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CHARACTERIZATION OF A RECYCLED POLYMER BY A GRINDING-MOLDING PROCESS

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Abstract: HDPE bottles were characterized after being recycled through a Crushing-Moulding process. The Infrared Spectroscopy (FTIR) technique was used to identify the type of Polymer resulting from recycling. Shore D hardness (S_D) and uniaxial stress tests were performed to characterize the mechanical properties of the samples. Micrographs were taken by Scanning Electron Microscopy (SEM) to analyze the behavior of the fracture after the stress tests. The mechanical properties in tension were compared with finite element simulation data to predict the maximum tensile strength of different polymer designations. The FTIR analysis classified the recycled material as a Low Density Polyethylene (LDPE) of bands associated with talc. The results of both the mechanical tests and the numerical model agree with this type of material. Fractographic analysis showed intragranular deformation and dimpling caused by poor adhesion of talc particles to the laminar microstructure.
Keywords: LDPE, FTIR Spectroscopy, Scanning Electron Microscopy (SEM), Finite Element Analysis (FEA), Recycling, Hardness.

INTRODUCTION

Polyethylene in its simplest form consists of a long chain of an even number of covalent bonds linked to the carbon atoms with a pair of hydrogen atoms linked to each of them; ending with methyl groups at the end of the chain. Chemically, pure polyethylene resins consist of alkanes with the formula shown in Eq. (1)

$$C_{2n}H_{4n+2}$$

High Density Polyethylene (HDPE) is chemically closest in structure to the polyethylene chain presented in Eq. (1). Its structure consists of molecules with few branches and with few defects that mar their linearity. With extremely low level of defects to hinder the organization of the molecules and a high degree of crystallinity; which allows resins with densities of 0.95±0.02 g/cm3; compared against Low Density Polyethylenes (LDPE) that present densities of 0.90±0.2 g/cm3. The above-described properties of HDPE make it amenable to applications that include large-scale packaging; such as industrial tanks and barrels for chemical use. For commercial and domestic use, these are expected to have good stiffness, low permeability, and high strength; which makes them ideal for containing liquids for domestic, industrial and automotive use: chlorine bleach, car oil, antifreeze, among other commercial products. When making containers with thin thicknesses, its greatest application is in milk bottles and butter tubes. (1) (2) The National Institute of Statistics and Geography (INEGI) reported that 107,056 tons of garbage generated in homes, buildings, streets, and recreational places were collected in Mexico in 2019. (3) This waste reaches a collection place where recyclable garbage is separated into different families; plastics being the ones with the greatest presence with 48% of the total mass collected. Plastic bottles represent a serious danger to the ecosystem of the northeastern region of Mexico, since the previously shared data talks about collected garbage and does not mention those that do not reach the collection center and are found in the environment. (4) Pollution by plastic waste is a common problem in the world and Mexico has been affected by it. As a result of the above, civil associations, academic groups and private investment groups have proposed investing in new and easily accessible recycling processes, prohibiting single-use plastics and proposing a change in habits as plastic users. (5) Due to the above, the objective of this study is to evaluate the mechanical and microstructural properties of a polymer recycled from HDPE bottles.

EXPERIMENTAL PROCEDURE

MATERIAL

Half-gallon HDPE bottles for milk, found in different cleaning campaigns in the northeast region of Mexico, were collected. The cap rings were removed from these; discarding them from this process, to make batches of 150 bottles, see Fig. 1a. The polyethylene obtained was cleaned using a stainless steel container by immersion and shaking, in a 10% solution of household detergent with distilled water, heating at 70 °C for 0.5 h. After the above, after time, the polyethylene was washed with distilled water and left to dry in an oven at 100 °C for 2 h.

