



MECHANICAL EVALUATION OF CASTOR OIL BASED POLYURETHANE BIOCOMPOSITES REINFORCED BY EUCALYPTUS WOOD RESIDUE

AVALIAÇÃO MECÂNICA DE BIOCOMPÓSITOS DE POLIURETANO À BASE DE ÓLEO DE RÍCINO REFORÇADOS POR RESÍDUO DE MADEIRA DE EUCALIPTO

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Abstract - Wood has been used as a structural material for a long time. It can be understood as a natural composite material with polymer matrix reinforced by natural lignocellulosic fibers. In Brazil, the Eucalyptus species has been widely used due to its rapid growth and great adaptability, making this variety one of the most cultivated. During wood processing, even using modern techniques, about 30% of the material is not used, which causes a large amount of waste and which is normally burnt. Thus, the objective of this work was to investigate mechanical properties of biocomposites with castor oil-based polyurethane matrix reinforced with Eucalyptus obtained as a residue from the wood industry. Composites were produced from 10 up to 40 vol% of residues obtained in three different processing stages. The biocomposites were subjected to flexural, tensile, and charpy and izod impact tests. The results showed that for both flexural and tensile strength, low volumetric fractions of the residues cause a drop in performance mostly associated with the appearance of bubbles in the matrix. As the volumetric fraction of the residue increases, there was a tendency for an increase in the performance of the composites. Regarding the impact tests, the biocomposites show almost no significant difference from the pure resin, with a tendency of lower impact resistances. Moreover, it became clear that both the volume fraction and the geometry of the reinforcement influences the biocomposites mechanical behavior. Indeed, the longer the residue the greater was its tensile and flexural performance.

Keywords: Mechanical Properties; Residue; Eucalyptus; Vegetable polyurethane.

Resumo - A madeira é utilizada como material estrutural há muito tempo. Esta pode ser entendida como um material compósito natural com matriz polimérica reforçada por fibras lignocelulósicas naturais. No Brasil, a espécie de eucalipto tem sido amplamente utilizada devido ao seu rápido crescimento e grande adaptabilidade, tornando esta variedade uma das mais cultivadas. Durante o processamento da madeira, mesmo utilizando técnicas modernas, cerca de 30% do material não é aproveitado, o que gera uma grande quantidade de resíduos e que normalmente é queimado. Assim, o objetivo deste trabalho foi investigar as propriedades mecânicas de biocompósitos com matriz de poliuretano à base de mamona reforçada com eucalipto obtido como resíduo da indústria madeireira. Foram produzidos compósitos de 10 a 40 vol% de resíduos obtidos em três diferentes etapas de processamento. Os biocompósitos foram submetidos aos ensaios de flexão, tração e impacto charpy e izod. Os resultados mostraram que tanto para resistência à flexão quanto à tração, baixas frações volumétricas do resíduo causam uma queda no desempenho principalmente associada ao aparecimento de bolhas na matriz. À medida que a fração volumétrica do resíduo aumenta, houve uma tendência de aumento no desempenho dos compósitos. Em relação aos ensaios de impacto, os biocompósitos quase não apresentam diferença significativa em relação à resina pura, com tendência a menores resistências ao impacto. Além disso, ficou claro

que tanto a fração volumétrica quanto a geometria do reforço influenciam o comportamento mecânico dos biocompósitos. De fato, quanto maior o resíduo, maior foi seu desempenho de tração e flexão.

Palavras-chave: Propriedades Mecânicas; Resíduo; Eucalipto; Poliuretano vegetal.

1. INTRODUCTION

In the past century, there has been a decline in the interest in traditionally used natural fibers, due to discovery of new technologies. Indeed, synthetic materials emerged and completely changed the way human beings relate to the environment. This replacement occurred due to technical advantages, such as longer life cycle, easier large-scale production or even superior mechanical performance (Nurazzi et al., 2021; Lau et al., 2018; Pickering et al., 2018; Pickering et al., 2016).

