

# PRODUCTION OF PLA/EE-g-GMA NANOCOMPOSITES REINFORCED WITH CARBON NANOTUBES FOR ANTISTATIC APPLICATION

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**ABSTRACT:** Poly(lactic acid) (PLA)/elastomeric ethylene grafted with glycidyl methacrylate (EE-g-GMA) nanocomposites were prepared, using carbon nanotubes (MWCNT) with conductive nanofiller. The PLA/EE-g-GMA/MWCNT nanocomposites were processed in a twin-screw extruder and injection molded. The properties of impact strength, tensile strength, electrical conductivity ( $\sigma$ ), and morphology were

determined. The impact strength of the PLA/EE-g-GMA blend increased by 442.5% compared to pure PLA, suggesting toughening occurred. Incorporating MWCNT into PLA/EE-g-GMA promoted a reduction in impact strength in the PLA/EE-g-GMA base-system; however, the nanocomposites are more tough than pure PLA. As the MWCNT content increased in the PLA/EE-g-GMA/MWCNT nanocomposites, the elastic modulus increased, and the tensile strength reduced, compared to PLA/EE-g-GMA. Scanning electron microscopy (SEM) indicated a good interaction between PLA and EE-g-GMA, forming well-adhered and refined particles, justifying the high performance under impact of the PLA/EE-g-GMA blend. In contrast, MWCNT negatively affected the morphology of the nanocomposites, generating many voids with increasing MWCNT content. The PLA/EE-g-GMA/MWCNT (5 phr - part per hundred resin) nanocomposite has technological potential, considering it increased impact strength by 63.8% concerning pure PLA. In addition, it presented an electrical conductivity of  $3.91 \times 10^{-06} \text{ S.cm}^{-1}$ , an adequate value for possible antistatic application.

**KEYWORDS:** Poly(lactic acid), impact modifier, carbon nanotubes, nanocomposites, antistatic.

**RESUMO:** Nanocompósitos de poli(ácido láctico) (PLA)/etileno elastomérico enxertado com metacrilato de glicidila (EE-g-GMA) foram preparados, utilizando nanotubos de carbono (MWCNT) com nanocarga condutora. Os nanocompósitos PLA/EE-g-GMA/MWCNT foram processados em extrusora dupla rosca e moldados por injeção. As propriedades de resistência ao impacto, resistência à tração, condutividade elétrica ( $\sigma$ ) e a morfologia foram determinadas. A resistência ao impacto da blenda PLA/EE-g-GMA aumentou em 442,5% em comparação ao PLA puro, sugerindo que houve tenacificação. A incorporação de MWCNT em PLA/EE-g-GMA promoveu uma redução na resistência ao impacto em relação ao sistema base PLA/EE-g-GMA, porém os nanocompósitos são mais tenazes do que o PLA puro. À medida que aumentou o teor de MWCNT nos nanocompósitos PLA/EE-g-GMA/MWCNT, o módulo elástico aumentou e a resistência à tração reduziu, em comparação com PLA/EE-g-GMA. A microscopia eletrônica de varredura (MEV) indicou uma boa interação entre o PLA e o EE-g-GMA, formando partículas bem aderidas e refinadas, justificando o alto desempenho sob impacto da blenda PLA/EE-g-GMA. Em contraste, o MWCNT afetou de forma negativa a morfologia dos nanocompósitos, gerando grande quantidade de vazios com o aumento da concentração de MWCNT. O nanocompósito PLA/EE-g-GMA/MWCNT (5 pcr - partes por cem de resina) tem potencial tecnológico, tendo em vista que aumentou a resistência ao impacto em 63,8% em relação ao PLA puro. Em adição, apresentou uma condutividade elétrica de  $3.91 \times 10^{-06} \text{ S.cm}^{-1}$ , valor adequado para possível aplicação antiestática.

