REVISTA DE GESTÃO E SECRETARIADO
MANAGEMENT AND ADMINISTRATIVE PROFESSIONAL REVIEW
ISSN: 2178-9010

Revista GeSec
São Paulo, SP, Brasil v. 14, n. 7, p. 10734-10748, 2023

DOI: http://doi.org/10.7769/gesec.v14i7.2424

Biotechnology management in the Amazon and the production of polypropylene / Brazil Nut Shell fiber biocomposite

Manejo biotecnológico na Amazônia e a produção de biocomposto polipropileno/fibra de casca de Castanha-do-Brasil

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Abstract

This study seeks to produce and analyze the characteristics of a polypropylene composite with vegetable fiber derived from Brazil nut shell. The objective is to develop a biocomposite material based on polypropylene fiber from the brazil nut shell, aiming at a lower environmental impact and promoting sustainability in the industrialization process of biodegradable plastic and/or derivatives. Physical and chemical characterization of vegetable fiber derived from Brazil nut shell (1); characterization of PP and composites with brazil nut shell fibers (PP/FC) (2); characterization of PP and PP/FC/RGO hybrid nanocomposites (3).

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The methodology used is exploratory research with laboratory tests in order to reach the most efficient result. The results obtained allowed us to conclude that the use of Brazil nut shell vegetable fiber as a reinforcement for polypropylene can improve the mechanical properties of materials derived from this process, which contributes to the industrialization of biodegradable plastic materials aiming at sustainability.
Keywords: Amazon. Environmental. Biocomposite. Polymer. Vegetable.

Resumo

Este estudo busca produzir e analisar as características de um compósito de polipropileno com fibra vegetal derivada da casca da castanha do Brasil. O objetivo é desenvolver um material biocompósito à base de fibra de polipropileno da casca da castanha-do-brasil, visando um menor impacto ambiental e promovendo a sustentabilidade no processo de industrialização de plástico biodegradável e/ou derivados. Caracterização física e química da fibra vegetal derivada da casca da castanha do Brasil (1); caraterização do PP e de compósitos com fibras da casca da castanha do Brasil (PP/FC) (2); caraterização do PP e de nanocompósitos híbridos PP/FC/RGO (3). A metodologia utilizada é a pesquisa exploratória com ensaios laboratoriais a fim de alcançar o resultado mais eficiente. Os resultados obtidos permitiram concluir que o uso da fibra vegetal da casca da castanha-do-brasil como reforço para o polipropileno pode melhorar as propriedades mecânicas dos materiais derivados desse processo, o que contribui para a industrialização de materiais plásticos biodegradáveis visando à sustentabilidade.
Palavras-chave: Amazônia. Ambiental. Biocomposto. Polímero. Vegetal.

Introduction

Society's concern for the environment and its yearning for ecologically correct products has led industries and academies to develop studies and research for materials that are renewable, that demand less natural resources and that do not generate waste after use, to such as biodegradable ones.

Studies with sustainable use of natural resources to generate degradable products in the plastics and derivatives industry, it becomes an important sustainable solution for the planet. It was observed during the research that one of the major problems faced when combining materials from different origins in a composite is the interaction between them. In this study, maleic anhydride was used, an agent commonly used with polypropylene (PP) as

a reference and irradiated polypropylene as an alternative. The objective of this study is to analyze the characteristics of the combination of polypropylene, one of the main plastics used by industry, and vegetable fiber from Brazil nut shell in a biocomposite. Specific objectives are to prove the feasibility of producing the composite by mechanical mixing in an extruder, initially characterizing different coupling agents, and to obtain mechanical gains in the resulting biocomposite. The methodology used is exploratory research with laboratory tests. The partial result points to gains in mechanical tests, indicating good processing and interaction between the polymeric matrix and the vegetable fiber load.

Theoretical-Conceptual Review

This work aligns with the UN Agenda, and its Sustainable Development Goals (SDGs), mainly with goal 12, or SDG 12 on Responsible Consumption and Production. This alignment is presented in the proposal to use an agribusiness residue that would become garbage in a renewable raw material. As reported by Arenhardt *et al.* (2018), waste management in the Amazon region is a major challenge, which is aggravated by the distance from large industrial centers.

