Development of composite materials from recycled irrigation tape and corn stover

Desarrollo de materiales compuestos de cintilla de riego reciclada y rastrojo de maíz

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Abstract

In this work, the formulation of composite materials of Recycled Irrigation Tape and Corn Stover (r-IT/CS) with additives, were studied. The variables analyzed were the size effect and the percentage of fibers. The r-IT/CS composites were processing in a twin screw extruder and compression molded. The materials characterization were by tension, bending, impact, hardness, and DSC measurements. As the fiber concentration and length increased, modulus, resistance (tension and bending), and hardness increased, but impact resistance decreased in comparison with the r-IT matrix. The percentage of crystallinity decreased by 4% with the addition of the CS fibers. The properties of the composite materials were similar to those of the polymeric matrix. These results open the possibility of using them as packaging.

Stover, Processing, Characterization

Resumen

En este trabajo se estudió el análisis de la formulación de materiales compuestos de Cintilla de riego Reciclada y Rastrojo de maíz (r-IT/CS) con aditivos. Las variables estudiadas fueron el efecto de tamaño y el porcentaje de fibras. Los r-IT/CS fueron procesados en un extrusor doble husillo y moldeados por compresión. Los materiales fueron caracterizados mediante el análisis de tensión, flexión, impacto, dureza y DSC. El incremento de la concentración y de la longitud de las fibras aumentó el módulo, la resistencia (tensión y flexión) y la dureza con respecto a la matriz de r-IT, sin embargo, en el caso de la resistencia al impacto disminuyó. El porcentaje de cristalinidad bajó 4% con la adición de las fibras de CS con respecto a la matriz de r-IT. Las propiedades de los materiales compuestos estudiados fueron similares a la matriz polimérica. Estos resultados abren la posibilidad de que puedan ser utilizados como empaques.

Rastrojo de maíz, Procesamiento, Caracterización

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Introduction

Agriculture in Mexico is one of the principal activities from the perspective of economic, cultural, and environmental. Agricola production occupied approximately 18 million hectares of the national territory in 2021 (Instituto Nacional de Estadística y Geografía (INEGI), n.d.). The state of Guanajuato has been characterized as one of the largest agricultural producers in the Mexican Republic. In the case of corn production, Guanajuato has about 400 thousand active hectares (Servicio de Información Agroalimentaria y Pesquera, n.d.) of which each 1kg of corn grain produces approximately 1 kg of corn stover as a by-product (gob.mx, n.d.).

The use of plasticulture is one of the most efficient ways to increase the yield of crops or to convert infertile land into one that is productive. It consists of the use of plastic material for protection or irrigation. Although these technologies offer benefits, they could end up in special handling waste at the end of their useful life. The amount of waste generated by plasticulture in Guanajuato is estimated at 135000 tons per year (de Medio Ambiente y Ordenamiento Territorial, n.d.), of which 50% corresponds to Drip Irrigation Tape (IT). A typical formulation for IT is a blend of LDPE/HDPE/LLDPE (~25/~50/~25) and carbon black (Tynys & Fawaz, 2020). LDPE provides good processability, HDPE provides mechanical strength, LLDPE provides good weldability with the drippers and sufficient ESCR and carbon black is added for UV protection.

One of the most significant problems on our planet is environmental pollution since it puts biodiversity at risk and consequently affects the benefits it provides us. In addition, there are various types of contamination, such as the dioxins released by the burning of corn stover and the lack of control in the disposal of plastics at the end of their useful lives. Therefore, it is necessary to develop research to generate scientific knowledge on how to recycle plastics following the global circular economy initiative.

A number of investigations are conducted on natural fiber-polymer composite materials. In order to improve the chemical interaction between the fiber and the matrix, different strategies have been used. The most reported chemical treatments are those involving surface modification (increase the surface roughness, alkaline methods, etc.) and coupling agent addition (chemical bonding using grafted fibers, Maleic Anhydride-grafted PE) (Vigneshwaran et al., 2020) (Nurazzi et al., 2021) (Aravindh et al., 2022). The main objective is to improve the compatibility between natural fibers (hydrophilic) and polymer matrix (hydrophobic). This improvement is reflected in the mechanical properties of the composite materials. In the majority of cases the tensile strength and flexural strength increases due to enhanced stress transfer between the fiber and the polymeric matrix. The fiber's composition, length (Gandhi et al., 2019), cellulose content, lignin content (Serra-Parareda et al., 2020), and L/D ratio mainly determine their performance as reinforcement.

