

MECHANICAL BEHAVIOR OF CASTOR OIL-BASED POLYURETHANE COMPOSITES: REINFORCEMENT COMPARISON

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Abstract. *The use of natural fibers as reinforcement in polymeric composites for technical applications has been a research subject of scientists. Nowadays, there is a great interest in the application of sisal fiber as substitutes for glass fibers, motivated by potential advantages of weight saving, lower raw material price, and ecological advantages of using green resources which are renewable and biodegradable. Castor oil, a triglyceride vegetable that has hydroxyl groups, was reacted with 4,4' diphenylmethane diisocyanate (MDI) to produce the polyurethane matrix. As reinforcements woven sisal fiber and woven fiberglass were used in separate, and the composites were processed by compression molding. Sisal fibers were used untreated and thermal treated at 60°C for 72h. The present work study tensile and flexural behavior in three composites: dry sisal/polyurethane, humid sisal/polyurethane, glass/polyurethane resin. The moisture content influences of sisal fibers on the mechanical behaviors were analyzed. Experimental results showed a higher tensile strength for the glass/polyurethane composite followed by polyurethane/sisal (dry or humid) and a higher stiffness for the glass/polyurethane composite followed by dry sisal/polyurethane and humid sisal/polyurethane, respectively. In this research, composites were also characterized by scanning electron microscopy.*

Keywords: *sisal fiber; castor oil based polyurethane; polymeric composites, tensile strength, mechanical behavior*

1. INTRODUCTION

Natural fibers, classified as lignocellulosic materials, have been used as reinforcement material once present innumerable advantages, such as: low specific mass, low superficial consuming in equipment as extrusions, easy handle, biodegradability and renewable resource, insulator thermal, electric and acoustic, aesthetic aspects, non-toxic, beyond low cost (Saheb and Jog, 1999, Carvalho and Calli Jr, 2002, Silva, 2003, Mothé and Araújo, 2004).

In the automobile industry it is considered to have a great potential to be applied in the internal covering area of cars, bus and trucks; in the civil construction to reinforce cement; as textile material, characterizing a market in expansion (Mothé and Araújo, 2004). The raw material from renewed resources is of great interest due to the replacement possibility of fibers originated from petrochemical derivatives, which may contribute to the environmental control.

Brazil has a great production of sisal and export for the entire world, with a yearly production of 111.600 tons, in 2005. It is also responsible for 58% of the worldwide production (Andrade, 2009). The main producer's states are Bahia - 93.5%, Paraíba - 3.5% and Rio Grande do Norte - 3%, the semiarid region (APEX, 2006).

Sisal fibers are extracted from *Agave sisalana perrine* leaves, monocotyledon originated from Mexico. A large-scale production of sisal started from 1888 being propagated tropical and subtropical regions. There are now 57 species of sisal catalogued (Saheb and Jog, 1999, Jacob *et al.*, 2006a, 2006b). Each leaf of sisal provides, in weight, 4% of staple fibers, 0.75% cuticle, 8% dry matter and 87.25% water (Saheb and Jog, 1999, Mattoso *et al.*, 1997, Li *et al.*, 2000). The fibers are removed for mechanical process after the leaves are cut, because dry fibers adhere to the pulp (EMBRAPA, 2006).

The microstructure of the natural fibers are constituted from cellulose fiber reinforcing an amorphous matrix of the hemicellulose and lignin. These celluloses consist of microfibrils dispersed all along the length of the sisal fiber. The fiber characteristics depend on the properties of each constituent besides their source, age (Li *et al.*, 2000, Mishra *et al.*, 2004).

Natural fibers moisture content in general is placed between 5 to 20% and due to the hydrophilic characteristic cellulosic fiber. This characteristic affects the mechanical properties of the composites because the moisture can lead to poor processability and porous products during processing of composites Li *et al.*, 2000, Mishra *et al.*, 2004).

According Li, Mai and Ye (2000), to modify the fiber surface structure in order to enhance the bond strength between fiber and matrix and reduce water absorption of sisal fiber chemical and thermal treatment are used. A thermal treatment or hydrothermal treatment at temperatures about 120°C isn't appropriate to natural fibers since increases fiber brittleness and reduced strength. So a thermal treatment process higher than 120°C reduce moisture absorption but with deterioration of other fiber properties (Frollini *et al.*, 2004).

Studies of lignocellulosic materials as composite reinforcements are usually used with thermoset or thermoplastic matrices so that thermoplastic matrix composites are normally used in non-structural applications and thermoset matrices can be used in structural materials (Frolline *et al.*, 2004).