Replacing only part of the non-renewable fossil raw material with renewable plant matter can generate social and economic gains, helping to combat problems of waste control, environmental pollution, among others (LOPES, 2017).

According to Callister (2007) composite materials are combinations of elements, which by chemical reactions, can generate substances with different characteristics of the constituents. Composites are combinations of materials with an intensity change in existing characteristics. The composite structure is formed by a dispersed phase, called filler, allocated internally in the continuous phase, called matrix.

For Jayavani (2016) biocomposite materials are conventional or biodegradable polymers reinforced with microfillers of renewable origin, that is, ecologically correct composite materials. Fotouh (2015) points out that eco-composites or biocomposites can reduce the amount of fossil and non-biodegradable polymers in a final product, without reducing its properties.

According to Yan *et al.* (2014) and Fonseca- Valero *et al.* (2015) vegetable fibers are mainly composed of cellulose, hemicellulose and lignin, have high mechanical strength, low density, carbon dioxide (CO^2) neutralization, biodegradability, abundant availability. Its ease of processing allows its use as reinforcement in polymeric composites (GUVEN *et al*., 2017).

Agrawal *et al*. (2000) and Rozman *et al*. (2000), state that the high lignin content makes the fiber very flexible; in addition, the hydroxyl groups and aromatic rings present in lignin provide good compatibilization between the fiber and the matrix.

According homma *et al.* (2014) the Brazil nut tree (Bertholletia excelsa Bonpl) is a tree native from the Amazon region that produces spherical fruits, which contain between twenty and twenty-five seeds, the almonds (Brazil nut) covered by a rigid shell. According to Vaisänen *et al*. (2016), the Brazil nut processing industry generates a waste of around two tons/month of chestnut shell. The rational reuse of this residue can contribute to the strengthening of the Brazil nut production chain and to the sustainable economic development of the Amazon region.

According to GhasemI *et al*., (2016) polypropylene (PP) is a thermoplastic polymer that stands out as a matrix of composite materials due to its high chemical resistance, easy molding, high mechanical strength, good thermal stability and miscibility. For Güven and Zengin (2011) irradiation-degraded polypropylene (PPI) is an efficient compatibilizing agent for PP nanocomposites.

Materials And Methods

The materials were processed at the facilities of the Institute for Energy and Nuclear Research (IPEN) and subsequently subjected to various tests in equipment at the laboratories of IPEN and the University of São Paulo (USP). The results were analyzed by statistical techniques of Analysis of Variance (ANOVA).

3.1 Materials for the Research

Polypropylene (PP) is a thermoplastic polymer, a hydrocarbon because it is made up of hydrogen and carbon. The grade used was H301, with properties of high resistance to impact and high rigidity. It has good thermal stability, low moisture absorption, and is easily processed by injection molding. It was provided by Brasken S/A;

Fiber, also known as lingocellulose fiber, due to its constituents (lignin, cellulose and hemicellulose), was extracted from the shell of the Brazil nut (FC). Because there is no financially viable application, it is considered waste and discarded. The material used was donated by the Brazil nut processing industries through Amazon Brazil Nuts.

Maleic anhydride, also known as MAPP (Maleic anhydride Polypropylene), is a chemical compound used as a polypropylene modifier. It is added to the polymer to improve its mechanical and adhesive properties, making it more resistant and compatible with other materials. MAPP is widely used in the automotive industry and in the manufacture of high-performance plastic parts.

3.2 Preparation Technique

Fragmentation of Brazil nut shell fibers was carried out, washed with distilled water, dried to remove moisture using an oven with air passage, grinding in ball mills and separation by granulometry of FC particulates with size ≤ 125 μm. Part of the polypropylene was subjected to gamma irradiation, and used as a compatibilization agent between the PP and the filler. Irradiation was performed in the presence of air, with a radiation dose of 40 kGy. The effects of compatibilization of hybrid composites with irradiated PP (PPI) was compared to that with maleic anhydride (MAPP).