Youssef et al. (2015), evaluated corn husk fibers reinforced recycled low density polyethylene composites (Corn husk fiber/ R-LDPE). They reported that increment in fiber loading caused an increment in moduli and tensile strength, whereas hardness reduced. DSC analysis revealed that the addition of corn husk fibers by different loadings to R-LDPE matrix leads to decline the crystallinity of the R-LDPE and the incorporation of filler to polymeric matrix slowed crystallization time. Finally, FTIR results indicated that the polymer matrix and corn husk fibers had good compatibility with the increasing fiber loading.

To the best of our knowledge, there are no published papers analyzing the effect of Corn Stover size and composition in recycled Drip Irrigation Tape matrix composites on mechanical and thermal properties.

The objective of the current study was to investigate the effect of Corn Stover fiber length and composition on the mechanical (tensile, flexural, impact and hardness) and thermal (DSC) properties of Recycled Drip Irrigation Tape/ Corn Stover Composites (r-IT/CS).

Methodology

Materials

A commercial waste Drip Irrigation Tape was used as the polymer matrix material. Composite filler was Corn Stover from local production in Juventino Rosas, Guanajuato. Two additives were used in this work. A reactive coupling agent maleic anhydride grafted polyethylene, MAPE (POLYBOND 3029, Brenntag) and a processing aid (Struktol TPW 104).

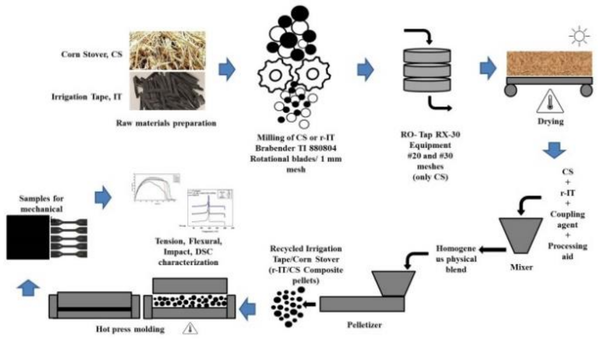

Figure 1 Methodology for the preparation of raw materials and compounding of composites used in this work
Source: Own elaboration

Preparation of materials

Figure 1 presents the methodology for the preparation of raw materials and the compounding of composite materials. The Drip Irrigation Tape (IT) was washed with cold water and soap to remove contaminants (dust, etc.), then dried at room temperature.

Afterward, IT was pulverized over a 1 mm screen to obtain recycled Irrigation Tape (r-IT) powder. The Corn Stover (CS) was ground and sieved over meshes # 20 and # 30 to obtain particle sizes between 0.3 mm (CS1) and 0.5 mm- 1 mm (CS2). Coupling Agent and Processing Aid additives were milled with a screen plate of 1 mm holes in diameter.

Composite Materials

For the composites, the CS and the Coupling Agent were dried at 70°C for 24 h in a convection oven. **Table 1** shows the different percentages of the raw materials used for the compounding. All of the components were dry-blended in a horizontal mixer with a helical agitator (model ML-5; Intertécnica Co., Mexico City, Mexico).

The resulting blends were fed into a conical twin-screw extruder (model BT-25, Brabender®) for melting and mixing with a 20 rpm screw speed. The temperature profile along the heating zones was between 100 °C and 180 °C. The composite strips were pelletized with a laboratory pelletizer machine (type 12-72-000, C.W. Brabender® Instruments, Inc., South Hackensack, NJ, USA). After processing, the composites were compression-molded in a CARVER (USA) semi-automatic laboratory press at 190 °C. The sheets obtained were 16*16 cm^2, 3 mm thick, and the pressing time was 25 minutes.

