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## **SUSTAINABLE APPROACH BY USING 3D PRINTING POLYLACTIC ACID RESIDUES AS MATRIX OF GRAPHENE**

**Gabriel da Cunha Cotrim**  
**Maria del Pilar Hidalgo**  
**Sandra Maria da Luz**

Graduate Program in Engineering Materials Integrity, University of Brasília, Brasília-DF  
[gabrielccotrim@gmail.com](mailto:gabrielccotrim@gmail.com) , [pilar@unb.br](mailto:pilar@unb.br) and [sandraluz@unb.br](mailto:sandraluz@unb.br)

**Abstract:** *The manufacturing and material shaping industry is experiencing a gradual transformation with the diffusion of Industry 4.0 technologies. Additive manufacturing (AM) technology revolutionizes this industry by enabling agile techniques, customization, and complex geometries. Among the various AM techniques, 3D printing by melting and material deposition (FDM) is a popular and cost-effective technique that involves the superimposition of layers of thermoplastics to form the desired geometry. Polylactic acid (PLA) is the most used polymer in FDM owing to its ease of printing, biodegradability, and mechanical properties, making this material very useful to the industry. As several studies in the literature demonstrate, PLA also is used as a matrix material in nanocomposites. One such reinforcement material that can be used in PLA nanocomposites is graphene, which is a relatively new nanomaterial. Graphene is a simple sheet of graphite with exceptional properties that can improve the behavior of its polymeric matrix, such as electrical conductivity and increased mechanical strength. This study aimed to develop a sustainable approach to utilizing PLA residues from 3D printing by incorporating graphene as a reinforcement material. In this work, several PLA residues were aggregated and ground and then mixed using a twin-screw extruder at 190 °C with 0.18% by weight of graphene. After mixing, the materials were injected in the form of specimens for tensile tests, and the materials were also characterized by thermogravimetry (TG), differential scanning calorimetry (DSC), and infrared spectroscopy with Fourier transform (FTIR). The study found that adding 0.18 wt.% graphene to PLA residues did not significantly affect the mechanical behavior of the samples. Furthermore, Fourier-transform infrared (FTIR) spectroscopy confirmed the presence of graphene. Also, the graphene addition did not affect the thermal behavior, and no conductive properties were added to the nanocomposite. These results have important implications for developing sustainable and high-performance materials for the manufacturing industry.*

**Keywords:** Additive manufacturing, recycling, PLA, 3D printing, FDM, graphene, nanomaterials.

### **1. INTRODUCTION**

The advancement of technology has been transforming the way of manufacturing, shortening development, and improving industry connectivity. The growth of additive manufacturing is one of the keys to the Industry 4.0 revolution. The concept is based on overlapping layers of one or more materials, varying their position in 3 dimensions, forming the desired geometry (Kumar et al. 2022; Yang and Gu 2021). 3D printing is based on fusion and material deposition (FDM), the most accessible technology for the public. In this process, a thermoplastic, in the form of a thread (filament), is heated to its melting point. In sequence, the material is extruded through the nozzle, which guides the material along the X, Y, and Z axes to the formation of layers in the required position, as shown in the scheme of Figure 1. 3D printing is ease of use, presents low cost and a wide variety of applications make this production technique widespread in different areas of society. Also, with the increase in adherence to the FDM technique, the consumption of inputs for this technology follows its growth (Kumar et al. 2022).

Despite having little waste, 3D printing manufacturing still generates a reasonable amount of waste material, failed parts, print media, and unused material (Mazzanti et al. 2019). The UNEP report (United Nations Environment Program) states that 3D printing waste can pollute direct ocean microplastics. The type of thermoplastic used and the way of disposal can influence the degree of impact caused by 3D printing in the oceans.

The materials used in a 3D FDM printer are thermoplastic polymers, such as Acrylonitrile -butadiene-styrene (ABS), polylactic acid (PLA), Nylon, Polycarbonate (PC), Polystyrene (PET), and among these, most popular is PLA (Zhao et al. 2018). PLA is a semi-crystalline polymer produced with monomers from fermentation sugar or starch derived from renewable organic resources, such as corn, sugarcane, wheat, and potatoes (Mathew, Oksman, and Sain 2005).