**PALAVRAS-CHAVE:** Poli(ácido láctico), modificador de impacto, nanotubos de carbono, nanocompósitos, antiestática.

## INTRODUCTION

Due to the need for new and modern materials for engineering applications, polymers are constantly being improved to expand the range of mechanical properties, aiming to suit specific applications (Shameem et al., 2021; Kamal et al., 2022). Given this, polymer nanocomposites have gained considerable prominence in the scientific and industrial community. They are hybrid materials made up of a polymeric matrix containing an inorganic phase dispersed at a nanometric level (Sun et al., 2021; Darwish et al., 2022; Luna et al., 2022). In general, producing polymeric nanocomposites requires a low concentration of nanofillers (1-5%). However, achieving adequate dispersion of nanofillers in the polymer matrix directly improves mechanical, thermal, electrical, barrier, and dimensional stability properties (Oliveira et al., 2015; Tejjido et al., 2022; Han et al., 2023; Pinto et al., 2023). Much attention has recently been paid to polymer nanocomposites reinforced with carbon-based nanofillers, especially for applications requiring conductive, flexible, and lightweight materials (Ryan et al., 2022; Zahid et al., 2023; Liu et al., 2023; Silva et al., 2024).

Carbon nanotubes are widely used as nanofillers, given their excellent characteristics of high resistance, high elastic modulus, electrical conductivity, good thermal properties, and good chemical stability (Anzar et al., 2020; Rathinavel et al., 2021). This has aroused great interest in its use in the preparation of polymeric nanocomposites, which are used to make products for the electronics sector (Anderson et al., 2021; Huang et al., 2023; Kausar

and Ahmad, 2023; Luna et al., 2023). At the same time, the reduction in the price of carbon nanotubes, especially multi-walled nanotubes (MWCNT), has contributed to their use on larger scales (Volder et al., 2013; Lopes et al., 2016). Currently, progress has been made in producing conductive nanocomposites using polymer matrices from renewable sources, with emphasis on poly(lactic acid) (PLA) (Kuang et al., 2023; Bikiaris et al., 2023).

PLA is one of the main biodegradable polymers used commercially, processed by extrusion and injection. It has good transparency, high elastic modulus, good tensile strength, and good Shore D hardness (Jem and Tan, 2020; Taib et al., 2023). Considering this, PLA is being investigated to produce nanocomposites with carbon nanotubes, aiming to produce new tailor-made materials (Bai et al., 2020; Norazlina and Kamal, 2021; Vidakis et al., 2021). However, PLA main drawback is low impact strength, causing limitations in the production of products requiring toughness. This problem has been solved through the production of nanocomposites obtained from PLA polymer blends, which form a tough material and expand its application possibilities (Brito et al., 2014).

There are reports in the literature on nanocomposites of blends based on PLA and carbon nanotubes using polycaprolactone (PCL) (Urquijo et al., 2017), poly(hydroxybutyrate-co-hydroxyvalerate) (PHBV) (Silva et al., 2021), poly(butylene adipate-co-terephthalate) (PBAT) (Sarul et al., 2021) and ethylene vinyl acetate (EVA) (Wang et al., 2022). However, the number of publications on PLA/carbon nanotubes nanocomposites with impact modifiers functionalized with glycidyl methacrylate still needs to be increased, generating the need for new investigations. Given the above, poly(lactic acid) nanocomposites were prepared with carbon nanotubes (MWCNT) using elastomeric ethylene grafted with glycidyl methacrylate (EE-g-GMA) as an impact modifier. The nanocomposites were processed in a co-rotating twin-screw extruder and injection molded, aiming for mechanical, electrical, and morphological characterizations.

## EXPERIMENTAL PROCEDURE

### Materials

- Poly(lactic acid) (PLA) was used as a polymeric matrix, with  $1.24 \text{ g/cm}^3$  and a melt flow index (MFI) of  $6.0 \text{ g/10 min}$  (ASTM D1238 -  $190^\circ\text{C}/2.16 \text{ kg}$ ), in the form of granules, supplied by 3DLab.
- The impact modifier was elastomeric ethylene grafted with glycidyl methacrylate (EE-g-GMA), with the commercial code Coace® W5D. The grafting content of glycidyl methacrylate is greater than 0.8%, with a density of  $0.92 \text{ g/cm}^3$  and a melt flow index of  $8-16 \text{ g/10 min}$  (ASTM D1238 -  $190^\circ\text{C}/2.16 \text{ kg}$ ), in the form of granules and supplied by Xiamen Coace Plastic Technology Co., Ltd.