In recent decades, however, the scientific community has grown concerned with the human impact on the environment, which led to the development of ideas such as environmentally friendly materials and sustainable development. In this new scenario, researches proposing natural lignocellulosic fibers (NLFs) as an alternative to synthetic materials emerged, especially as polymer composites reinforcement (Madhu P *et al.*, 2020; Singh *et al.*, 2020; Garcia Filho *et al.*, 2020; Robledo-Ortíz Jr. *et al.*, 2020; Martulli *et al.*, 2019; Wu Y *et al.*, 2018; Güven *et al.*, 2016; Sanjay *et al.*, 2018; Pickering *et al.*, 2016).

The use of natural fibers as reinforcement in composite materials is justified by several arguments, among which we can highlight: ecological materials, relatively low cost and weight, social benefits and good mechanical properties (Akdoğan and Vanli 2020; Kotik, 2019). In addition, the use of NLFs not only improves the mechanical strength of the composites, but also reduces the amount of oil derived polymeric material (ALSUBARI et al., 2021; Sanjay et al., 2018; Varanda et al., 2018).

Eucalyptus is a plant native to Australia, and is easily cultivated in several countries with temperate and tropical climates due to its adaptability. However, the use of the fibers from the trunk of this tree are relatively limited since it requires several processing steps to separate the fibers (Oliveira *et al.*, 2005). Therefore, the Eucalyptus fibers are most commonly used as wood. In this way, it can be said that eucalyptus “fiber” is one of the most cultivated FNLs in Brazil (Teixeira et al., 2020; De Oliveira et al., 2017).

Another important issue regarding the environment is waste management. The increase in the world population that has occurred in recent decades has caused a growth in the demand for goods, leading to a greater production of waste and consumption of non-renewable natural resources. Brazil alone, produced around

79 million tons of urban solid waste in 2018 (Souza, 2020), corresponding to an increase of 1.66% compared to 2017. Since the residues from eucalyptus wood processing are hydrocarbon-based materials, they usually are burnt for energy production (Teixeira et al., 2020).

The use of wood waste as reinforcement in composites represents a more ecologically correct solution to burning, because in addition to adding value to an industrial waste, it also corresponds to a carbon sequestration that will not be released into the atmosphere in the form of CO₂.

As an alternative to petroleum-derived polymeric resins, resins of plant origin represent a more sustainable solution as they come from a renewable source (Cifarelli et al., 2021). Regarding vegetable polyurethanes, when compared to those derived from petroleum, they present some application limitations. This is justified by its heterogeneous composition which may vary by geographic area, but also by the limited compatibility with conventional polyols and other ingredients in polyurethane formulations (Chakraborty et al., 2020; Marcovich et al., 2017).

Still within this context and as an alternative to conventional polymers, biocomposites have reached prominent positions, combining natural fibers with biodegradable polymers. An industrial example of application is the automotive companies that are recently focusing on different types of polymeric biocomposite materials to obtain a combination of good properties, in addition to lighter components, which directly interferes in the reduction of fuel consumption in automobiles (Abdel -Hamid et al., 2019; Fiore et al., 2018).

Thus, the objective of this work is to present the mechanical characterization of biocomposites produced with polyurethane matrix derived from castor oil reinforced with eucalyptus residues from the wood industry.

2. MATERIALS AND METHODS

In this work, eucalyptus chips obtained as waste from the timber industry in the region of Campos dos Goytacazes - RJ were used as reinforcement for the composites. Which in turn were separated from three different stages of wood processing, with size varying significantly at each stage. The first residue (RE1) consists of chips of relatively large length and thickness, while the second (RE2) consists of smaller and finer chips and the third (RE3) of a fine powder. All residues were washed (separately) in running water with the aid of suitable sieves and dried in an oven at 80 °C for 48 hours. The three types of tailings are of the same origin, only changing their size and morphological characteristics. Thus, the initial characterization of this material was carried out by determining the density of the residue by liquid pycnometry with distilled water and the characterization

of the microstructure of the surface of the fibers, carried out by means of Scanning Electron Microscopy.

In Figure 1 the residues used in this study are presented, it is noted that both the morphology and the size of the residues differ visually. The largest of the eucalyptus residues (RE1) has a length greater than its width and is obtained as tangled skeins. The second residue (RE2), of intermediate size, does not have chips that pass through a 4-mesh sieve (approximately 4.8 mm opening). Finally, the last residue (RE3) is a powder passed through 4 mesh sieves.

