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# VALORIZATION OF BEAN AGRICULTURAL WASTES FOR THE PRODUCTION OF POLYMERIC COMPOSITES

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**Abstract:** The objective of this study is to evaluate the characteristics of polymeric composites containing different levels of addition of bean residue. To this end, agricultural residues were first reduced to particles and characterized "in natura" through tests for moisture content, bulk density, content of extractives, lignin and ash and by thermogravimetry technique. Then, the particles were dried and selected based on their granulometric fractions to make low-density polyethylene (LDPE) composites with addition levels of 0, 10, 20, 30 and 40% bean residues. The polymeric material and the bean waste particles were hot-extruded to obtain strands and then pellets; the resulting materials were then molded and hot-pressed. After fabrication, the specimens were subjected to physical testing for humidity, density, water absorption, and flammability and to mechanical testing for tensile strength. Transmission electron microscopy (TEM) also was performed. The results showed that the lignocellulosic material had decreased density and mechanical properties and increased water absorption properties after 24 h of immersion, and the moisture content in the samples increased as a function of bean waste substitution level in the polymer composite materials. These results may be related to the intrinsic chemical characteristics demonstrated by this material in the physicochemical characterization: low density and low contents of holocellulose, lignin and extractives. The mechanical properties were also influenced by the pores created by the bean particles, which may have contributed to the creation of larger zones of weakness in the samples.

**Keywords:** Byproducts, LDPE, flammability.

## GRAPHICAL OVERVIEW
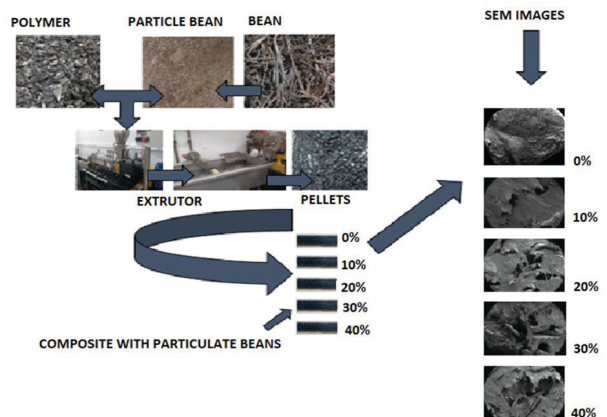


## INTRODUCTION

Production of polymeric items containing lignocellulosic materials has been increasing in the last ten years in several sectors of the world economy. Common and everyday materials produced from these components are pergolas, decks, furniture, decoration items, brises, baseboards, materials for the automotive industry, among others. (Carus et al. 2015; Chaudemanche et al. 2018).

Large amounts of polymeric waste are, however, generated from the production of these composites or other polymeric materials, and the improper disposal of these materials causes environmental pollution, especially in riverbeds and oceans. Therefore, there is a need to decrease the production and use of nonrenewable products or, at the very least, promote greater recycling of these materials. Brazil, for instance, was responsible for generating 11.3 million tons of polymeric wastes in 2016, and only 1.28% of these total polymers were recycled (Kaza et al. 2018).

Although the physical, mechanical, and chemical properties of low-density polyethylene (LDPE) are compatible with widespread industrial use, it can accumulate in the environment as a pollutant. In this context, the use of recycled polymeric materials is a potential alternative for reducing the environmental impacts resulting from the manufac-

turing of polymeric materials and reverse logistics after polymer use (Thomas et al. 2022; Kaza et al. 2018).

Another economical option with less environmental impact that has been growing in recent years in the polymeric composite sector is the use of these polymeric materials in conjunction with lignocellulosic wastes, that is, products manufactured by injection-molding or extrusion-disposal of discontinuous granular thermoplastic polymers with the addition of fibers (Brenken et al. 2019). Such insertions solve two bottlenecks presented by these "in natura" materials: the low stiffness of some polymers and the high water absorption of most plant fibers (Mertens et al. 2017).