Material	IT (wt%)	CS1 (wt%)	CS2 (wt%)	Polybond 3029 (wt%)	TPW 104 (wt%)
r-IT	100				
9010CS1	90		10	5	5
9010CS2	90	10		5	5
8020CS1	80		20	5	5
8020CS2	80	20		5	5

Table 1 Percentages of the raw materials used for the compounding
Source: Own elaboration

Mechanical characterization

The mechanical properties of r-IT and the composites were measured by tensile, impact hardness, and flexion tests. Before testing, all the samples were conditioned for 40 h at 23 ± 2 °C and 50 ± 5% relative humidity. The tensile and flexion tests of composite materials were performed according to ASTM-D638 and ASTM-D790 standards respectively in a universal testing machine (model AGS-X, Shimadzu Scientific Instruments, Columbia, MD, USA) with a constant strain rate of 5 mm/min. The impact strength of the composites was measured using an Impact tester Pendulum type, CEAST, and test specimens were prepared according to the ASTM-D256 method. Finally, hardness measurements for the materials were based on the Shore D scale.

Thermal Characterization

The thermal transitions of IT, r-IT and the composites r-IT/CS (901OCS2) were analyzed by a differential scanning calorimeter analyzer (DSC Q100, TA Instrument, USA). The samples were heated to 200 °C with subsequent cooling to 30 °C both at 10 °C/min in a nitrogen atmosphere.

Results

3.1 Tensile Properties

To study the performance of CS fiber-like reinforcement, the mechanical properties of r-IT/CS composites were examined. Many studies reported that surface modification and coupling agent addition promote the interaction between the polymer matrix and the filler fibers.

It is well known that strong interfacial interaction allows the effective stress transfer from the polymeric matrix to rigid filler fibers (Rangappa et al., 2021).

Figure 2 illustrates the possible reaction coupling agent-fiber surface. The fiber is composed of cellulose/hemicellulose/lignin and the coupling agent for reactive Maleic Anhydride (MA) groups grafted. During melt blending, the Anhydride group present in the Coupling Agent could react with the OH- present in the fiber (forming an ester bond) or could be formed a hydrogen bond (forming an intermolecular force) (Rowell, 2007).

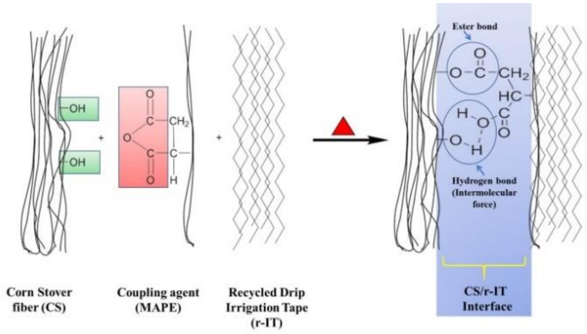

Figure 2 Mechanism of compatibilization proposed between Coupling Agent (MAPE) and Corn Stover fiber surface (CS)
Source: Own elaboration.

Figure 3 presents the stress-strain curves of r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composites with different fiber length. In general, in all composite materials, it is noted that the tensile strength increased **in** comparison with the r-IT matrix. This result could be related to the stresses transferred between fiber-matrix that had been favored by the coupling agent added to the composites (Rowell, 2007) (Nurazzi et al., 2021). Moreover, the Strain values for 9010CS2 and 8020CS2 are higher and in the case of 9010CS1 and 8020CS1 are similar when they are compared with the r-IT matrix.

It can be concluded by examining the improvements in the tension mechanical properties during melt blending that MAPE contributes to better compatibilization.

Figure 4 and Figure 5 illustrates the results tendencies of tensile tests for the composites in comparison with the r-IT matrix. In the case of tensile strength, (**Figure 4a**) the increment was independent of the fiber length. In the case of 9010CS1 and 9010CS2 composites, the tensile strength increased from 13.72 MPa to 14.3 MPa with 10 wt% CS added with 0.3 mm of fiber length.

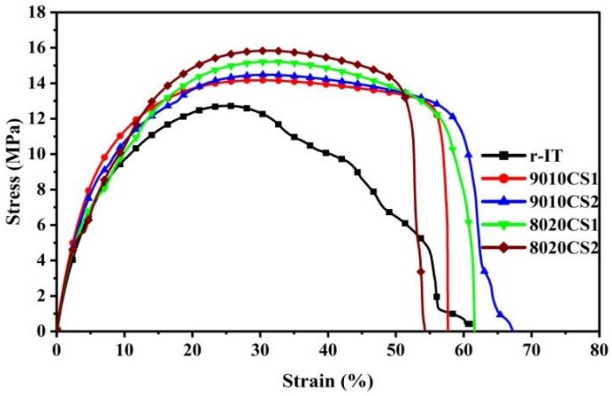

Figure 3 Stress-Strain curves for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials
Source: Own elaboration [OriginLab]