- Multi-walled carbon nanotubes (MWCNT) were utilized as nanometric fillers, with HQNANO CNTs-010 code. These were prepared through chemical vapor deposition (CVD), which was provided by Advanced 2D Materials Co., Ltd. The specifications include 95% purity, inner diameter of 3-5 nm, outer diameter of 8-15 nm, specific surface area greater than 233 m<sup>2</sup>/g, and density of 0.15 g/cm<sup>3</sup>.

## Extrusion processing

Before processing the nanocomposites, the PLA, EE-g-GMA, and MWCNT were dried in a vacuum oven at 60 °C for 24 hours. To obtain the nanocomposites in the molten state, PLA/MWCNT concentrates were initially prepared using the melt intercalation method (laboratory mixer from Thermo Scientific), with roller-type rotors, operating at 60 rpm at a temperature of 180 °C, for 3 minutes. The PLA/MWCNT concentrate was crushed in a knife mill, giving rise to Flakes for dilution in the extruder. The compositions developed were PLA/EE-g-GMA + MWCNT (70/30% by mass + 1 phr; 70/30% by mass + 3 phr; and 70/30% by mass + 5 phr). MWCNT was used as a complementary additive to induce electrical conductivity in the nanocomposites, and therefore, phr (parts per hundred of resin) was adopted.

The PLA/EE-g-GMA/MWCNT nanocomposites were mixed manually and subsequently prepared by mixing in the melt state in a modular co-rotational twin-screw extruder from Coperion Werner & Pfleiderer, with screw diameters (D) of 18 mm and L/D = 40. The following process parameters were used: screw speed of 250 rpm, flow rate of 4 kg/h and temperature profile of 160°C – 170°C - 170°C - 180°C – 180°C - 190°C - 190 °C. Pure PLA and the PLA/EE-g-GMA blend were processed under the same conditions as the nanocomposites, aiming for a comparative effect. After processing, the pure PLA, PLA/EE-g-GMA blend, and the PLA/EE-g-GMA/MWCNT nanocomposites were granulated in the extruder, dried at 60 °C in a vacuum oven for 24 h for subsequent injection molding.

## Injection/compression molding process

The injection molded samples were produced on an Arburg injection molding machine, Model Allrounder 207C Golden Edition. Test specimens were produced for impact testing and tensile strength, following ASTM D256 and ASTM D638 recommendations. The molding parameters adopted were injection pressure = 1000 bar; holding pressure = 500 bar; temperature profile = 160°C, 170°C, 170°C, 180°C and 190°C; mold temperature = 20°C; and cooling time in the mold = 25 s.

The extruded granules were used to develop films for electrical conductivity testing. A mold approximately 1 mm thick was used in a hydraulic press at 190°C for 4 min and pressure of 5 tons. The film was cooled at room temperature for 10 min under a load of 50 N.

## Nanocomposite characterization

The Izod impact strength test was carried out on notched specimens, according to the ASTM D256 standard, in a Ceast model Resil 5.5 J machine, operating with a 2.75 J hammer, at room temperature ( $\sim 23^\circ\text{C}$ ). The results were analyzed using an average of seven specimens.

The tensile test was conducted on the injected specimens following the ASTM D638 standard. The Oswaldo Filizola BME universal testing machine was used with a speed of 5 mm/min and a load cell of 20 kN, at room temperature ( $\sim 23^\circ\text{C}$ ). The results were analyzed using an average of seven specimens.

The electrical conductivity analysis was carried out on PLA/EE-g-GMA/MWCNT nanocomposite films, using the Keithley electrometer, model 6517B, using the volumetric method as equipment.

Scanning electron microscopy (SEM) analyses were carried out on the fracture surfaces of the specimens subjected to the impact test. A scanning electron microscope, VEGAN 3 TESCAN, was used at a voltage of 30 kV under high vacuum. The fracture surfaces of the samples were coated with gold.