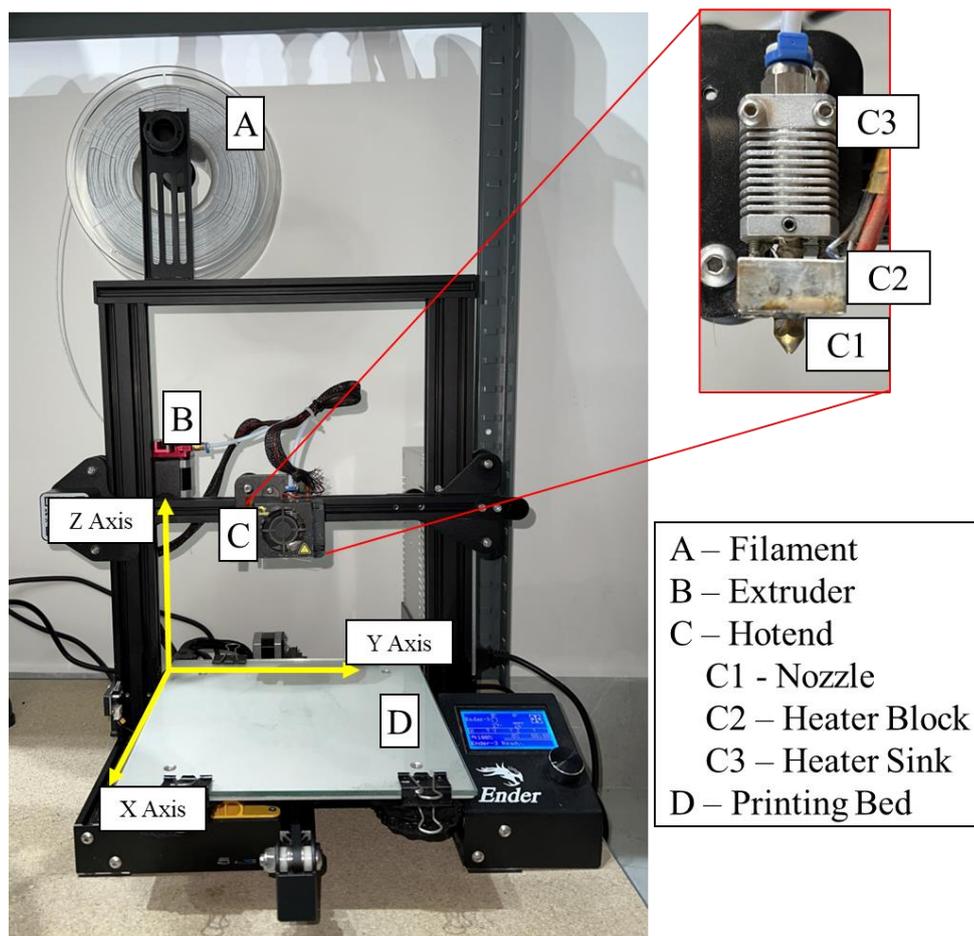


Figure 1. Representative schematic of a 3D FDM printer.

In its polymeric chain are branches of oxygen double bonds and the carboxylic acid functional group, as exemplified in Figure 2. This polymer has good mechanical and thermal characteristics, and its applications range from plastic supermarket bags to medical prostheses (Luis-Pérez, Buj-Corral, and Sánchez-Casas 2021). PLA is biodegradable, which means that under favorable conditions, it undergoes a natural degradation process, thus reducing the damage to the environment caused by the accumulation of plastic in ecosystems (Duval 2014; Mathew et al. 2005).

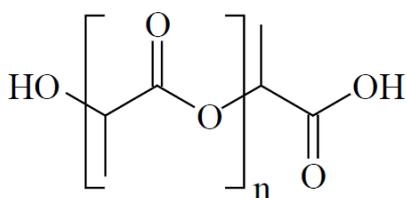


Figure 2. PLA polymer structural unit (Pillin, Montrelay, and Grohens 2006).

Although PLA is a biomaterial with a reduced impact on the environment compared to polymers from fossil sources, reprocessing and recycling the material is essential to increase its life cycle. Reducing the consumption of renewable resources and energy makes reprocessing interesting for industry and the environment (Badia and Ribes-Greus 2016; Botta et al. 2018). The reprocessing of thermoplastics exposes the material to high temperatures, degrading it and modifying its mechanical and thermal properties (Badia and Ribes-Greus 2016). To mitigate the problem of mechanical properties loss, one of the techniques used in producing nanocomposites have PLA as a matrix and graphene as reinforcement (Bustillos et al. 2018; García et al. 2020; Scaffaro et al. 2017). The presence of graphene in the nanocomposite can increase tensile strength by up to 25% (Gao et al. 2017).

Nanomaterials are structures with at least one dimension on the nanometer scale. They have several applications, such as in textiles, cosmetics, toys, the medical area, advanced materials, and the environmental area (Mazari et al. 2021).

Graphene is a carbon-based nanomaterial, a single-layer sheet of graphite. Graphene has several interesting properties: high electronic mobility, low resistivity, and high mechanical strength (Kulkarni 2014).

Although 3D printing waste has already been reused, there is a gap in the literature related to thermal and mechanical properties after reprocessing. In this way, the main objective of this work is to evaluate the behavior and properties of 3D printing PLA residues when used as a matrix with graphene.

## 2. MATERIAL AND METHODS

The steps to reprocess the 3D printing waste and add graphene as reinforcement are illustrated in Figure 3. First, the PLA waste with different additives and types of pigments was aggregated, grounded, extruded, granulated, and injected into the form of tensile specimens. The addition of graphene occurred in the extrusion stage. This work produced two types of materials: Recycled PLA and Recycled PLA with 0.18 wt.% graphene.