There are many reports about sisal fibers composites, Paiva and Frollini (2006) studied unmodified and modified surface sisal fibers by mercerization (alkali treatment) using NaOH 10%, esterification (succinic anhydride) and ionized air treatment on phenolic and lignophenolic matrixes. The study showed an improvement on fiber/matrix interfacial adhesion by mercerization and esterification when compared to ionized air treatment. Mwaikambo and Ansell (1999, 2002) and Rong *et al.* (2001) also observed the fiber surface topography and crystallographic index changes with mercerization treatment. However they observed yet that depending on the NaOH concentration a reduction of fiber thermal resistance is promote.

Bismarck *et al.* (2001) reported that the sisal fibers thermal stability is not affected by dewaxing treatment while the mercerization and methyl methacrylate grafting increases the maximum decomposition temperature by 10°C as compared to untreated fibers. Jacob *et al.* (2006b) investigated the influence of mercerization (NaOH 4%), silane and thermal (150°C for 8h) treatments on the tensile strength in woven sisal fabric reinforcing natural rubber composites. The study shows that the highest values are exhibited by thermally treated composites besides provide better adhesion and a stronger interface than other chemical modifications.

Castor oil is a vegetable triglyceride, not employed for feeding. The molecule is, characteristically, formed by hydroxyl groups and applied as a polyol in the synthesis of cross-linked polyurethane (Trân *et al.*, 1997). Figure 1 shows the representative composition of the oil. Viscous liquid castor oil is obtained from expressing or extracting with solvent from *Ricinus communis* plant seeds, which belong to the *Eurphorbiaceae* family (Vilar, 2006, Ogunniyi, 2006).

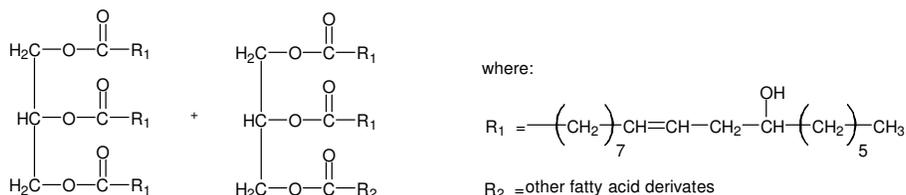


Figure 1. Castor oil triglyceride (Ogunniyi, 2006)

Castor plant grows in countries with tropical and subtropical climates that present average temperatures about 20-26°C and low air humidity. Main producers are India, China and Brazil, which were responsible for 92% of the worldwide production in 2001 (Biodiesel, 2006). The yearly worldwide production of seeds is about 1 million tons; consequently, the oil production remains around 500,000 tons (Ogunniyi, 2006, Santos and Barros, 2000).

There are different varieties of castor seeds, but in average it is possible to obtain 46 to 55wt% of oil (Ogunniyi, 2006). This oil is a natural resource which is considered to be closer to a pure compound, where 87-90% of the fatty acid presents in their constitution is the ricinoleic acid. About 10-13% are non-hydroxylated fatty acids which include linoleic and oleic acids (Trân *et al.*, 1997, Silvestre Filho, 2001). The average composition of the castor oil is indicated in Tab. 1. The use of this oil on the synthesis of polyurethane produces a polymer less aggressive to humans and environment (Fiorelli, 2002).

Table 1. Average composition of castor oil (Ogunniyi, 2006)

Components	Concentrations (%)
Ricinoleic acid	89.0
Linoleic acid	4.2
Oleic acid	3.0
Stearic acid	1.0
Palmitic acid	1.0
Dihydroxystearic acid	0.7
Linolenic acid	0.3
Eicosanoic acid	0.3

Silva (2003) studied the behavior of slabs from castor oil based polyurethane resin with coconut and sisal as plain weaves; unidirectional short fibers (10 mm of length) and unidirectional long fibers. Results show sisal fibers present the best results when compared to coconut fibers. The raise of fibers content fraction increase tensile strength, stiffness and water absorption but decrease the flexural strength.

Polymeric laminates of epoxy and castor oil based polyurethane resins reinforced with unidirectional sisal fibers were studied by Carvalho (2005) to reinforce timber structures. It was observed that the use of these composites is

technically viable to application as reinforcement. Mercerization treatment (NaOH 10%) on the sisal woven fabric for an hour reduces the composites variability on tensile behavior, however decreases stiffness and tensile strength.

Fiorelli (2002) analyzed the utilization of polymeric composites composed by epoxy, polyurethane or phenolic resins reinforced with glass or carbon unidirectional textiles when used to reinforce lower pieces of timber structures. He concluded that the increase of stiffness and flexural strength is proportional to the number of layer textile and the use of one carbon layer is equivalent to three fiberglass layers.