## RESULTS AND DISCUSSION

A Figuras 1 (a-d) mostra a superfície de fratura sob o teste de impacto, realizado à temperatura ambiente, da blenda PLA/EE-g-GMA e dos nanocompósitos. In Figure 1 (a), the PLA/EE-g-GMA blend has a biphasic structure with spherical EE-g-GMA particles dispersed in the PLA matrix. This observation indicates the formation of an immiscible blend with different phases. The EE-g-GMA particles are well adhered to the PLA matrix, which indicates that the interface did not fracture during the impact test. The good adhesion in the morphology suggests that the EE-g-GMA copolymer promoted a good interaction with the PLA, possibly due to low interfacial tension, thus making the interface resistant. Brito et al. (2014) indicated that the epoxy group is capable of reacting with the carboxyl and/or hydroxyl terminal groups of PLA. Therefore, the stability in the morphology of the PLA/EE-g-GMA blend was probably due to interactions between the EE-g-GMA epoxy ring and the PLA terminal groups, which generated stabilization in the morphology and minimized coalescence. The compatibility between PLA and EE-g-GMA, visualized through the morphology of the fracture surface, corroborates the high-impact performance presented later. Adding MWCNT to the PLA/EE-g-GMA blend promoted the formation of a coarser morphology. As the MWCNT content increased, voids appeared in the nanocomposites, indicating that EE-g-GMA particles were detached from the PLA matrix during the impact test. The most dramatic effect was for the PLA/EE-g-GMA/MWCNT (5 phr) nanocomposite, as it severely increased the number of voids. This indicates that MWCNT negatively affected the interaction of the epoxy group of EE-g-GMA with PLA, resulting in a reduction in interfacial adhesion.