Several researchers have studied, in this context, the use of biomass wastes from different agricultural branches in conjunction with polymers, and they have highlighted combinations of hybrid polymer composites with the following plant fibers: bamboo (Subash et al. 2022), pinecone (Şahinöz et al. 2022), sugarcane bagasse (Madhoushi et al. 2021), jute (Ganesh; Anand, 2022), hemp (Bhowmik et al. 2022), pineapple (Dilli Babu et al. 2020), banana pseudostem (Jordan and Chester 2017), coconut peel (de Araújo Veloso et al. 2021), and coffee wastes (García-García et al. 2015), with the primary objective of improving the biodegradability and chemical and mechanical properties of the composite material (Brenken et al. 2019; Peças et al. 2018).

Residues from bean plantations are among the agricultural residues with application potential still little explored in this scientific area. The Brazilian bean grain crop in 2023/2024 is projected to reach a production of almost 3.3 million tons. Bean production is accompanied by a large generation of waste that is not widely used. (CONAB 2023).

Therefore, the main purpose of this research is to evaluate the physical-mechanical properties of polymeric low-density polyethylene (LDPE) matrix composites in which bean waste particles have been introduced at different polymer mass addition levels (0, 10, 20, 30 and 40%).

## MATERIALS AND METHODS

### MATERIAL

The bean residues used in the experiment originated from the leftover *Phaseolus vulgaris* harvest of the Pimentas farm (Lavras, Minas Gerais, Brazil) (latitude 21°15′58.5″ S and longitude 45°03′ 31.5″ W). The residues consisted of stems and pods that were collected after natural drying. To produce the composites, the material was ground and separated according to granulometry. The particles that passed through a 40 mesh (0.42 mm) sieve and were retained on a 60 mesh (0.25 mm) sieve were selected.

The recycled low-density polyethylene (LDPE) used for the development of the composites was obtained from a recycling industry located in the city of Lavras, Minas Gerais, Brazil, and it had dimensions of 1.5 x 4.0 cm. Both materials could be fed into the extruder without pretreatment.

The bean residues and the LDPE were dried in an oven at a temperature between 50-75 °C. The drying was finished when the particles reached a moisture content between 3-4 %, a value necessary not to hinder the following procedures.

## METHODS

### MATERIALS CHARACTERIZATION

Bean waste particles, granulometrically separated, as previously mentioned, and homogeneously distributed, aiming to homogenize the physicochemical characteristics of the three parts of the plant, were characterized by the tests shown in Table 1. All tests were performed in triplicate.

| Test | Standard |
|------|----------|
| Apparent density | NBR 14810-2 (ABNT 2018) |
| Moisture | NBR 14810-2 (ABNT 2018) |
| Extractives | NBR 14853 (ABNT 2010) |
| Acid-insoluble lignin | NBR 7989 (ABNT 2010) |
| Ash content | NBR 13999 (ABNT 2017) |

**Table 1.** Characterization of the bean residues.

The holocellulose content was obtained by percentage difference based on the other chemical constituents (Equation 1).

H (%) = 100(%) - total extractives (%) - lignin content (%) - ash content (%)     (1)

Thermogravimetric analyzes (TGA) were also performed to characterize the residues. The test was performed in an open alumina crucible, and the samples were heated from 25 to 500 °C at a nitrogen flow of 20 mL/min, with a heating rate of 10 °C/min. The degradation temperature was determined from the inflection of the baseline of the differential thermogravimetric (DTG) curve.

### COMPOSITE PRODUCTION

After the characterization of the materials, the polymeric composites were produced through the gradual mass addition of bean waste particles in a low density polyethylene (LDPE) matrix, totaling five treatments. The addition levels were 0, 10, 20, 30 and 40% (de Araújo Veloso et al. 2021).