The study of sisal fiber and castor oil based polyurethane as raw material in the production of composites contribute to environmental preservation once these materials are derived from natural, biodegradable and renewable resources.

The objective of the present study is compares the tensile and flexural properties of natural composites with synthetic composites, manufactured by compressing molding. The main purpose of using a thermal treatment in the sisal fibers is to promote the fiber surface modification and improve fiber/matrix adhesion.

2. EXPERIMENTAL

2.1. Fibers

As reinforcement, sisal and glass fibers were used.

Woven sisal fabric was obtained from the Northeast region of Brazil, shown in Fig. 2, and received in form of plain weave (ASM International, 1993). The fabric presents two millimeters of thickness and Tab. 2 presents sisal physics characteristics.

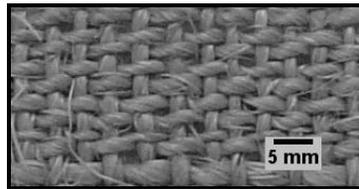


Figure 2. Plain weave sisal fabric

Table 2. Physics properties of woven sisal fabric

Average Yarn distance (weft), in mm	2.00
Average Yarn distance (warp), in mm	3.50
Diameter of fiber *, in μm	100 - 200

* data by SEM

Fibers in natural condition, denominated “humid”, and fibers thermally treated in the oven at 60°C for 72 hours, denominated “dry”, were used as reinforcement.

Determination of moisture regain (h) in the sisal fibers was calculated according to the ASTM D1348 standard (2003), method A, using the Eq. (1):

$$h (\%) = \frac{M - D}{D} \times 100 \quad (1)$$

Where h is the moisture regain, in percent; M is the original mass of specimen and D is the mass of oven-dry specimen at 105°C.

The bi-directional woven fabric of fiberglass was donated by Owens Corning. The fabric for manual lamination T366B is obtained by weaving continuous fiber 111A produced with Advantex glass and has weight mass of 350 \pm 60 g/m² (Owens-Corning, 2004).

2.2. Resin

The castor oil based polyurethane resin was used as matrix and is cured at room temperature. Kehl Industry Ltda from Brazil provides these reagents and has its patent. It is a bi-component formed by a polyol and a pre-polymer commercially designated as D045 and IC201, respectively and only the polyol components can be available as bio-based materials (Ajjithkumar *et al.*, 1998).

The polyol D045 is liquid at room temperature, presents yellow color and density in the range 1.0-1.2 g/cm³, according to the manufacturer. It is soluble in water and produces biodegradable residue (KEHL, 2006).

The pre-polymer IC201 is composed by 4,4'-methylenediphenyl diisocyanate (MDI), which structure is displayed in Fig. 3. It is liquid at room temperature, with dark brown color and a density at 1.24 g/cm³, as informed by the manufacturer. This reagent is insoluble in water; however, it reacts producing CO₂ and an inert and not biodegradable residue (KEHL, 2006).

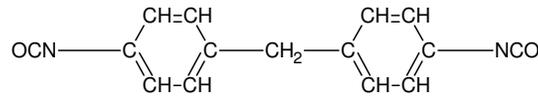


Figure 3. 4,4' Methylenediphenyl diisocyanate

2.3. Composites

Woven sisal fabric composites with 33-44 wt% of reinforcement and fiberglass composites with 31-35 wt% of reinforcement were prepared by compress molding at room temperature.

The components of resins were manually mixed and shed upon fibers. Laminate thickness is the equivalent of a fabric layer and samples were removed from the mold after 4-8 days processed.

The fibers were arranged between flat plates of glass pressed after extraction of the bubbles with a metal roller. Plastic sheets were used as release agent to polyurethane laminates.

2.4. Tensile test

Tensile specimens of composites were prepared in agreement to the ASTM D3039 (2008) and resin specimens were prepared according to ASTM D638 (2008).

Tensile specimens of polyurethane (PU) resin, PU/sisal fibers and PU/glass composites were tested at a rate of 10 mm/min, 5 mm/min and 5 mm/min, respectively. Minimum of five specimens per test condition were tested.

Samples fracture surface used in the tensile tests of PU resin and laminates were analyzed by scanning electron microscopy (SEM).

Elongation was calculated by the following Eq. (2) for \mathcal{E} :

$$\mathcal{E} (\%) = \frac{\Delta L}{L_0} \cdot 100 = \frac{L - L_0}{L_0} \cdot 100 \quad (2)$$

Where \mathcal{E} is the elongation (%), L is the final length and L_0 is the original length.

