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Characterization and Analysis of Commercial Polyethylene Bags as a Prior Step to their Mechanical Recycling: A case Study in Granada, Spain

M A Martín-Lara*, V Godoy, J A Moreno, M Calero and J M Soto

Chemical Engineering Department, University of Granada, Avda, Fuentenueva, s/n 18071 Granada, Spain

***Corresponding author**
M.A. Martín-Lara, Chemical Engineering Department, University of Granada, Avda, Fuentenueva, s/n 18071 Granada, Spain, Tel: +34 958240445; E-mail: marianml@ugr.es

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Abstract
Increasing world plastic production generates million tonnes of waste. Flexible packaging bags specially suppose a challenge in mechanical recycling because of their singular properties. This research aims to provide a physical-chemical characterization of commercial polyethylene bags in order to facilitate a future improvement or adaptation of recycling processes to them. Firstly, properties such as dimensions and density were measured in regular and garbage bags samples purchased in different establishments of Granada (Spain). Then, a chemical classification in three groups (HDPE, LDPE, mixture) was achieved according to Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC) tests. Finally, ash content was also determined.

Keywords: Calorimetry, Characterization, Flexible Plastic Bags, Infrared Spectrum, Landfill, Plastic Waste, Polyethylene

Introduction
Plastic is an essential material in many areas of everyday life, whose production has grown exponentially since the 1960s. World plastic production was estimated at 335 million tonnes in 2016, 3.8% higher than production in 2015. Europe is currently the world's third largest producer of plastics, with 19% of production [1]. This translated into 60 million tonnes of plastic produced in 2016. Spain is among the top five countries in Europe in terms of plastic consumption, reaching 3.8 million tonnes in 2015 [2].

Today, the most demanded polymers by society belong to the polyolefin group, and are high density polyethylene (HDPE), low density polyethylene (LDPE) and polypropylene (PP). These three polymers account for 55% of total global plastic demand [3]. The most acclaimed sectors by industry for the use of these polymers are packaging sector, construction, automotive, electronics or agriculture [4]. In Europe, packaging sector accounts for approximately 40% of the plastic used, followed by construction (20%), automotive (9%) and electronics (6%). In Spain the trend is similar, although more plastic is used for packaging (48%) and agriculture (6%) [3]. In packaging sector, we must highlight the production of single-use plastic bags, which are dispensed daily in shops around the world and are used in households in the most developed countries.

According to the amount of plastic produced, large amounts of waste are generated and often managed incorrectly. In 2015, approximately 300 million tonnes of plastic waste were generated worldwide with polyethylene waste being the most abundant [5, 6]. In Europe, the amount of plastic waste generated in 2014 reached 25.8 million tonnes [3]. Currently, 80% of the European populations dispose of all their waste in the same container, without separation. This fraction is known as municipal solid waste, and most of the flexible packaging (including plastic bags) ends up in this waste stream. Approximately 40% of municipal solid plastic waste is flexible packaging.

Plastic waste can follow three different paths: mechanical recycling, whereby waste is transformed back into a usable product similar to the original one; energy recovery, which consists of the incineration of waste for energy; and finally landfills, where waste that is not recycled, accumulates [7]. In 2016, mechanical recycling rate in Europe reached 31%, energy recovery 42% and land filling approximately 27%. These percentages vary according to the infrastructure available in each country and its economic and political situation. For example, the amount of plastic waste deposited in landfills in Spain in 2016 was well above the European average, approximately 45%. Therefore, almost half of the plastic waste generated in Spain ends up in landfills [1].

Flexible packaging (including plastic bags) is one of the most difficult materials to recycle. It tends to be problematic during collection and sorting because of its low weight/volume ratio, and presents difficulties to be handled in recycling facilities [8]. This makes it uneconomic for companies to invest in collection and recycling. In addition, the properties of the recycled product such as colour, mechanical properties or impact resistance are sometimes not the most suitable for certain applications. The main objective of this article is, therefore, to analyze the physical-chemical properties of plastic bags in order to obtain information that will allow the improvement of recycling facilities and machinery, as well as to obtain a final product with better properties. This would also prevent the film from being land filled.