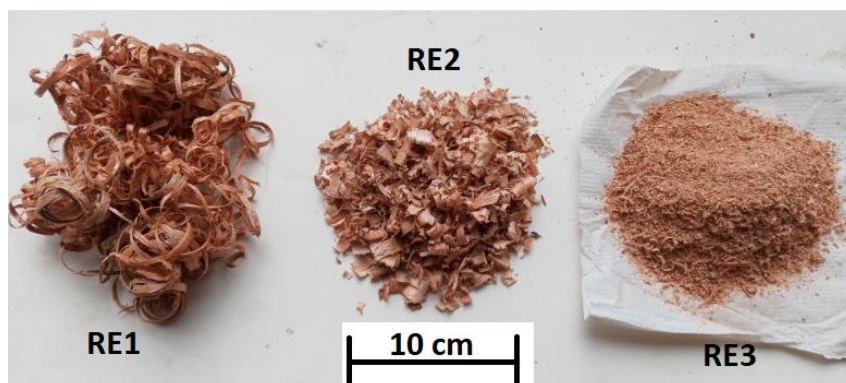


Figure 1: Eucalyptus wood waste.

For the production of the composite, bicomponent polyurethane resin derived from castor oil, produced by the company Imperveg Polímeros Indústria e Comércio Ltda located in the city of Aguaí - São Paulo, was used as a matrix. The resin polymerization reaction is obtained by mixing the prepolymer that contains isocyanate groups (component A) and a polyol derived from castor oil (component B), in the proportion of 120% by mass of B to A. It takes about five days to complete the resin after mixing components A and B, however the resin already has enough rigidity so that the specimen can be demolded after 24 hours.

The composites were made by inserting the already homogenized mixture of component A and B together with the type of residue inside a closed metallic mold and then subjected to a constant pressure, approximately 4 MPa, for a period of 24 hours. With the density value of the eucalyptus chips, it was possible to calculate the amount of eucalyptus mass for each volumetric fraction. The plates were produced in volumetric fractions of 10, 20, 30 and 40%, for the three types of waste.

Composite panels were produced for three types of bending, tensile and impact tests. For the bending test, the plates were produced in the dimensions of 150x128x7mm. After manufacturing, they were cut to dimensions of 150 x 15 mm and subjected to 3-point bending tests according to ASTM D790-17 in a universal machine. For the tensile test the plates were produced in the dimensions of 150x128x2 mm. Then they were cut to dimensions of 150 x 15 mm and subjected to tensile tests based on ASTM D3039 / D3039M-17 in a universal machine. The

bending and tensile tests were carried out in a universal machine of the brand Instron model 5582 present in the Laboratory of Advanced Materials of the Universidade Estadual do Norte Fluminense (LAMAV / UENF). For the impact test, the plates were produced in the dimensions of 150 x 128 x 10 mm. The specimens were cut in accordance with ASTM D256-10 for the Izod test and in accordance with ASTM D6110-18 for the Charpy impact test. The notch was performed with the aid of a manual carver with a depth of 2.54 mm and an angle of 45° and the test was performed using a PANTEC instrumented pendulum, Model XC-50 present in the Laboratory of Advanced Materials of the Universidade Estadual do Norte Fluminense (LAMAV / UENF).

3. RESULTS AND DISCUSSIONS

The eucalyptus fiber had its density determined, by the pycnometric method, as being $0.74 \pm 0.01 \text{ g/cm}^3$. The density of eucalyptus fibers varies dramatically with their moisture content, and factors such as local plant growth time (Teixeira et al., 2020; Soares et al., 2018) and the common method of determining this property is the calculation of wood density. Density was used to calculate the fiber masses to be included in the volumetric fraction calculations of the composites. Figure 2 shows the surface microstructure of eucalyptus fibers (RE1) where the main characteristics of this residue can be observed. This is actually composed of agglomerates of fibers in the form of pieces, or splinters, of wood.

