Morpho-structural and thermo-mechanical characterization of recycled polypropylene and polystyrene from mixed post-consumer plastic waste

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ABSTRACT

In this work, a complete sorting and characterization of dirty non-recyclable mixed plastic (plasmix fraction) from municipal solid waste was performed. The procedure comprised a visual inspection and identification of the materials presented in the mixed plastic bales, and subsequent moisture and dirt content determination. Afterwards, both polypropylene and polystyrene fractions obtained from the sorting process were recycled and physico-mechanically and thermally characterized for the assessment of recycled material quality. The dirty non-recyclable mixed plastic from municipal solid waste was composed of polypropylene (28.89 %), polyethylene terephthalate (22.02 %), polystyrene (9.65 %) and rigid polyethylene (4.68 %). Regarding the moisture and dirt content of the mixed plastic fraction, the results were highly variable, mainly due to the heterogeneity of the material, its origin, atmospheric conditions, etc. The average sum of both parameters (moisture and dirt) was around 15 %. The mechanical properties were within the expected range for each type of polymer, noting that polystyrene recycled materials are more brittle than recycled polypropylene. The recycling of non-food polypropylene wastes yielded a material with higher impact resistance (70 kJ m^{-2}) and Young's modulus (1934 MPa) as compared to that obtained from the recycling of food packages. Concerning the polystyrene recyclates, it is worth mentioning that recycled materials obtained from foamed residues (expanded polystyrene and extruded polystyrene showed approximately twice the tensile strength with respect to the recycled materials from high impact polystyrene and general-purpose polystyrene residues. In some cases, infrared and thermal analyses revealed cross-contamination with traces of other polymers.

1. Introduction

Modern society needs many different types of materials, and often these materials are chosen based on their efficiency or their synergies within a combination of components for a system or a product. One of the most widely used materials is plastic, which has become indispensable due to its versatility and capability, offering customized solutions for a wide variety of products, applications and sectors [1].

In 2020, global plastics production reached approximately 369 million tonnes, with China being the largest producer (32 % of global production). In Europe, plastics production almost reached 55 million tonnes in 2020. The most demanded type of plastic is polypropylene (PP), followed by polyethylene (PE) in all its forms: low density polyethylene (LDPE), high density polyethylene (HDPE), etc. In terms of demand by sector, packaging and construction are the largest consumers of plastic [2].

Over the last 60 years, plastic has brought economic and social benefits. However, the excessive use of disposable products, together with their non degradability, has led to an exponential increase in the amount of plastic waste resulting from land-based and maritime activities, giving rise to frequent episodes of economic, environmental and social concern. Therefore, concerted actions from authorities on a global scale are required to mitigate these plastic-related issues [3].

According to data published by the association PlasticsEurope, 29.5 million tonnes of plastic are collected annually from post-consumer waste, but only 34.6 % is recycled. The rest is landfilled (23.4 %) or incinerated (42.0 %) [2]. This means that around 6.9 million tonnes of plastic waste are landfilled, taking 1000 years to degrade without the

possibility of recycling or reuse, as well as negatively affecting the environment and public health due to microplastics in the air, soil, water and food.

In response to this problem, the first EU-wide Plastics Strategy lays the groundwork for a new, more sustainable plastics economy since its adoption on 16 January 2018. The aims of this initiative are to make all plastic packaging on the European Union (EU) market recyclable or reusable by 2030, to reduce the consumption of disposable plastics and to restrict the deliberate use of microplastics. In other words, the reduction and prevention of environmental pollution by plastics becomes essential to achieve a more circular economy, thus promoting growth and innovation [4,5].

In line with this strategy, six European organizations - Plastics Recyclers Europe (PRE), Petcore Europe, European Carpet and Rug Association (ECRA), Polyolefin Circular Economy Platform (PCEP Europe), European Plastics Converters (EuPC) and VinylPlus® - have adopted a framework of voluntary commitments aimed at continuing and expanding existing plastic recycling activities, in collaboration with the European Commission. The overall objective is to achieve a 50 % recycling rate of plastic waste by 2040 [6].

Depending on their quality, recycled plastics can be used in multiple sectors, such as building and construction (46 %), packaging (24 %) and agriculture (13 %). A special case is represented by food packaging since, according to the European Regulation 282/2008, only those recycled plastics from authorized recycling processes can be marketed [7]. To preserve food safety, the producers of recycled materials used for food packaging must ensure that unregulated chemicals or contaminants are not present in the package and/or do not migrate into the food matrix. Therefore, any packaging component that comes into contact with food must be of a purity suitable for its intended use. In Spain, these limits are regulated by the Royal Decree 846/2011 (amended by Royal Decree 517/2013 and Royal Decree 1025/2015), which establishes the conditions that raw materials based on recycled polymers must meet for their use in applications and articles intended to come into contact with foodstuffs [8].

Nowadays, mechanical recycling has become indispensable to prolong the shelf life of plastic materials and mitigate their effects on environment. In general, mechanical recycling procedures consist in a series of operations able to provide secondary plastic materials, with high quality grade, suitable to be processed by conventional technologies. But some of these operations, such as extrusion, may induce the rearrangement of polymer molecules and, therefore, changes in the thermo-mechanical properties of the materials. During extrusion, not only scission of polymer chains occurs but also simultaneous cross-linking reactions may be significant. Hence, the balance between these two competing processes would determine the properties of mechanically recycled plastics [9,10].

The residual part remaining after the mechanical treatment of urban wastes is called plasmix, which is composed of the undersieve from the size separation equipments and the final residues from the whole mechanical sorting operations [11]. Nowadays, there is no universal solution for the recycling of these mixed plastics and, in general, the available solutions either cannot separate all the plastics or are not cost effective. In line with this, Rigamonti et al. [12] could not identify an optimal option to improve the environmental performance of mixed plastics among several management scenarios. Therefore, plasmix waste is normally landfilled, incinerated, stored or used for energy recovery as a substitute to coal burning, except for easily identifiable polymers such as high density polyethylene (HDPE) and polyethylene terephthalate (PET) that can be separated for high value applications [11,13,14]. Incineration after mechanical separation from residual waste, landfilling after washing or substitution of coke in a blast furnace have been reported to be favorable options to lessen the environmental impact of mixed plastic wastes [11,12]. Nonetheless, the emission of heavy metals and other toxic compounds should be deemed when plastics are subjected to thermal treatments [11].

This work focuses on the sorting and characterization of plasmix fraction contained in municipal solid waste (MSW), as well as on the revalorization of PP and polystyrene (PS) residues. The different types of plastics contained in this fraction were analysed. In addition, the classification took into account, where possible, the different colors and whether the waste came from food contact materials or any other application. Subsequently, both PP and PS wastes in its different forms (general-purpose, GPPS; high impact, HIPS; expanded, EPS; and extruded, XPS) were recovered and recycled into high quality pellets. Finally, the recycled PP and PS materials were physico-mechanically and thermally characterized to assess their processability using conventional plastic transformation technologies. The main novelty of this work is the material used, plastic waste contained in municipal solid waste that is difficult to recycle mechanically due to its dirt and moisture content. The obtained results can be used for defining the processing conditions for an optimal sorting of plasmix fraction, as well as for the recycling of plastic wastes at larger scale.