These values were similar to the r-IT (13.03 MPa). In the case of 0.5 mm -1 mm of fiber length in the 8020CS1 (15.0 MPa) and 8020CS2 (15.67 MPa) composites the tensile strength improved 20% with respect to the r-IT matrix. Differences in tensile stress with different fiber lengths could be explained by the interaction fiber-matrix. It is known that the mechanical performance of composite materials depends if the fiber is well dispersed in the matrix. In this work, 5% of MAPE coupling agent was utilized to improve the interfacial interaction between the hydrophobic r-IT matrix and the hydrophilic Corn Stover matrix.

It has been reported that a fiber length of 1mm promotes higher mechanical properties (Nestore et al., 2013) (Gandhi et al., 2019). In general, the tensile strength is expected to decrease with the addition of short fibers, but our results are consistent with Zhang et al. (2020). They analyzed PIW/ MAPE / Lubricant / LLDPE composites and concluded that had higher tensile strength and Young's modulus with the addition of PIW.

This could be explained by the reactive compatibilization during extrusion compounding since the Coupling Agent settles at the fiber-matrix interface and serves as a medium for providing effective interfacial bonding with the r-IT matrix. In consequence, stress is transferred without the failure of the matrix. In the case of Yield strain results, the highest value was for 8020CS1 composite, i.e., 30.13%, and the lowest value was for 9010CS1 composite, i.e., 22.25%. This effect could be explained by the fiber length. The first case was 0.3 mm and the second case was between 0.5-1mm (**Figure 4b**). The results of Young´s modulus are presented in **Figure 4c**. The addition of CS makes the composites stiffer than the recycled Irrigation Tape matrix.

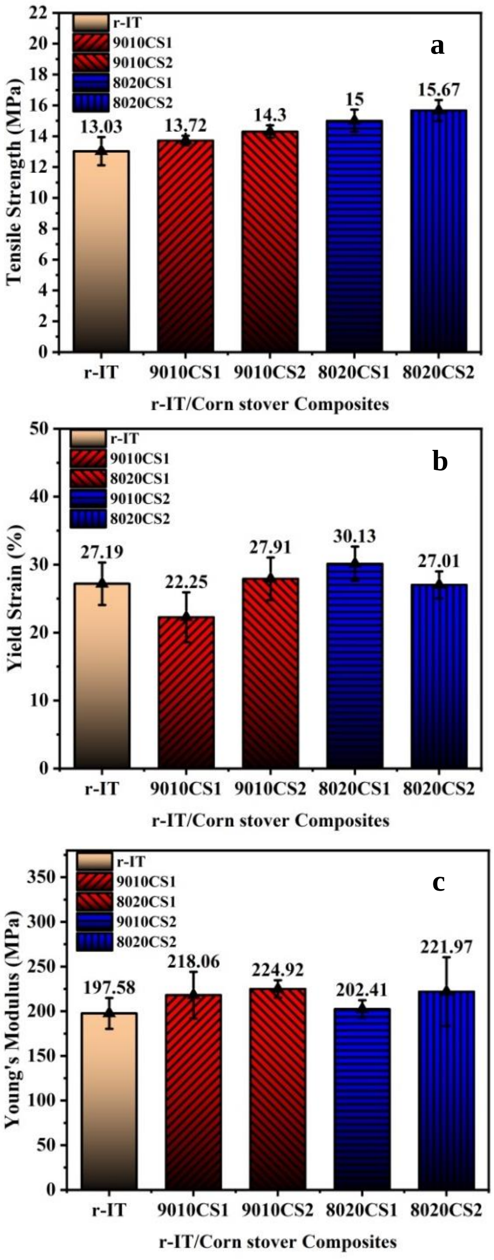

Figure 4 Tensile strength (a), Yield strain (b), Young´s Modulus (c) results for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials

Source: Own elaboration [OriginLab]

The modulus is related to the stress transfer at the interface during loading. It is worth mentioning that the fibers used in 9010CS1, 9010CS2, 8020CS1, and 8020CS2 composites are considered short natural fibers. All the composites had a higher Young´s Modulus than the r-IT matrix. Due to the contribution of Corn Stover to the reinforcing effect (limited mobility and deformability of the r-IT matrix) and its interaction with the MAPE coupling agent, the composites presented favorable stiffness values.