2.5. Flexural test

Flexural tests of resins and composites were performed using a universal machine, INSTRON, model 4301 and samples were carried out at room temperature. Three point bending tests were performed according to the ASTM D790 standard (2003), at a load-cell of 500 Kgf. Minimum of six specimens per test condition was tested.

Fiberglass laminates used a crosshead speed of 1.5 mm/min, while PU resin and sisal fibers laminates used a crosshead speed of 2.0 mm/min.

The ratio used between the supports distance and the thickness of samples in the test was 16. Flexural stresses tested are calculated following Eq. (3):

$$\sigma_f = \frac{3 \cdot P \cdot L}{2 \cdot b \cdot d^2} \quad (3)$$

Where σ_f is the stress in the outer fibers at midpoint (MPa), P is the load (N), L is the support span (mm), b is the width of beam tested (mm) and d is the depth of beam (mm).

3. RESULTS AND DISCUSSION

3.1. Tensile test

Table 3 shows on the tensile strength and its respective elongation for polyurethane resin and laminates specimens tested. The polyurethane laminates specimens were prepared with humid sisal and dry sisal, treated for 72 hours at 60°C in the oven. Humid sisal presents a moisture regain about 17.3% while dry sisal presented a moisture regain about 4.1%.

Table 3. Tensile data of resin and laminates

Material Properties	Resin	Laminates		
	PU	PU/humid sisal <i>h</i> =17.3%	PU/dry sisal <i>h</i> =4.1%	PU/glass
Tensile strength at yield (MPa)	2.5	17.0	16.9	95.4
Standard deviation	0.4	2.0	1.0	7.4
Coefficient of variation (%)	17.8	11.9	5.86	7.80
Elongation at yield (%)	29.4	8.0	6.2	2.0
Standard deviation	2.2	0.4	0.3	0.22
Coefficient of variation (%)	7.62	4.82	5.52	10.9
Elongation at break (%)	31.2	11.3	8.0	2.0
Standard deviation	3.9	2.2	1.2	0.21
Coefficient of variation (%)	12.4	19.3	15.2	10.6

Tensile strength of polyurethane (PU) resin is 2.5 MPa and its elongation at maximum load and break are 29.4% and 31.2%, respectively. Results show low precision on the tensile strength, which is observed by the high coefficient of variation produced. The PU resin presents high elongation until the rupture showing to be a ductile-fragile material.

Low tensile strength values are consequence a lot of voids that are formed during the cure process, Fig. 4(a). These voids act as stress concentration and are responsible to the propagation of cracks during tensile tests, Fig. 4(b).

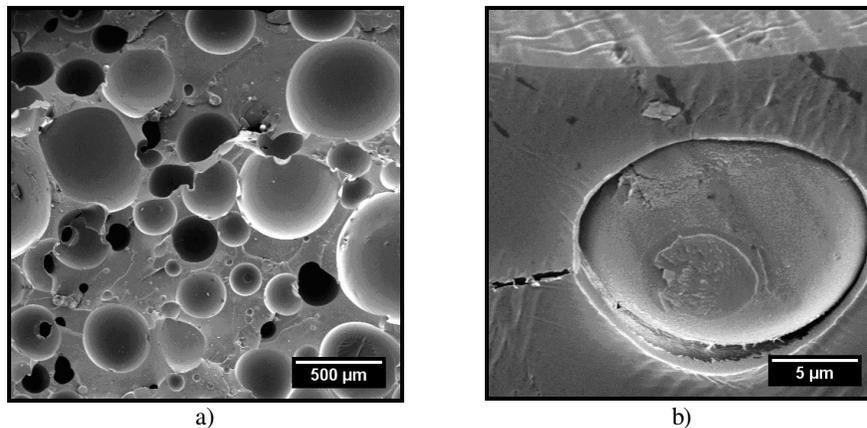


Figure 4. SEM of PU fracture surface: a) Presence of voids (50x),
b) Presence of insoluble substance (5000x)

Voids presence shows a great influence on the tensile strength, which can be confirmed by the decreasing in this property according to the increase in the voids content. However, the influence on the elongation is not significant to the data indicated in Tab. 3. Polyurethane resin fracture surface can be visualized in the Fig. 5. The presence of river marks that indicate of the crack propagation direction and scarps that represent the plane deviation were observed.