SELECTION OF MATERIALS Polypropylene Brazil nut shell fiber Irradiated polypropylene/MAPP	
EXTRUSION Mechanical mixing of materials in the extruder feeder , for uniform composition and production of pellets	
PELLETS Granules in standard size for processing	

INJECTION Feeding the injection molding machine injector with the pellets for the production of test specimens	
TEST SPECIMENS Injected in standardized dimensions for later characterization	

Table 1 – Composite processing sequence.
Source: The authors.

Several formulations were produced to evaluate the influence of inserting vegetable fiber and which was the most efficient coupling agent, as shown in Table 2.

composites	pure PP (%) [b]	PPI [a] (%) [b]	Fiber (%) [b]	MAPP (%) [c]
PP	100	-	-	-
PP/MAPP/FC	94.7-89.5	-	5-10	0.25
PP/PPI/FC	90-6	5-20	5-20	-

Table 2 - Composite formulation
(a) Irradiated PP; (b)% by mass; (c) % by mass of fiber
Source: The authors.

3.3 Characterization of Pure PP, Vegetable Fiber and Biocomposites

The following analyzes were carried out: Physical and chemical characterization; X-ray fluorescence spectrometry – WDXRF; vibrational absorption spectroscopy in the infrared with Fourier transform – FTIR; scanning electron microscopy (SEM);

Results and Discussions

Within the focus of the objectives of this research, the tests sought to identify the possible mechanical gains and explain through which physical and chemical mechanisms the gains occurred. In this sense, the test of resistance to mechanical traction was the main guideline of the research. The organic and inorganic composition of the plant fiber was made by WDXRF.

In addition to the elements present in the sample, the chemical bonds they form, directly affect the mechanical behavior of the material, the FTIR test identifies which bonds are present in the PP, and how they are affected by the addition of fiber and coupling agent.

4.1 Characterization of Brazil Nut Shell Fiber

While the composition of PP is abundantly documented in the literature, plant fiber is strongly influenced by the soil, region and climate where it was cultivated. In this sense, the physical chemical and WDXRF tests selected sought to identify the organic and inorganic composition of fibers, polymers and composites. The microscopies bring visual aspects and their analysis is done by analogies to the literature observing possible anomalies in the physical structure.

4.1.1 Physical and chemical characterization of the organic composition under study

Next, Table 3 shows the results of the chemical analysis of the organic composition for the vegetable fibers of the Brazil nut shell. The results showed 54% lignin, a large amount when compared to other vegetable fibers commonly used as reinforcements in composites, such as jute with ~13% lignin and sisal with 11% lignin. The cellulose level shown in the table is 38.2%, this level is below sisal fiber with 73.1% and above piassava fiber with 31.6%.

Compound	Amount (%)
Cellulose	38.2 ± 1.0
lignin	54.1 ± 1.1
Extractives	3.9 ± 0.2

Table 3 - Organic composition of Brazil nut shell fiber
Source: INAMURA *et al.* (2013)

Moisture content is a factor of great influence for plant fibers used as reinforcement in composites, high levels can impair all mechanical properties (SPINACE *et al.*, 2009). The moisture content of 14.7% found for the Brazil nut shell fiber is close to that found for jute (10%) and sisal (11%) fibers commonly used as reinforcement in polymeric composites (JAYAVANI *et al.*, 2016).

The density value of 1.33 g/cm³ shown in Table 4 for the fiber is close to the values recorded for other vegetable fibers, such as ramie fibers (1.50 g/cm³) and jute (1.45 g/cm³).

cm³). Compared to synthetic fibers, the fiber density of these vegetable fibers is close to that recorded for aramid (1.4 g/cm³) and much lower than that recorded for glass fiber, type E (2.5 g/cm³). The moisture content and fiber density are shown in Table 4.