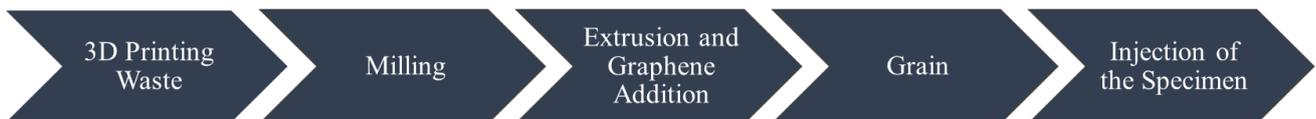


Figure 3. Flowchart showing the main stages to re-use the 3D printing PLA.

### 2.1. Waste description and preparation

The 3D printing residues PLA from Creality Ender printers used in this work are provided in different colors. There was no addition of any other polymer. The residues underwent a batch milling process using the analytical mill (Quimis Equipamentos Científicos LTDA). After milling, the material was sieved to remove fine particles and dried in an oven at 60 °C to remove moisture absorbed during storage. The mixture obtained, and the milling steps are shown in Figure 4.

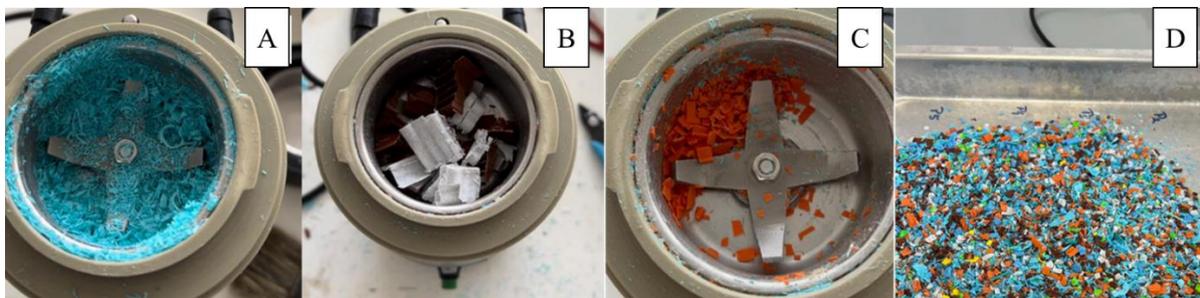


Figure 4. Milling process - A: Batch milled material. B: Pre-grinding residue. C: Batch milled material. D: Finished blend ready to use.

### 2.2. Graphene addition and extrusion

The extrusion process consists of shaping the thermoplastic employing a heated chamber that, through an endless screw, pushes the material against an opening. Under pressure, the material was mixed and molded according to the application needs (Callister and Rethwisch 2012). Using a twin-screw extruder aims to increase the dispersion of the filler added to the polymer, increasing the homogeneity of the resulting material (Pandey et al. 2021).

The extrusion was carried out using a *Thermo Scientific Model Process 11 twin-screw extruder* Figure 5. The temperature of the last stage of the extruder heating ramp was 190°C (Correia et al. 2022). The equipment has 3 mixing zones, and the graphene was manually added through an opening before the last mixing zone to ensure greater material homogeneity. The heating chamber was separated into 8 temperatures zones: 50°C, 120°C, 130°C, 150°C, 160°C, 170°C, 180°C, and 190°C.

The milled 3D printing waste was fed into the feeder constantly. After the material left the heated zone, it passed through a tank containing water for cooling, and soon afterward, it was granulated and separated, as shown in Figure 5.

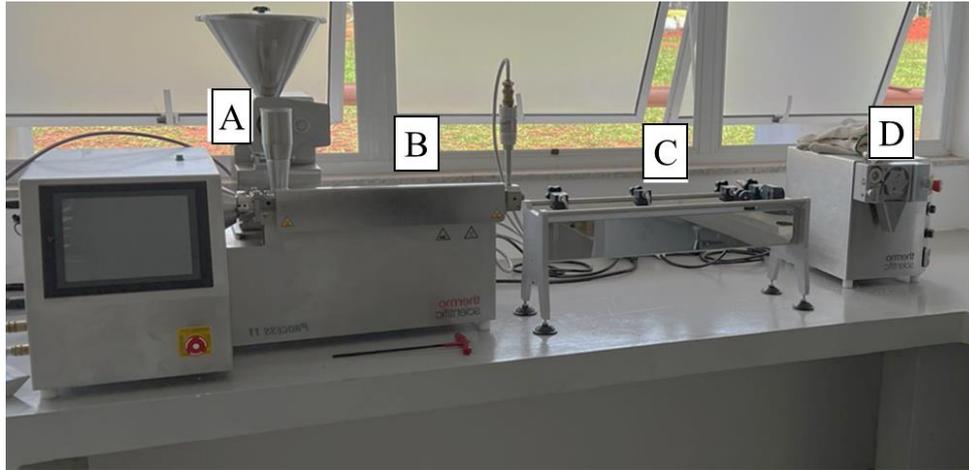


Figure 5. Process 11 Extruder – A: Feeding. B: Heating chamber. C: Cooling. D: Granulation

### 2.3. Injection of Test Specimens

A plastic injection molding machine injects molten thermoplastic material into a die to produce molded parts. The material is heated to its viscous form and pressed at high speed into the die. The die is closed to mold the material to the desired shape. Then the die is cooled so the molded part can be removed (Alvarado-Iniesta, Cuate, and Schütze 2019).