The clean bottles were fed into a mill; Gumar brand model 1200-70 with 6 fixed blades and 9 rotary blades with a power of 70 hp, with the purpose of reducing the size of the raw material to be recycled, this was repeated two more times to have homogeneous sizes of polymer for the process casting and casting, see Fig. 1b. A final cleaning was carried out by immersion and agitation of the crushed material in a 20% Sodium Hydroxide (NaOH) solution at 75 °C for 1 h. The final drying was carried out by washing the polyethylene with distilled water and drying it at 100 °C for 2 h in an oven. (6) With a Cincinnati Milacron brand plastic injection machine; see Fig. 1c, the granulated plastic was heated to 200 °C to achieve melting and the plastic was molded into frames for 3 cm thick plates, see Fig. 1d. Loctite NC55 liquid was used as release agent. Finally, the plates were rectified to take care of their straightness and that they had parallel faces.

Figure 1 Recycling process of the collected bottles: a) Batch of clean bottles, b) crushed, c) Injector/Moulder and d) Molding on racks.

MECHANICAL PROPERTIES

To characterize the mechanical properties of the material, Uniaxial Tensile Tests were performed. Ten square specimens were tested with a test section width (w) of 13 mm and a calibrated length (L) of 50 mm, Table 1. These specimens were machined in the longitudinal direction of the plate, taking care to maintain parallelism between faces. and to maintain a surface free of imperfections. conforming to ASTM D638-10 standard. Finally, the machining marks were removed by sanding the specimens with 800 grade SiC sandpaper, see Fig. 2.

W	D	LO	L	G	R	WO	T	Wc
14	115	165	57	50	76	19	13	13

Table 1 – Dimensions of the Uniaxial Tension Specimen (mm).

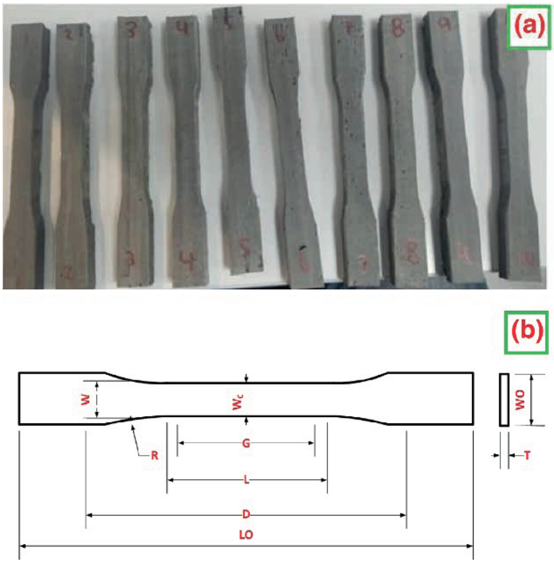

Figure 2 Uniaxial Tensile Specimen used in the investigation: a) Prepared specimens and b) Diagram of the Specimen.

The hardness of the material was also characterized by Shore D hardness tests; following the ASTM D2240-03 standard, measuring five times in three different points of the test pieces: in the central, upper and lower part of the test specimen. The results of the mechanical tests were averaged, with the purpose of a better understanding of the recycled material. (7) (8) (9)

ANALYSIS BY THE FINITE ELEMENT METHOD (FEM)

An analysis was made using the Finite Element Method (FEM) to simulate the uniaxial tension test and estimate the maximum stress of the material (UTSsim). Solidworks Simulation Premium version 202 software was used; with Institutional license, to solve a 3D model through static simulation. The specimen was considered as a deformable element with a triangular mesh, with an element size between 1.50-34.00 mm base. The mesh quality was 86,000 elements and 127,000 nodes, see Fig. 3. The test specimen was clamped at one end and a force was applied at the other to simulate the conditions of a universal tension machine. Table 2 shows properties of designations corresponding to HDPE and LDPE materials found in the literature. This with the purpose of comparing the simulation results for both designations against the properties of the recycled material. The maximum Von Mises stress obtained by the model was recorded for the proposed analysis. (2) (11) (12)

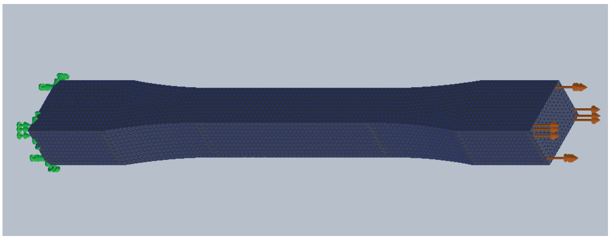

Figure 3 3D model with mesh quality for FEM analysis.