	Moisture content (%)	Density (g/cm³)
FC	14.7 ± 0.1	1.33 ± 1.0

Table 4 - Moisture content and fiber density of Brazil nut shell (FC)
Source: INAMURA *et al.*, (2013)

4.1.2 X-ray fluorescence spectrometry – WDXRF

The metabolism and growth of plants are directly linked to the presence of 16 fundamental nutrients, three non-minerals (C, H, O) and thirteen minerals, these divided into primary, secondary and micronutrients. Among the three highest percentages found in the samples, around 70% of the total, are potassium (K), primary nutrient, with 46.5%, calcium and sulfur, secondary nutrients, with 12.6% and 9.0 %, respectively. These elements are essential for plant growth. Plants absorb these nutrients directly from the soil through their roots. If the soil is deficient in these nutrients, it is necessary to correct it by means of fertilization according to the Brazilian Association for Potash and Phosphate Research - Potafos (1998). Next, in Table 5, the results of the X-ray fluorescence analysis for the Brazil nut bark fibers are presented.

Compound	Amount (µg/g)	Uncertainty (µg/g)	Amount* (%)
K_2O	34381	± 500	46.5
OS_3	9356	± 100	12.6
Dog	6684	± 100	9.0
Al_2O_3	6533	± 100	8.8
Fe_2O_3	6380	± 100	8.6
MgO	3214	± 100	4.3
OTHERS	7445	-	10.0

Table 5 - Inorganic composition of Brazil nut shell fiber
*Among inorganic compositions found
Source: The authors.

4.1.3 Scanning Electron Microscopy – SEM

Some vegetable fibers have a smooth surface, due to a layer of wax, which covers the fiber, this wax impairs adhesion, as it directly affects the interface region between the fiber and the matrix, which is the weakest area of the composite, impairs the transfer of efforts between the matrix and the reinforcement (MULINARI et al., 2010). This problem was not observed in this study. The particles used had a morphology like that shown in figure 1b, where the highlighted particle has a rough surface, with pores and protuberances that, according to Campos (2015), help mechanical anchoring. Figures 1a and 1b show the images obtained by SEM of the Brazil nut shell fiber, with magnification 1,000x and 2,000x, respectively.

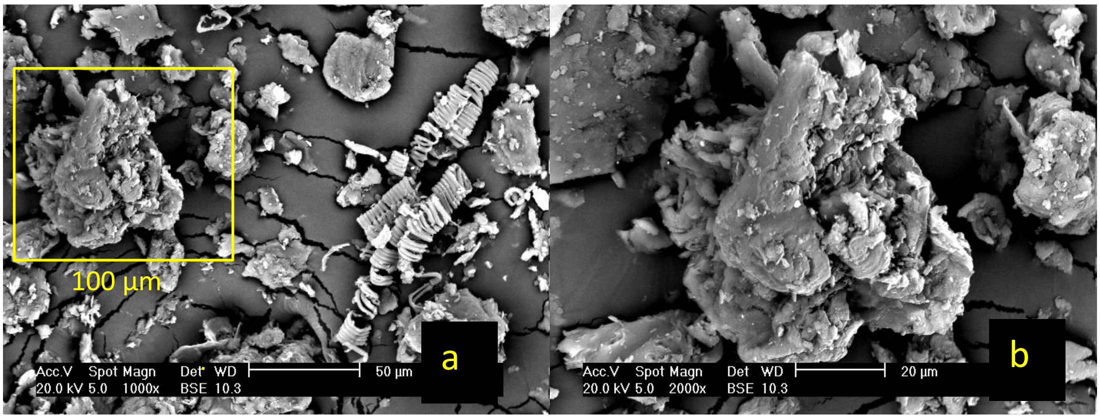

Figure 1 – Micrographs obtained by SEM of Brazil nut shell fibers, with 1,000x magnification (a) and 2,000x magnification (b)
Source: The authors.

4.2 Characterization of PP and Composites with Brazil nut Shell Fibers (PP/FC)

In order to characterize the composites produced, tensile strength tests were carried out to identify the mechanical gains. The SEM tests demonstrated the interaction between the fiber and the polymer, while the FTIR test characterized the behavior of the molecular bonds of the PP, before the addition of the fiber in its composition.

4.2.1 Tensile strength

Table 6 and figure 2 show the results of the tensile strength tests of pure PP samples and PP composites containing vegetable fiber from Brazil nut shell (FC), irradiated polypropylene (PPI) and maleic anhydride (MAPP).