To produce the composites, 600 g of material was processed in a twin-screw extruder with a manual feed. In the extruder, this material went through seven hot zones (120 °C - 130 °C - 140 °C - 140 °C - 140 °C - 140 °C - 150 °C), generating a strand that was later cooled in water (24 ± 2 °C) and cut into small pellets that were placed in metal molds with dimensions of 120x12x3 mm (length, width and thickness). The molds were transferred to an automatic hydraulic press (MA 098) for a pressing cycle at 135 °C and 40 MPa for 15 minutes. After the samples were pressed, they were cooled in water (24 ± 2 °C) and stored in a controlled, weather-free environment (22 ± 2°C and RH 65 ± 5%) for a period of 7 days (de Araújo Veloso et al. 2021).

### COMPOSITE CHARACTERIZATION

Evaluation of the polymer matrix composites containing bean residues was performed by the tests presented in Table 2. All tests were performed in triplicate.

| Test | Standard |
|------|----------|
| Tension | ASTM D638-01 (ASTM 2002) |
| Water Absorption | ASTM D570-98 (ASTM 2018) |
| Flammability | UL-94 (UL 1986) |

**Table 2.** Tests performed on the composites

The moisture content of the composites on a dry basis was determined by weighing them before and after thermal processing in an oven (105 ± 5 °C) for 24 h. The bulk density was measured by dividing the mass by the volume of each sample.

The flammability test was carried out with the aid of a fixed test piece and a Bunsen burner. The composites were fixed to the material and a flame was lit through the Bunsen burner. After ignition, the time for self-extinguishing of the material and whether there was dripping are measured, in order to correlate the flammability classification according to the standard UL-94 (UL 1986).

The tensile test was carried out in a universal testing machine with a load cell of 2000 kgf, test speed of 5 mm/min and distance between the claws of 9.76 cm.

Transmission electronic microscopy (TEM) tests were also performed using a Zeiss microscope (model DSM 940ª) that belongs to the Department of Plant Pathology of the Federal University of Lavras (UFLA) to evaluate the region of tensile rupture. To this end, the samples were dried in an oven and then plated with gold.

### EXPERIMENTAL DESIGN

The experimental design was entirely randomized with 5 treatments and 3 replicates. Linear regression tests and analysis of variance (ANOVA) were performed to compare and evaluate the effect of bean residue addition on the polymeric matrices.

## RESULTS AND DISCUSSION

### BEAN RESIDUE CHARACTERIZATION

Table 3 shows the results of the chemical and physical properties of the "in natura" materials, namely, bean particles.

The measured moisture value for the bean residue was 9.98%. This value is higher than the 4% value reported for coffee parchment (Scatolino et al. 2017), and since the lignocellulosic and polymeric material particles were dried to a moisture content of 3-4% before the production of the composites, both materials had a moisture content lower than 5%, close to that recommended by Barbosa et al. (2019), who stated that a 5% moisture content in lignocellulosic materials is ideal for composite applications because it avoids gas evaporation and the appearance of bubbles.

The measured apparent density for the bean residue was 240 kg/m³; since the value is lower than 500 kg/m³, it is considered a low-density material according to the Technological Research Institute (IPT 1985). This value agrees with that obtained in other studies of the same residue (Miranda et al. 2022a). It is also equal to the particle density of *Pinus taeda* (Miranda et al. 2022b) and is lower than the apparent density obtained by Wong et al. (2020) for sugarcane bagasse (620 kg/m³). According to Iwakiri and Trianoski (2020), low density values are desirable for composite manufacturing because a greater number of particles is present within the same volume of the multiphase material, and the resulting material is lighter.

For extractives and lignin, hydrophobic compounds of low molecular weight in the bean residues presented respective values of 8.16% and 8.13%. The values for lignin were lower than those obtained for soybean residues (22.04%) (Borges et al. 2022) and coffee wastes (29.28%) (Santos et al. 2022). The values of extractives were lower than those obtained from sugarcane bagasse (19.59%) (Soares et al. 2017) and soybean residues (13.69%) (Borges et al. 2022). These data reveal that the use of this product in polymeric matrices is favorable (Iwakiri and Trianoski 2020).