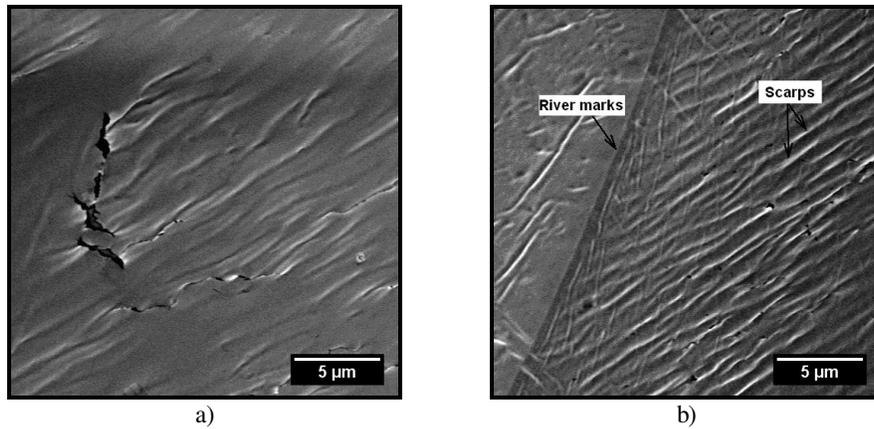


Figure 5. SEM of PU fracture surface (5000x)

Tensile strength of PU/humid sisal laminate is 17.0 MPa and its elongation at maximum load and break are 8.0% and 11.3%, respectively. PU/humid sisal laminate presents elongation at break higher than 11%, characteristic of ductile material. Tensile strength of PU/humid sisal laminate shows an increase about 7 times with the addition of humid sisal fibers when compared to the PU resin.

Figure 6 shows the fracture surface to the PU/humid sisal laminate. In Figures 6(a) and 6(b) the presence of voids in the resin that were formed during polymerization process is effective. Pull-out fibers of laminate is observed in the Fig. 6(b). Regarding fiber surfaces, Figs. 6a and 6c, was not observed the presence of matrix adhered to the fibers, showing the low adhesion between fiber/matrix. Fibers broken ends of sisal can be seen in the Fig. 7.

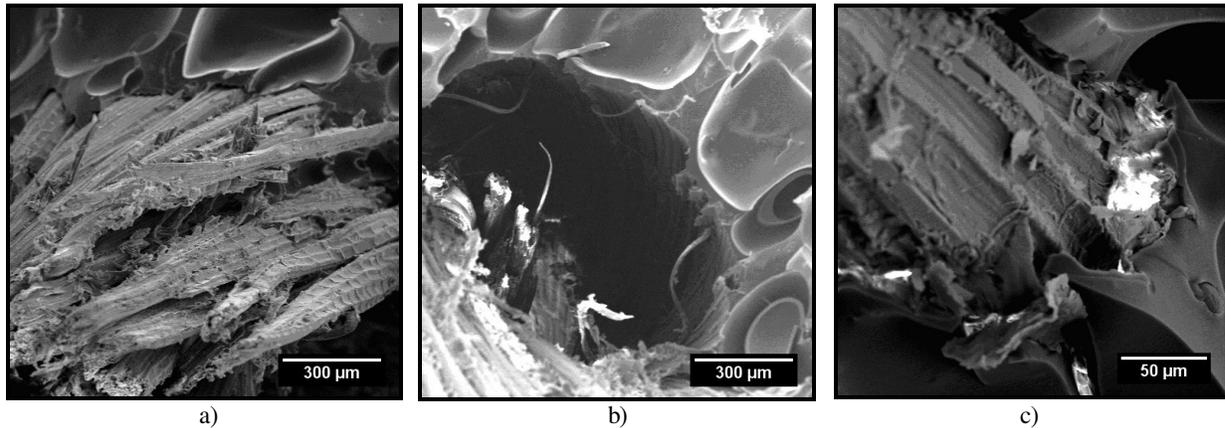


Figure 6. SEM of fracture surface on the PU/humid sisal laminate: a) absence of matrix on the sisal fibers (100x), b) pull-out and voids on the resin (100x) and c) adhesion fiber/matrix (500x)