Materials and Methods

Materials

The study carried out in this paper has focused on the analysis of bags for commercial use made mainly of polyethylene. To this end, a set of 40 single-use plastic bags, purchased from 40 different retailers (both large and small) in Granada (Spain), was firstly analysed. These bags can be differentiated into three types as shown in Figure 1: T-shirt bag, die-cut bag and bag with handles. Secondly, a set of 19 types of garbage bags was analyzed, purchased in 9 large stores in Granada. These bags had different sizes but the same shape.

Figure 1: Types of commercial plastic bags analyzed

For characterization of each kind of bag, a template was prepared with the different properties to be measured, as shown in (Table 1), for a type of bag. The different methodologies used to determine the parameters indicated in the table are described below.

Table 1: Template for data collection from a T-shirt bag

Number of bag	
Bag image	
Manufacture material indicated on the bag	
Average thickness (microns)	
Weight (g)	
Dimensions: 	
Density (g/cm^3)	
Type of bag	
Infrared spectrum: Image of the infrared spectrum obtained	
Differential scanning calorimetry (DSC): Image of the DSC obtained	
Solid waste (%):	

Methods

Determination of weight and dimensions

Weight was determined using an Adventurer Pro balance, model AV114C. It has a weighing range of 110 to 0.0001 g. Dimensions were taken with a standard meter according to each type of bag.

Determination of thickness and density

Thickness was determined using a Baxlo Precision micrometer, which has a measuring range of 12.5 to 0.001 mm. To measure density, a Mettler densiometer was used, based on the Archimedes Principle using ethanol as fluid. Density of the solid is determined by the following equation:

$$\rho = \frac{A}{A-B}(\rho_0 - \rho_L) + \rho_L \quad \text{(Eq. 1)}$$

Where ρ = density of solid sample; A = weight of sample in air; B = weight of sample in ethanol; ρ_L = density of ethanol (0.78 g/cm^3); ρ_L = density of air (0.0012 g/cm^3).

Identification of polymer type by Fourier Transform Infrared Spectroscopy (FTIR)

In order to identify as accurately as possible the plastic materials that make up each bag, FTIR analysis was performed on a Perkin-Elmer spectrophotometer, Spectrum-65 model, equipped with a Total Dimmed Reflectance (ATR) device that allows the measurement of very absorbent, very thick or irregular samples, in most cases without the need for prior preparation. It measures in the range of 4000-400 cm^{-1} and with a resolution of 2 cm^{-1}.

Determination of melting point using Differential Scanning Calorimetry (DSC)

Another technique used to determine the materials present in the composition of the bags analyzed is Differential Scanning Calorimetry (DSC). It is a technique that makes it possible to determine, in the case of plastic materials, the melting and glass transition temperatures, among other characteristics. In this study, it was used to identify the materials that make up the bags by obtaining the melting point of the different components. The technique was performed in accordance with ISO 11357-3:2011.

The analysis was carried out on a Perkin-Elmer TG-DSC thermo gravimetric balance, model STA 6000, following a procedure suitable for this type of material and taking into account the correction for memory effect.

Determination of ash content

Determination of ash content in plastics allows determining the quantity of impurities present in each bag, normally due to the incorporation of inks and additives during the manufacturing process. This analysis was carried out by calcination at 600 °C in a Selecta brand muffle furnace with a residence time of 30 minutes in accordance with the international standard ISO 3451-1:2008.

Results and Discussion

Single-use bags

In this paper a wide characterization of single-use plastic bags that can be found in different shops in Granada was carried out. Before starting the characterization study, estimation was made of the different kinds of bags found, obtaining that 60% of the bags analyzed were t-shirts, 30% were die-cut bags and 10% were bags with handles (Figure 2). It should be noted that all large supermarkets and most small shops sell T-shirt bags, while die-cut bags are usually found in other types of shops, such as clothing or accessory stores.