Several researchers have reported that only a certain wt% of randomly distributed natural fibers with short length improve properties (20-40 wt%) (Arumugam et al., 2022). The short fiber characteristics provide poor resistance due to the high-stress concentration when the fiber content is low, resulting in non-reinforcing fibers in a diluted matrix. The behavior of Young´s Modulus data could be understood from the better extent of stress transmittance caused by the interfacial bond and interactions fiber/matrix and the good dispersion of the CS fibers. This enhancement in their values is independent of the content but it is dependent on the fiber length (Zhang et al., 2020). The Modulus increases as fiber content and length increase, i.e., 218 MPa, 224.92 MPa, 202.4 MPa and 221.97 MPa for 9010CS1, 9010CS2, 8020CS1 and 8020CS2 respectively.

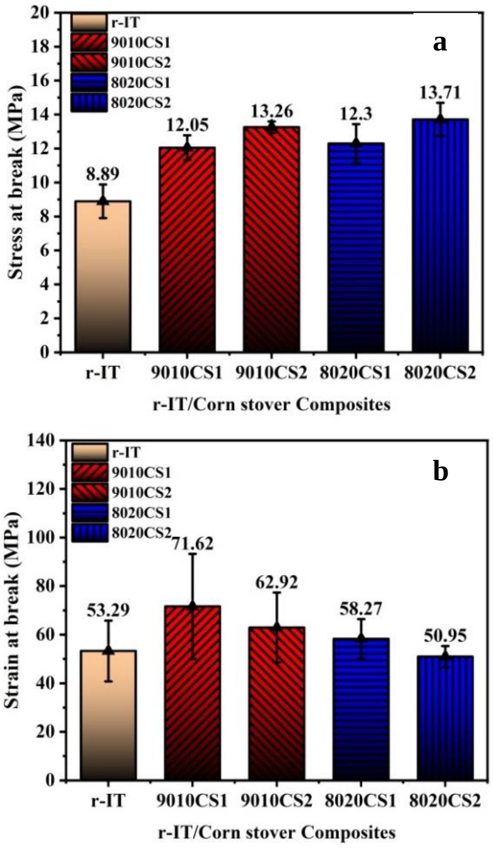

Figure 5 Stress at break (a), and Strain at break (b), results for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials
Source: Own elaboration [OriginLab].

Figures 5a and 5b present the mechanical data of the stress and strain at break respectively. Similarly, the average results show an increment with respect to the r-IT matrix. Generally, short natural fibers in the thermoplastic matrix will produce sites of stress concentrations and in consequence induce crack initiation and potential failure of the material (Gupta & Ramkumar, 2021). In the present study, as the fiber length increases, it is observed that the stress at break increases but the strain at break decreases. Similar results were reported by Delgado-Aguilar et al. (2018). This observation could be explained by improvements in fiber-matrix bonds and for the addition of rigid CS fibers.

Flexion properties

The data of flexural mechanical properties of r-IT, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials are presented in **Figure 6**. A fiber's properties determine how effective the flexion strength is, so they are the principal factor defining the structural applications of fiber-reinforced composites. According to Lu et al., (2021) fiber distribution and chemical nature may determine its mechanical performance.

Vigneshwaran et al. (2020) reported that if the fiber is brittle, the flexion strength is higher than the tensile strength. It is related to the fiber components: lignin, hemicellulose, and cellulose. Due to its crystalline nature, cellulose enhances mechanical properties (Young's modulus and tensile strength) of composite materials. In the case of Corn Stover, the content of cellulose was 52.62%, based on the study by Tarrés & Ardanuy (2020), and 50.57% in the work of Delgado-Aguilar et al. (2018). According to their conclusions, flexural strength and modulus tend to increase while flexural strain tends to decrease.

Figure 6a shows that the flexural strength depends on the concentration and length of the fibers. In the former, the increase is more evident at higher fiber contents (19.2 MPa for 9010CS1, 24.04 MPa for 9010CS2, 25.4 MPa for 8020CS1 and 25 MPa for 8020CS2).These observations are attributed to improved interactions at the CS fiber/r-IT matrix interface.
In the latter case, the flexural strength is increased in 9010 composites; this could be due to the fiber distribution in the Irrigation Tape matrix.