FTIR SPECTROSCOPY TESTS

Identification and characterization tests of the functional groups of the polymer were carried out using the Fourier Transform Infrared Spectroscopy (FTIR) technique. A Thermofisher model Nicolet is20 spectrophotometer coupled with an Attenuated Total Reflectance (ATR) accessory was used. Polymer analysis was performed directly on the samples without the need to prepare Potassium Bromide (KBr) pellets. The type of polymer was identified after the recycling process by plotting Transmittance (%) – Wave Number (cm-1) with a spectral resolution of 4 cm-1. The number of scans for each acquisition was set at 16 shots. To compare the recycled material with respect to the original material: 10 x 10 x 0.6t mm samples were taken from the bottles before recycling. The samples of the original material and the recycled material were analyzed by plotting Transmittance (%) – Wave Number (cm-1) in the FTIR spectrophotometer. The bands chosen for the analysis of the polymer were those found in the regions: 3000-2800, 1550-1400 and 750-650 cm-1.(16) The components within the spectrum were manually identified, using the procedure proposed by R Chércoles in his work Analytical characterization of polymers used in conservation and restoration by ATR-FTIR spectroscopy (16) and by Prof. G. Sócrates in the book Infrared and Raman Characteristic Group Frequencies and by means of the procedure proposed by. (17)

Material	Young's modulus, AND (MPa)	Yield Strength (MPa)	Poisson coefficient, ν
LDPE1 (1,2)	227.50	11.73	0.420
LDPE 2 (10)	207.26	26.78±0.64	0.420
HDPE1 (1,2)	1072	28.6±2.00	0.420
HDPE2 (1, 2)	750	18±0.95	0.420
Recycled material	281.33	18.4	0.420

Table 2 – Mechanical properties used for modeling by FEM.

SCANNING ELECTRON MICROSCOPY (SEM)

An FEI Quanta FEG 650 Field Emission Scanning Electron Microscope (SEM) was used to observe the morphology of the recycled polymer and analyze the fracture surface after the tensile test. The observation areas were cut to sizes suitable for movement within the vacuum chamber of the microscope and an Au film was deposited on them with a Quorum EMS 150-R model evaporator. The observation was made in the Secondary Electron mode, with an Acceleration Voltage (HV) of 10 kV, a Working Distance (WD) of 29 mm, a beam size (Spot Size) of 3.0 and with magnification of 250X. 500X and 1500X. The observation of fracture zones was carried out using images at 250X using image analysis software and determining the ductile and brittle percentage proportion of the invoice.