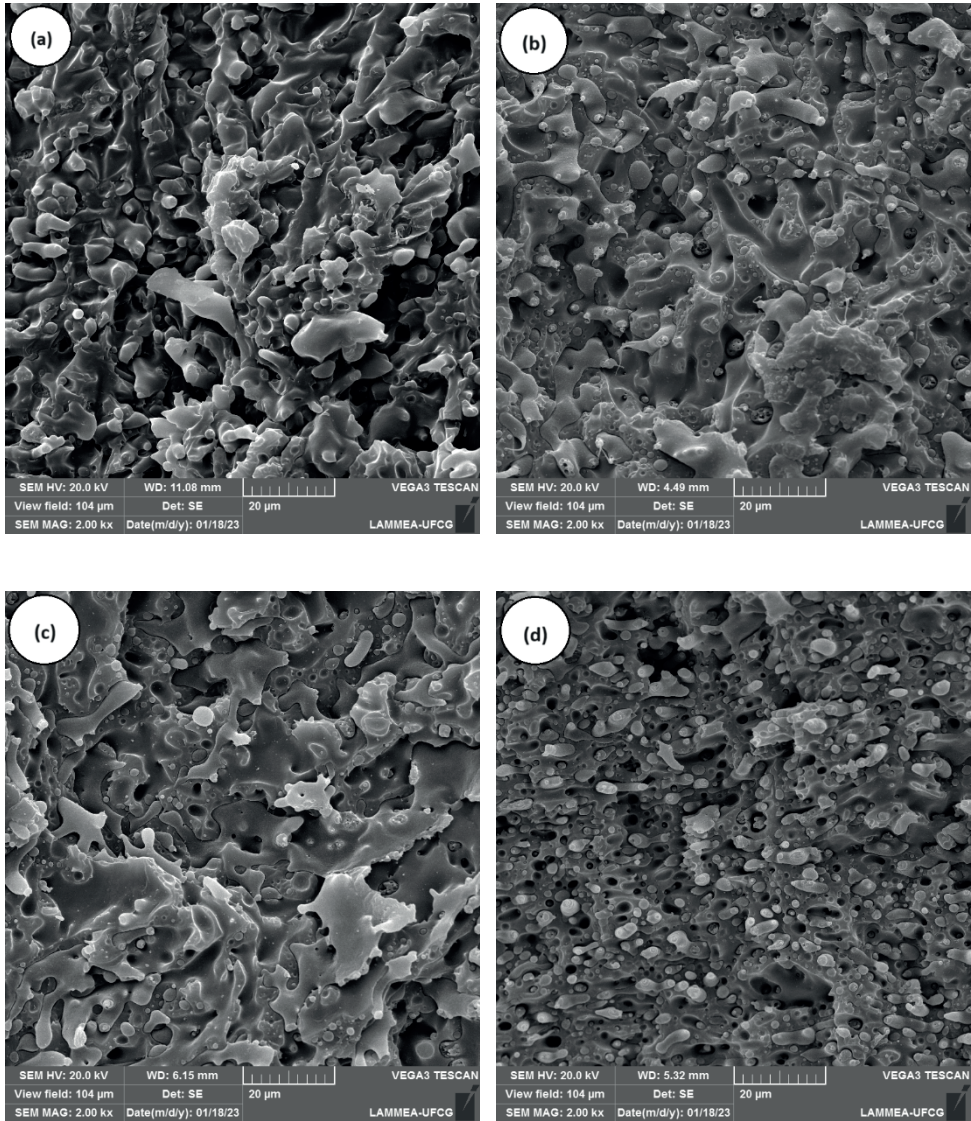


Figure 1. Evolution of morphology at 2000x magnification, for: (a) PLA/EE-g-GMA; (b) PLA/EE-g-GMA/MWCNT (1 phr); (c) PLA/EE-g-GMA/MWCNT (3 phr); (d) PLA/EE-g-GMA/MWCNT (5 phr).

Figure 2 shows the morphology of the PLA/EE-g-GMA/MWCNT nanocomposites at 22000x magnification, aiming to evaluate the distribution of carbon nanotubes. For higher MWCNT content in nanocomposites, there was a tendency to increase the distribution of carbon nanotubes, especially with 5 phr. Consequently, it favored the formation of a conductive network in nanocomposites, resulting in the formation of materials with electrical conduction capacity, as presented later.

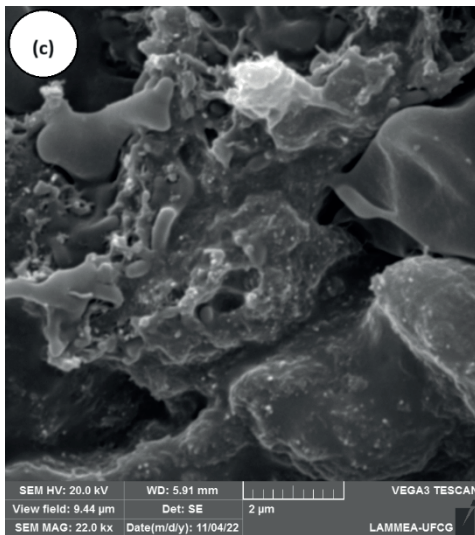
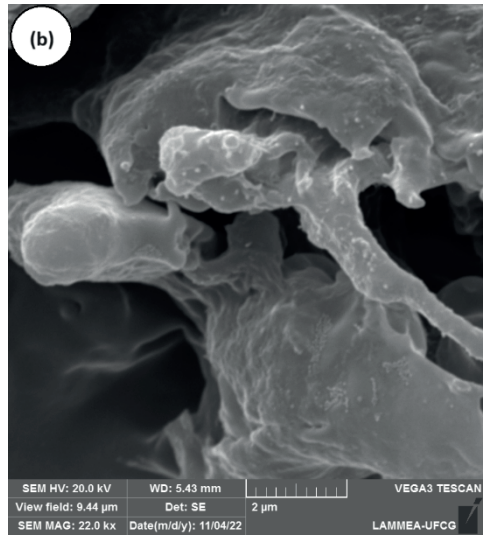
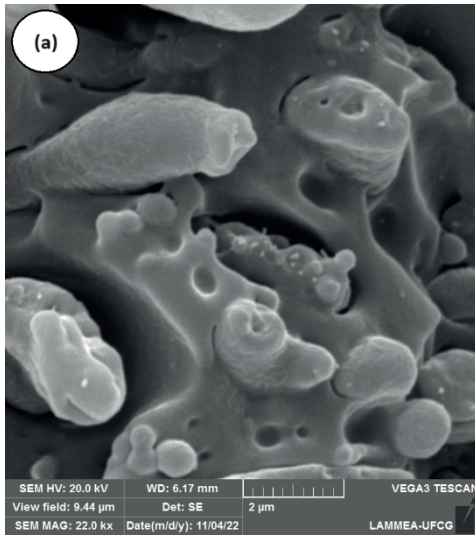


