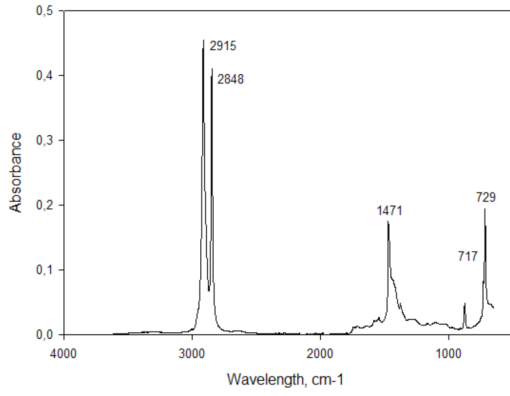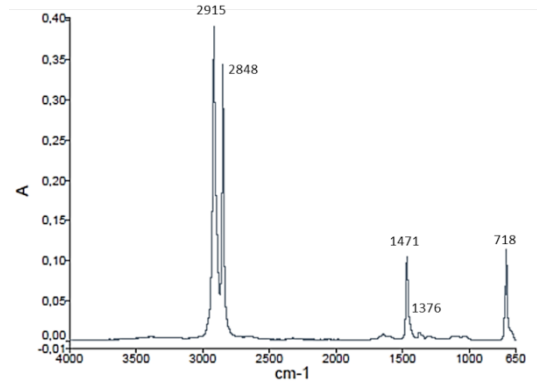

FIGURE 5: NIR spectra of the different plastic waste identified in MSW of Granada (Spain).

A) FTIR HDPE

B) FTIR LDPE

C) FTIR PET
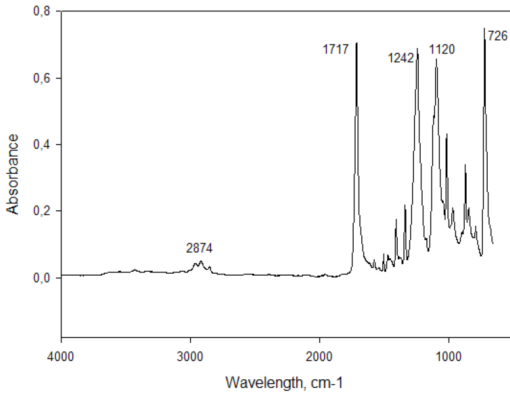
D) FTIR PP
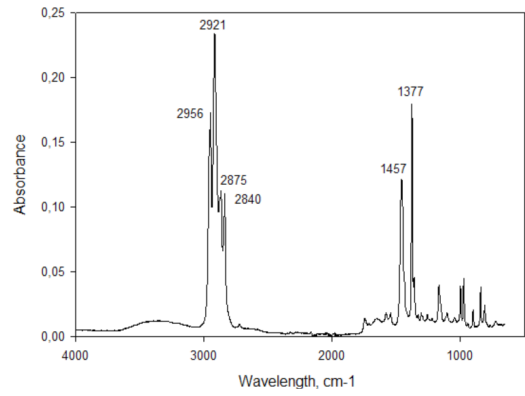
E) FTIR PS/EPS
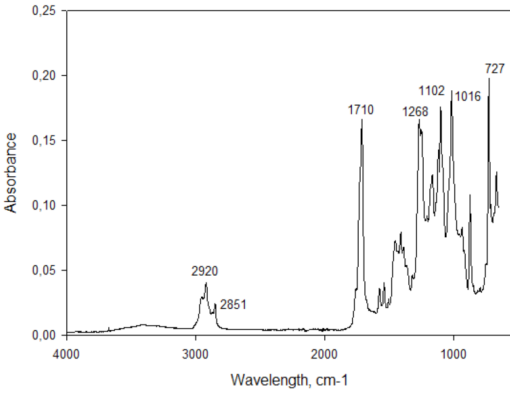
F) FTIR PA