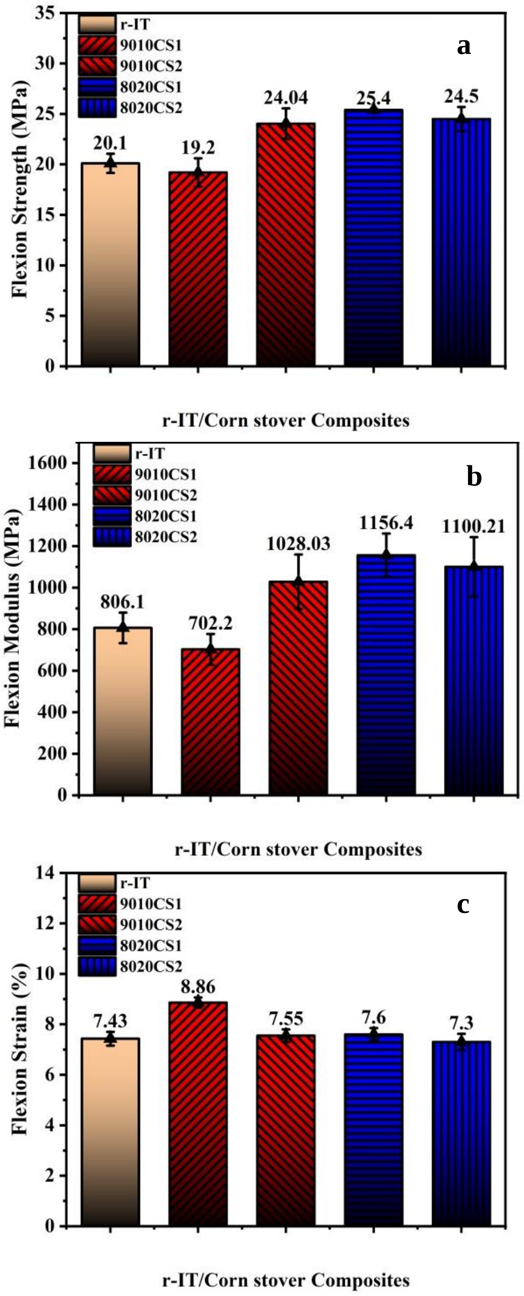

Figure 6 Flexion strength (a), Flexion Modulus (b) and Flexion strain (c) results for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials
Source: Own elaboration [OriginLab]

The flexural modulus showed that the materials are more rigid, and the increase is more notorious when the fiber content increases from 10% to 20%. This effect could be associated with the cellulose content in the Corn Stover (**Figure 6b**). Finally, **Figure 6c** presents the flexural strain values. The values were similar to those of the r-IT matrix except for the 9010CS1 (8.86%). Considering these results, it can be concluded that the interface fiber matrix is superior.

Impact Properties

The absorbed energy and the impact strength of CS composites are presented in **Figure 7 (a-b)**. Irrigation Tape is a ductile material because it is a polyethylenes blend (Zhu et al., 2018) (Tynys & Fawaz, 2020). In this study, the recycled Irrigation Tape exhibited impact strength of 23.5 kJ/m^2 after extrusion. According to the impact test results, 9010CS1, 9010CS2, 8020CS1, and 8020CS2 composite materials, are influenced by the content and the fiber length. The impact strength levels off with the increase in the concentration of CS but improves with the fiber length (**Figure 6a**). This effect is the consequence of the extent of fiber-matrix bonding.

Figure 7b shows that the lowest absorbed energy were the composites with CS1 (0.3 mm). The 9010CS1 (9.98 MPa) and 8020CS1 (9.01 MPa) are therefore more brittle than the r-IT matrix. The 9010CS2 (15.21 MPa) and 8020CS2 (11.58 MPa) are more ductile. Furthermore, the length of the fiber contributes to enhanced impact resistance. It could be the result of high load-carrying ability caused by the potential interactions with Corn Stover fibers and the irrigation Tape matrix that can transfer strength more efficiently. Another reason could be associated with the crystalline structure in the r-IT matrix.