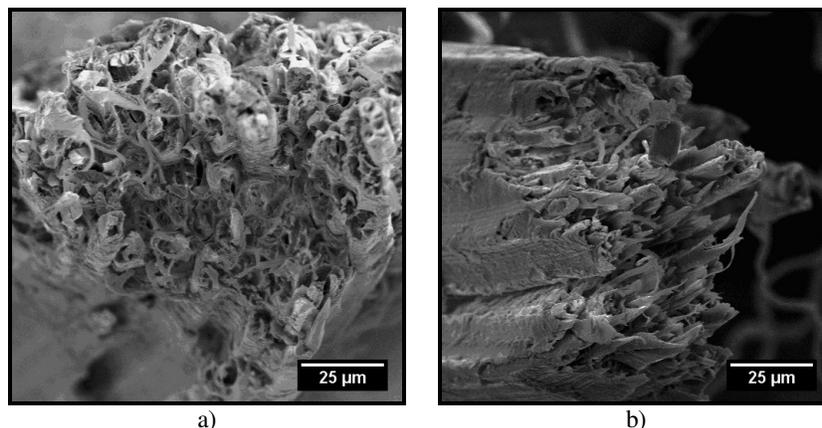


Figure 7. SEM of sisal fibers broken ends on the PU/humid sisal laminate (1000x)

Tensile strength of PU/dry sisal laminate is 16.9 MPa and its elongation at maximum load and break are 6.2% and 8.0%, respectively. PU/dry sisal laminate presents elongation at break higher than 8%, characteristic of ductile material. Tensile strength value of PU/dry sisal laminate shows an increase about 7 times when compared to the PU resin, which can be associated to the reinforcement by dry sisal fiber exposed at 60°C to 72 h. Figure 8 shows the fracture surface to the PU/dry sisal laminate.

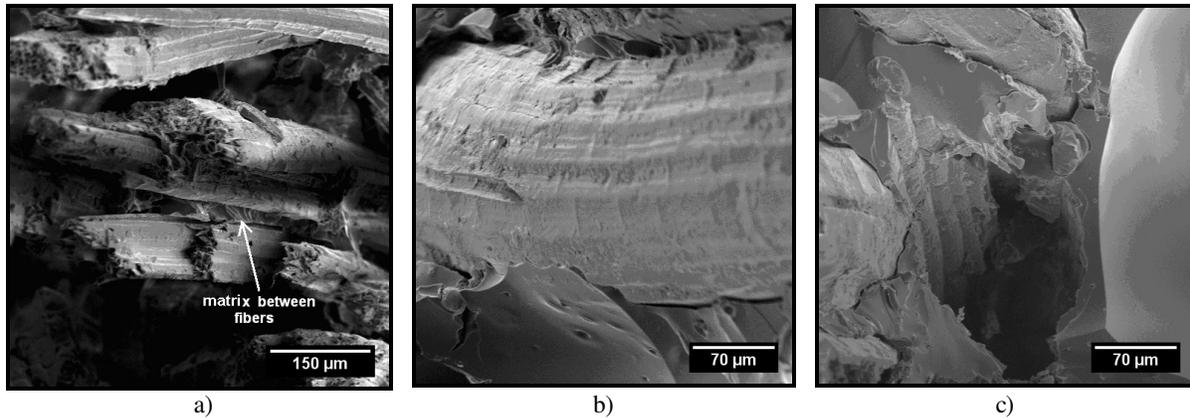


Figure 8. SEM of fracture surface on the PU/dry sisal laminate: a) presence of matrix between fibers (200x), b) adhesion fiber/matrix (350x), c) pull-out (350x).

It is important to visualize on Figs. 8(a) and 8(b) the presence of matrix adhered on fibers surface, phenomena attributed to the fibers drying process. Due to this fact, it was concluded that the absence of water in the fibers collaborates to sisal fiber polyurethane matrix interface.

The tensile strength of PU/humid sisal and PU/dry sisal laminates, Tab. 3, present similar values. Moisture decreasing proves, in consequence, a reduction in the elongation, which was not considered suitable to application as PU/sisal composite in structures, otherwise, can be observed a reduction in the standard deviation for tensile strength, favorable factor to this material.

In the Fig. 9, fibers broken ends of dry sisal can be seen with a size amplification of 750 times. A combination of interlaminar and intralaminar fractures and large amount of resin adhered on to the fibers surface can be observed in both SEM pictures, indicating high fiber/matrix adhesion.

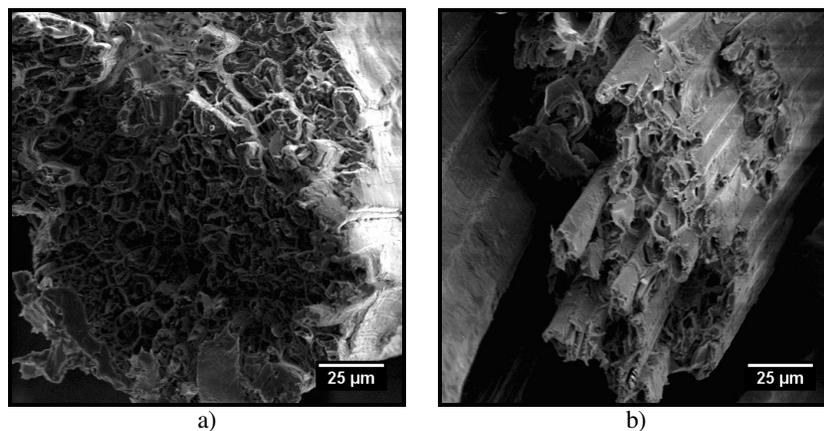


Figure 9. SEM of sisal fibers broken ends on the PU/dry sisal laminate (750x)