Composites with PPI as a coupling agent showed gains in Young's modulus when compared to pure PP, while those using maleic anhydride had losses in maximum stress and in Young's modulus. According to Jarukumjorn and Suppakarn (2009) these losses may occur due to poor adhesion, MAPP inefficiency and fiber agglomeration, which create fragile points in the specimens.

All composites showed sharp drops in elongation. Elongation is associated with rigidity, mobility of polymer chains and crystallinity of the material. Decreased elongation is indicative of good adhesion and increased stiffness of the composite, this can be observed for composites with PPI. For cases with MAPP, there was a reduction in elongation and mechanical resistance, characteristic of poor adhesion and increased rigidity leading to a more fragile fracture, similar results are found by Padilha *et al* (2010).

Materials	Maximum Tensile Strength (MPa)	Tensile Strength at Rupture (MPa)	Elongation at Rupture (%)	Young's modulus (MPa)
PP	33.5± 1.0	20.9± 1.8	392.7 ±35	364 ±11
PP/MAPP/FC5	32.1± 1.7	30.8± 1.8	16.6 ±1.2	311 ±16
PP/MAPP/FC10	29.2± 2.4	28.0± 2.7	16.6 ±1.5	280 ±23
PP/PPI5/FC10	34.0± 1.6	32.5± 1.4	13.0 ±1.2	448 ±21
PP/PPI10/FC10	34.3± 1.7	33.5 ± 1.6	13.5 ±1.3	407 ±20
PP/PPI20/FC20	35.6 ± 3.1	34.2 ± 2.2	15.0 ±1.3	429 ±37

Table 6 - Tensile strength of PP and composites with PP
Source: The authors.

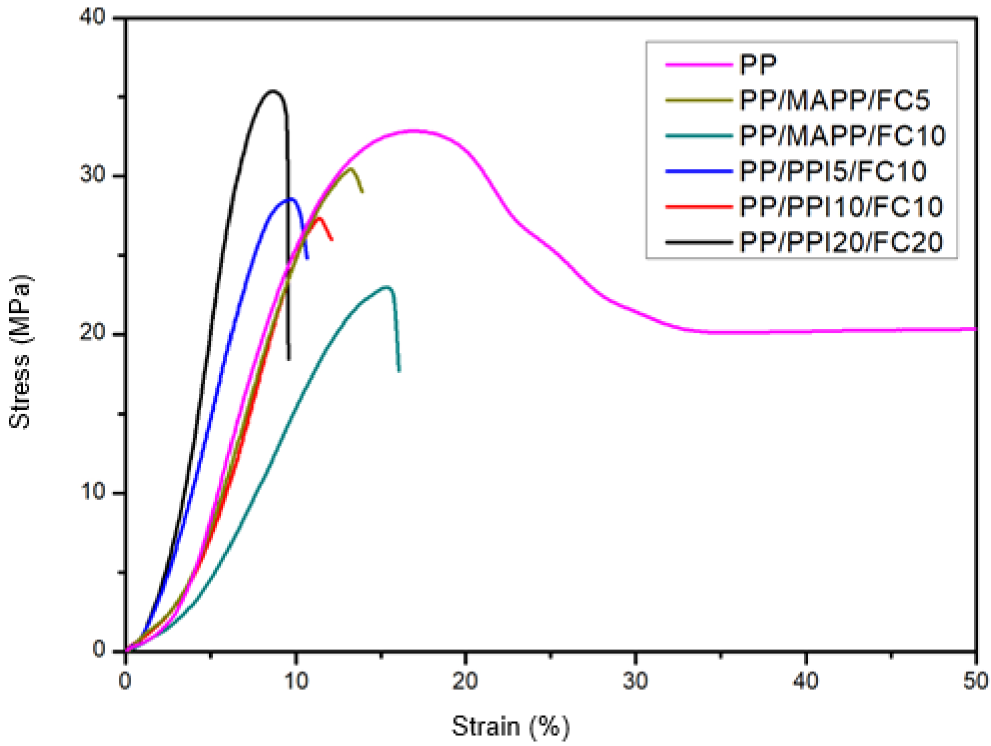

Figure 2 - Stress x Strain Diagram for PP and Brazil Nut Shell Fiber (FC) composites.
Source: The authors.