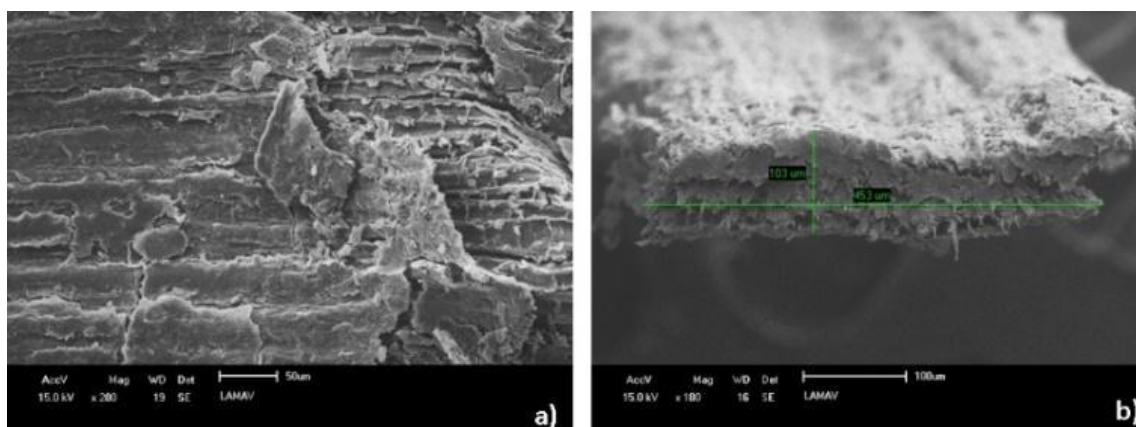


Figure 2: Cutting tool marks on the eucalyptus chips (a) and fibers joined at the tip of the chip (b).

As seen in Figure 3, RE1 is a sliver of wood where the marks of the cutting tool can be observed (figure 3 a) and the superimposed layers of eucalyptus fibers (figure 3 b). Residues RE2 and RE3 have a microstructure similar to RE1 where there is only variation in the dimensions of the chips, where RE2 is an intermediate residue between 1 and 3 where RE1 are the chips of the first cut of the wood, RE2 are chips removed from the surface finish of the eucalyptus wood and, finally, RE3 is a fine powder from the final cuts of wood.

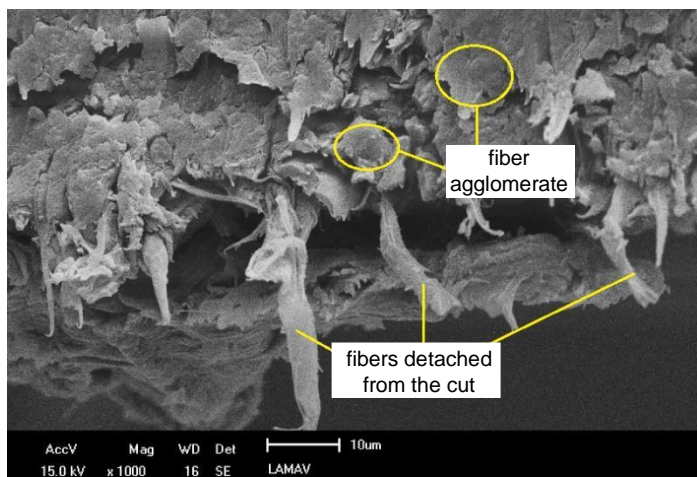


Figure 3: Scanning electron microscopy of the surface of RE1 showing clumps of fibers and some fibers highlighted in the section

Table 1 presents the result of the mechanical properties obtained in the tests carried out according to the type and volumetric fraction of the eucalyptus wood residue.

Table 1: Mechanical properties according to the type and volumetric fraction of the waste.