RESULTS AND DISCUSSION

RECYCLING PRODUCT IDENTIFICATION

Figure 4(a-b) shows the spectrum of the original and recycled material, zoomed in the 1400-1300 cm-1 region. The literature mentions that there are bands assigned to identify the type of Polyethylene (PE) associated with the CH2 and CH3 groups: Band I at ≈1377 cm-1, Band II at ≈1366 cm-1 and Band III at ≈1351 cm -1. (14) The spectrum for the parent material is shown in Fig. 4a; In this, a highly intense Band II is observed at position 1366.85 cm-1 and Band III at position 1352.39 cm-1. The shape of Band III; a defined hill, is characteristic of High Density Polyethylenes (HDPE). The spectrum of the recycled material is observed in Fig. 4b, where there is the presence of Band I at position 1375.74 cm-1 and Band III located approximately at position 1359.24 cm-1. Unlike the unrecycled material, Band III found in the recycled material shows a plateau shape, without much band definition. This is attributed to the low density of Polyethylene, which identifies the recycled material as Low Density Polyethylene (LDPE). Fig. 5 presents the full range spectra (4,000-600 cm-1) of the original sample (Fig. 5a) and the recycled sample (Fig. 5b). It can be seen in Fig. 5a, marked with a green ovoid, that the material matches the characteristic bands of high-density polyethylene. Bands of asymmetric tension (νasymmetric) and symmetric tension (νsymmetric) were identified for the -CH2- bonds, at positions 2914.08 cm-1 and 2846.46 cm-1, respectively. Bands of asymmetric doubling (δasymmetric) were found in 1471.50 cm-1 and symmetric doubling (δsymmetric) in 1461.29 cm-1; both represent C-H bonds.

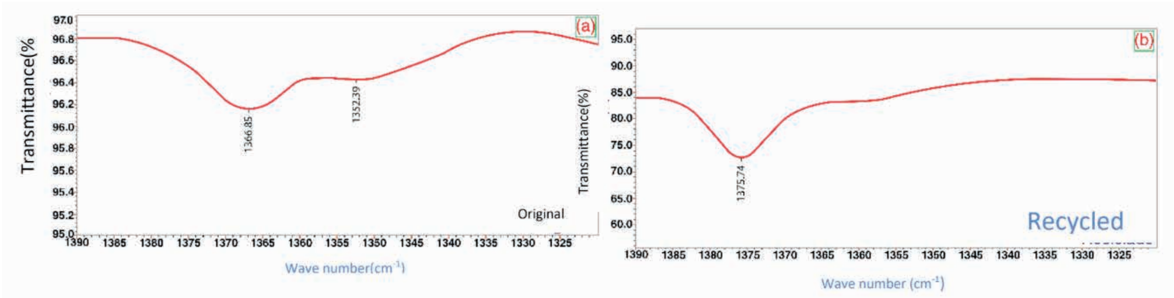

Figure 4 Spectrum obtained by the FTIR technique of the material: a) Original and b) Recycled.

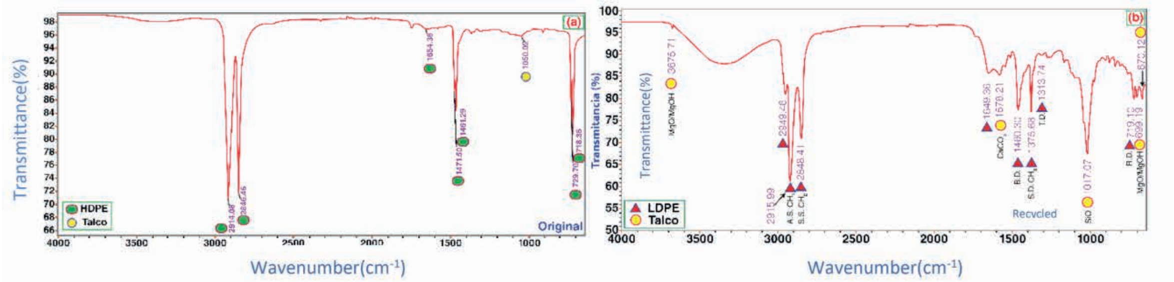

Figure 5 Infrared spectrum obtained by full range FTIR for the material: a) Original and b) Recycled.