4.2.2 Scanning electron microscopy with field emission source - SEM-FEG

Figure 3a and 3b, presents the images of the freeze-fractured surfaces, obtained by SEM-FEG for the PP/PPI/FC composites. From the image obtained by SEM-FEG at 1,000x and 5,000x magnification of the PP/PPI/FC composite (Figure 3a) it is possible to observe a minimal detachment at the edges between the fiber and the matrix, and that the fracture occurs in the fiber, indicating good adhesion between the Brazil nut shell fibers (FC) and the matrix (PP).

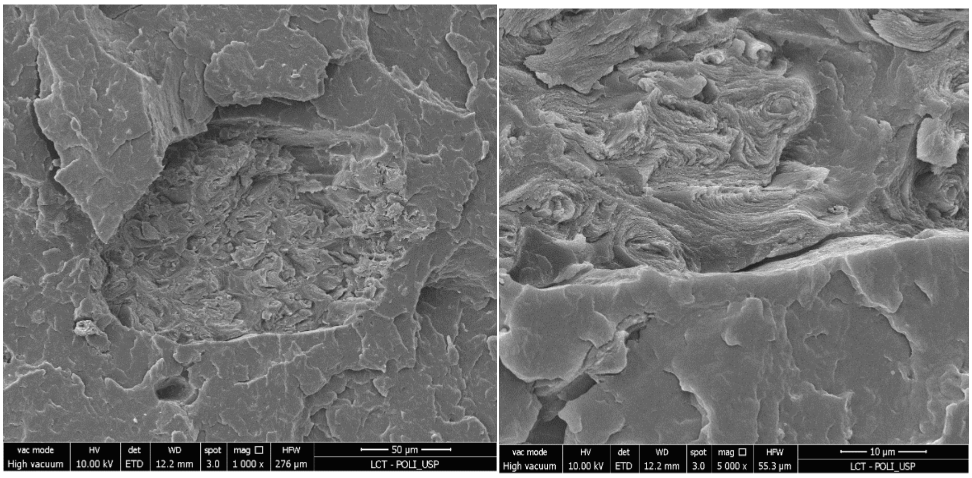

Figure 3a and 3b: Micrographs obtained by SEM-FEG of the freeze-fractured surfaces of the PP/PPI/FC composite at 1,000x(a) and 5,000x(b) magnification
Source: The authors.

4.2.3 Fourier transform infrared absorption vibrational spectroscopy – FTIR in attenuated total reflection (ATR) mode

In Figure 4, some wavelength regions are highlighted, such as the region between the 2947-2835 cm^{-1} bands, characteristic of the symmetric CH stretching; the band 1453 cm^{-1} refers to the angular deformation of the CH_2 group; the region between the 1375-997 cm^{-1} bands that correspond to symmetrical (1375 cm^{-1}) and asymmetrical (997 cm^{-1}) deformation of the CH_3 group; and the 972 cm^{-1} band associated with asymmetric angular strain vibrations of the CH_2 group. The addition of the PPI coupling agent and the FC load did not significantly affect the molecular bonds present in the sample.