Tensile strength of PU/glass laminate is 95.4 MPa and the elongation at maximum load and break, both are 2.0%.

PU/glass laminate has low elongation at break, according Tab. 3, characteristic of fragile material. Tensile strength value of PU/glass laminate shows an increase about 38 times when compared to the PU resin, with the addition of fiberglass. The fracture surface of respective laminate can be visualized in Fig. 10.

Figures 10(a) and 10(b) presents a lot of voids on the resin that are formed during the resin polymerization, while the Fig. 10(c) shows the presence of pull-out.

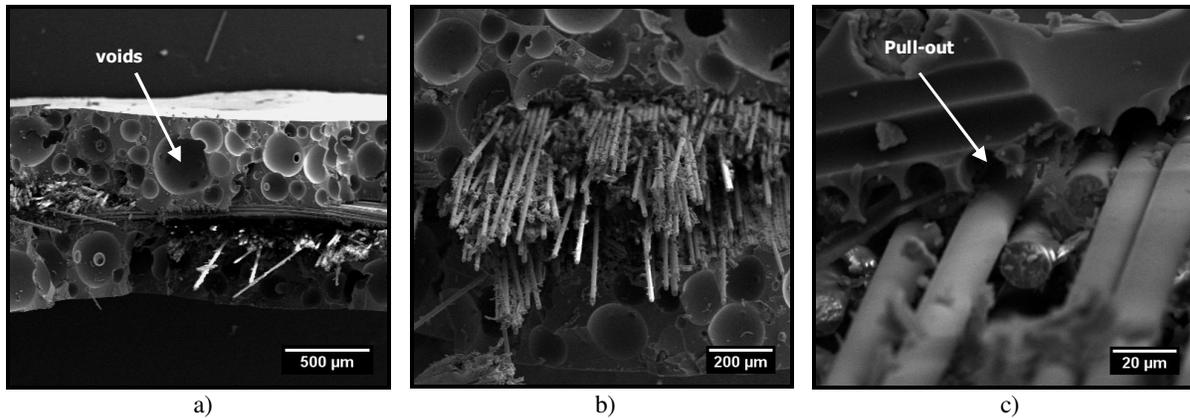


Figure 10. SEM of fracture surface on the PU/glass: a) voids on the resin (50x), b) adhesion between fiber/matrix (100x), c) pull-out and fiber fracture (1000x)

The fracture surface of fiberglass can be seen on the Fig. 11. Observing fiber surface, Fig. 11(a), it is clear the low adhesion between fiber/matrix which can be confirmed by Fig. 11(b) that shows the limited contact between resin and fibers. Figure 11(c) shows the fracture surface on the fiberglass with the indication of crack initiation and the direction of crack propagation.

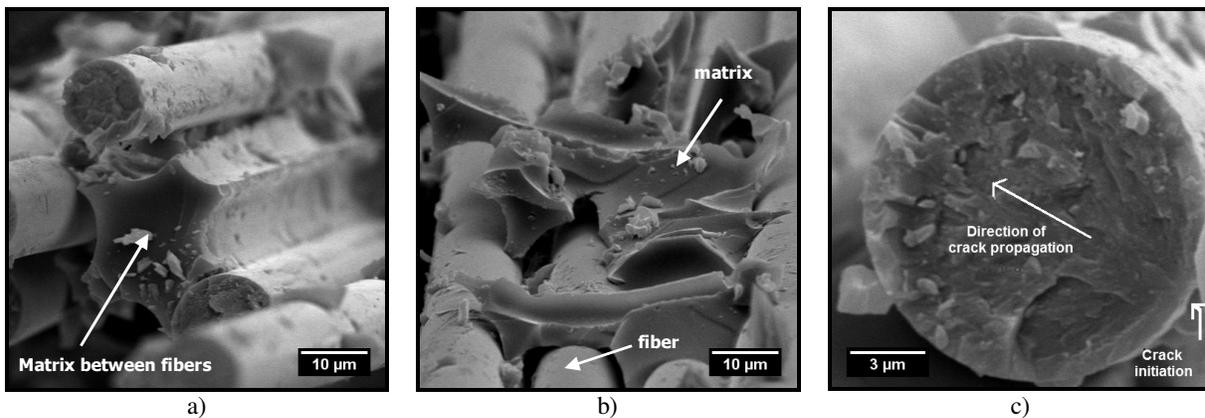


Figure 11. SEM of fracture surface on the PU/glass laminate: a) adhesion between fibers (2000x), b) adhesion fiber/matrix (2000x), c) fiber fracture surface (7500x)