The last characteristic bands identified were the rolling doubling bands (δrolling) at positions 729.70 cm^{-1} and 718.36 cm^{-1}; these represent C-H (-CH2-)n≥26 bonds. At position 1050.02 cm^{-1} a weak band was found, representative of SiO bonds in Talc. (15) (16) The complete spectrum of the recycled material is presented in Fig. 5b. The characteristic bands of polyethylene identified in the recycled material were marked with a red triangle. The asymmetric tension band (νasymmetric) was found at 2915.99 cm^{-1} and symmetric tension (νsymmetric) at 288.41 cm^{-1} for the -CH2- bonds. In the positions 1460.30 cm^{-1} and 1375.68 cm^{-1}, the bands of asymmetric doubling (δasymmetric) and symmetric doubling (δsymmetric) were found in the plane for the C-H bonds. The last characteristic band identified was the roll doubling (δroll) at position 719.10 cm-1 for the C-H(-CH2-)n≥6 bonds. The presence of MgO/MgOH bands at positions 3675.71 cm^{-1} and 699.19 cm^{-1}, SiO bands at positions 1017.07 cm^{-1} and 670.12 cm^{-1} and a band at position 1578.21 cm^{-1} for the group were also identified. CaCO3. These bands were identified with a yellow circle in Fig. 5 and are characteristic of Talc. (18)(19) With the analysis of FTIR spectra obtained in this work, it is concluded that after recycling, the recycled material presented characteristics of Low Density Polyethylene (HDPE) with the presence of a contaminant that shows MgO/MgOH and SiO bonds, associated with talc.

MECHANICAL CHARACTERIZATION AND FINITE ELEMENT ANALYSIS (FEA)

Fig. 6 (a-b) shows the graphs of the mechanical properties obtained by hardness tests on the Shore D (SD) scale; see Fig. 6a, and uniaxial tension tests; see Fig. 6b. The average hardness of the material; obtained from the ten manufactured specimens was 58 SD, this is observed in Fig. 6a. This value coincides with what has been reported in the literature and in conventional low-density polyethylene mechanical property sheets, since these show an average of 55 SD. (1) (22) (23) Regarding the properties obtained by the tensile tests, the material presented a maximum resistance to average tension (UTSprom) of 19.1 ± 0.67 MPa. FEM modeling; Fig. 7, showed that the areas of greatest effort are represented in the model with a lemon green hue. Analyzing this region, the zone of possible fracture was estimated to be found in a position between 125.00-142.00 mm along the axial load axis from the origin of the specimen (0,0,0). Measuring the positions of the fracture zones in the specimens tested by the tensile test, the real distance was found between 90.75 and 114.58 mm, see Fig. 7b.

Fig. 8 shows the comparison of the Stress (σ) – Strain (ε) curve obtained by the uniaxial stress tests and by the finite element simulation. The LDPE1 designation presented a curve with a UTSsim value = 12.01. In the case of the uniaxial test on the recycled material, the curve shows UTSreal = 19.1 MPa. The curve was simulated with the properties obtained by the tensile tests and the value obtained was UTSsim = 18.39 MPa. The correlation coefficient (R2) was obtained between the double logarithm graph of the real data against the simulated values; see Fig. 9, this being 97.49%.

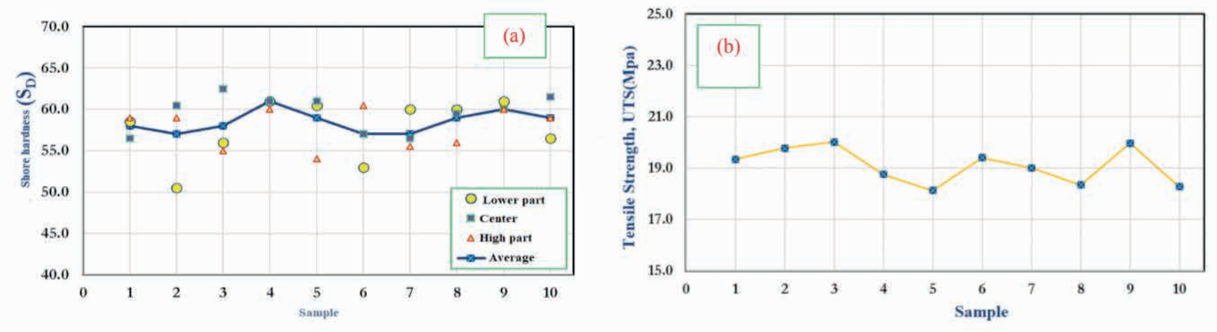

Figure 6 Mechanical properties obtained in the recycled material by: a) Shore D Hardness Tests and b) Uniaxial Tensile Tests.