The ash content was 16.94%, and although this is a relatively high value compared with other lignocellulosic materials, such as cocoa residue (3.17%) (de Araújo Veloso et al. 2021) and bamboo fibers (0.22%) (Gomes et al. 2021), there are no literature reports on the influence of this component on polymeric composites (Iwakiri and Trianoski 2020).

The holocellulose value for the bean residue was 56.55%, which is lower than that reported for corn cobs (76.70%) by Scatolino et al. (2013). Due to the hydroxyl groups (OH) present in holocellulose, the resulting lignocellulosic materials are highly hydrophilic. This can hinder the use of these materials in various applications by negatively impacting physical properties related to water and moisture absorption (Iwakiri and Trianoski 2020).

| Moisture (%) | Apparent density (g/cm³) | Extractives (%) | Lignin (%) | Ashes (%) | Holocellulose (%) |
|---|---|---|---|---|---|
| 9.98 [(0.83)*] | 0.24 [(0.03)] | 8.16 [(1.60)] | 8.13 [(0.79)] | 16.94 [(0.43)] | 56.55 [(3.97)] |

**Table 3.** Chemical characterization of bean residue

*Standard deviation of means

The results of the thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) of the bean residues are presented in Figure 1.
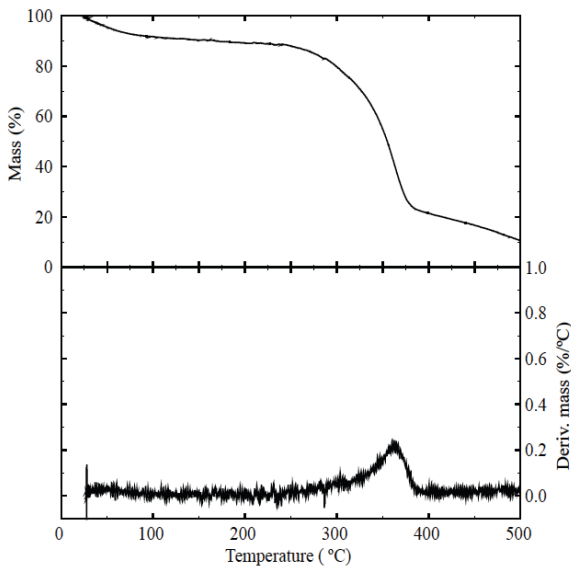


**Fig. 1.** TGA and DTG curves of the bean residues.

Initially, a 9.01% loss of adsorbed water is observed below 120 °C. Between 200 and 400 °C in the TGA plot, there is a region of holocellulose decomposition, corresponding to 67.66% of the composition. A peak in the decomposition of these components is noted at ~350 °C in the DTG curve. Lignin decomposes between 400 and 500 °C (10.92%), and above this temperature, the mass loss is negligible (Acchar et al. 2013). The decomposition of these lignocellulosic components is consistent with the chemical characterization shown in Table 3.

## PHYSICAL PROPERTIES OF THE COMPOSITES

The results of the physical properties, moisture (W), water absorption at 2 h (WA2 h) and 24 h (WA24 h) after water immersion and bulk density (ρ) are displayed in Figure 2. The equilibrium moisture between the relative humidity and room temperature of the composites increased linearly, with a significant $R^2$ coefficient of 0.7914 and an Fc of 0.7914, from 0.16% in the reference sample to 3.26% in the sample with 40% beans, as observed in Figure 2a.

The water absorption after 24 h of immersion (Figure 2c) of the composites also followed a linear trend in growth as a function of increasing lignocellulosic residues in the polymeric com. However, the linear regression coefficient $R^2$ was smaller (0.5124), and the coefficient of analysis of variance was 2.0675. This trend is similar to that found by de Araújo Veloso et al. (2021), in which the moisture of the composites in the study varied from 0.03% in the LDPE reference sample to 2.68% in the sample with 40% cocoa waste and the water absorption after 24 h varied from 0.17% in the reference sample to 2.68% in the sample containing the highest amount of lignocellulosic waste.