Hardness Properties

Based on the hardness mechanical properties of r-IT and the composite materials, **Figure 8** illustrates the results. The incorporation of Corn Stover fibers raised the hardness values experimented on based on the Shore D scale in all materials compared with r-IT. The better adhesion between the CS fiber and the r-IT matrix supports these results. The fiber of CS promoted an enhancement of 5% for 9010CS1 and 9.2% for 9010CS2 (the highest increment), 6.8% for 8020CS1 and 7.4% for 8020CS2.

These observations clearly state that the major reason for the increase in hardness was the concentration of the Corn Stover in the composite. Our results agree with those presented by Gupta & Ramkumar (2021). In their investigation, they concluded that adding hard and brittle natural fibers to LLDPE/Coir composites increases their hardness and fiber-matrix interaction. It is important to mention that according to an earlier study, Drip Tape's hardness increased by 13% after seven months of irrigation (Zhu et al., 2018).

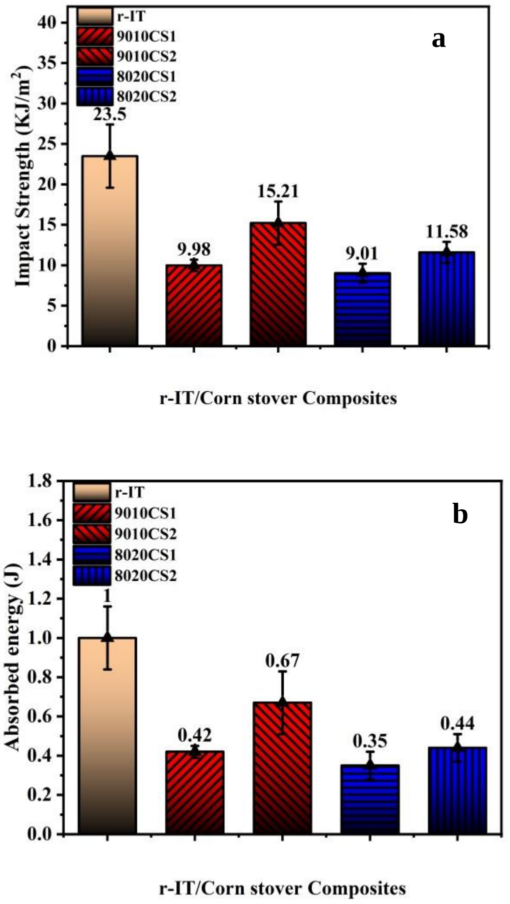

Figure 7 Impact strength (a) and Absorbed energy (b) results for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials
Source: Own elaboration [OriginLab].

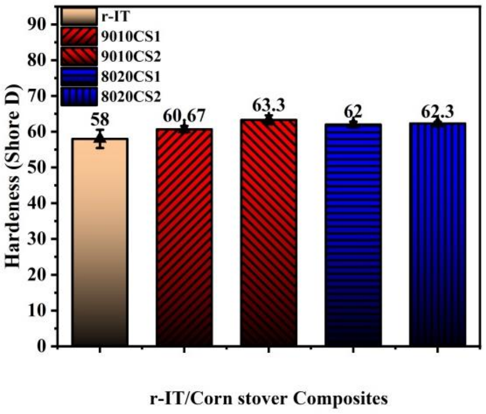

Figure 8 Hardness (Shore D) results for r-IT matrix, 9010CS1, 9010CS2, 8020CS1 and 8020CS2 composite materials
Source: Own elaboration [OriginLab].

Thermal Properties

The principal calorimetric temperatures of the Irrigation Tape Original (IT), recycled Irrigation Tape after extrusion processing and compression molding (r-IT), and the 9010CS2 composite are presented in **Figures 9 (a –b)**. **Figure 9a** shows the thermogram curves and the table insert shows data of the melting temperature (Tm) and the fusion enthalpy.

Corn Stover fibers present in the composite affect the Tm, essentially above of 1°C for IT, r-IT, and 9010CS2 composite (127.2 °C, 128.5 °C, and 128.8 °C respectively). The melting peaks for the r-IT and the 9010CS2 composite were less deep than those for the original IT. Exposure to the environment (temperature, solar irradiation, humidity, etc.) promotes photooxidation or thermal oxidation in the amorphous phase of r-IT, i.e., it leads to a chain scission reaction. The UV light directly affects the LDPE and LLDPE components of the r-IT. The increase in Tm indicates that the presence in r-IT of the non-PE components decreased (Zhu et al., 2018). It is interesting to notice that the principal difference is the melting enthalpy that passes from 103.2 J/g to 93.9 J/g for the r-IT and 9010CS2, respectively. This decrease is related to the reduction of the composite crystallinity consequence, the heat required for melting the composite material decreases.