The injector used in this work was the Thermo Scientific MiniJet Pro, Figure 6. The injection was carried out with a pressure of 500 bar for 5 s and a temperature of 175°C in a specimen mold number V according to ASTM D638. The holding time was 30 s at a pressure of 250 bar in the mold at room temperature.

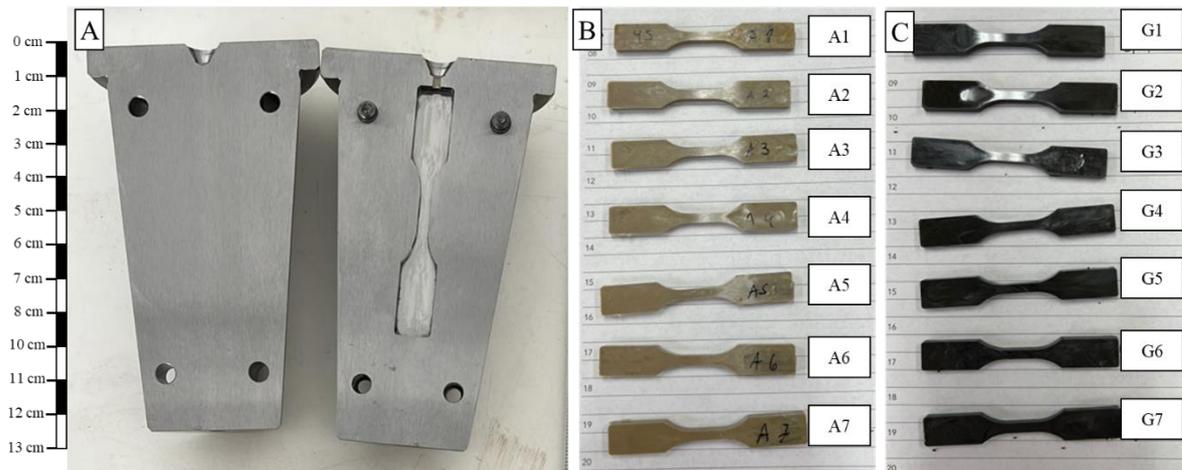


Figure 6. The mold used for injection molding (A) and the injected specimens (B).

### 2.4. Characterization of Filaments by Fourier Transform Infrared Spectroscopy (FTIR)

To carry out this test, a Thermo Scientific NICOLET IS10 spectrophotometer was used, with a diffuse reflectance accessory (DRIFT), configured to operate between the 4000 and 400  $\text{cm}^{-1}$  bands at a 4  $\text{cm}^{-1}$  interval and 64 scans.

### 2.5. Thermogravimetry (TG) and Differential Scanning Calorimetry (DSC)

The equipment used to perform the TGA and DSC analyses was the TA Instruments SDT Q600. Ten mg of each filament sample was previously cut into suitable parts for the test. An alumina crucible was used, and the temperature varied from room temperature to 600°C at a step of 10°C/min under an atmosphere of  $\text{N}_2$  at 100 mL/min.

With the results of the DSC, it was possible to calculate the degree of crystallinity of the polymers. The calculation compares the enthalpy of fusion of 100% crystalline PLA, 93.1 J/g, with that obtained in the samples according to Equation 1, adapted from (Mendes 2020).

$$\%X_c = \frac{\Delta H_f * 100}{\Delta H_{f100\%}} \quad (1)$$

Where: %  $X_c$  = percentage of polymer crystallinity;  $\Delta H_f$  = enthalpy of fusion of the sample;  $\Delta H_{f100\%}$  = theoretical melting enthalpy of the polymer with 100% crystallinity.

## 2.6. Mechanical characterization by tensile test

Fourteen samples were analyzed, 7 from recycled PLA and 7 from recycled PLA with graphene. The dimensions of the specimens followed the Group V format outlined by the ASTM D638 standard. The equipment used for the tensile test was the Instron 8801, with a load cell of 100 kN, a pressure of 20 Psi in the gripper, and a 1 mm/min speed. An extensometer was used to measure the deformation of the sample. The parameters that will be provided in this characterization are: Resistance Limit, the material's ability to withstand an applied force without breaking; Elongation at break, the measure of the material's ductility or flexibility; Young's Modulus, it quantifies the resistance of a material to elastic deformation.

## 2.7. Characterization of electrical properties

The test of electrical properties aims to assess whether the addition of graphene in the nanocomposite added conductive properties to the material. For this purpose, two HP brand multimeters – Hewlett Packard model 34401A and a direct current source from Agilent Technologies, model 6015 A, with a maximum voltage capacity of 500 volts and a current of up to 5 amps.