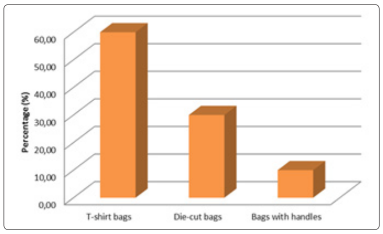
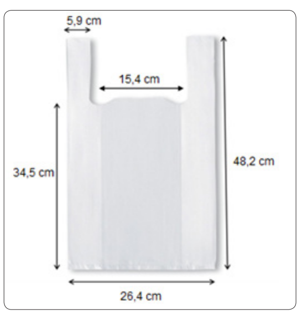


Figure 2: Percentage of every kind of plastic bags analyzed

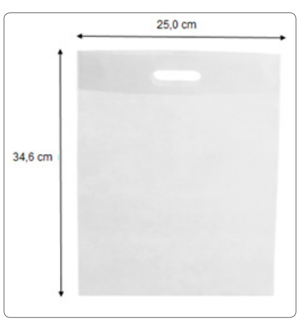
Determination of weight and dimensions

The range of weight values obtained was very wide (between 3 and 30 grams) and depended a lot on the type of bag, size, thickness, etc. The lighter bags weighed between 3-4 grams, such as those commonly used in bakeries. On the other hand, the heaviest bags weighed between 20 and 30 grams, and are usually the most common in clothing, footwear or accessory stores.

In terms of dimensions, the t-shirt bags were all very similar, with variations of 5-10 cm between the smallest and the largest. In general, the length of T-shirt bags was between 47-59 cm, the width of the bags between 18-32 cm and the width of the handles among 5-7.5 cm. In the group of die-cut bags there were bigger differences and, in terms of length, bags from 26 cm long to 59 cm were found. With the width something similar happened, bags with a width of 23 cm to 45 cm were found. Within the bags with handles the differences were smaller; the width values were among 34-46 cm while the length measurements were among 34-42 cm. In any case, it is important to take into account that the variety of shops and bags in the province of Granada is very large; there are even shops that sell the same type of bag with different sizes. In this work an effort has been made to choose as varied a sample as possible. An example of each type of bag and its dimensions is shown in (Figure 3).



A) T-shir bag



B) Die-cut bag

C) Bag with handles

Figure 3: Example of dimensions obtained for every kind of bag

Determination of thickness and density

Thickness values obtained were very variable. The thinnest bags were those dispensed in bakery type shops, also according to the weight described in section 3.1.1. These bags were approximately 13 to 16 microns thick. The supermarket bags were about 30 microns thick. The rest of the bags analyzed had very variable thicknesses, most of which were found to be between 25 and 50 microns thick, with some exceptions of 70 or 80 microns.

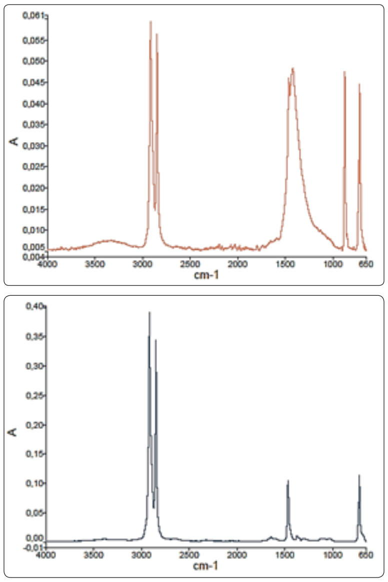
With regard to density polyethylene has values between 0.91-0.93 g/cm^3 and high density polyethylene between 0.94-0.97 g/cm^3 [9]. These values refer to pure polyethylene. Another author referred to the density of medium density polyethylene, between 0.93-0.94 g/cm^3 [10].Taking these values as a reference, the density of each of the bags was calculated and it was found that the range of densities obtained for each material was very wide and did not exactly follow the values reported by the literature for pure polyethylene. It should be kept in mind that plastic products that have already been processed often contain fillers and additives that may vary in their physical and mechanical properties, including density [11]. These additives are added to give color and provide greater strength, generally. The multi-layer film also exists, which is usually composed of three or four layers of different polymers depending on the properties to be changed or improved [12]. This means that each layer may have a different density. The range of minimum-maximum values obtained for each type of material is shown in (Table 2).