Figure 2. Morphology obtained by SEM at 22000x magnification, for: (a) PLA/EE-g-GMA/MWCNT (1 phr); (b) PLA/EE-g-GMA/MWCNT (3 phr); (c) PLA/EE-g-GMA/MWCNT (5 phr).

Figure 3 shows the impact strength of pure PLA, the PLA/EE-g-GMA blend, and nanocomposites with different MWCNT contents.

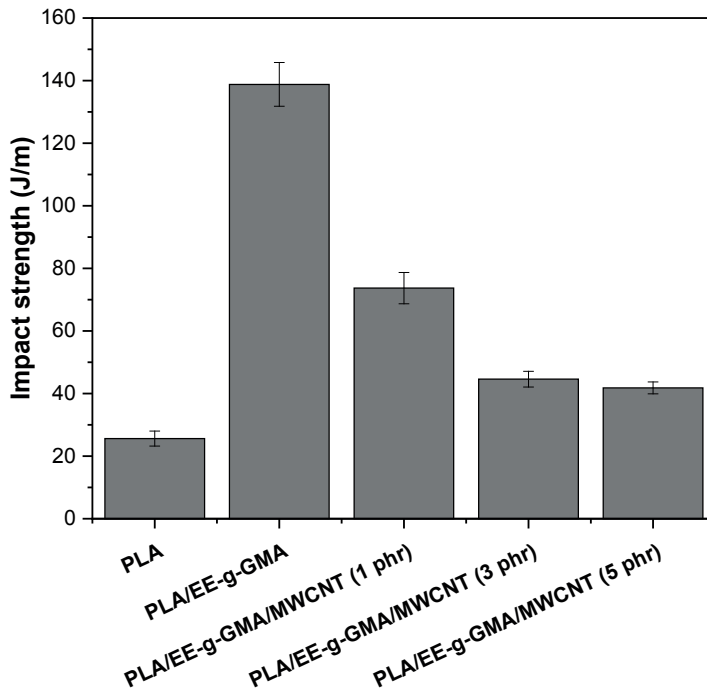


Figure 3. Impact strength of pure PLA, the PLA/EE-g-GMA blend, and nanocomposites.

As shown in Figure 3, pure PLA presented a low impact strength (25.6 J/m), a value considered to be a brittle polymer. Similar results were also reported in the literature (Wang et al., 2017; Ferreira et al., 2023). The PLA/EE-g-GMA blend exhibited an impact strength of 138.9 J/m, corresponding to a gain of 442.5%, compared to pure PLA. This demonstrates that the EE-g-GMA copolymer efficiently toughened the PLA, possibly due to the good interaction between the components, as seen in the SEM. The epoxy ring in the glycidyl methacrylate group of EE-g-GMA promoted good interaction with the carboxyl and/or hydroxyl terminal groups of PLA, forming a strong interface (Araújo et al., 2019). Consequently, there was a good stress transfer at the interface of the PLA/EE-g-GMA blend, favoring better dissipation and distribution of energy in the PLA matrix and resulting in a more effective toughening mechanism. Adding MWCNT negatively affected the impact strength of nanocomposites, indicating a stress-concentrating effect. This is because MWCNT is a rigid nanofiller that restricts molecular mobility, increasing fragility. It was observed that as the concentration of MWCNT increased, the impact strength of the nanocomposites continuously reduced. The incorporation of 1 phr of MWCNT reduced impact strength by 46.9% compared to the PLA/EE-g-GMA blend. Upon increasing the MWCNT content to 3 phr and 5 phr, the blend's impact strength reduces by around 67.8% and 29.8%, respectively, compared to the base blend. However, the results of PLA/EE-g-GMA/MWCNT nanocomposites were higher than those of pure PLA, suggesting that they are more tough. For example, the PLA/EE-g-GMA/MWCNT (5 phr) nanocomposite achieved an increase in impact strength of 63.8% when compared to pure PLA.