## 2.8. Morphological characterization by Scanning Electron Microscopy (SEM)

The SEM aims to analyze the morphological characteristics of the surface of the samples obtained in the extrusion process. The morphological characterization by microscopy was performed using the JEOL equipment, model JSM-7001F diode-based backscatter electron (BSE) detector. The specimens were metalized in the equipment EM SCD 500 using the method Sputter Coater, gold plate.

## 3. RESULTS AND DISCUSSION

Figures 7A and 7B show the recycled PLA pellets and recycled PLA nanocomposite with graphene, respectively. Before the injection, the samples had a granular shape of 1 mm. The recycled PLA stabilized in a dark green color, and the PLA presented a black color with the addition of graphene, even considering the low weight of graphene added to the system.

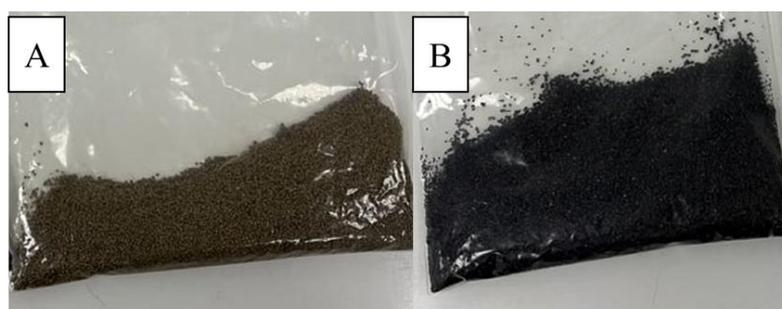


Figure 7. Samples after extrusion process: A) Recycled PLA; B) Recycled PLA with Graphene.

The results of the FTIR characterization test are shown in Figure 8. The x-axis values started at  $800 \text{ cm}^{-1}$  for better visualization of the results and removal of noise present in the graphene sample. In the graphene sample, it is possible to visualize the characteristic band of the C=C bond, which occurs at  $1648 \text{ cm}^{-1}$ . Other bands that appear correspond to the interaction of graphene with the ambient humidity, which is at  $3200\text{-}3500 \text{ cm}^{-1}$  and  $2900\text{-}3200 \text{ cm}^{-1}$ , respectively, for OH and CH bonds (Silverstein et al. 2005).

In the spectrum referring to the recycled PLA and the nanocomposite with graphene, in Figure 8 it is possible to highlight the characteristic bands present in PLA. The highest intensities were found at  $1780, 1465, 1380, 1280$  and  $1143 \text{ cm}^{-1}$ . The  $1780 \text{ cm}^{-1}$  band corresponds to the -C=O stretch. This group is a striking feature of the PLA polymer, the main path for PLA degradation (Silverstein et al. 2005; Vanin et al. 2004). The  $1380 \text{ cm}^{-1}$  band is associated with symmetric  $\text{CH}_3$  stretching. The band at  $1465 \text{ cm}^{-1}$  is associated with the asymmetric stretching of this same bond. Bands at  $1284$  and  $1143 \text{ cm}^{-1}$  are related to COC bonds (Silverstein et al. 2005; Sundar et al. 2021). These bands are relative to the polymeric matrix of the nanocomposite. The presence of graphene, although discreet, is perceptible. The band

referring to the C=C bond is found at  $1648\text{ cm}^{-1}$ . In the second spectrum of Figure 8, it is possible to compare the samples with and without graphene, and there is an increase in the intensity of the C=C bond band in the sample with the nanomaterial.