Table 2: Density ranges obtained for every polymer present in single-use bags

Material	Density range (g/cm^3)
HDPE	0.80-1.13
LDPE	0.85-0.86
Mixture	0.95-1.05

Identification of polymer type using FTIR

The identification of the type of material using this technique was difficult due to the similarity of the high density and low density polyethylene spectra and the presence of numerous additives. Figure 4A and 4B show two of the 40 infrared spectra obtained in the laboratory for HDPE and LDPE bags, respectively.



A)

B)

Figure 4: Infrared spectra of single-use bags analyzed. A) HDPE bag ; B) LDPE bag

The main difference between the two spectra at first glance was two absorption peaks: the first at 1420 cm^{-1} in Figure 4A, and the second at 873 cm^{-1} in the same spectrum. However, these two peaks do not always differentiate between HDPE and LDPE.

According to several authors, the absorption peaks that characterize the infrared spectra of pure HDPE and LDPE may be similar between the two materials. Tables 3 and 4 show the main peaks reported by these authors in their analyses. Each peak corresponds to a functional group, normally related to the vibrations of the C-H or C-O links [13-15].

Table 3: Frequency and functional group of the peaks corresponding to a pure HDPE infrared

Origin	Wavenumber (cm^{-1})	Functional group
CH_2	2919-2915	Asymmetric C-H stretch
CH_2	2850-2848	Symmetric C-H stretch
CH_2	1472	C-H link
CH_3	1368	C-H link
CH_2	720-730	CH_2 splitting

Table 4: Frequency and functional group of the peaks corresponding to a pure LDPE infrared spectrum. Sources: Modified from [13,15].

Origin	Wavenumber (cm-1)	Functional group
CH_2	2917-2915	Asymmetric C-H stretch
CH_2	2852-2848	Symmetric C-H stretch
C=O	1730-1725	Carbonyl group
CH_2	1472-1462	C-H link
CH_3	1377-1352	“Umbrella” mode
C-O-O-C	890-820	Peroxides
CH_2	718-729	CH_2 splitting

The differences between HDPE and LDPE are centered on the presence of a carbonyl group at 1730 cm^{-1} and a methylene group vibration at 1377 cm^{-1}, both corresponding to the LDPE spectrum. There is another peak that appears only in Table 4, located at 890-820 cm^{-1} that the authors identify as the presence of "peroxides" [13]. Point out that the presence of peroxides depends on the LDPE treatment process and need not always be present.

However, in the bags analyzed in the laboratory, much more peaks were obtained for some samples than those represented in (tables 3 and 4). This showed the presence of additives, pigments, loading agents, etc. that are usually present in processed plastic products and make it difficult to identify the material. The most readily recognized additives in the IR spectra of polymers are calcium carbonate ($CaCO_3$) and titanium dioxide (TiO_2), whose absorption peaks appear at 1420 cm^{-1} and 694-690 cm^{-1}, respectively. The following are the absorption peaks obtained for the spectra in (Figure 4 see Tables 5 and 6), and the corresponding functional groups [16-18].