2. Materials and methods

2.1. Identification and quantification of materials in plastic mix stream from MSW collection and sorting. Selection of different plastic fractions for mechanical recycling

The identification and quantification in terms of polymer types were carried out at the Ecocentral Waste Treatment Plant, located in Alhendín (Granada, Spain). In this plant, the plasmix fraction is currently being compacted in big bales prior to its landfilling. Plastic mix bales (Table 1) were supplied by the Ecocentral plant for the classification of the solid wastes by different techniques: (i) visual inspection by identifying the codes used by manufacturers; (ii) Near Infrared Spectroscopy (NIR) using a portable equipment and a portable analyser model Thermo Scientific, microPHAZIR AG; (iii) Fourier Transformed Infrared Spectroscopy (FTIR, Perkin-Elmer, Spectrum 65 equipment); and (iv) Differential Scanning Calorimetry (DSC, Perkin-Elmer, STA6000 TG-DSC simultaneous analyzer).

The following fractions were identified in the bales: Food PP (F-PP), including bottles, containers and any other food contact PP parts such as caps or lids; Non-Food PP (NF-PP) represented by bottles, containers and other packaging parts not suitable for food contact, as well as by other PP objects not used for packaging); GPPS; HIPS; EPS, XPS; Other PS (unidentifiable, undifferentiated or indistinguishable PS); PET; rigid HDPE; multilayer packaging; film; paper/cardboard; organic matter; others (non-plastic or plastic not included in any of the preceding

Table 1
Characteristics of the mixed-plastic bales analysed.

BALE 1			
Weight		407.5 kg	
Dimensions	Width	1.25 m	
	Height	1.12 m	
	Length	1.60 m	

BALE 2			
Weight		322.0 kg	
Dimensions	Width	1.22 m	
	Height	1.10 m	
	Length	1.25 m	

fractions); and remains, including small pieces of miscellaneous materials that are very difficult to identify). Once this sorting process was completed, F-PP and NF-PP fractions were separated by color. Furthermore, their differentiation into rigid, flexible and multilayer was performed. The fraction of metallic elements was also quantified, differentiating between ferrous and non-ferrous materials by in situ separation with a magnet.

2.2. Determination of moisture and dirt content in the selected fractions

F-PP, NF-PP, GPPS, HIPS, EPS and XPS fractions collected from the bales were selected for the determination of moisture and dirt content.

The moisture content (MC) of the materials was immediately determined by drying a known amount of each plastic at a temperature $\leq 90\,^{\circ}$C until constant weigh. The value of MC was determined as the difference in weight of the samples before and after drying according to the Eq. (1):

$$MC(\%) = \frac{(m_{bd} - m_{ad})}{m_{bd}} \times 100 \qquad (1)$$

where m_{bd} (g) and m_{ad} (g) are the weights of the samples before and after drying, respectively.

To determine the dirt content (DC) the materials were previously shredded and washed. The washing step was performed with aqueous NaOH solutions at variable temperature. As the goal was to remove the maximum amount of dirt and impurities, neither the solid/liquid ratio nor the amount of water used to rinse the washed samples were controlled. Hence, the number of washing and rinsing cycles were those required to leave the samples free of impurities. Subsequently, the samples were dried at a temperature below 90 °C as indicated above and the DC was calculated by weight difference according to the Eq. (2):

$$DC(\%) = \frac{(m_{bw} - m_{aw})}{m_{bw}} \times 100 \qquad (2)$$

where m_{bw} (g) and m_{aw} (g) are the weights of the samples before and after washing respectively.

Four determinations of both moisture content and dirt content were made, also differentiating between PP and PS. Samples weighing around 350–500 g of material were used in each test. The results obtained are an average of different measurements taken in the different samplings carried out for the characterization of the material.

2.3. Transformation of the selected fractions in recycled pellets

After completing the collection, separation and classification stages, the 6 selected fractions (F-PP, NF-PP, GPPS, HIPS, EPS and XPS) were appropriately processed in order to obtain pellets from recycled material. A process similar to that normally described for the mechanical recycling of plastics was followed. Firstly, the dirt materials were shredded in a knife mill (model WSGM-250, J. Purchades; Spain) to obtain a suitable particle size for subsequent, washing and extrusion

(Fig. 1). The equipment features a set of sieves to control the maximum size of the resulting pellets.

Before this, the shredded wastes were subjected to several washing and rinsing cycles in a process similar to that described for the determination of DC. Briefly, the materials were immersed two times in abundant water with vigorous agitation to remove coarsest particles and the more accessible impurities. Subsequently, they were immersed in diluted aqueous NaOH (ca. 3 % w:v) for 24 h with occasional stirring. Finally, the materials were rinsed with abundant water until a clear solution was obtained, and dried at room temperature for 48–72 h.

In order to obtain homogeneous materials, each plastic fraction was fed into an air-circulating dehumidifier (model KKT 75, Koch Technik; Germany) at 50 °C overnight to ensure complete dryness, and then processed in a co-rotating twin-screw extruder (model SHJ-20, Siepla; Spain) using a suitable temperature profile to avoid polymer degradation. This equipment consists of a 20 mm diameter twin-screw extrusion system with a capacity to process up to 10 kg·h^{-1} at temperatures of up to 400 ºC. It also has a degasser with a vacuum pump, main and side twin-screw feeders, and a 1.2 m long cooling bath. Finally, the extruded and homogenized materials were pelletized by using the WSGM-250 knife mill with an 8 mm diameter stainless steel mesh (Fig. 2).

2.4. Injection molding and characterization of recyclates

2.4.1. Injection molding of test specimens

The pellets of the different samples were dehumidified and injected using an injection molding machine (model Victory 28, Engel Holding GmbH; Austria). Two different molds were used to obtain the two different types of samples required: (i) type 1BA tensile test specimens according to ISO 527 standard (75 mm·5 mm·2 mm), and (ii) 80 mm·10 mm·4 mm specimens according to ISO 178 standard (Fig. 3).

2.4.2. Mechanical properties

Tensile, impact and Shore hardness tests were performed to obtain the mechanical properties of the recyclates. Prior to these tests, the specimens were conditioned in the laboratory at 23 ± 2 °C and 50 ± 5 % RH for 48 h. Tensile experiments were determined using a Universal Testing Machine (model H10KS, Tinius Olsen Testing Machine Company; USA) with a load capacity of up to 10 kN and equipped with an extensometer. The experiments were carried out using type 1BA tensile test specimens (5 replicates) following the method described in ISO 527–2 standard.