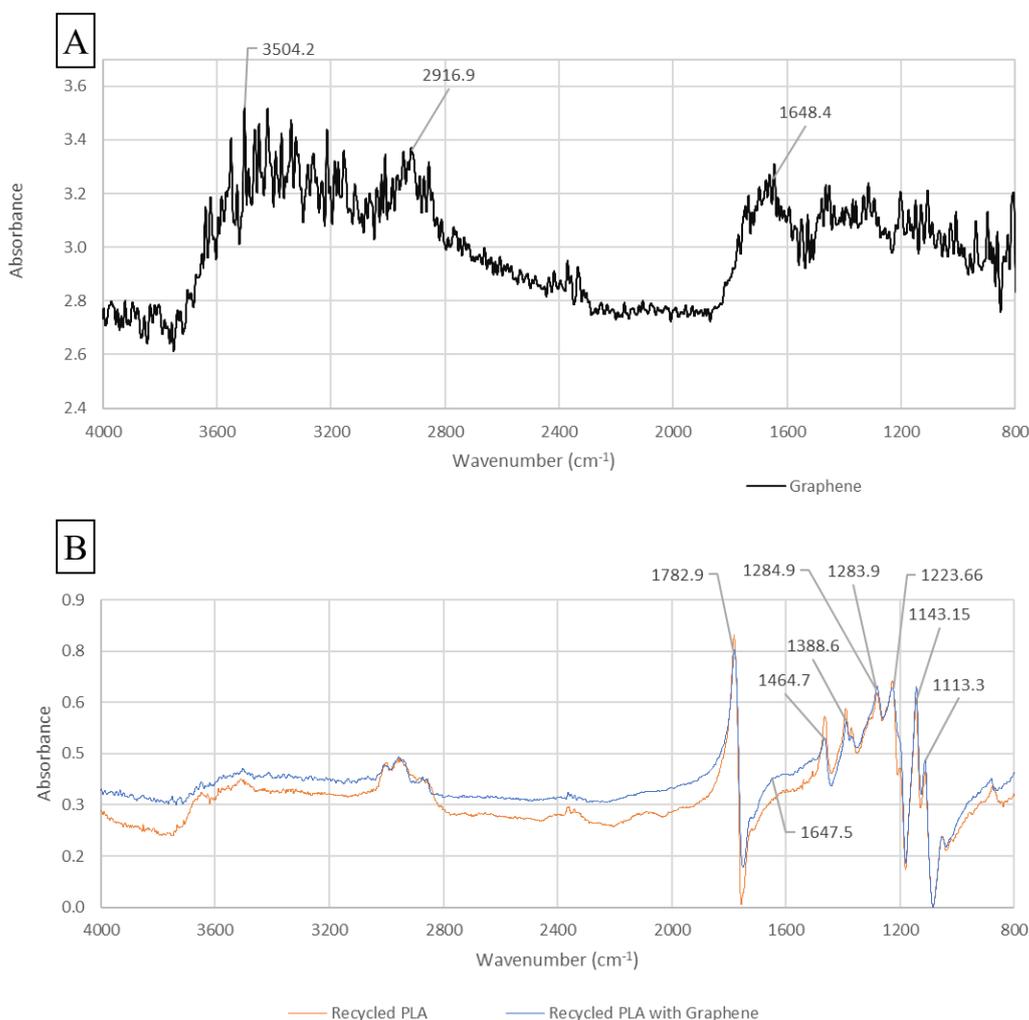


Figure 8. FTIR spectra of samples: A, Graphene sample; B, Recycled PLA, and Recycled PLA with Graphene

The results of the TG, DTG, and DSC curves were compiled in Figure 9. The TG and DTG curves show little thermal differences between the recycled PLA and Recycled PLA with graphene nanocomposite samples. This is due to the low amount of graphene added, which, although it intensified the material's color, did not affect the thermal properties, a behavior also found in the literature (Botta et al. 2018). The maximum degradation temperature was  $351^{\circ}\text{C}$ .

Through the DSC curves, it is possible to list 3 main regions where critical processes occur for the thermal characterization of the material, and circles indicate them. The black circle is the characteristic region of the material degradation process. In the red circle, it is possible to observe the typical region of the fusion process, where the intermolecular bonds are broken, and the material has the characteristic of a viscous fluid. The region marked by the yellow circle refers to the material's glass transition process, where it leaves the solid state for a malleable and moldable state.