Table 5: Absorption peaks obtained in the infrared spectrum of a HDPE bag

Origin	Wavenumber (cm^{-1})	Functional Group
CH_2	2915	Asymmetric C-H stretch
CH_2	2847	Symmetric C-H stretch
CH_2	1461	C-H link
$CaCO_3$	1420	Calcium carbonate
C-O-O-C	873	Peroxides
CH_2	729	CH_2 splitting
CH_2	717	CH_2 splitting

Table 6: Absorption peaks obtained in the infrared spectrum of a LDPE bag

Origin	Wavenumber (cm^{-1})	Functional Group
-	3393	-
CH_2	2915	Asymmetryc C-H stretch
CH_2	2848	Symmetric C-H stretch
-	2321	-
-	1645	-
CH_2	1471	C-H link
CH_2	1463	C-H link
CH_3	1376	“Umbrella” mode
-	1301	-
-	1096	-
CH_2	729	CH_2 splitting
CH_2	718	CH_2 splitting

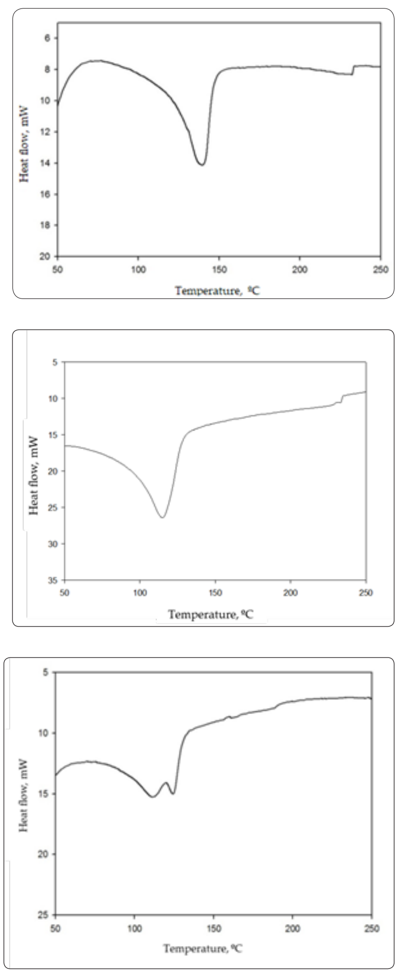
The first important observation was the presence of peroxides in the infrared spectrum of the HDPE sample, which was not consistent with what the authors stated. This spectrum also contained calcium carbonate. The other peaks agreed with Table 3. However, the infrared spectrum of the LDPE had numerous peaks that were not identified with those described in Table 4 and that probably corresponded to additives. The only peak that would differentiate in this case the infrared spectrum of HDPE and LDPE is the one that appeared at 1376 cm^{-1}, characteristic of LDPE.

It should be noted that, in some cases, this identification may be more difficult if the bag has a mixture of both polyethylenes in its composition.

Identification of polymer type using DSC

This technique is more decisive when differentiating between HDPE, LDPE and mixture. Commercial high density polyethylene may have melting temperatures between 120-130 °C, but in most cases the values are closer to 130 °C or even higher, such as those obtained by some authors and in the analyses carried out in this paper [19-21]. On the other hand, the melting temperature of low-density polyethylene is usually between 115 -125 °C, but it can vary above or below these values, since other authors have obtained melting temperatures of 113 °C or 127 °C [22-24].

In the laboratory bag analyses, melting temperatures of 113-115 °C for LDPE and 126-139 °C for HDPE were obtained (Figures 5A and 5B). In the case of the bags that were composed of a mixture of both, the graph showed two clear melting points (Figure 5C) and the melting temperature of HDPE decreased a little with respect to the rest of the bags, presenting temperatures of 124 -125 °C.



A)

B)

C)

Figure 5: DSC of single-use bags analyzed. A) HDPE bag; B) LDPE bag; C) Mixture

Taking into account these results, an estimate was made of the types of bags found, according to the polymer used in their manufacture. Thus, 87.5% of the bags were made of HDPE, 7.5% of the bags were made of a mixture of both and 5% of the bags were made of LDPE.