The Charpy impact strength of unnotched samples was determined for each material using a pendulum impact-tester (model Charpy-Izod IMPats 15, ATS FAAR Industries; Italy) following the 1 eU method described in ISO 179–1 standard. Prior to the tests, the specimens were stabilized at a temperature of 23 ± 2 °C and RH of 50 ± 5 % for at least 16 h. Impactors of 5 J and 2 J were used for recycled PP and recycled PS respectively. The results obtained were expressed as the average of 10 replicates.

Shore hardness of the different materials was assessed in a universal micro-durometer (model Bareiss Digi Test, Neurtek Instruments; Spain),

Fig. 1. Materials after shredding in the knife mill: A) F-PP; B) NF-PP; C) HIPS; D) GPPS; E) EPS); F) XPS.
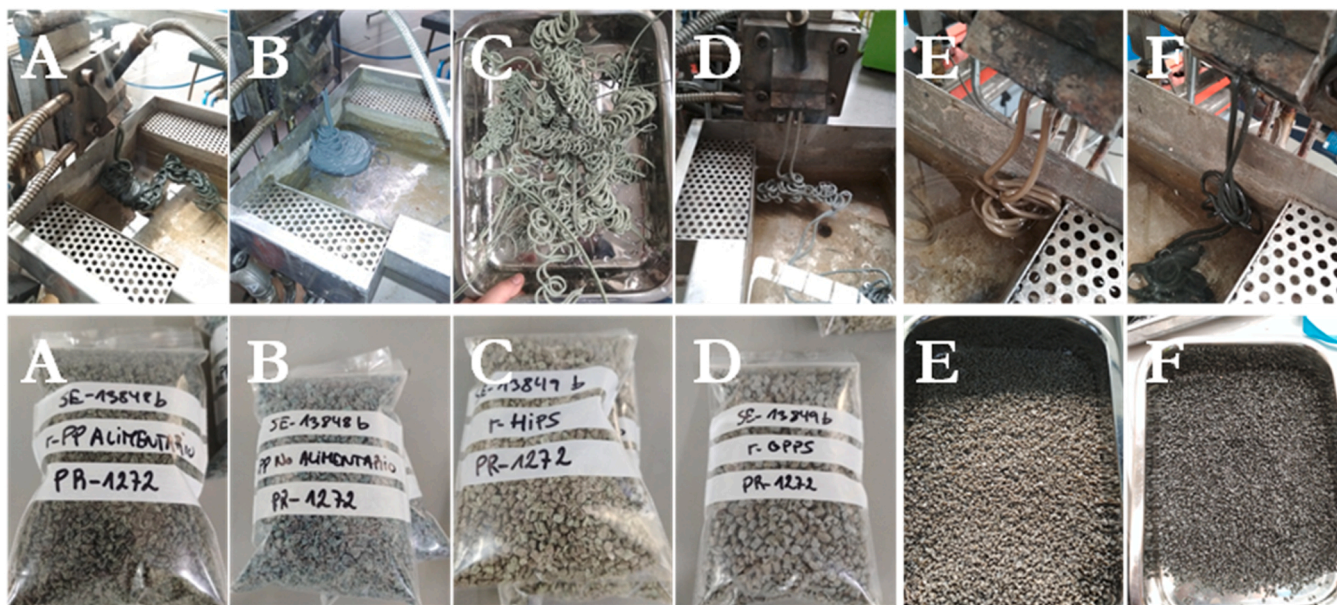
Fig. 2. Extruded (top) and pelletized (bottom) recycled materials: A) F-PP; B) NF-PP; C) HIPS; D) GPPS; E) EPS; F) XPS.

Fig. 3. Test specimens for the determination of recycled materials properties: A) F-PP; B) NF-PP; C) HIPS; D) GPPS; E) EPS; F) XPS.

equipped with a type D hardness tester, after 15 s of contact between the indenter and the specimen. As specified in ISO 868 standard, the results were expressed as the mean of 5 replicates by using 80 mm·10 mm·4 mm specimens.

2.4.3. Thermal properties

Both Heat Deflection Temperature (HDT) and VICAT softening temperature of the materials were measured with an HDT/VICAT instrument (model MP-3, ATS FAAR Industries; Italy). In this case, the samples were previously conditioned at 23 ± 2 °C and 50 ± 5 RH for at least 88 h. HDT was analyzed according to the ISO 75–1 and 75–2 standards, subjecting the normalized test specimens (80 mm·10 mm·4 mm) to a constant load, preferably in the plane position, to produce a bending stress as the temperature increases. A silicone oil bath was used as a heating system, with a heating rate of 120 ± 10 °C h^{-1} and a load of 0.45 MPa. The results were expressed as the

average of 3 replicates. VICAT softening temperature was determined as described in the method B of the ISO 306 standard. More specifically, the samples were exposed to a heating rate of 50 °C h^{-1} and a permanent load of 50 N. Three replicates of the 80 mm·10 mm·4 mm specimens were used.

Differential scanning calorimetry (DSC) (model Star System, Mettler-Toledo; Spain) was used to determine glass transition temperature (T_g), melt temperature (T_m), melting enthalpy (ΔH_m) and crystallinity (X_c). The different samples of recycled plastics (around 10 mg) were analyzed from -35 °C to 200 °C at a rate of 10 °C min^{-1} and under nitrogen flow (50 mL min^{-1}). The samples were heated, cooled and reheated under the same conditions. While the first heating ramp erased the thermal history of the materials, the second one was used to determine T_g, T_m, and ΔH_m. Since PS is a highly amorphous polymer, X_c was calculated only for the recycled PP materials by using the Eq. 3:

$$X_c(\%) = 100 \times \frac{\Delta H_m}{\Delta H_m^0} \tag{3}$$

where ΔH_m^0 corresponds to the melting enthalpy of a 100 % crystalline PP. A value of $\Delta H_m^0 = 205$ J g^{-1} was used in agreement with Fischer [15].

2.4.4. Other physico-chemical properties

In addition to the above-mentioned mechanical and thermal tests, the density (ρ) and the melt flow index (MFI) were also determined. Moreover, the purity of the recyclates was confirmed by Infrared (IR) spectroscopy.

The ρ of the final materials was measured in an electronic density meter with a resolution of 0.001 g cm^{-3} and a weight capacity of 300 g (model MD-300S, Alfa Mirage Co. Ltd.; Japan). The measurements were conducted at a temperature of 23 ± 2 °C by immersing the sample in distilled water as described in the method A of the ISO 1183–1 standard. The ρ, which was expressed as the mean of 3 replicates, was calculated by the Eq. (4):

$$\rho_s = \frac{m_{air}}{m_{air} - m_{dw}} \times \rho_w \tag{4}$$

where ρ_s (g cm^{-3}) is the density of the sample at the specific temperature, m_{air} (g) and m_{dw} (g) are the weights of the sample in air and in distilled water, respectively, and ρ_w (g cm^{-3}) is the density of distilled water.