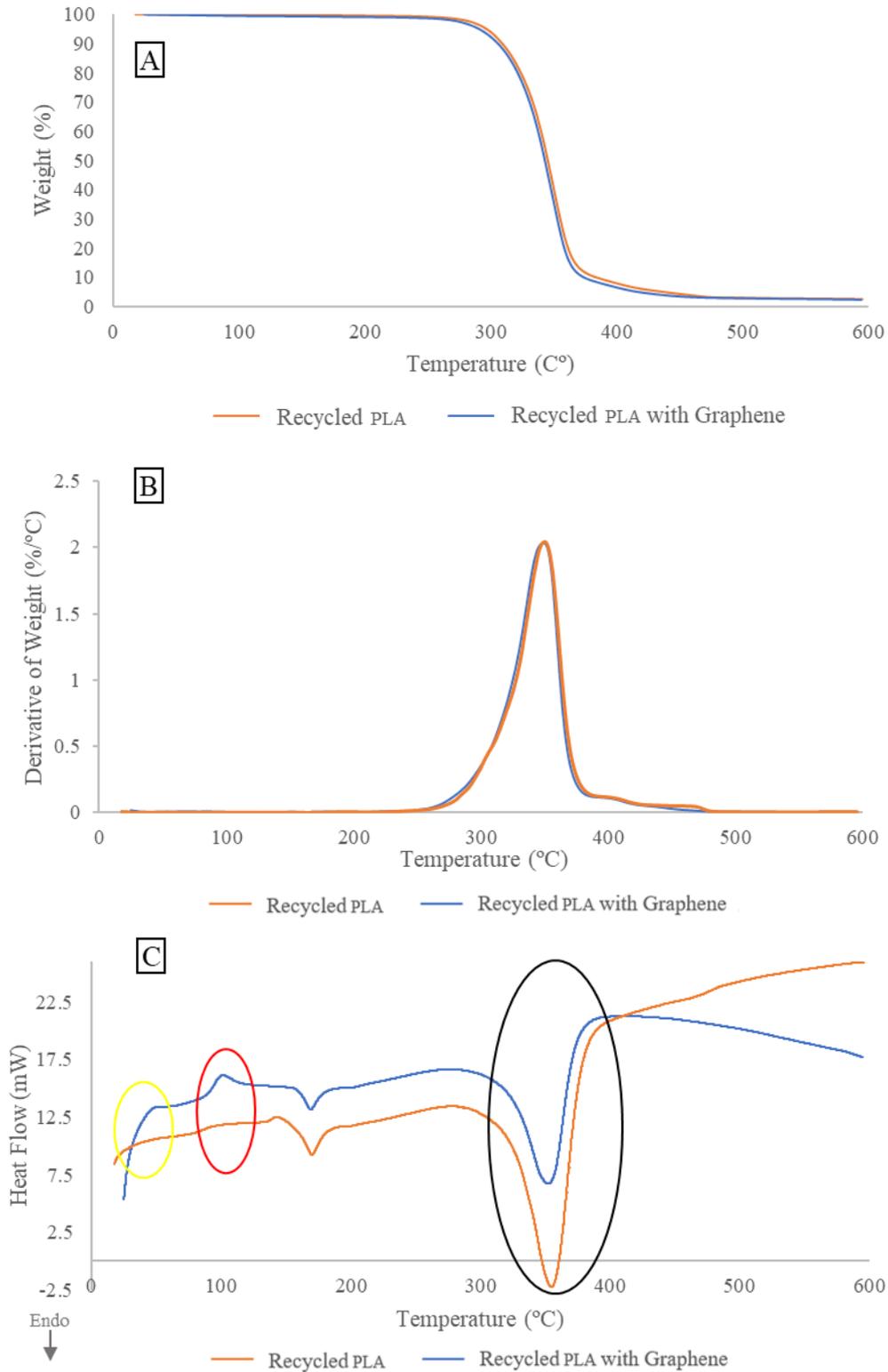


Figure 9. TG (A), DTG (B), and DSC (C) curves for recycled PLA and recycled PLA with graphene, respectively.

Table 1 is a summary of the thermal analysis of the samples. The addition of graphene to the nanocomposite generated a crystallinity variation of 5% in relation to the reprocessed polymer, this phenomenon can be attributed to the disruption of polymer chain packing, enhanced heat dissipation, and strong interfacial interactions between graphene and the polymer, collectively inhibiting the formation of crystalline structures within the composite. The T<sub>g</sub> and T<sub>m</sub> values were slightly lower than those found by (Botta et al. 2018; Gao et al. 2017), for Recycled PLA and Recycled PLA with

graphene addition. However, these authors did not evaluate the behavior of 3D printing residues, which could cause the difference between the values.

Table 1. Thermal characteristics from TG, DTG, and DSC curves.

Sample	Tg (°C)	Tm (°C)	Tdegradation (°C)	Crystallinity (%)
Recycled PLA	54.77	157.27	270.51	20.63
Recycled PLA with Graphene	55.81	159.74	275.95	15.10

### 3.1. Mechanical characterization by tensile test

Figure 10 shows the tensile and deformation curves representing the Recycled PLA and Recycled PLA with graphene samples. The specimens presented an average tensile strength limit of 82.37 and 82.95 MPa, respectively, for Recycled PLA and Recycled PLA with graphene, as shown in Table 2. The average breaking strain of the samples was 1.70% and 1.76%, respectively, for Recycled PLA and Recycled PLA. The strength threshold values are slightly higher than that found in the reprocessed PLA behavior (Santana et al. 2018).

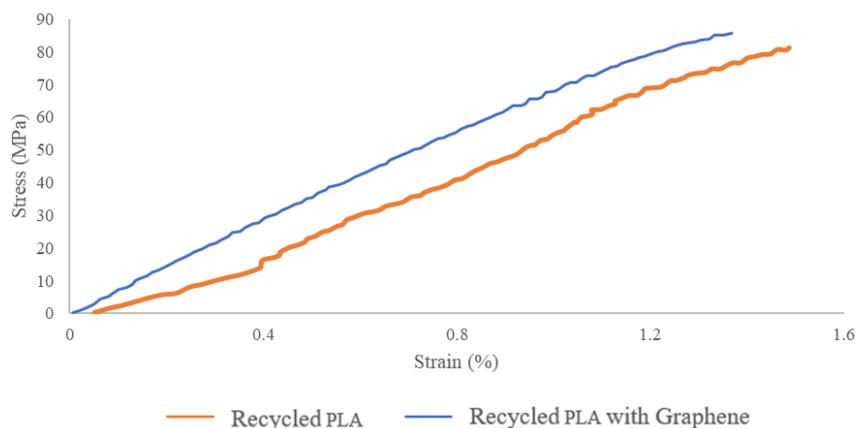


Figure 10. Example of resistance limit curves for the materials.

Table 2. Mechanical tensile properties for recycled materials.

Parameter	Recycled PLA	Recycled PLA with Graphene
Resistance limit (MPa)	82.37±0,01	82.95±0,01
Elongation at break (%)	1.70	1.76
Young's Modulus (GPa)	3.51±0,021	3.570±0,023

The samples showed brittle fracture characteristics. Despite the deformation above 1%, there was no formation of “necks” in the specimen (CP), or plastic behavior before rupture.