Type and Volumetric Fraction of waste (%)	Bending Strength (MPa)	Tensile Strength (MPa)	Notching Resistance (J/m)	Impact Resistance (KJ/m ²)	Notching Resistance (J/m)	Impact Resistance (KJ/m ²)	
							Izod
- 0%	12,10 ± 3,42	8,90 ± 1,26	314,6 ± 52,7	23,6 ± 3,6	184,1 ± 45,1	14,8 ± 3,3	
RE1	10%	7,51 ± 2,63	7,36 ± 2,15	141,0 ± 19,7	11,5 ± 1,7	71,2 ± 15,9	6,1 ± 1,2
	20%	23,03 ± 0,86	11,35 ± 0,83	190,3 ± 21,8	14,3 ± 1,5	140,8 ± 48,8	10,0 ± 3,0
	30%	36,11 ± 3,91	12,64 ± 0,41	113,4 ± 13,2	9,1 ± 1,3	42,6 ± 5,7	3,5 ± 0,4
	40%	54,80 ± 7,51	11,11 ± 1,68	126,4 ± 6,0	11,0 ± 1,1	31,9 ± 16,2	2,6 ± 1,3
RE2	10%	5,53 ± 0,54	8,34 ± 2,39	324,1 ± 101,1	23,4 ± 6,5	214,5 ± 64,8	17,4 ± 3,3
	20%	14,59 ± 1,68	4,81 ± 0,53	126,6 ± 11,0	10,4 ± 0,6	57,3 ± 9,8	5,0 ± 0,9
	30%	17,31 ± 2,38	4,03 ± 0,56	141,6 ± 19,9	10,9 ± 1,4	59,1 ± 6,8	4,5 ± 0,6
	40%	10,57 ± 1,46	6,25 ± 0,57	74,8 ± 5,2	5,8 ± 0,4	74,5 ± 10,3	5,7 ± 0,8
RE3	10%	3,98 ± 1,03	3,28 ± 0,75	188,6 ± 52,7	14,5 ± 4,0	162,1 ± 73,1	12,6 ± 5,8
	20%	5,78 ± 0,48	3,17 ± 0,74	69,6 ± 19,6	5,5 ± 1,5	54,8 ± 10,2	4,7 ± 0,8
	30%	7,64 ± 2,11	3,84 ± 0,81	97,3 ± 17,7	7,8 ± 1,5	129,8 ± 15,1	10,3 ± 1,3
	40%	8,42 ± 3,07	8,15 ± 0,94	77,0 ± 15,9	5,7 ± 0,9	50,8 ± 5,3	4,0 ± 0,5

Figure 4 presents the flexural strength curves for each residue as a function of the volumetric fraction inserted in the composite.

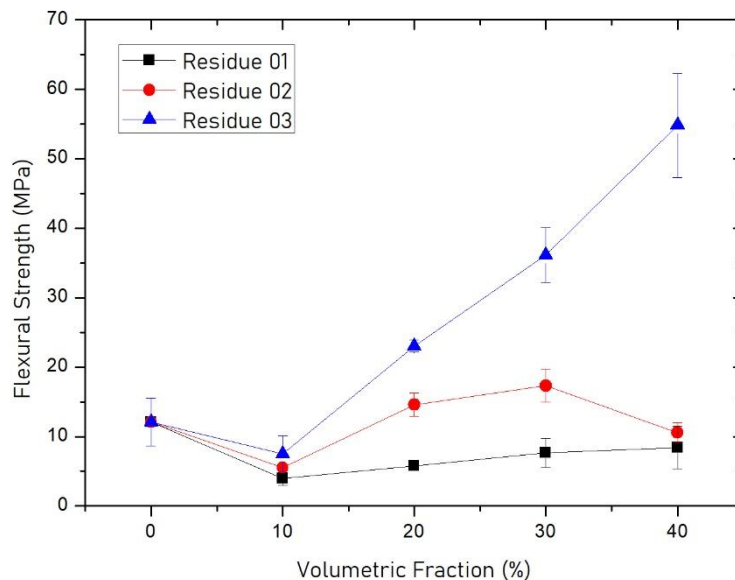


Figure 4: Flexural strength curves for composites according to the volumetric fraction of waste used.

When analyzing the data, it is possible to observe that for all types of eucalyptus wood residues, the 10% increase in volumetric fraction in the composite causes a decrease in flexural strength compared to pure polyurethane resin. In larger volumetric fractions, above 10%, the RE1 presented a higher performance among the three studied with an increase proportional to the amount of reinforcement. Compared with the pure resin, the composite reinforced with 40% RE1 showed an approximately sevenfold improvement in flexural strength. It is also observed that RE2 presents an increase in resistance to 20 and 30% followed by a decrease to 40% in volumetric fraction. Finally, RE3 shows a small tendency to increase strength, on average, with increasing powder, however, all values are below the strength of pure resin.