The MFI determination of the recycled pellets was performed in an extrusion plastometer (model MP600 Tinius Olsen Testing Machine Company; USA) according to the method A of the ISO 1133 standard. MFI is defined as the extrusion velocity of a molten resin through a die of specific length and diameter. It is calculated as the amount of material extruded in a given period of time and under specific load and temperature conditions. For PP recyclates the experimental conditions were load 2.16 kg and temperature 230 °C, whereas 5 kg load and 200 °C temperature were used for recycled PS materials.

In addition, infrared spectra of the materials were measured in a Fourier-transformed IR (FTIR) spectrometer equipped with a DTGS detector (model Tensor 27, Bruker; Spain). Data in the region 4000–600 cm^{-1} with a wavenumber resolution of 4 cm^{-1} by using the attenuated total reflectance (ATR) method were recorded and analysed with the software OPUS v6.5 (Bruker, Spain). The spectra of the recycled materials were compared with those of virgin counterparts and commercial plastics by using an internal digital library.

3. Results and discussion

3.1. Identification and quantification by type of polymer

The results obtained in the sorting process carried out on the plastic mix bales from MSW are shown in Table 2. The main conclusion is that almost 65 wt% of the bale (on average) is composed of different plastic wastes, mainly PP (28.89 wt%), PET (22.02 wt%), PS (9.65 wt%) and rigid PE (4.68 wt%). As the main objective of the project is to increase the recyclability of those plastics that are most likely to end up in landfill, this work focused on the study of the PP and PS fractions, which accounted approximately for 38.54 wt% of the bale. Despite this, it is also noticeable that above 20 wt% of plasmix bales consisted of PET wastes.

Although it is difficult to compare the results with other studies given the geographical variability in terms of waste collection systems, separation and classification systems, etc., there are studies that obtained similar results to those obtained in this work. Dahlo et al. [16] carry out a study on the recycling potential of post-consumer plastic waste in Finland. These authors found that, in the composition of MSW, most of the plastic waste was packaging (around 90 %), being PP and LDPE the

Table 2
Results of the sorting and characterization of the plastic mix bales.

Fraction			Bale 1, wt%	Bale 2, wt%
PP	F-PP	Rigid	9.72	9.81
		Multilayer	4.23	2.34
		Flexible	6.54	4.89
	NF-PP	Rigid	10.20	8.57
		Multilayer	0.00	0.00
		Flexible	0.73	0.76
PS		GPPS	1.38	1.89
		HIPS	3.69	2.01
		EPS	4.01	2.69
		XPS	1.21	1.22
		Other PS	0.33	0.88
PET	Bottles		15.68	14.55
	Multilayer		5.74	8.07
Rigid PE	High density		4.77	2.65
	Multilayer		1.07	0.88
Film			11.72	15.04
Paper and cardboard			3.45	6.99
Organic matter			1.01	0.69
Remains			5.10	3.29
Others	Other materials		7.35	11.36
	Plastics with N°7		0.25	0.19
Ferrous metals			0.07	0.12
Non-ferrous metals			1.74	1.11
Total			100.00	100.00
Total F-PP			20.49	17.03
Total NF-PP			10.93	9.32
Total PP			31.42	26.36
Total PS			10.62	8.69
Total PET			21.43	22.61
Total Rigid PE			5.84	3.53

most common plastic types present followed by PET, PS and HDPE. In Italy, plasmix fraction has been reported to be composed of 57 % plastic, 10 % paper and cardboard, 3 % wood, 3 % textiles, and 27 % in erts and others including metals [17].

3.2. Determination of moisture and dirt content

Table 3 shows both MC and DC for PP and PS wastes. A high variability was found among samples due to their heterogeneity, as well as to the different climatic conditions (temperature and rainfall) that occurred at the different sampling times.

Regarding the DC, it was determined that dirt mainly corresponded to particulate material readily removable during handling (soil, leaves, branches, etc.), grease and strongly adhered organic matter that is mostly represented by rests of food. It is important to remark that PP and PS wastes lost 12.84 and 18.58 wt% respectively of their initial weights due to moisture and dirt (Table 3).

Gala et al. [18] found a moisture content that varied between 3.0 % and 15.9 % in different types of municipal plastic wastes after manual and automated sorting processes. Additionally, Calero et al. [19] reported MC values of 1.58 % for PP and 20.98 % for PS, and DC values of 2.65 % and 13.79 % for PP and PE respectively, for plastic materials contained in MSW.

3.3. Material characterization tests

Recycled plastics from MSW come from multidiverse sources with different properties due to their variate grades and degradation extent

Table 3
Moisture content (MC), dirt content (DC) and average moisture + dirt loss of PP and PS fractions collected from plasmix bales.

Plasmix fraction	MC (wt%)	DC (wt%)	Average moisture + dirt loss (wt%)
PP	4.84	8.12	12.84
PS	6.97	8.11	18.58

during life use. Therefore, the sorting stage in mechanical recycling of plastics is crucial to achieve recycled materials with satisfactory mechanical properties [20]. Moreover, high temperatures and shear forces during extrusion lead to different degradation processes within the polymer matrices [21]. As a consequence, recycled plastics normally exhibits lower mechanical and thermal resistance as compared with their virgin counterparts. This is the reason why addition of virgin polymers is a common practice in plastic recycling plants. Hence, comparison with either virgin polymers or recycled plastics that have suffered a different lifeuse and/or have been subjected to different recycling processes may be challenging. Nevertheless, in the following sections we have tried to compare or results with those in similar works.

3.3.1. Mechanical properties

Fig. 4 shows the values of the main mechanical properties obtained from the tensile, impact strength and hardness tests for the six recyclates under study.

In general, NF-PP yielded better mechanical properties than F-PP,

with Young's modulus and impact strength 1.6 and 2.3 times higher respectively. This relatively great difference between both PP recyclates has been previously ascribed to stress-concentrating defects, as well as to variability in polymer architecture [22]. Similar results were obtained for PP by Momanyi et al. [23] at low test temperatures (22–23 ºC). These authors carried out an interesting comparative test in terms of tensile properties at different temperatures, between 23 and 80 ºC, elucidating that the tensile strength of recycled PP decreased by up to 75 % at the highest temperature in comparison with the lowest one. About recycled PS, the same work reported lower values of tensile strength (14 MPa at 23 ºC) than those found in our study (Fig. 4b). Campomanes Santana and Manrich [24] stated that recycled PP presents higher tensile stress than recycled HIPS, which was attributed to the intrinsic nature of the polymers.

