

# Characterization of Polymeric Composite Reinforced with *Moringa oleifera* Pod Fibers

## Caracterização de Compósito Polimérico Reforçado com Fibras de Vagem de *Moringa oleifera*

Murillo de Moraes Valentim<sup>1</sup> , Janaina Fracaro de Souza Gonçalves<sup>2</sup> 

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### ABSTRACT

The concern for the rational use of natural resources and the search for sustainability is a hallmark of the 21<sup>st</sup> century. Manufacturers from various sectors aim to diversify the materials used in their projects, which predominantly rely on non-renewable resources. An alternative involves composite materials based on natural fibers, known as biocomposites, that reinforce currently used high-performance polymers. In the present study, composites were prepared based on a commercial polypropylene (PP) matrix and reinforced with *Moringa oleifera* pod fibers. These compounds were characterized by tensile tests, three-point bending, differential scanning calorimetry (DSC), thermogravimetry (TGA) and fracture analysis by optical microscopy. The addition of vegetable fibers increased the initial degradation temperature of the composite by 4 °C compared to PP while the melting temperature decreased by 11 °C, indicating compatibility between the matrix and the reinforcing agent. Although there was little dispersion in the sampling of mechanical properties data, the results indicate that these properties were inferior to similar biocomposites cataloged to date and even to commercial polypropylene. These variations were investigated through optical microscopy analysis focusing on the distribution of plant fibers in the matrix, particle size and analysis of fractured surfaces to assess fiber-matrix adhesion. The use of plant fibers such as *Moringa oleifera* not only provides an alternative to non-renewable materials but also promotes global sustainability by repurposing biodegradable natural waste.

**keywords** biocomposites, *Moringa oleifera*, natural fiber, green manufacturing

### RESUMO

A preocupação com o uso racional dos recursos naturais e a busca pela sustentabilidade é uma tendência do século XXI. Fabricantes de diferentes setores buscam diversificar os materiais utilizados em seus projetos em contraste com os atuais, não renováveis. Uma alternativa são materiais compósitos baseados em fibras naturais que reforçam polímeros de alto desempenho atualmente utilizados, os chamados biocompósitos. No presente trabalho, compósitos foram preparados com base em uma matriz comercial de polipropileno (PP) e fibras de vagem de *Moringa oleifera* aplicadas como reforço. Esses compostos foram caracterizados por ensaios de tração, flexão em três pontos, calorimetria exploratória diferencial (DSC), termogravimetria (TGA) e análise de fratura por microscopia óptica. A adição de fibras vegetais aumentou a temperatura inicial de degradação do compósito em 4 °C em relação ao PP e a temperatura de fusão foi deslocada 11 °C para baixo, indicando compatibilidade entre matriz e agente de reforço. Houve pouca dispersão na amostragem dos dados de propriedades mecânicas, porém os dados indicam que as propriedades mecânicas foram inferiores aos biocompósitos similares catalogados até o momento e ao próprio polipropileno comercial. Explicações para essas variações foram buscadas por meio de análises de microscopia óptica em relação à distribuição das fibras vegetais na matriz, tamanho das partículas e análise de superfícies fraturadas investigando a adesão entre fibra e matriz. O benefício do uso de fibras vegetais como a *Moringa oleifera* está relacionado ao reaproveitamento de resíduos naturais biodegradáveis, fortalecendo a sustentabilidade global.

**palavras-chave** biocompositos, *Moringa oleifera*, fibra natural, manufatura verde

<sup>1</sup>Master's Student, PPGEM-CP/PG, UTFPR, Cornélio Procópio, Paraná, Brazil. murillovalentim@alunos.utfpr.edu.br

<sup>2</sup>Dr. Prof., PPGEM-CP/PG, UTFPR, Cornélio Procópio, Paraná, Brazil. janainaf@professores.utfpr.edu.br

## Introduction

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The search for sustainable and environmentally friendly materials has driven research in the field of composites that use natural fibers as reinforcement. Natural fiber composites emerge as environmentally sustainable materials that have aroused growing interest in the field of product manufacturing engineering (Bocci et al., 2020; Corrado & Polini, 2019).