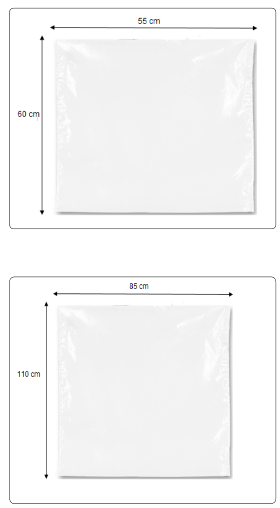
Determination of ash content

Ash content obtained by fully calcining a bag sample is indicative of the content of additives, impurities and other substances added in the manufacturing process of the bag. The average value obtained from the solid waste content for single-use bags is 15.54%. However, the range of values was very wide, from bags containing only 0.81% to bags with 38.88% of waste content. No direct relationship was found between this variation in residue content and the type of bag or other property analyzed.

Garbage bags

Determination of weight and dimensions

Weight of garbage bags varies depending on whether they were household or industrial bags. Domestic bags had an average weight of 14.00 grams, while industrial bags had an average weight of 50.23 grams. Similarly, the main difference in the dimensions of these bags is between domestic and industrial type bags. In domestic type bags, length was from 55 to 73 cm, while the width varied among 47-56 cm. In industrial-type bags length was between 100-110 cm and width among 80-95 cm as shown in (Figure 6).



A)

B)

Figure 6: Dimensions of garbage bags. A) Household bags; B) Industrial bags

In addition, bags which, according to the manufacturer's indication, are made from recycled material were also counted. This is usually quite common in this type of bag. Approximately 42% of garbage bags were made from or contained recycled material. It is important to note that this information is usually provided by the manufacturer on the characteristics of bag package, but there may be bags that contain recycled material and is not indicated.

Determination of thickness and density

Thickness values obtained varied depending on whether they were domestic or industrial bags. Domestic bags had an average thickness of 24 microns, while industrial bags had an average thickness of 32 microns. However, within the industrial bags there were significant differences in thickness, with bags ranging in thickness from 25 microns to 44 microns.

Density of the garbage bags was calculated and compared with the same values described in section 3.1.2. As with single-use bags, densities of the garbage bags covered very wide ranges that were not always close to the data provided in the literature for pure materials [9,10]. Density ranges are shown in (Table 7). The analysis of the garbage bags did not find any that were made entirely of LDPE, so this density has not been indicated in the table.

Table 7: Density ranges obtained for garbage bags

Material	Density range (g/cm^3)
HDPE	0.81-1.05
LDPE	-
Mixture	0.85-1.07

Identification of polymer type using FTIR

Analysis of the garbage bags by infrared spectroscopy provided infrared spectra for HDPE bags and for mixture bags such as those shown as examples in (Figures 7A and 7B), respectively. Likewise, the absorption peaks obtained for the previous spectra were as follows (see Tables 8 and 9).

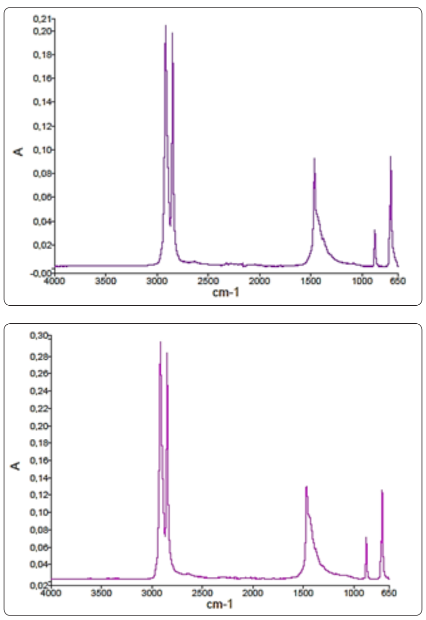


Figure 7: Infrared spectra obtained for garbage bags. A) HDPE bag; B) Mixture.

Likewise, the absorption peaks obtained for the previous spectra are expressed in Tables 8 and 9.