Elongation at break is also affected by the degradation of polymer chains during mechanical recycling due to, among other things, the reduction in the molecular weight [25]. Elongation at break was 51 % for F-PP and approximately 6-fold higher for NF-PP. In the case of the
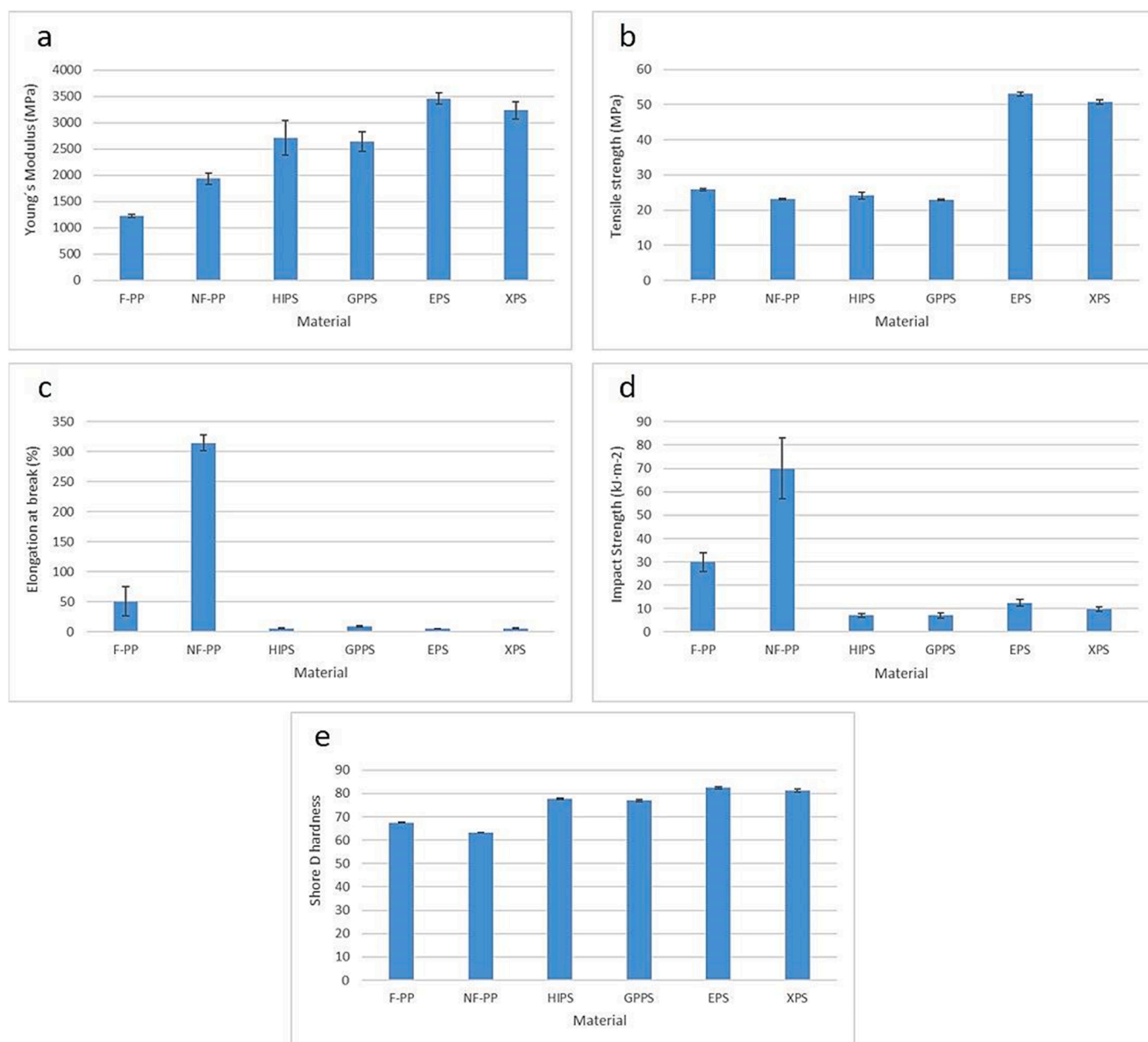

Fig. 4. Mechanical properties of PP and PS recyclates: a) Young's modulus; b) Tensile strength; c) Elongation at break; d) Impact strength; e) Shore hardness. Values are presented as mean ± standard deviation.

four PS recyclates, elongation at break was rather similar since it oscillated in the range 5.2–9.1 % (Fig. 4c). Momanyi et al. [23] reported that the stress of recycled PP continuously decreased after the yield point until breakage. They recorded an elongation at break for recycled PP in the range 15–150 % depending on the test temperature. In the study by Campomanes Santana and Manrich [24], recycled PP exhibited an elongation at break, of almost 300 %, that was higher for virgin PP (below 200 %). Moreover, the same authors found similar elongation at breaks for virgin and recycled HIPS of around 25 %. These differences were attributed to the different grades of the polymers used. In contrast, elongation at break of HIPS from disposable cups was progressively reduced from above 30 % to approximately the half after several recycling cycles [26]. From our results, it is especially noticeable the low elongation at break obtained for HIPS recyclates (5.9 %), which points out to an extensive degradation of the elastomeric polybutadiene phase and an embrittlement of the polymer matrix during the lifeuse and/or the recycling process.

It is observed in Fig. 4d the difference in impact strength for both types of polymers, in accordance to their relative brittleness. Impact strength values were in the range 30–70 kJ m^{-2} and 7.1–12.5 kJ m^{-2} for PP and PS recyclates respectively. All the specimens for both PP and PS recycled materials exhibited complete break. From our results, it is worthy to mention the low impact strength of recycled HIPS, which is consistent with the above mentioned embrittlement, since other works reported values oscillating in the range 40–60 kJ m^{-2} for HIPS recycled from waste electric and electronic equipment [20] or disposable cups [26]. This later work also reported a slight increase in the impact strength of HIPS after consecutive recycling cycles. The authors explained these results by a likely plasticizing effect of the short polymer chains after scission and to a better distribution of the polybutadiene phase.

In general, those materials from the recycling of foamed wastes (EPS and XPS) presented higher mechanical parameters as compared with those from the recycling of rigid plastics (HIPS and GPPS), with Young's modulus about 1.3 times higher (Fig. 4a), tensile strength values more than twice (Fig. 4b), and impact strength up to 1.8 times higher (Fig. 4d). This can be related to the observation of bubbles inside the HIPS and GPPS specimens, which likely acted as stress-concentrating defects and caused irregularities in the stretching (as evidenced by the higher SD values in the Young's modulus (error bars in Fig. 4a)) and fracture of specimens. As the recyclates came from post-consumer wastes, the presence of impurities in the recycled materials such as pigments, printing inks, labels, or lubricants, as well as different degradation extent of the polymer matrices during life use and recycling operations should be also considered [20,21]. The processing parameters during extrusion or injection molding and the number of thermal reprocessing cycles have been reported to be of paramount importance in the properties of recycled plastics and their blends with virgin polymers, especially concerning their mechanical performance [10,21,27]. As discussed above, the degradation of the butadiene rubber phase during extrusion has been reported to cause embrittlement of HIPS [20]. Eventually, the inclusion of foreign particles such as aluminium from metallized lids or paper labels in the EPS and XPS specimens may have also altered mechanical properties. This is in agreement with Wang et al. [28] who concluded that the properties of recycled polymers are often altered by the frequent presence of organic, inorganic or biological residues in plastic wastes.