Recent research and developments have demonstrated the effectiveness of natural fibers as reinforcing agents in the composites industry, being applied in diverse sectors, such as transportation, internal components, civil construction, aeronautics, and structural engineering (Alsubari et al., 2021; Amir et al., 2021; Asyraf et al., 2020; Sapuan et al., 2021). Furthermore, these composites offer significant advantages, such as lower cost compared to synthetic composites, in addition to being biodegradable, widely available, renewable, and lightweight (Asyraf et al., 2021; Elanchezhian et al., 2018; Ilyas et al., 2021), which makes them a viable solution to several contemporary challenges, including reducing energy consumption during the production process, resulting in almost no carbon emissions and mitigating issues related to improper disposal (Dicker et al., 2014; Omran et al., 2021).

Natural fibers can be classified into three main groups: cellulose-based, protein-based, and mineral-based (Azman et al., 2021). The use of natural fiber-based materials has become increasingly prevalent in the manufacturing industry, attracting the attention of numerous researchers in search of sustainable alternatives. In this context, several natural fibers have been investigated with the aim of improving the mechanical properties of composites, among which the fiber of *Moringa oleifera* stands out.

*Moringa oleifera*, often called the "tree of life", is a plant native to tropical and subtropical regions, known for its numerous nutritional and medicinal properties (Muangnoi et al., 2012). Its leaves, seeds, and pods are rich in vitamins, minerals, and antioxidants, making it a valuable food in several cultures (Saucedo-Pompa et al., 2018). In addition to its food applications, *Moringa* has been explored for its fibers, which present promising mechanical characteristics for the production of composites.

*Moringa* pod fibers, in particular, have shown potential to be used as reinforcements in materials, offering a sustainable and renewable alternative to synthetic composites, thus contributing to reducing environmental impact and promoting the circular economy (Ahmad et al., 2015).

Polypropylene (PP), one of the most widely used thermoplastic polymers in industry, is valued for its chemical resistance, lightness, and ease of processing (Costa et al., 2021). However, growing concerns about plastic pollution and sustainability have motivated research on the incorporation of natural fibers into PP, aiming to improve its mechanical properties and reducing its environmental impact (Jubinville et al., 2021).

Previous studies have shown that the addition of natural fibers to PP can result in composites with improved mechanical and thermal performance. The use of fibers from *Moringa oleifera* pods not only enhances the properties of PP, but also contributes to the valorization of agricultural by-products and circularity (Saucedo-Pompa et al., 2018).

The characterization of natural fiber composites, such as those derived from *Moringa oleifera* pods, represents a significant advance in the search for sustainable and eco-efficient materials. *Moringa oleifera* is a plant widely recognized for its nutritional and medicinal properties, but its potential as a source of fibers for composites has not yet been widely explored.

By investigating the interaction between these fibers and polypropylene (PP), this work contributes to expanding the knowledge about new materials that can replace conventional plastics, promoting sustainability and reducing environmental impact.

Furthermore, the use of natural fibers, such as those from *Moringa* pods, can open up new possibilities for the composites industry, encouraging more responsible and innovative practices.

The dissemination of these results is essential to stimulate future research and foster interest in the application of renewable materials.

In this context, the present study aims to characterize the composite material of natural fiber from *Moringa oleifera* pods and polypropylene, seeking to contribute to the understanding of the interactions between these materials and their potential industrial applications.

## Material and methods

The procedure began with the manufacture of specimens that were later characterized through mechanical tensile and three-point bending tests, thermal analysis of Differential Scanning Calorimetry (DSC), Thermal Gravimetric Analysis (TGA) and optical microscopy.