This may be related to the holocellulose content found in the lignocellulosic materials (Table 3). Lignocellulosic materials are hydrophilic due to the hydroxyl groups (OH) present in holocelluloses. This characteristic may restrict the use of these materials for several applications (Coutinho et al. 2003; Iwakiri and Trianoski 2020).
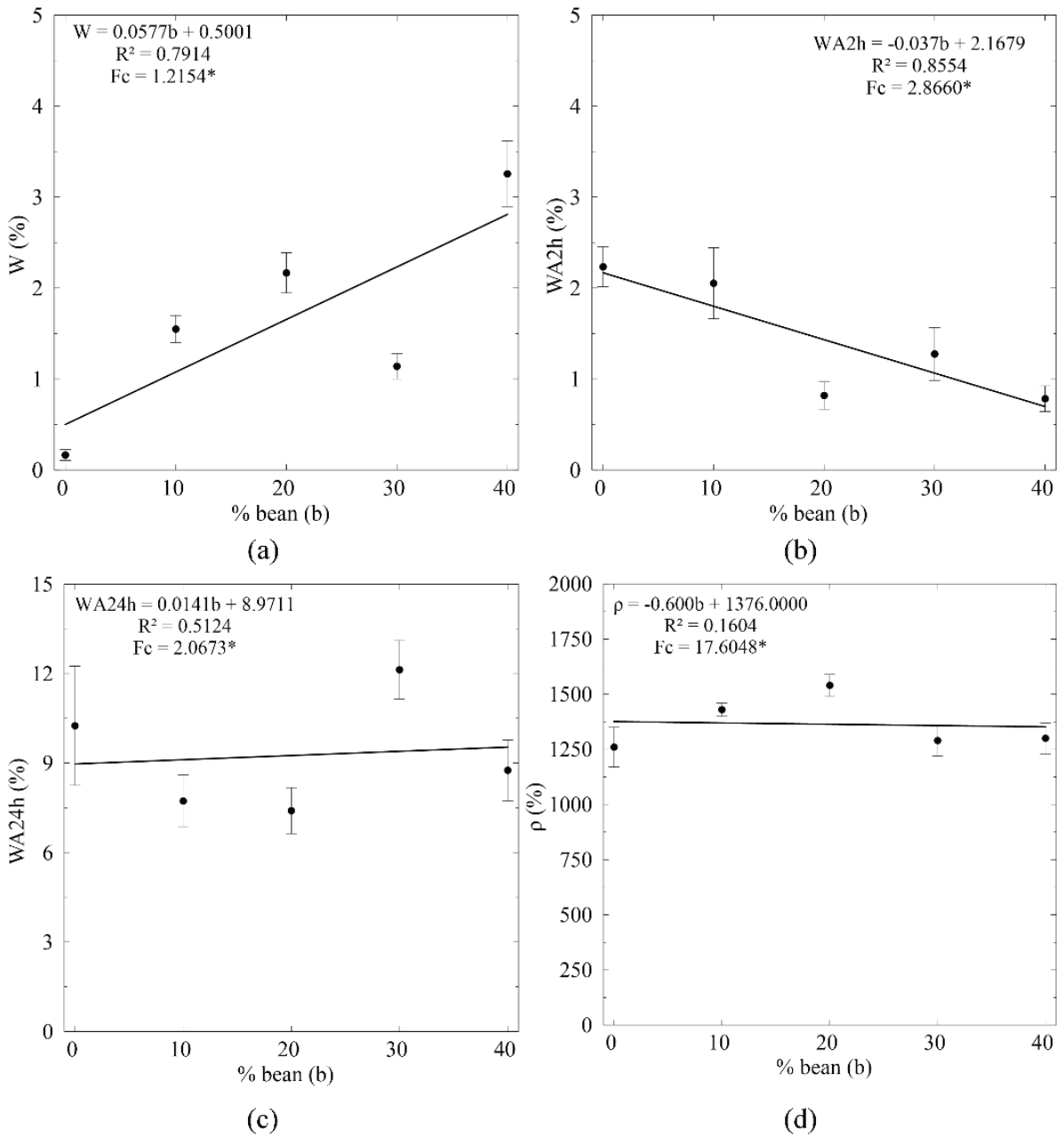
**Fig. 2.** Moisture (a), water absorption after 2 h (b), water absorption after 24 h (c) and bulk density of the composites (d). * Significant regression analysis at 5% significance.