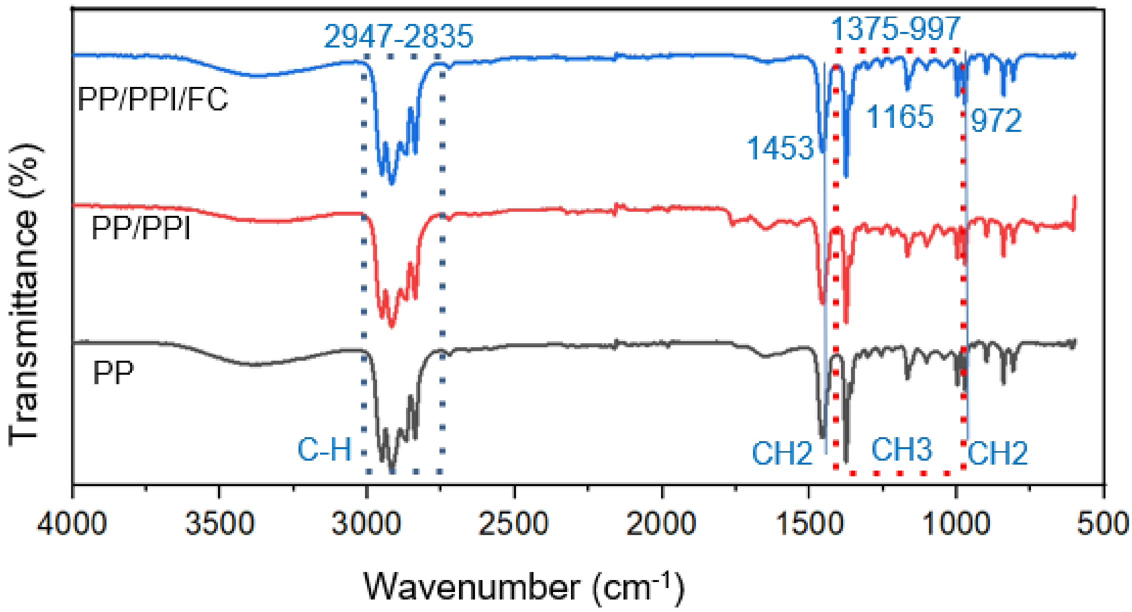

Figure 4: FTIR-ATR spectra for pure PP, PP/PPI, PP/PPI/FC composite.
Source: The authors.

4.2.4 Calculation of the crystallinity index isotacticity index

Isotacticity and crystallinity index, both associated with the organization of molecular bonds, between PP and its composites. High indices are indicative of high rigidity and mechanical resistance to deformation. The indices were calculated using the Origin program, OriginPro 2018 version, based on the spectrograms. The results are shown in Table 7.

	Isotacticity		crystallinity	
	Index (%)	Variation (%)	Index (%)	Variation (%)
PP	74.5	-	70.1	-
PP/PPI	64.2	-13.7	68.8	-1.8
PP/PPI/FC	69.9	-6.2	69.9	-1.2

Table 7: Results of the isotacticity and crystallinity index calculations for pure PP, PP/PPI, PP/PPI/FC composite.
Source: The authors.

From the results presented in Table 7, it is possible to infer that the PP crystallinity index was practically not affected by the insertion of the PPI and the load (FC). However, the isotacticity index of PP showed a decrease, both due to the addition of PPI to PP, as well as due to the insertion of FC. The reduction in the isotacticity index was more pronounced for the PP samples processed with irradiated PP (13.7%), and smaller for the PP/PPI/FC sample (6.2%). But as shown in the mechanical tests, the losses resulting from the drop in isotacticity were attenuated or compensated by the transfer of efforts to the load.

Conclusions

In conclusion, the use of composites of polypropylene and Brazil nut shell as a vegetable fiber showed positive results in line with the objectives outlined. Of the four factors analyzed in the mechanical test (maximum tensile strength, tension at break, elongation and elastic modulus), the PP/PPI/FC option remained stable in the first and was superior in the others. Showing that the goal of mechanical gains was achieved.

In the case of formulations with MAPP, there was a slight decrease in the maximum tensile strength and in the modulus, indicating that the option to use irradiated PP as an alternative to the traditional MAPP was an effective option in terms of the use of coupling agent. The processing of the material by mechanical mixing in the molten state was effective, confirmed by the result of microscopy that showed good dispersion and adhesion of the fiber/polymer.

The application of this type of composite has a very important environmental bias, as the vegetable fiber used as reinforcement is a renewable and biodegradable material, which can replace synthetic materials of fossil origin in several applications. In addition, the use of plant residues as a source of raw material contributes to the reduction of waste and the promotion of a circular economy.

Therefore, the results obtained demonstrate that composites of polypropylene and Brazil nut shell are a viable and sustainable alternative for the production of materials with adequate mechanical properties and low environmental impact. This technology has great potential to be applied in several areas, contributing to reducing dependence on synthetic materials and promoting a more sustainable and circular economy.

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Submetido em: 02.06.2023

Aceito em: 04.07.2023