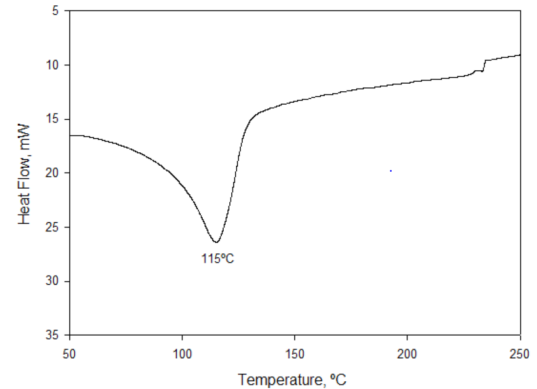
G) DSC HDPE

H) DSC LDPE

I) DSC PET

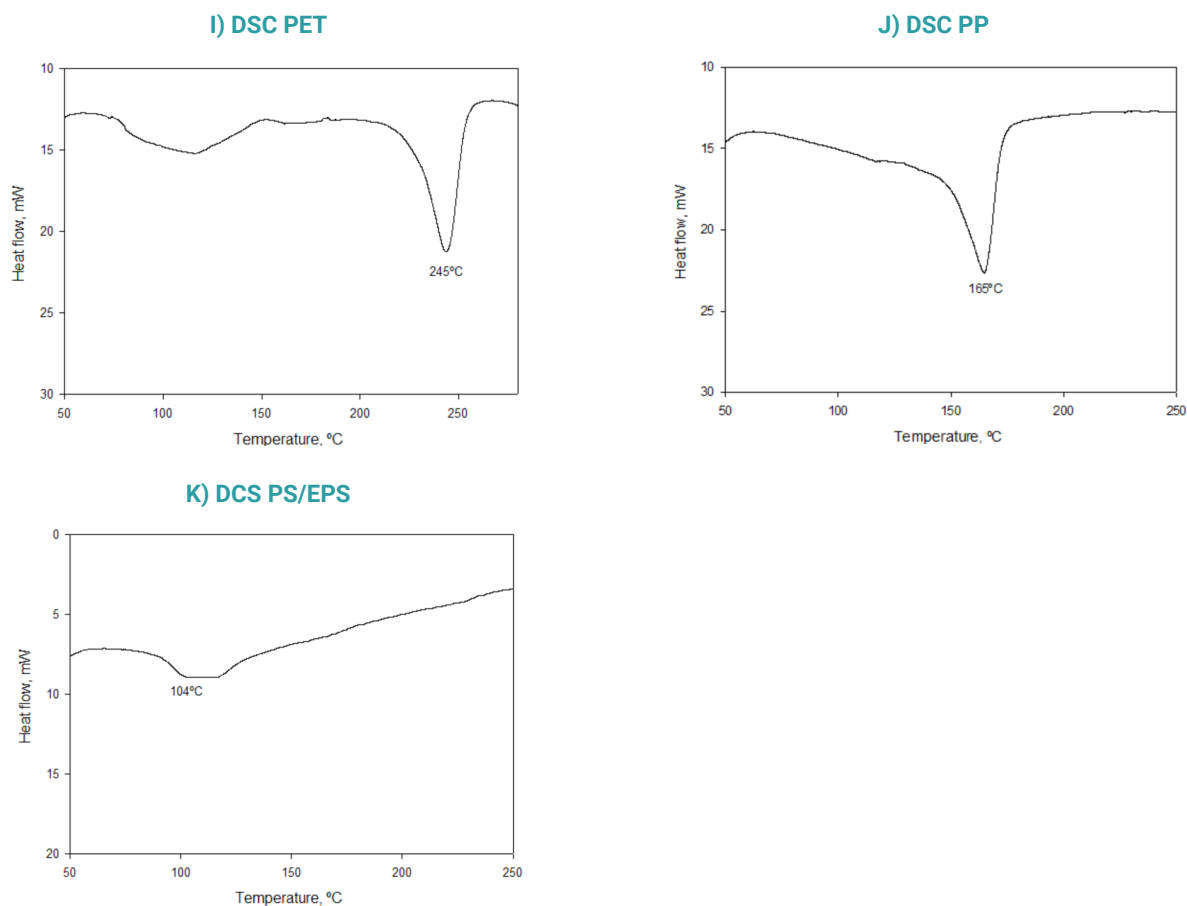## J) DSC PP

K) DCS PS/EPS
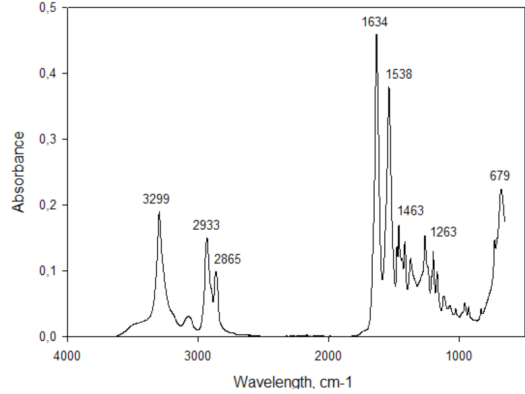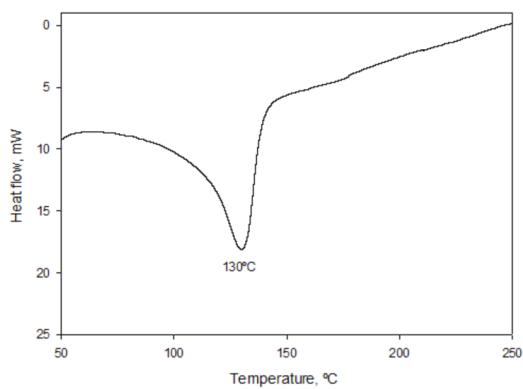
FIGURE 6: A-E) FTIR spectra obtained in laboratory of plastic waste present in MSW of Granada (Spain); F-I) DSC obtained in laboratory of plastic waste present in MSW of Granada (Spain).

recycling among plastic waste from mixed municipal solid waste. Mechanical recycling is a good alternative today to reduce the amount of waste that ends up in landfills in order to reduce the amount of oil needed to make virgin polymers and to contribute to reducing greenhouse gas emissions.

The characterization of the raw material carried out with various Infrared Spectroscopy and DSC techniques showed that the plastic fraction of the MSW from Granada (Spain) was composed mainly of PE, PET, PP, PS and PA.

With regard to the data found during the washing process, moisture and dirt in the waste were key factors in mechanical recycling, as it can account for up to 30% of its weight. This affects the performance of the process depending on the waste in each case. PS is the material that contains the most moisture and PE the most dirt. PP is a material with less moisture and dirt in general and for that reason it loses little weight during the process. It is important to take these parameters into account because depending on the final treatment that the recycled product will have (injection, blowing, extrusion, etc.), the previous stages of washing and drying may be decisive. It was also concluded that temperature was a non-significant factor in the amount of dirt loss in terms of weight, but it did allow better removal of paint, fat and glue. From the point of view of feasibility, materials such as polyethylene film can

be washed at room temperature to reduce energy costs, since their main source of dirt is organic matter (plant debris and soil). Finally, it should be noted that, in most cases, the waste water from the plastic washing process would not require further treatment for discharge in Granada (Spain). However, it would be necessary in the case of the PE.

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