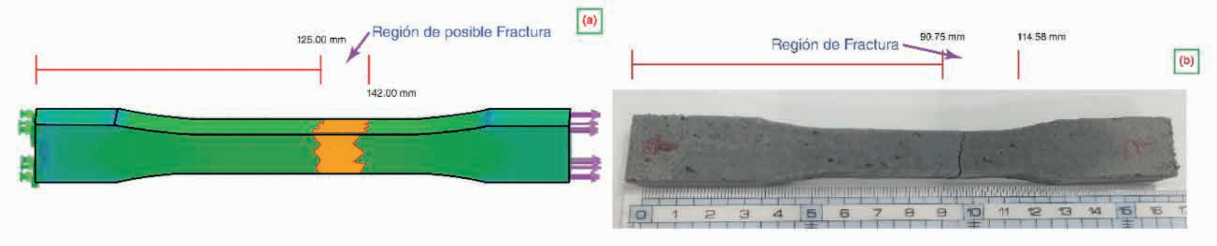

Figure 7 FEM modeling: a) Solved geometry and b) Fractured specimen.

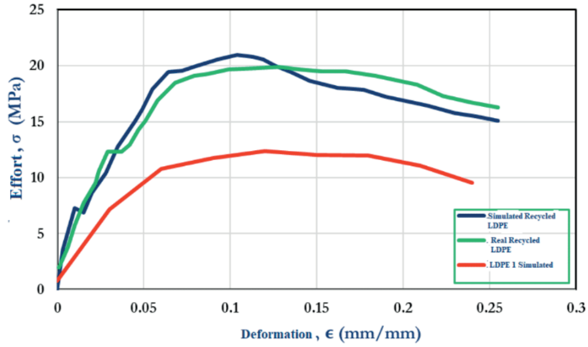

Figure 8 Some Effort (σ) – Deformation (ε) graphs obtained in the study.

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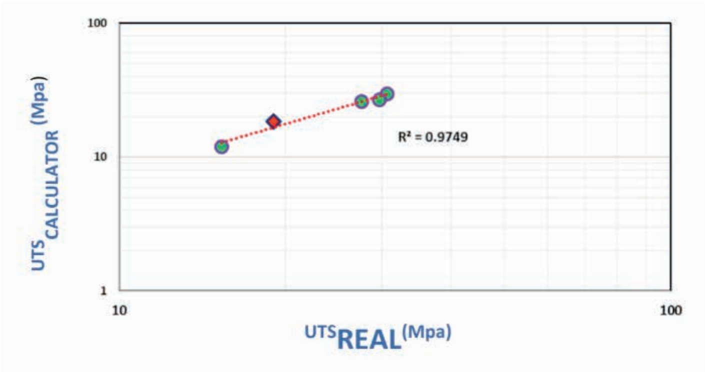

Figure 9 Graph of comparison of the Maximum Effort at UTSreal Tension against the Maximum Effort at $UTS_{calculated}$ Tension.