After 2 h of immersion (Figure 2b), an opposite trend was observed, meaning that initially, as the polymer had high hydrophobicity, these characteristics prevailed over short periods. At longer periods, however, the hydrophilicity of the bean particles caused an increase in water absorption (Adegoke and Bello 2015).

In the results shown in Figure 2d, a slight decrease in bulk density is noted with the increase in bean particles in the multiphase material, but it is not significant at the level of linear regression and analysis of variance. A similar trend was found by de Araújo Veloso et al. (2021) after cocoa waste was incorporated into LDPE matrices, and this may be related to the lower density of the waste in relation

to the polymer. LDPE has a density ranging between 912 and 925 kg/m³, while lignocellulosic materials have much lower densities (240 kg/m³), allowing the resulting multiphase materials to be lighter (Coutinho et al. 2003).

### FLAMMABILITY OF THE COMPOSITES

The results of the flammability test can be found in Table 4.

| Addition | Time to self-extinguish after ignition (s) | Was there drip? | UL 94 Classification |
|---|---|---|---|
| 0 | 405 | Yes | H-B |
| 10 | 321 | Yes | H-B |
| 20 | 285 | Yes | H-B |
| 30 | 191 | Yes | H-B |
| 40 | 155 | Yes | H-B |

**Table 4.** Flammability test results.

Although all composites showed equal flammability ratings (H-B, highly combustible), the materials containing more bean residue impacted the time to self-extinguish the fire, meaning that such materials are more flammable than the polymers and therefore impair flame spread resistance (UL-94, UL 1986).

Kim et al. (2018) claimed that the high concentrations of levoglucosan generated with the thermal decomposition of lignocellulosic fibers can increase flammability levels. This fact was also suggested by Chai et al. (2012) when they compared the flammability of polymer composites containing glass fibers and composites with flax fibers. The authors observed that composites containing natural fibers have higher heat release rates and higher burning rates than those containing glass fibers.

The low fire resistance of natural fibers and consequently the higher flammability of these materials can hinder their use in environments with stricter safety regulations.

### MECHANICAL AND MICROSTRUCTURAL CHARACTERIZATION OF THE COMPOSITES

From the tensile test results, it was obtained the following properties: modulus of elasticity (E), modulus of rupture (σ) and tenacity (T), that can be seen in Figure 3.

The E ranges from 102 to 240 MPa; the σ of this material is reported to have values between 6.9 and 16 MPa; the value for the reference sample was compatible with those reported for it in the literature (Coutinho et al. 2003).

The insertion of bean residues led to a significant decrease in the E and σ values, from 117.87 MPa to 101.50 MPa and 20.07 MPa to 8.97 MPa, respectively, in the reference sample for the sample with higher lignocellulosic material content (Fig. 3a and b). This decrease may be related to the low concentration of cellulose found in the bean particles compared with other lignocellulosic wastes, which acts to reinforce the composites (Table 3) (Oliveira et al. 2017).

The tenacity (T) values also decreased as the lignocellulosic residues were added (Fig. 3c) (4678.34 to 511.16 kJ/m²). The same behavior was reported in literature for high impact polystyrene composites (HIPS) reinforced with green coconut shell fibers (Pereira et al. 2017).