In the case of composites reinforced by 10% in volumetric fraction, the loss of performance can be justified due to a large number of bubbles in the polymer matrix. As the amount of reinforcement increases, the pressure exerted by the press on the die is more efficient while the resin is still liquid. In this way, the quantity and size of the bubbles in the composites decreases significantly, which justifies the increase in performance presented in residues 1 and 2, since the resin is placed in abundance in the mold and the pressure expels the excess out of the mold and keeping the amount of fiber constant inside, thus causing the expulsion of part of the bubbles. As RE3 is a very fine powder, the bubble removal process is not as efficient as in the case of RE1 and RE2, leading to a greater emergence of bubbles in any volumetric fraction. Figure 5 shows examples of the bubbles observed in the specimens, where with 10% of RE2 small bubbles are visible in large quantities along the specimen, with 20% of RE1 the bubbles are concentrated on one of the faces of the body. and at 30% RE1 the bubbles are no longer visible to the naked eye.

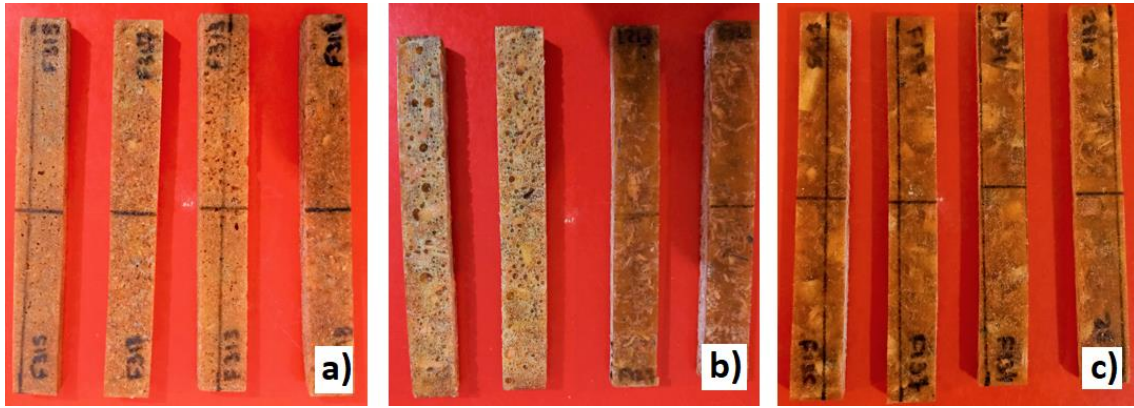


Figure 5: Composite reinforced by 10% RE3 (a), 20% RE2 (b) and 30% RE1 (c).

Figure 6 presents the curves of the results for the tensile strength of composites according to the volumetric fraction, together with the tensile strength value of the pure polyurethane resin (0% volumetric fraction).

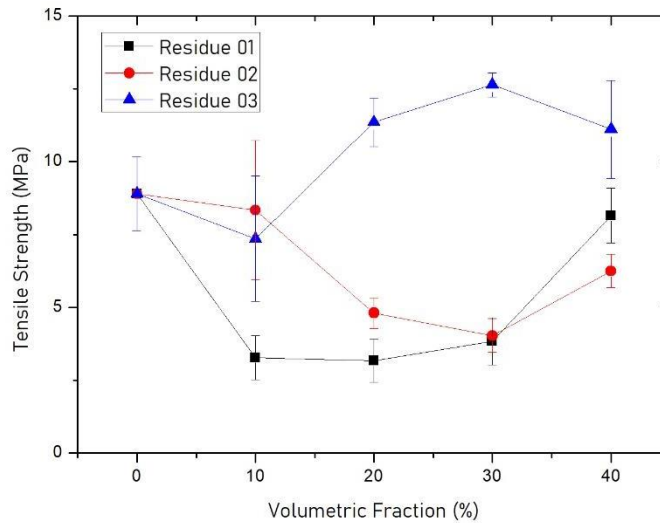


Figure 6: Tensile strength curves for polyurethane matrix composites reinforced by eucalyptus wood residues.