The *Moringa oleifera* pod was dried in an oven at 70 °C for 24 hours, crushed in a willye knife mill, sieved through two sieves, mesh 28 and 35 at a frequency of 6 Hz, dried again in an oven at 60 °C for 4 hours. The compound in pellet format was obtained by mixing 85 % polypropylene with 15 % fiber and processing in a bench extruder. The material obtained is shown in Figure 1.

**Figure 1** - Biocomposite of *Moringa* pod fiber and palletized commercial virgin polypropylene (PP).



## Mechanical test

The test specimens were manufactured by thermal pressing of the composite pellets under a pressure of 76.38 kgf/cm<sup>2</sup> and a temperature of 200 °C in a mold with standardized test cup geometry in accordance with the ASTM D638 standard.

Ten specimens (CP1, CP2, CP3, CP4, CP5, CP6, CP7, CP8, CP9 and CP10) were produced for tensile test. The value of the modulus of elasticity in tension ( $E$ ) was obtained through the slope of a straight line with linear regression  $R^2$  equal to 0.99. The value of the yield stress ( $\sigma_e$ ) was obtained graphically by drawing a straight line with a slope equal to the modulus of elasticity ( $E$ ) at 0.2 % deformation. The tensile strength limit (LRT) was obtained with the maximum stress value for each test.

For the tensile test, the deformation was measured by a mechanical extensometer coupled directly to the test piece. For the three-point bending test, the deformation was captured through the vertical displacement of the machine as the load was applied.

The three-point bending test was performed in accordance with ASTM D5023. The distance between the supports was 40 mm and the loading speed was 5 mm.min<sup>-1</sup>. Five specimens were produced for this test (CP1, CP2, CP3, CP4, CP5). Equations (1), (2), and (3) were used to determine the modulus of elasticity, flexural strength, and maximum deformation, respectively:

$$E_b = \frac{L^3 m}{4bd^3}, \quad (1)$$

$$\sigma_f = \frac{3PL}{2bd^2}, \quad (2)$$

$$\varepsilon_{\max} = \frac{6Dd}{L^2} \times 100, \quad (3)$$

where the modulus of elasticity in bending ( $E_b$ ) is measured in MPa;  $L$  represents the distance between supports (mm);  $b$  is the specimen width (mm);  $d$  is the specimen thickness (mm);  $m$  is the linear coefficient of force-deflection curve linear region measured in N/mm;  $\sigma_f$  denotes the flexural strength (MPa);  $P$  is the maximum applied load (N);  $\varepsilon_{\max}$  is the maximum strain (%);  $D$  is the maximum deflection at the center of the specimen (mm).

## Thermal analysis

Through thermal analysis, it is possible to monitor physical and chemical properties of a substance, as a function of time or temperature.

The transformation phenomena of the studied material can be analyzed and quantified: decomposition, dehydration, phase transformation, chemical reactions related to endothermic and exothermic effects.

In the present study, a 3.8 mg sample was subjected to a heating rate of  $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$  from  $30\text{ }^{\circ}\text{C}$  to  $250\text{ }^{\circ}\text{C}$  under a flow of  $50\text{ mL}\cdot\text{min}^{-1}$  of nitrogen purge gas using the DSC heat flow method. For the thermogravimetry (TGA), a 6.918 mg sample was subjected to a heating rate of  $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$  from  $25\text{ }^{\circ}\text{C}$  to  $700\text{ }^{\circ}\text{C}$  under a flow of  $50\text{ mL}\cdot\text{min}^{-1}$  of nitrogen purge gas.