The electrical properties test was conducted as an investigation. The sample with graphene was tested up to the maximum source voltage of 500V, and there was no variation or electrical conduction signal through the material. Graphene did not add conductive properties to the nanocomposite in the amount of graphene added.

The results obtained in the Scanning Electron Microscopy (SEM) are shown in Figure 11. It is possible to verify the presence of material agglomerate points in the recycled PLA with a graphene sample. These intrusions are formed by the agglomeration of graphene embedded in the material (Botta et al. 2018). It is also possible to see gaps formed around the graphene clusters indicating a lack of adhesion of the matrix to the reinforcement. The Recycled PLA sample has fewer agglomerated points, but in the 5000x zoom, it's clear that the recycled PLA has some intrusions, possibly occurring by intrusion of contamination in the storage, milling and processing steps.

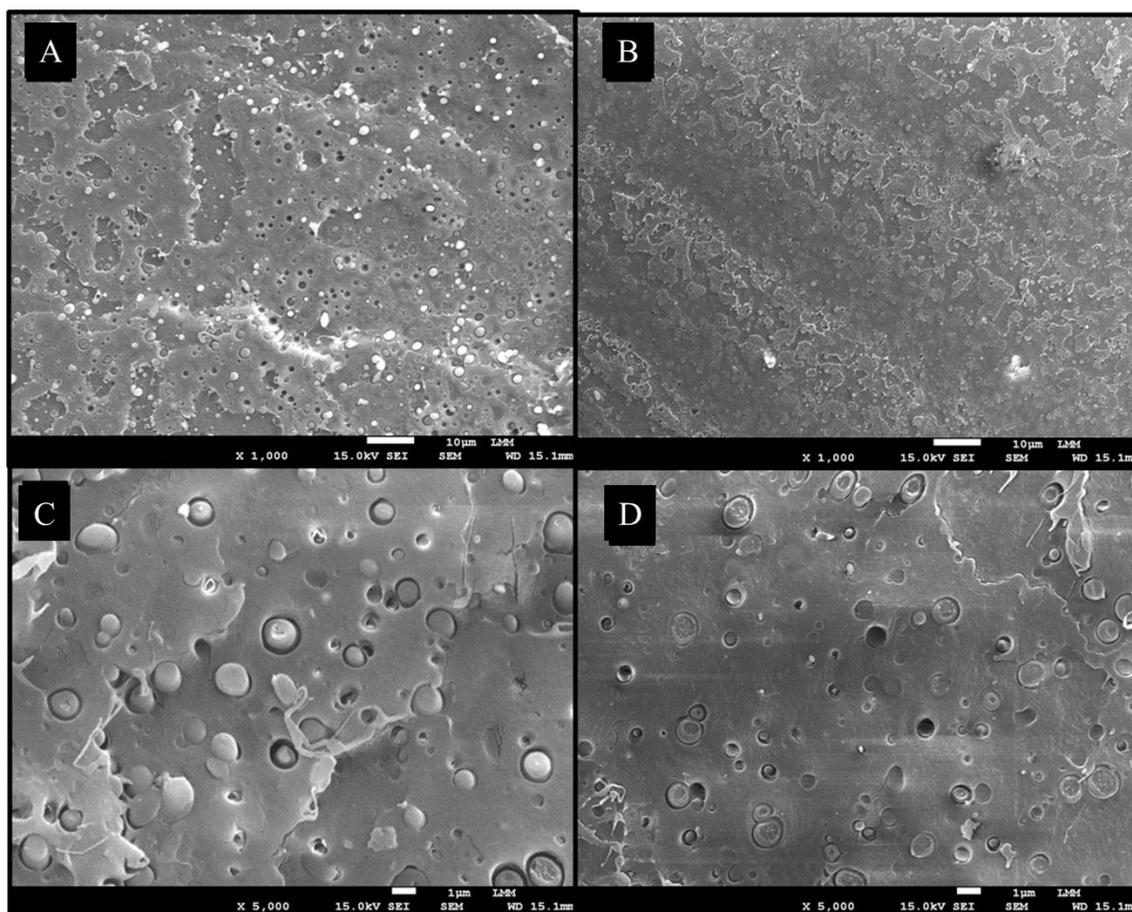


Figure 11. Images obtained by SEM: A, Recycled PLA with Graphene, 1000x zoom; B, Recycled PLA at 1000x zoom; C, Recycled PLA with Graphene zoom 5000x and D, Recycled PLA zoom 5000x.

#### 4. CONCLUSION

The twin screw extrusion process homogenized the samples, presenting homogeneous colors depending on the type of material: recycled PLA or recycled PLA with graphene. Although the resulting coloration of the materials differed, the low percentage of added graphene did not result in electrical conductivity, thermal stability, or mechanical properties changes. The only difference observed was the decrease in the degree of crystallinity for the nanocomposite. For future research, it would be interesting to study different levels of graphene addition, which may result in other properties. Another positive aspect of the work is that the material maintains its properties regardless of adding graphene.

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