For Veloso et al. (2021), the reduction in tenacity values is associated with intrinsic factors to both materials (polymer and waste). In addition, low tenacity values can directly affect the impact resistance of the composite (Coutinho et al. 2003; Pereira et al. 2017).

Low degrees of interaction or degradation at the interface between the fibers and matrices may also have influenced the decrease in the mechanical property values of the composites (Mertens et al. 2017).

(a)



(b)



(c)
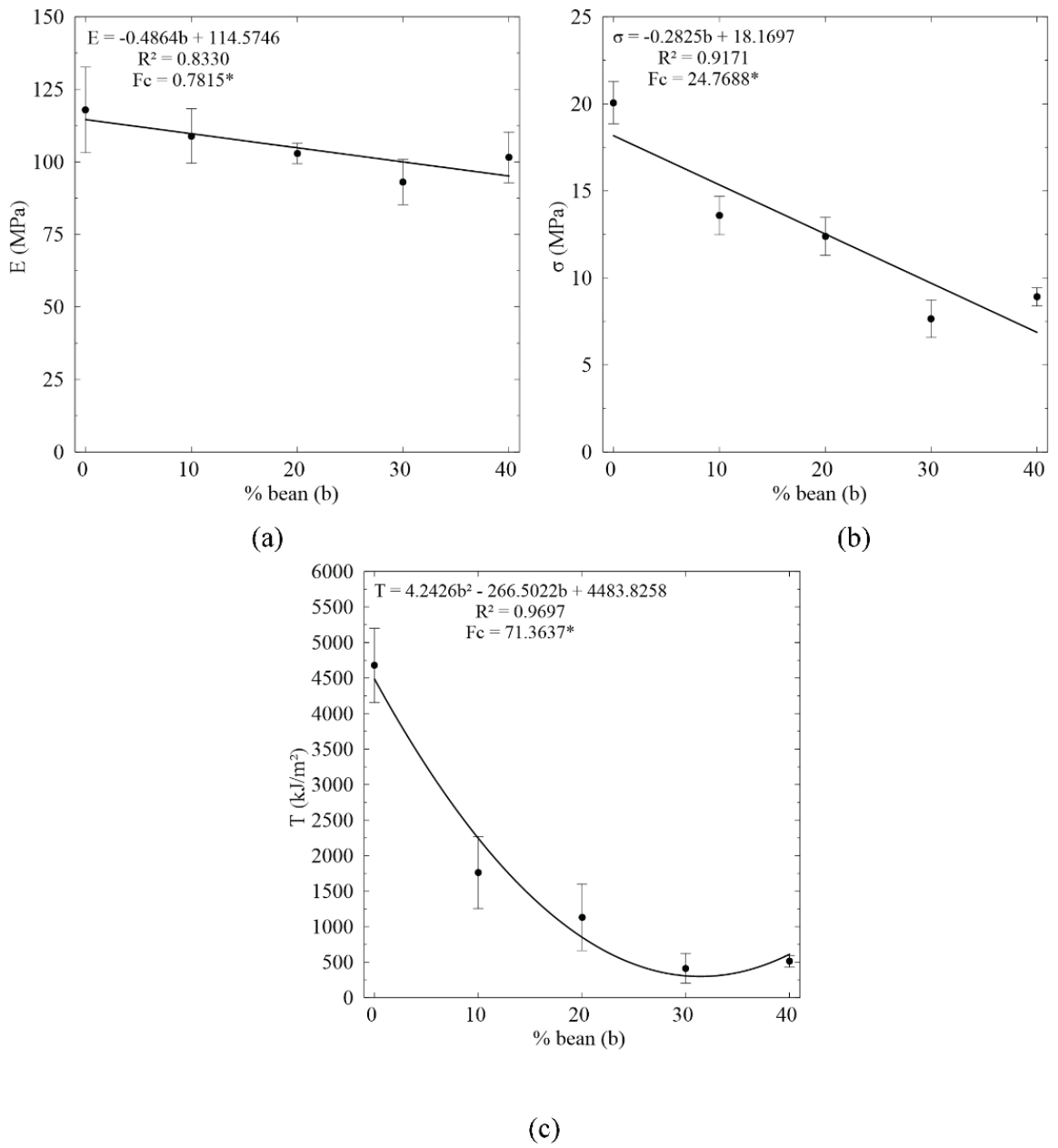
**Fig. 3.** Result of the tensile tests: modulus of elasticity (E) (a), modulus of rupture (σ) (b) and tenacity (T).