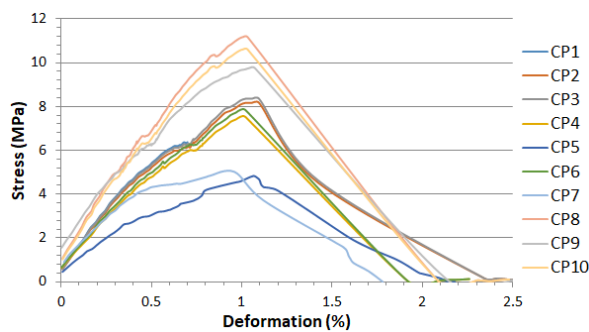
## Optical microscopy

Microscopy analyses of the fractured sections of the specimens from the three-point tensile and flexural tests were carried out using an OLYMPUS optical microscope model BX53M.

## Results and discussion

The curves obtained from the tensile test of the ten specimens are plotted in Figure 2.

**Figure 2** - Overlay of tensile test curves.



The dispersion of data indicated in Figure 2 is an indication that an error may have occurred in the execution of the three-point bending test or even in the production of the test specimens.

Yield stress statistics ( $\sigma_e$ ), tensile strength limit (LRT), and modulus of elasticity ( $E$ ) are compiled in Table 1, considering statistics for all ten specimens of the tensile test (CP1, CP2, CP3, CP4, CP5, CP6, CP7, CP8, CP9, and CP10).

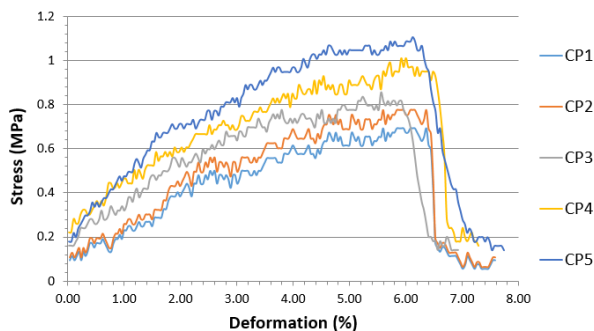
**Table 1** - Tensile test statistics.

	$\sigma_e$ (MPa)	LRT (MPa)	$E$ (MPa)
Average	7.56	7.74	1018.26
Standard deviation	2.43	2.45	270.79
Confidence level for average (95%)	1.740	1.752	193.714

There is a 95 % probability that: the mean will be between the intervals of 5.13 and 10.29 MPa in relation to the yield stress ( $\sigma_e$ ), the mean will be between the intervals of 5.29 and 10.19 MPa in relation to the ultimate tensile strength (LRT), and the mean will be between the intervals of 747.47 and 1289.05 MPa in relation to the modulus of elasticity ( $E$ ).

The curves obtained from the bending test of the five specimens are plotted in Figure 3.

**Figure 3** - Overlay of bending test curves.



The statistics of bending resistance ( $\sigma_f$ ), bending elasticity ( $E_b$ ), and maximum deformation ( $\epsilon_{\text{máx}}$ ) are compiled in Table 2, considering statistics for all five specimens of the three-point bending test (CP1, CP2, CP3, CP4, CP5).

**Table 2** - Bending test statistics.

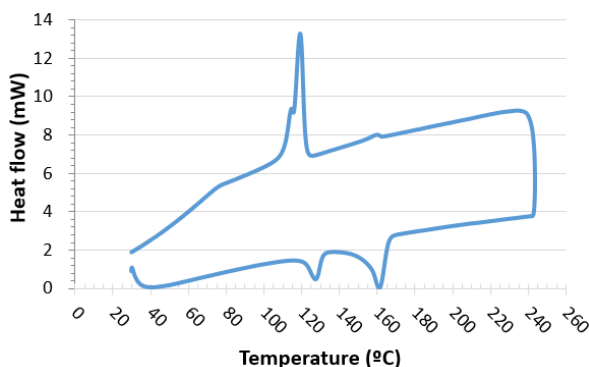
	$\sigma_f$ (MPa)	$E_b$ (MPa)	$\epsilon_{\text{máx}}$ (%)
Average	16.17	1609.53	3.66
Standard deviation	3.07	486.43	0.18
Confidence level for average (95%)	3.81	603.98	0.22

There is a 95 % probability that: The mean is between the intervals of 13.1 and 19.24 MPa in relation to flexural strength ( $\sigma_f$ ), the mean is between the intervals of 1123.1 and 2095.96 MPa in relation to flexural modulus of elasticity ( $E_b$ ) and the mean is between the intervals of 3.48 and 3.84 % in relation to maximum deformation ( $\epsilon_{\text{máx}}$ ).