The hardness of a material can be defined as its resistance to localized plastic deformation. As can be observed in Fig. 4e, PS materials have higher hardness than PP materials, which is related to their higher Young's modulus and tensile strength. No significant differences were found in terms of hardness by polymer type, with values between 60 and 70 for recycled PPs and around 80 for PS recycled. Hardness is usually related to the crystallinity of the polymer, and it is important to consider that amorphous polymers are harder than crystalline polymers. The parameters of the measuring method (shape of the indenter, applied

force and force application time required) also influence the hardness of a material in such a way that it is difficult to find a simple relationship for the results obtained by using different types of hardness testers and other measuring equipments. In addition, the temperature is another influential factor, since the higher the temperature the lower the hardness because materials become soft elastic [23]. These authors obtained similar results for hardness of recycled PP and PS to those obtained in the present study.

3.3.2. Thermal properties

The HDT temperature represents the resistance of a material to distortion caused by a physical load at an elevated temperature. In the HDT test, the specimen (which is clamped at both ends) is subjected to a physical load in the middle as the temperature is progressively increased. The temperature at which the specimen deforms by 0.25 mm is recorded as its HDT value. In other words, the HDT test reports the stiffness of the material as its temperature increases. Therefore, HDT allows, by means of a simple test, to assign a value to the performance of the material in those applications that imply high temperatures in the short term. In addition, the HDT value is useful in the injection moulding process as an indicator of the maximum temperature at which the manufactured part can be ejected from the mold without deformation. The HDT data obtained for each material (Table 4) are within the expected range for each specific type of polymer, ranging from 85 ºC to 120 ºC for PP depending on whether it is a copolymer or homopolymer, and in the range of 75–100 ºC for PS [29]. The high difference in the average value for the two PP materials can be explained by the high deviation in the results obtained for NF-PP, which is probably related to a higher variability of PP types in this fraction. Our results are slightly higher than those found in other studies, being these deviations attributable to the slight differences in the methodology followed for both sorting and processing stages [30]. For PS, some studies demonstrated that HDT temperature varies between 77 and 87 ºC depending on the type of PS and the mixture with other plastics or additives [31].

VICAT test results vary greatly depending on the heating rate and the load applied to the sample. For example, the VICAT temperature of a commercial PP with a heating rate 50 ºC·h^{-1} and 50 N load can increase from 70 to 80 ºC to approximately 150 ºC when the load is reduced to 10 N. For the conditions used in this study (50 ºC·h^{-1}, 50 N), the values are within the expected range without large variations for each type of polymer (Table 4). The values obtained for recycled PP and PS are lower than those previously reported, these variations being probably related to the high heterogeneity of the samples, since they come from a MSW stream [30,31]. Furthermore, degradation and shortening of polymer chain length by mechanical or thermal decomposition during use and recycling operations may have also occurred [32,33].

Figs. 4 and 5 depicts the DSC curves of the 6 recycled materials. T_m values of 163 °C and 162 ºC were obtained for F-PP and NF-PP respectively (Table 4), thus confirming that the materials are composed of PP

Table 4
Thermal properties and crystallinity of PP and PS recyclates. Values of HDT and VICAT are presented as mean ± standard deviation.

Material	HDT (°C)	VICAT (°C)	T_g (ºC)	T_m (ºC)	ΔH_m (J g^{-1})	X_c (%)
F-PP	83 ± 3	84 ± 35		163	88	43
NF-PP	108 ± 37	72 ± 7		162	70	34
HIPS	88 ± 7	86 ± 1	95			
GPPS	82 ± 1	84 ± 2	94			
EPS	91 ± 5	96 ± 1	97			
XPS	88 ± 4	92 ± 1	94			

T_g: glass transition temperature
T_m: melting temperature
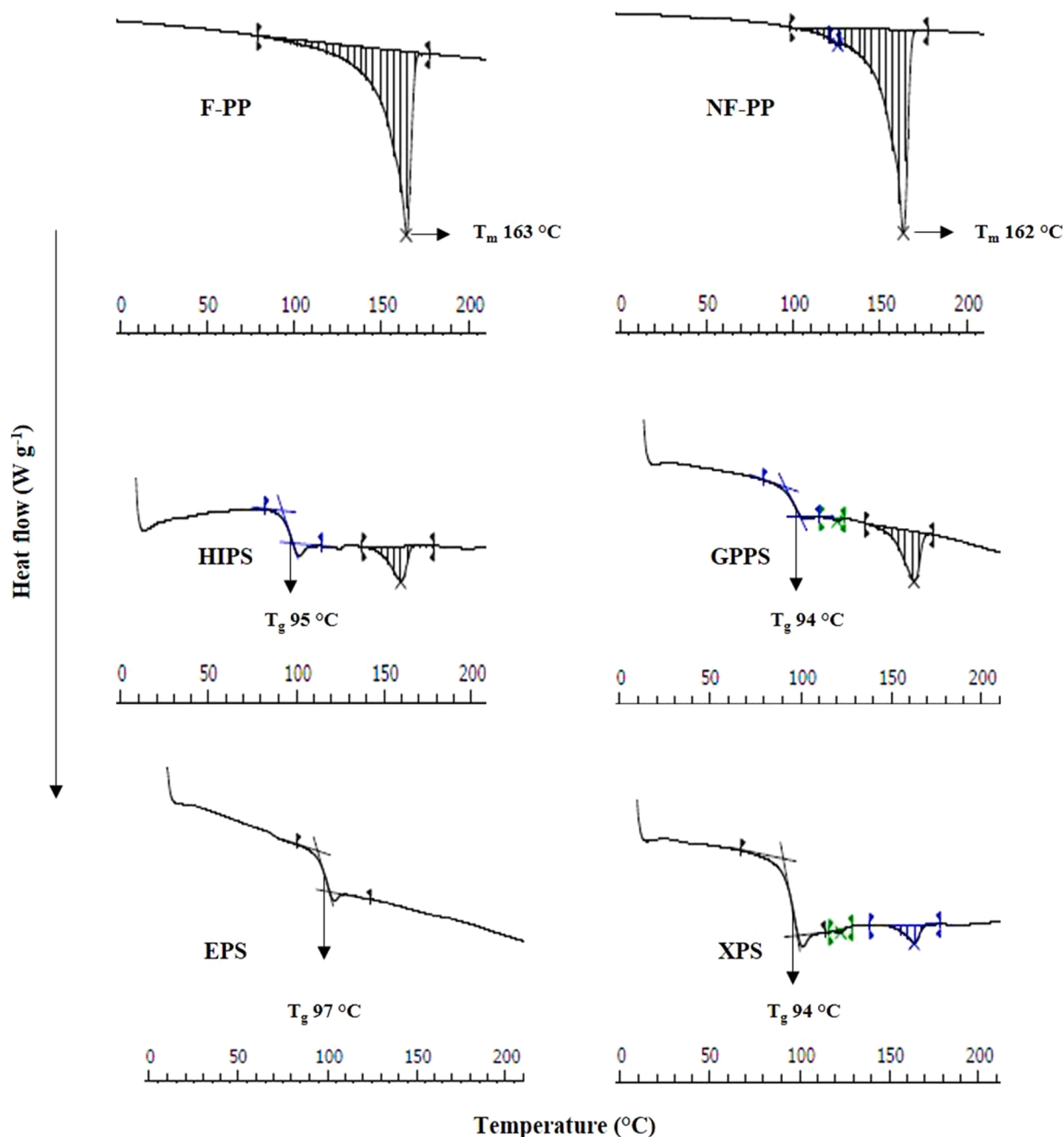ΔH_m: melting enthalpy
X_c: crystallinity.