Figure 4 reports the elastic modulus (E) and tensile strength (TS) of pure PLA, the PLA/EE-g-GMA blend, and the nanocomposites.

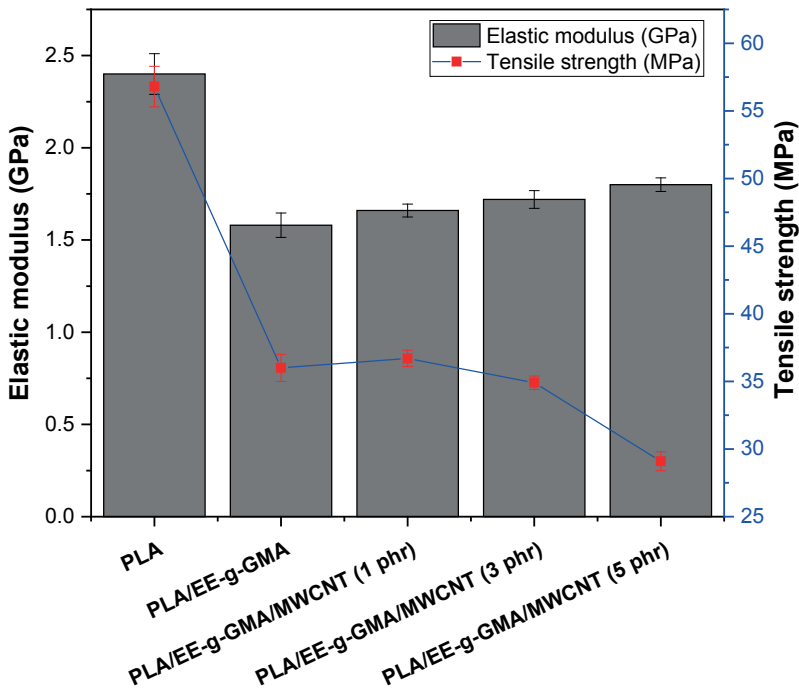


Figure 4. Mechanical properties under tensile for pure PLA, the PLA/EE-g-GMA blend, and the nanocomposites.

The highest elastic modulus value was for pure PLA with 2.4 GPa, indicating the highest stiffness among all materials. Incorporating 30% of EE-g-GMA into the PLA matrix had a deleterious effect on the elastic modulus, reducing it by 34.5%. This reduction in the elastic modulus was due to the addition of EE-g-GMA, considering that it has elastomeric behavior and, therefore, increases the flexibility of the PLA/EE-g-GMA blend. Regarding the PLA/EE-g-GMA/MWCNT nanocomposites, there was a continuous increase in the elastic modulus compared to PLA/EE-g-GMA. This is attributed to MWCNT being a rigid nanofiller, thus generating restriction in molecular mobility and increasing rigidity. The recovery in elastic modulus was more evident in the nanocomposite containing 5 phr of MWCNT, with a gain of 14% in relation to the PLA/EE-g-GMA system.

The tensile strength of PLA, 56.8 MPa, indicated that it required a greater load to deform. As expected, the greater flexibility of the PLA/EE-g-GMA blend reduced the tensile strength to 36 MPa. It was observed that the tensile strength reached a maximum value for the nanocomposite with up to 1 phr of MWCNT, with a value of 36.7 MPa. However, the value is very close to that of the PLA/EE-g-GMA blend, given the margin of experimental

error. The tensile strength decreased smoothly above a concentration of 1 phr of MWCNT, suggesting stress concentration. The results indicate that carbon nanotubes (MWCNT), at 3 phr and 5 phr levels, did not act as reinforcement in the PLA/EE-g-GMA system, given the harmful effect on tensile strength.