This tendency also may be related to the greater number of pores in the samples, as confirmed by TEM analysis (Fig. 4). The fracture regions in the samples show a nonhomogeneous distribution of bean materials at the points of weakness. Voids are also present, resulting from the detachment and removal of the fibrous residue grain, indicating that there are weak interactions between the reinforcement and the matrix. In this sense, stress transfer is reduced when unfilled pores are present at the interfaces and at some imperfections, which aids in the propagation of cracks in the material (Mertens et al. 2017).
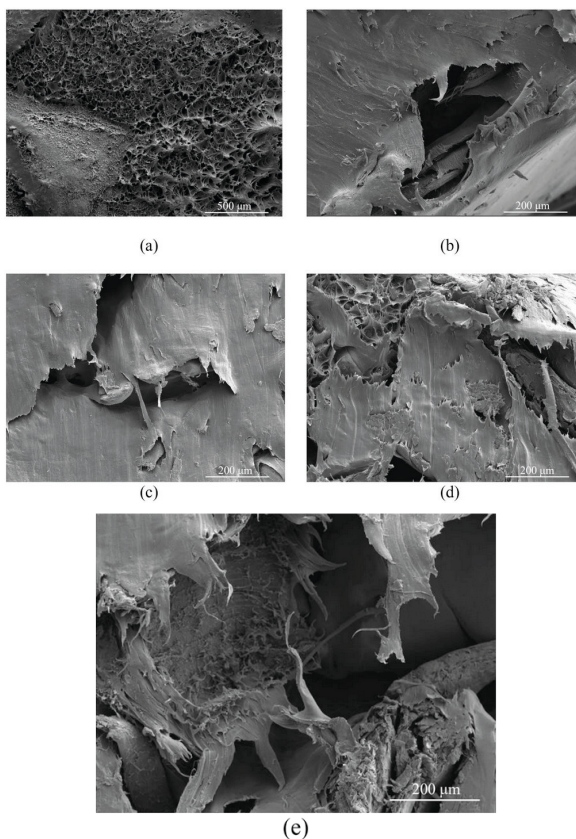


**Fig. 4.** TEM images of materials: reference (a) and composites with bean particles addition levels of 10% (b), 20% (c), 30% (d) and 40% (e).

## CONCLUSIONS

The objective of this study was to analyze the influence of bean particles incorporation on the physical, mechanical and microstructural properties of polymer composites.

Regarding physical properties, the results showed that the incorporation of lignocellulosic material caused uniformity in the apparent density of the panels. Furthermore, moisture and water absorption at 24 hours increased proportionally to the higher polymer substitution content per residue, 0.16 to 3.26% and 8.76 to 10.25%, respectively, due to the holocellulose content of waste (56.55%). The bean residue also contributed to faster flame propagation in the flammability test.

Regarding the mechanical properties arising from the tensile test, there was a downward trend in all properties analyzed, modulus of elasticity 117.87 to 101.50 MPa, modulus of rupture 20.07 to 8.97 MPa and tenacity, 4678.34 to 511.16 kJ/m², with the incorporation of bean residues. This may be related to low compatibility between the matrices, interface degradation and a large number of voids in the samples confirmed in TEM analyses.

Therefore, it was not possible to prove the feasibility of applying polymer composites reinforced with bean waste, requiring further studies with new formulations, particle pre-treatments and other parameters to overcome the problems analyzed.

## ACKNOWLEDGMENTS

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