According to the data presented in Table 1 and plotted in Figure 4, in all cases, in addition to a decrease in tensile strength for a 10% reinforcement in volumetric fraction of waste, a large dispersion of results can be observed in relation to the average. Due to the method of making the specimens, for the cases of 10%vol, there is a large amount of bubbles along the specimen, which causes a loss of performance in addition to the decrease, on average, of the tensile strength. As the volumetric fraction of reinforcement is added, there is a decrease in the dispersion of the results in relation to the mean. The variation in the amount of bubbles can be seen in Figure 7 which shows the surface of two composites, one reinforced with 10 and 40% in volumetric fraction of RE2.

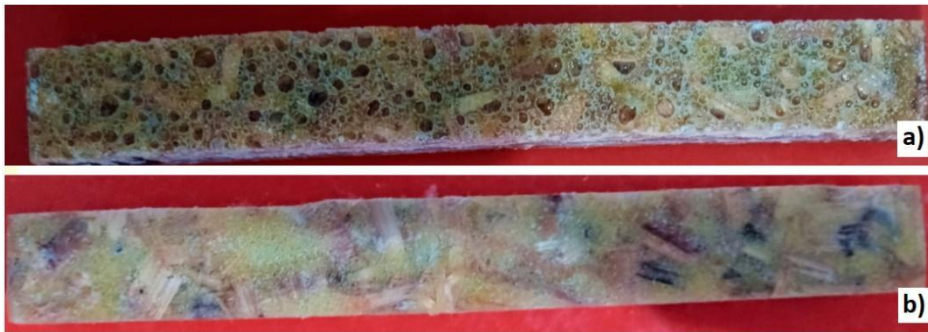


Figure 7: Composite reinforced by 10%vol (a) and 40%vol (b) of RE2.

In Figure 7, it is evident that, with the increase of the volumetric reinforcement fraction, there is a significant decrease in the amount of bubbles in the composites. In the case of composites reinforced by RE1, the increase in the volumetric fraction of residue in the matrix from 10% causes, in addition to the decrease in the amount of bubbles, an increase in tensile strength in relation to the pure resin.

The absence of bubbles for larger volumetric fractions is justified by the method of making the specimens. With the metal mold closed with the resin and reinforcement mixture inside, the pressure applied to the mold causes the excess resin to be expelled, along with the excess bubbles. Thus, a higher volumetric fraction of reinforcement causes, at first, a decrease in bubbles due to a greater efficiency of pressure transmission of the liquid resin on the surfaces of the reinforcement. The composites reinforced with 40% vol of RE1, however, present an inferior performance in relation to the composites reinforced with 20 and 30% in volumetric fraction in addition to an increase in the dispersion of the results. In this case, it was observed that the large amount of reinforcement used interferes in the bubble expulsion process and 30% in volumetric fraction is, therefore, the “optimal” limit of RE1 reinforcement to be added to the polyurethane matrix. Finally, residues RE2 and RE3 present unsatisfactory tensile strength performance, where they endure as a load in the matrix.

From the work carried out, it was possible to determine the value of the Energy absorbed by the composite samples during the impact test. The results were presented in Table 1 and in Figures 8 and 9 their curves for the Charpy and Izod impact tests are presented.