Table 1 presents the electrical conductivity ( $\sigma$ ) of pure PLA, the PLA/EE-g-GMA blend, and the PLA/EE-g-GMA/MWCNT nanocomposites as a function of the carbon nanotube content. As reported in the literature (Yadav et al., 2020), the characteristics of materials in electrical properties are insulating ( $10^{-12}$ - $10^{-22}$  S/cm), semiconductors ( $10^{-2}$ - $10^{-9}$  S/cm), conductors ( $10^2$  S/cm), and superconductors ( $10^{20}$  S/cm).

Samples	Electric conductivity (S.cm <sup>-1</sup> )
PLA	$7.24 \times 10^{-12}$
PLA/EE-g-GMA	$4.37 \times 10^{-12}$
PLA/EE-g-GMA/MWCNT (1 phr)	$7.11 \times 10^{-11}$
PLA/EE-g-GMA/MWCNT (3 phr)	$5.31 \times 10^{-08}$
PLA/EE-g-GMA/MWCNT (5 phr)	$3.91 \times 10^{-06}$

Table 1. Electrical conductivity results of PLA, the PLA/EE-g-GMA blend, and the nanocomposites.

Pure PLA showed an electrical conductivity of  $7.24 \times 10^{-12}$  S.cm<sup>-1</sup>, which remains unchanged in the order of magnitude in the PLA/EE-g-GMA blend ( $4.37 \times 10^{-12}$  S.cm<sup>-1</sup>), suggesting that they are insulating materials. Similar results for PLA were also observed in the literature (Wang et al., 2016; Silva et al., 2020). The nanocomposite with a content of up to 1 phr of MWCNT showed a slight increase in electrical conductivity to  $7.11 \times 10^{-11}$  S.cm<sup>-1</sup>, but maintained its insulating behavior. A higher proportion of MWCNT (3 phr and 5 phr) in the nanocomposites caused a more prominent increase in electrical conductivity, which indicates the formation of a three-dimensional conductive network. The addition of 5 phr of MWCNT detected a significant change in electrical conductivity,  $10^6$  times, reaching approximately  $3.91 \times 10^{-6}$  S.cm<sup>-1</sup>. As verified in the SEM, the higher MWCNT content promoted increased distribution in the PLA matrix. This led to the formation of a more efficient conductive path in the nanocomposite with 5 phr of MWCNT, resulting in a semiconductor behavior. For example, electrical conductivity greater than  $10^{-8}$  S.cm<sup>-1</sup> is required for antistatic application (Pascual et al., 2012). Given this, the PLA/EE-g-GMA/MWCNT (5 phr) nanocomposite indicates the potential to be used in applications where electrostatic dissipation is required.

## CONCLUSION

This work produced semi-biodegradable PLA/EE-g-GMA nanocomposites reinforced with carbon nanotubes (MWCNT), aiming at likely antistatic application. The EE-g-GMA copolymer promoted interaction with PLA, forming a tough blend at room temperature. The addition of carbon nanotubes to the PLA/EE-g-GMA blend, regardless of the MWCNT content, formed nanocomposites with impact strength superior to pure PLA. However, the toughening mechanism of the PLA/EE-g-GMA blend was inhibited in the presence of MWCNT, given the decline in the impact strength of the nanocomposites for higher MWCNT content. This behavior was sustained with the increase in the elastic modulus, indicating greater stiffness of the nanocomposites compared to the PLA/EE-g-GMA blend. Regarding tensile strength, it was found that up to 1 phr of MWCNT, there was a slight reinforcing effect, but above this value, the property began to suffer a harmful effect. The SEM morphology indicated that a higher concentration of MWCNT promoted inhibition of the interaction between the PLA and EE-g-GMA phases, generating many particles torn off during the impact test. On the other hand, a higher concentration of MWCNT provided a more accentuated distribution in the PLA/EE-g-GMA system, forming a conductive network, especially with 5 phr of MWCNT. Overall, the PLA/EE-g-GMA/MWCNT (5 phr) nanocomposite presented adequate electrical conductivity for static charge dissipation applications.

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## CONFLICT OF INTEREST

There is no conflict of interest and all authors have agreed with this submission and they are aware of the content.

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