According to de Sá (2013) the polypropylene composite with 15% *Moringa oleifera* seed shells showed an average of 33 MPa of flexural strength ( $\sigma_f$ ), slightly more than double the demonstrated resistance of 16.17 MPa. Regarding the flexural elasticity modulus, the same author presented an average of 1776.9 MPa, which is 10% higher than the demonstrated 1609.53 MPa. In relation to bending deformation, the composite presented an average of 2.5% with the highest average deformation of 3.66%.

The result of the differential scanning calorimetry (DSC) test is plotted in Figure 4.

**Figure 4** - Biocomposite DSC curve.



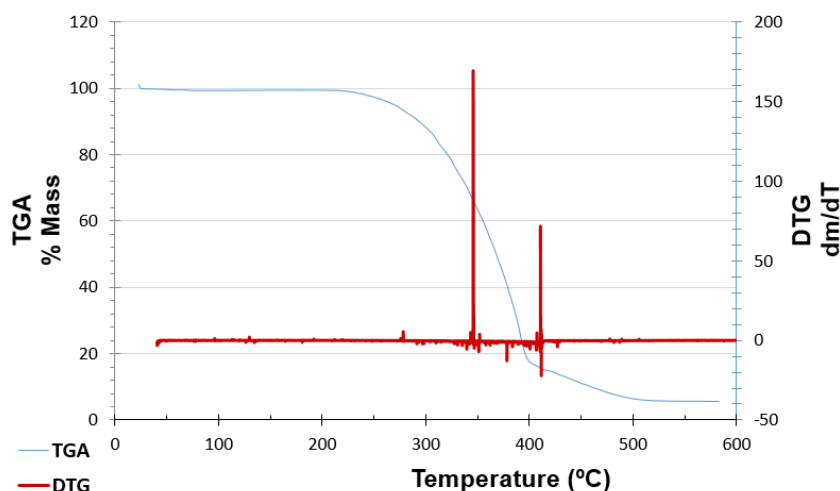
In the endothermic region of Figure 4, from 30 °C to 250 °C, we have two valleys demonstrating the melting temperatures ( $T_m$ ) of the sample, one at 161 °C where the heat flow was zero. This value compares positively with the research by de Sá (2013) for a polypropylene (PP) composite with 15% *Moringa oleifera* seed shells, whose sample presented a melting point of 169 °C, that is, 7 °C higher. This difference may be linked to the crystallinity of the vegetable fiber used.

The melting temperature of commercial polypropylene (PP) is around 172 °C, so it can be said that this valley is associated with the melting of polypropylene (PP). The downward shift in the melting temperature of the composite is associated with a decrease in the material's crystallinity, keeping it more amorphous so that there is less resistance to the breakdown of binders, facilitating melting.

The valley at 127 °C is associated with the melting of a first phase, confirming that it is a composite material composed of more than one phase. This valley is associated with the fusion of vegetable fibers.

Lignocellulosic materials degrade thermally in the range of 150 °C to 500 °C (Silva et al., 2008). Components of these materials volatilize more intensely in different temperature ranges: cellulose between 240 °C and 350 °C, hemicelluloses between 200 °C and 300 °C, and lignin between 350 °C and 500 °C (Leão, 2008), which can also be noted in the TGA/DTG curve in Figure 5.

**Figure 5** - TGA and DTG biocomposite curve.



Cellulose is the most abundant natural polymeric substance and present in the largest proportion in plants, making up to 40% to 50% of their weight (de Sá, 2013). Lignin is the most hydrophobic component, acting as a matrix material holding the fibers together, providing hardness and rigidity to the cell wall, confirmed as the last lignocellulosic material to degrade (Leão, 2008).