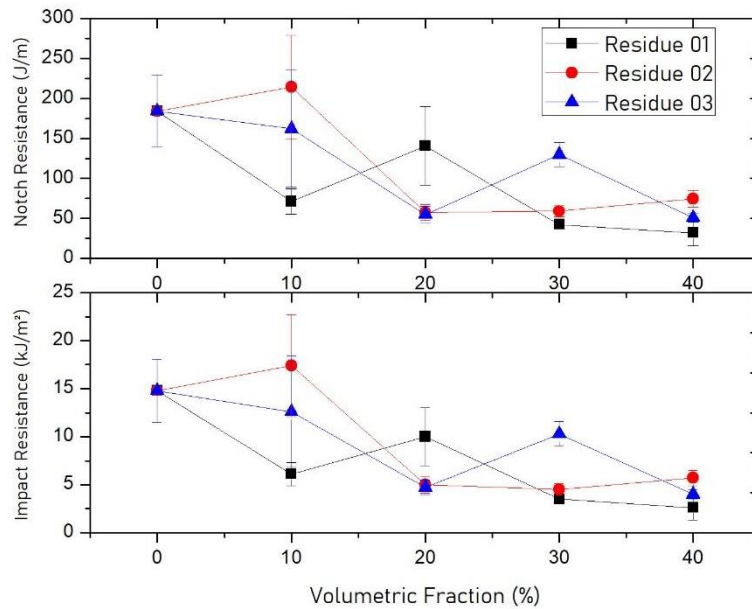


Figure 8. Notching strength and impact strength of samples analyzed by Charpy impact test.

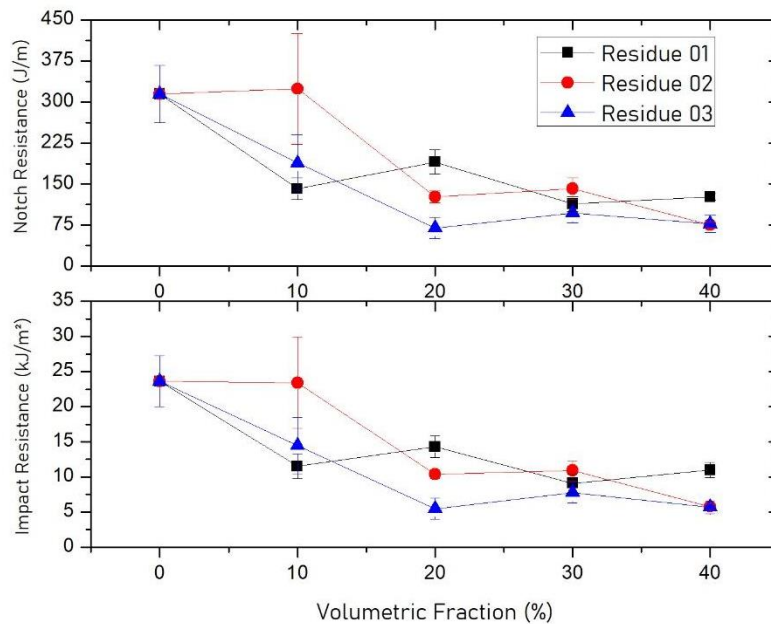


Figure 9. Notching strength and impact strength of samples analyzed by Izod impact test.

For the impact test by Charpy that for all volumetric fractions of eucalyptus wood residue cause a depreciation in their properties in relation to pure polyurethane, with the exception of composites of type RE2 and RE3 for volumetric fraction of 10%, where it can be considering the results equivalent to pure polyurethane.

In the impact test by the Izod method, it is possible to observe the same behavior, where the impact resistance decreases with the increase of the volumetric fraction of the residue. The composite with the volumetric fraction of 10% using

RE2, obtained approximately the same range of properties achieved by the pure polyurethane. This behavior was also observed in the study carried out by Mendes et al. (2020) where the increase in the volumetric fraction of eucalyptus fibers caused a decrease in impact resistance.

This result was expected, because with the addition of fibers in the polymer matrix, tension concentrating regions are created, and these regions need less energy to initiate the rupture process.

4. CONCLUSIONS

- Eucalyptus residues have great potential for use as reinforcement in polyurethane matrix composites. And that the size of the wood chips directly influences the flexural and tensile strength of the composite.
- The use of polyurethane resin derived from castor oil demonstrated technical feasibility, presenting itself as a bioecological alternative.
- For flexural strength, the insertion of 40% in volumetric fraction of RE1 showed a performance improvement of more than 700%, on average, when compared to pure resin.
- For the tensile strength of the composite, where the insertion of 30% in volumetric fraction of RE1 presented a performance improvement of more than 40%, on average, when compared to the pure resin.
- The presence of bubbles significantly decreases both in size and quantity with higher fractions of residue inserted as reinforcement.
- For the impact tests, there was a tendency to drop in strength as the volumetric fraction increased. This behavior corroborates the results presented for the tensile and flexural strength, where composites of lower strength have higher impact toughness.

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