Fig. 5. DSC curves of the recycled materials.

in agreement with the IR spectra in Section 3.3.3 (Fig. 6) and with the typical values for PP polymers in the range 160–170 ºC [22,34]. Nonetheless, the broad peaks indicated a relatively wide distribution of crystallite sizes, hence revealing the heterogeneity of the materials. In addition, a minor melting event appeared in the NF-PP curve at 125 ºC, indicating the presence of PE traces, probably by polyolefine cross-contamination [22]. The ΔH_m for these materials were 88 and 70 J·g^{-1}, leading to X_c values of 43 % and 34 % for F-PP and NF-PP recyclates respectively (Table 4). Nevertheless, ΔH_m values may be overestimated because of polymer contamination [22]. Crystallinity of PP may increase after recycling. For instance, Huang and Peng [10] found improved melting enthalpy and crystallinity of PP after several recycling cycles that resulted in increased tensile strength.

For those materials recovered from PS waste, the main observable event in the DSC curves is the T_g, which confirms the amorphous nature of these materials. Therefore, PS does not have a specific melting point

but softens from a defined temperature value, which is approximately in the range 90–100 ºC. The IR spectra below confirmed that these materials are composed exclusively or mostly of PS (Fig. 6). In addition to the T_g peak, the DSC curves of most recycled PSs showed different melting events indicating the presence of residual PP and PE. This was not observed for EPS recyclate, probably due to the fact that EPS is an easily identifiable plastic that is not usually combined or mixed with other polymers in its different applications.

3.3.3. Other physico-chemical properties

FTIR spectra of recycled PP and PS materials (red lines) resembled those of typical virgin polymers (blue and pink lines) (Fig. 6). In general, no significant differences in the spectra of both PP recyclates and the four recycled PSs were observed. The spectra of PP materials exhibited a broad and flat band approximately between 3100 cm^{-1} and 3600 cm^{-1}, which was especially visible for F-PP. This is indicative of the presence
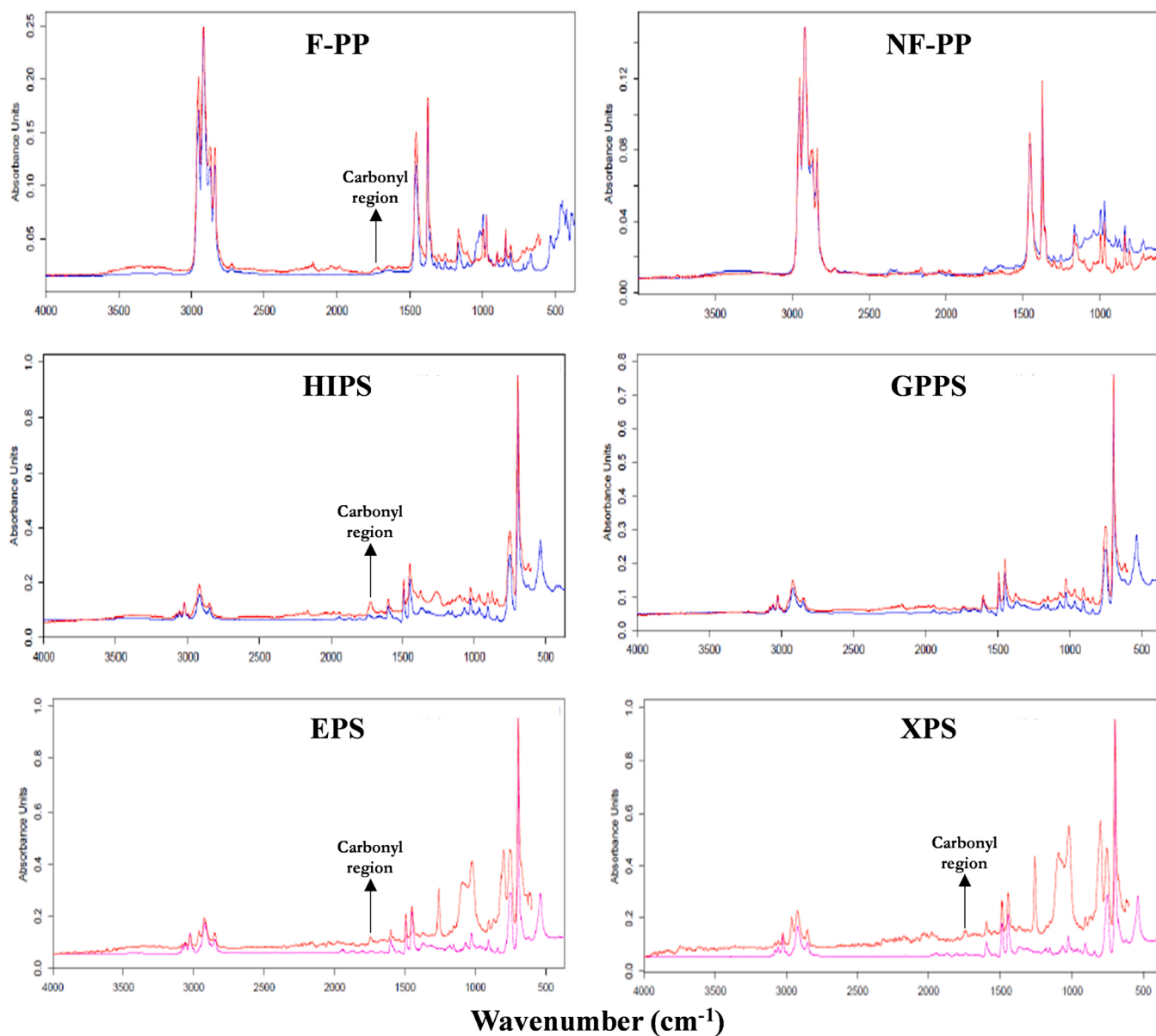
Fig. 6. IR spectra of the recyclates (red lines) in comparison with IR spectra from an internal digital library (blue and pink lines).