Table 8: Absorption peaks obtained in the infrared spectrum of a HDPE bag

Origin	Wavenumber (cm^{-1})	Functional group
CH_2	2915	Asymmetric C-H stretch
CH_2	2847	Symmetric C-H stretch
-	2163	-
-	2038	-
CH_2	1471	C-H link
CH_2	1462	C-H link
C-O-O-C	874	Peroxides
CH_2	729	CH_2 splitting
CH_2	718	CH_2 splitting

Table 9: Absorption peaks obtained in the infrared spectrum of a mixture bag

Origin	Wavenumber (cm^{-1})	Functional group
CH_2	2914	Asymmetric C-H stretch
CH_2	2848	Symmetric C-H stretch
-	2323	-
-	2019	-
CH_2	1470	C-H link
CH_2	1462	C-H link
C-O-O-C	874	Peroxides
CH_2	729	CH_2 splitting
CH_2	717	CH_2 splitting

The absorption peaks for both spectra were identical. Both had two unidentifiable peaks. These peaks, as already described in section 3.1.3 according to several authors surely corresponded to certain types of additives [13-15]. In general, the infrared spectra obtained for the garbage bags were well matched to the data of these authors, but in this case it would not be possible to differentiate between HDPE or mixture, as there is no peak characterizing either of the two spectra. At best, the only conclusion that could be drawn is that both spectra correspond to polyethylene.

Identification of type polymer using DSC

Analysis of the garbage bags showed that there were no bags made solely from LDPE. Only DSCs were obtained from bags made of HDPE and with a mixture of both (Figure 8A and 8B). The melting temperatures obtained were 126-130 °C for bags made of HDPE. For the bags with a mixture of both polyethylenes, the temperatures were 110-116 °C for LDPE and 123-128 °C for HDPE. These values were correctly consistent with those obtained in the literature already described in section 3.1.4 [19-24].

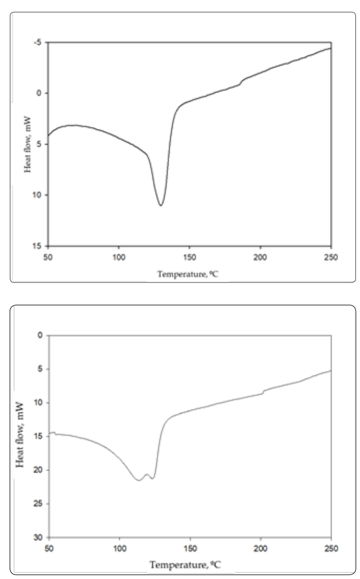


Figure 8: DSC of garbage bags. A) HDPE bags; B) Mixture bags

Finally, the percentage of garbage bags made of HDPE or polyethylene mixture was also analyzed. It can be noted that approximately 68% of the bags were made of HDPE and approximately 31% of the bags contained a mixture of HDPE and LDPE.

Determination of ash content

The average value for ash content in garbage bags was 16.71%. The range of values was very wide, with exchanges containing only 6.47% to exchanges with 27.54% of ashes. These are truly high amounts. These results show once again the presence of numerous additives in the bags.

Conclusion

In this work, a complete characterization and analysis of commercial polyethylene bags using in Granada (Spain) was performed. A continuous tendency, in terms of dimensions, was only detected in bakery plastic bags. Infrared spectra of the regular bags were compared with those obtained by authors for pure polymers and some differences were found. These differences were caused for the presence of additives detected as calcium carbonate and peroxides functional groups. As a result of FTIR and DSC tests, it can conclude that the majority of the analyzed bags were made up using HDPE. Regarding garbage bags two clear tendencies were found: the smallest and lightest ones corresponded with domestic use and the heaviest ones with industrial applications. No LDPE garbage bags were found in Granada (Spain) stores and infrared spectra showed that there were no differences between those classified as HDPE made up and the mixture bags (HDPE and LDPE). In both cases (regular and garbage bags) the ash content due to additives did not depend on the type of polymer used.

Declaration of Conflicting Interests

The Authors declare that there is no conflict of interest.

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