The crystallinity of the material was calculated according to the equation (1):

$$X_c = \left(\frac{\Delta H_m}{\Delta H_0}\right) * 100 \qquad (1)$$

Where:

ΔH_m is the melting enthalpy obtained by DSC analysis and ΔH_0 is the theory melting enthalpy of the PE with 100% of crystallization, 293 J/g (Zhu et al., 2018) (Zhou et al., 2019)

The data of the calculated melting enthalpy of IT, r-IT, and 9010CS2 composite were 32.6 J/g, 35.22 J/g, and 32.04 J/g respectively.

The fibers act as a diluting agent (plasticization effect) for the irrigation tape matrix and promote the melting of the crystals, which can reduce the crystallinity of the composite. Another factor could be the addition of the MAPE coupling agent to the composites because it could interrupt the crystallizable sequence of the matrix. A similar result reported by Zhang et al. (2020), wherein they mentioned that the increase in the contact area between LLDPE- short Straw fibers reduced the interaction with the matrix of LLDPE, decreasing the crystallinity of the composites.

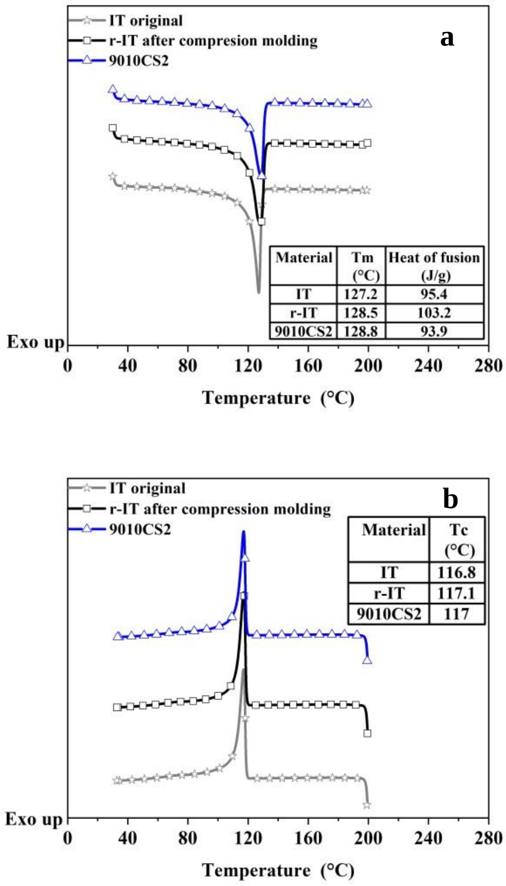

Figure 9. DSC thermograms, (a) Fusion temperature transition and (b) Crystallization temperature for IT original, r-IT after compression molding, and 9010CS2 composite material
Source: Own elaboration [OriginLab]

A slight increase in crystallization temperature (T_c) of the IT, r-IT, and 9010CS2 can be observe in **Figure 7b**. The results were 116.8 °C, 117.1 °C, and 117 °C respectively. CS fibers incorporation shows that the endothermic transition is unaffected at lower temperatures. This result indicates that CS fibers in the present research did not act as a nucleating agent like in other investigations (Kuram, 2021). In addition, the CS fibers conserve the thermal stability of the composite despite the photodegradation after its exposure, milling, extrusion reprocessing, and compression molding of r-IT. These results are according to Youssef et al., (2015).

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Conclusions

Polymer composites were prepared by extruding and compressing recycled Irrigation Tape (r-IT) and Corn Stover fibers (CS). The composite formulations consist of concentrations of 10% and 20% CS fibers with two different particle sizes (CS1 and CS2) and a 5% of MAPE Coupling Agent. Increased CS loading or length increases the tensile, bending, and hardness properties. However, there is a noticeable decrease in the impact of properties. DSC results showed that the CS fibers addition reduced the percentage of crystallinity. Rheology, morphology, and concentration of the coupling agent in composite materials analyses are additional variables that need to be studied in the future.

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