of fatty amide slip agents and/or surface bound water trapped by hygroscopic additives and polar contaminants [22]. The pattern of the CH_2 and CH_3 stretching vibrations region for both recycled PP materials (3000–2800 cm^{-1}) indicates the coexistence of PP and PE, as well as the intense peak in the 1500–1400 cm^{-1} region [35]. This contamination with PE traces was not observed in the DSC analysis of F-PP likely because it is masked by the broad band corresponding to the melting event of PP (Fig. 5). The high intensity of the band at 2950 cm^{-1}, together with the peak around 1380–1370 cm^{-1}, revealed that the material is mainly composed of PP. In addition, the small peaks between approximately 800 and 1200 cm^{-1} are typical of PP materials. Moreover, a small peak in the carbonyl region (appearing in the region 1780 1620 cm^{-1}) is also observed in the F-PP spectrum, which may suggest either degradation of the polymer by thermo-oxidative or photocatalytic mechanisms or the presence of ester-based additives [22]. Kazemi et al. [35] indicated that for recycled plastics oxidation is more likely to occur during the service life of the materials.

In the case of recycled PS, the most intense and narrow peak was observed at a wavenumber around 700 cm^{-1}, typical for PS polymer. Similarly to recycled PP, the small peak in the carbonyl region revealed

the presence of oxidation products generated by degradation during service life and recycling. In fact, a recent work reported a rapid methodology in which carbonyl peaks in 1780–1620 cm^{-1} region of FTIR spectra are used to distinguish between virgin and recycled PS containers [36]. These authors indicated that the intensity of FTIR spectra was not adequate to make distinctions, but they used the appearance of a peak at 1722 cm^{-1} as a marker band to identify recycled PS in blends with virgin polymers.

As shown in Table 5, MFI values of 17.1, 19.4, 8.8, 11.3, 13.8 and

Table 5

Melt flow index (MFI) and density (ρ) of recyclates. Values are presented as mean ± standard deviation.

Material	MFI (g·10 min^{-1})	ρ (g·cm^{-3})
F-PP	17.1 ± 0.2	0.944 ± 0.001
NF-PP	19.4 ± 0.23	0.918 ± 0.001
HIPS	8.8 ± 0.1	1.039 ± 0.001
GPPS	11.3 ± 0.2	1.067 ± 0.001
EPS	13.8 ± 0.2	1.059 ± 0.001
XPS	8.2 ± 0.2	1.066 ± 0.001

8.2 g·10 min^{-1} are obtained for F-PP, NF-PP, HIPS, GPPS, EPS and XPS respectively under the different test conditions, with no significant differences for the materials depending on the type of polymer. Therefore, the six recyclates can be referred as rather easy-flowing materials suitable for conventional plastic transformation processes [22]. Degradation throughout shelf life and disposal may influence MFI of recycled polymeric materials. Moreover, substantial shear forces during extrusion and recycling operations produce the scission of polymer chains and broaden the molecular weight distribution, thus leading to improved fluidity of recycled plastics with respect to virgin materials [10,26,37]. In this sense, an increase from approximately 14 g 10 min^{-1} to around 20 g 10 min^{-1} has been reported when raw PP is subjected to 5 recycling cycles [10]. In another recent work, a slight increase in the MFI values has been found for HIPS disposable cups after six consecutive reprocessing cycles simulating recycling [26]. Similarly, Shojaeiarani et al. [37] showed a varied increment (13–108 %) in the MFI of different bioplastics after 5 extrusion processes. In contrast to these findings, the MFI of recycled PS did not significantly change after extrusion with a small-scale extruder for the fabrication of 3D-printing filament [38]. Moreover, in the case of specific polymers such as PE, MFI normally decreases after recycling and extrusion [9,33]. For instance, Jin et al. [9] reported decreased ability of molten LDPE to flow after extensive mechanical recycling. In spite of chain scission, this was attributed to hampered intermolecular mobility by crosslinking of polymer chains, which was the prevalent mechanism after 40 consecutive extrusions.

In addition to being dependent on temperature, the density of a material can vary due to different factors such as changes in its crystallinity, loss of plasticizers, solvent absorption, presence of reinforcements, fillers, etc. The density of the recycled PS materials under study presented values that were comparable with those expected for virgin PS. However, they were slightly higher than those expected for virgin PP (0.900–0.910 g·cm^{-3}), especially F-PP, which could indicate the presence of reinforcements, fillers or other additives into the PP matrices studied, or that the materials may be contaminated with small amounts of other denser polymers such as PE (Table 5) [22,39].

4. Conclusions

This work addressed the sorting and characterization of the mixed plastic waste contained in Municipal Solid Waste, as well as the feasibility of its processability by injection moulding, extrusion and/or thermoforming. The plasmix fraction were composed of different polymers mainly PP (28.89 wt%), PET (22.02 wt%), PS (9.65 wt%) and rigid PE (4.68 wt%), being PP and PS fractions the objective of this work. After determination of the moisture and dirt content, PP and PS wastes exhibited losses of 12.84 and 18.58 wt% respectively of their initial weights.

According to the characterization of PP and PS materials after recycling, NF-PP yielded better mechanical performance than F-PP, with Young's modulus and impact strength 1.6 and 2.3 times higher respectively. Recycled PS from EPS and XPS residues presented higher mechanical parameters as compared with those from the recycling of HIPS and GPPS, with tensile strength values more than twice and Young's modulus about 1.3 times higher. In addition, no significant differences were found in terms of hardness by polymer type, with F-PP being slightly harder than NF-PP, and the material from EPS being slightly harder than the rest of the materials from the different PS wastes.

The HDT and VICAT data obtained for each material are within the expected range for each specific type of polymer for the conditions used in this study. The density values in agreement with the theoretical values for PS, and slightly higher for PP, especially F-PP, which could indicate the presence of reinforcements or other additives into the PP matrices studied, or that the materials may be contaminated with small amounts of other denser polymers. This fact has been confirmed with the results of the FTIR and DSC tests.

The results obtained in this work have shown that, in general, the plastic materials recovered from the rejected fraction of MSW, focusing on PP and PS, are potentially recyclable at larger scale, thus helping to avoid their deposit in landfills and to reduce the use of fossil resources and environmental impact.

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