Based on the reported mechanical properties and finite element modeling, the recycled material presented properties similar to low-density polyethylene (LDPE). The properties obtained in this report indicate that this recycled material can be used in containers for chemical products, packaging, toys, beverage bottles, basic plumbing objects, and insulating cable covers. (2) (10) (29)

MICROSTRUCTURE OF THE FRACTURE ZONE

The fracture zone of the tested specimens was analyzed by uniaxial tension tests, see Fig. 10 (a-d). A linearly oriented membrane matrix with the presence of porosity is observed, this being characteristic of LDPE Polyethylenes, See Fig. 10a. (24) Large dimples (Dimples) can also be seen, which are attributed to the presence of talc in the material; as well as an intragranular fracture mechanism, see Fig. 10(b-c). When performing the tensile tests on the material, after passing the elastic limit and beginning plastic deformation, the poor adhesion of these particles to the base material initiates the growth of the dimples until the breaking point is reached. Talc acts as a compound that interferes with the Van der Waals chemical bonds of polyethylene, causing the packing of possible talc particles and their interaction at the laminar interface of the polymer to lack good adhesion. On closer inspection; Fig. 10d, the fracture zone has a greater presence of plastic deformation due to intragranular deformation and microvoids, characteristic of ductile fracture (95±2% of the total area). Few zones show brittle fracture characteristics (5±1.5% of the total area), such as cleavage. (24) (25) (26) (27)

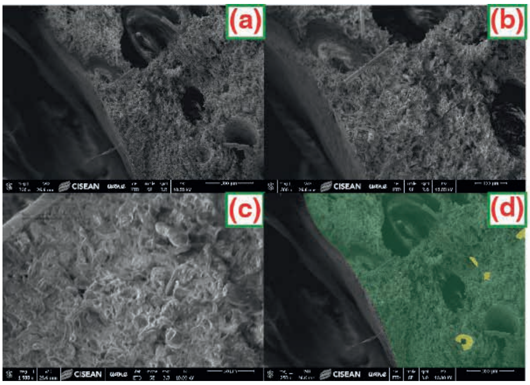

Figure 10 – Micrographs of the fracture zone of a tested LDPE specimen, obtained by Scanning Electron Microscopy: a) 250X, b) 500X and c) 1500X.

CONCLUSIONS

Using a recycling process by crushing-molding, it was possible to recycle High Density Polyethylene (HDPE). The conclusions reached in this work on the characterization of the material obtained through recycling are listed below.

- The FTIR spectrum obtained in

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the recycled material showed bands associated with Low Density Polyethylene (LDPE).

- In the recycled material, bands of MGO/MgOh and SiO were identified, associated with Talc.

- The mechanical properties obtained in the recycled material through Shore D hardness and Uniaxial Tensile tests show similarities with low density polyethylene designations.

- Finite element modeling showed that the recycled material has similarities to low-density designations on polyethylenes.

- Observation by electron microscopy showed characteristics of low-density polymers and the fracture zone presented an intragranular deformation.

THANKS

The process of collecting and cleaning the original material was a crucial stage for this work; since cleaning brigades were carried out to reduce the environmental impact of the bottles collected. This work was carried out in different areas of the city of H. Matamoros, in Tamaulipas, Mexico. A local plastics company was involved in the recycling process, with which the original material (HDPE) was crushed, processed and molded to obtain the recycled plates that were identified as LDPE. The Solidworks simulation and the mechanical tests of Shore D hardness and uniaxial tension were carried out at the Instituto Tecnológico de Matamoros. The tests carried out with the Infrared Spectrophotometer (FTIR) and the observation by Scanning Electron Microscopy (MEB) were made at the Northeast Environmental Sustainability Research Center (CISEAN).

All the people who participated in this project are part of the community and the main objective was to involve the inhabitants of the region to reduce the environmental impact of plastic waste affecting green areas or streams. It is expected that with this job; With the detailed description of the process and with the characterization of the recycled material, the population is encouraged to participate more in some of the recycling stages and try to use this process to reduce the footprint left in the environment with the waste. plastics. This is reinforced by the applicability of the recycled material due to the mechanical properties obtained in this study.

To conclude, thanks are extended to the Tecnológico Nacional de México (TecNM) campus Instituto Tecnológico de Matamoros and to the Center for Research in Energy and Environmental Sustainability of the Northeast (CISEAN) of ‘’ Universidad Autónoma del Nordeste” (UANE) for the support provided to development and conduct of this research.

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