The mass loss observed up to 100 °C is related to the loss of moisture, polysaccharides, and its components (mannose, xylose, glucose, among others) and volatile substances. Through the DTG in Figure 5, two stages were observed in which the highest variations in mass occurred on the peaks at 345 °C and 410 °C.

The proposed biocomposite with 15% *Moringa oleifera* seed shells (de Sá, 2013) loses mass in two stages, one from 223 °C to 395 °C and the other from 395 °C to 460 °C, whereas the one proposed in this study loses mass in a single stage from 240 °C to 500 °C. Since pure commercial polypropylene (PP) loses mass from 236 °C to 500 °C (de Sá, 2013), it is possible to state that the fiber addition of 15% to the *Moringa oleifera* pod in the matrix, through the proposed method, did not significantly influence, but positively increased somewhat the thermal resistance of PP, moving its starting point of degradation from 236 °C to 240 °C, demonstrating thermal compatibility between fibers and matrix.

The composite subjected to the Thermogravimetric test (TGA) showed approximately 100% mass loss.

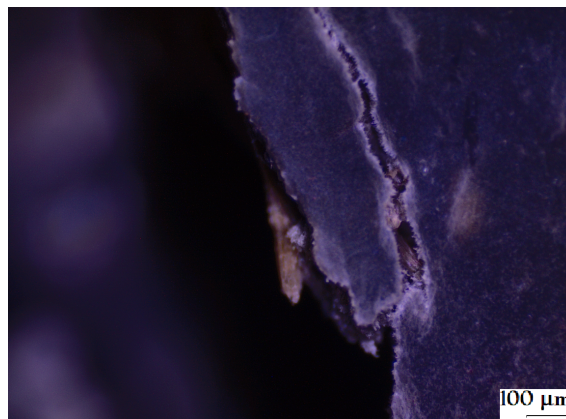
Figure 6 shows the microscopy of the fractured section of a specimen after a three-point bending test, in the same way as Figure 7 shows for the tensile test. It is observed that most of the fibers are orthogonal to the fractured section, and also well distributed.

Visually in the Figures 6 and 7, until the possible magnification in this microscopy, the polypropylene matrix material was well diluted around the fibers, however, compared to (de Sá, 2013) mechanical tests indicated lower resistance of specimens. One indication would be low fiber-matrix adhesion. What could be done would be microscopy with higher magnification to confirm the fiber-matrix interaction.

**Figure 6** - Fractured section of a specimen after a three-point bending test. Magnification 5X.



**Figure 7** - Fractured section of a specimen after a tensile test. Magnification 5X.



## Conclusions

This study performed mechanical characterization of a biopolymer composed of 15% *Moringa oleifera* pod fiber and 85% commercial polypropylene (PP), which presented 16.17 MPa of flexural strength ( $\sigma_f$ ), 1609.53 MPa of flexural modulus of elasticity ( $E_b$ ), 7.56 MPa of tensile yield stress ( $\sigma_e$ ), 7.74 of tensile strength limit (TRL) and 1018.36 of tensile modulus of elasticity ( $E$ ). These values were below those cataloged to date for this biocomposite, demonstrating that there was low mechanical adhesion between the fibers and the matrix. The addition of plant fibers increased the initial degradation temperature of the composite by 4 °C in relation to polypropylene (PP) and the melting temperature was shifted 11 °C lower, indicating excellent thermal compatibility between the matrix and the reinforcing agent.

### Author contributions

M.M. Valentim participated in the: Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Project Managements, Resources, Programs, Supervision, Visualization, Writing – Original draft, Writing – Revision and editing. J.F.S. Gonçalves participated in the: Project Managements, Supervision, Validations.

### Conflicts of interest

The authors declare no conflict of interest.

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