Comparing the sisal fibers reinforcement with fiberglass on the PU laminates, it is clear that the synthetic fiber has the biggest tensile strength, and consequently, the PU/glass laminate. Tensile strength of PU/glass laminate presents a value about 5.6 times of the PU/sisal laminate.

3.2. Flexural test

Table 4 shows flexural data of all tested materials: polyurethane (PU) resin, PU/humid sisal ($h=17.1\%$), PU/dry sisal ($h=6.0\%$) and PU/glass laminates.

Table 4. Flexural data of resin and laminates

Material \ Properties	Resin	Laminates		
	PU	PU/humid sisal $h=17.1\%$	PU/dry sisal $h=6.0\%$	PU/glass
Flexural strength (MPa)	0.45	1.6	3.7	14.2
Standard deviation	0.05	0.16	0.34	2.12
Coefficient of variation (%)	11.1	10.3	9.26	14.9
Deflection (mm)	18.4	17.2	16.8	7.3
Standard deviation	0.51	0.78	0.46	0.26
Coefficient of variation (%)	2.78	4.54	2.71	3.62

Flexural strength of PU resin is 0.45 MPa with a deflection at 18.4 mm. PU resin shows low flexural strength and high extension without break, which can be considered a ductile-fragile material that confirms with tensile test.

Flexural stress of PU/humid sisal laminate is 1.60 MPa and its deflection is 17.2 mm, while flexural stress of PU/dry sisal laminate is 3.7 MPa and its deflection is 16.8 mm. It was observed that the PU/humid sisal and PU/dry sisal specimens do not break at strains.

Flexural strength of PU/humid sisal laminates, with an addition of one layer, introduces an increase about 252% when compared to the PU matrix while flexural strength presented of PU/dry sisal laminates, with an addition of one layer, introduces an increase about 7 times (722%).

Flexural strength of PU/dry sisal, when compared to the flexural strength of PU/humid sisal was about twice bigger to the dry reinforcement. Regarding to the deflection of dry and humid fibers laminates it was not found significant difference between values that were about 17 mm.

Differences were found between the laminates with moisture reduction of fiber, such as increasing the standard deviation of flexural strength, unfavorable factors to use this material in civil structures. However, there were reductions in the coefficient of variation of flexural strength and displacement.

Based on the experimental results, it follows that the absence of moisture in the sisal favors the adhesion between fiber and polyurethane, was indicated heat treatment to fibers when laminate is used to reinforce structures, as timber structures.

The flexural stress of PU/glass laminate is 14.2 MPa and its deflection is 7.3 mm. Its specimens do not break at strains. Flexural strength of PU/glass laminates, with an addition of one layer, introduces an increase about 30 times when compared to the PU matrix.

Flexural strength of PU matrix was used as reference to the analysis of results. Flexure strength values of laminates were higher than values founded of respective matrix, in order that, the addition of reinforcement promoted the increase in flexural strength, different from presented to Silva (3).

As show that presented in Tab. 4, the best performance in flexure was the laminate PU/glass, followed by PU/dry sisal and PU/humid sisal, respectively.

When comparing the sisal fibers reinforcement with fiberglass on the PU laminates, flexural strength of PU/glass laminate presents a value about 3.8 times of the PU/dry sisal laminate. The value difference can be considered smaller and favor the use of sisal fiber as reinforcement.

4. CONCLUSIONS

Tensile strength at yield of polyurethane based on castor oil is 2.5 MPa with a correspondent elongation at 29%, which characterize a ductile material.

Thermal treatment on the PU/sisal laminates it was not suitable to application of this material as structural reinforcement when is considering its tensile behavior. Otherwise, thermal treatment was good to application of this laminate when used in flexural mode.

PU/glass fiber laminate presents the biggest tensile and flexural strength followed by PU/dry sisal and PU/humid sisal laminates, respectively.

Comparing the sisal fibers reinforcement with fiberglass on the PU laminates, flexural and tensile strength of PU/glass laminate presents a value about 3.8 and 5.6 times of the PU/dry sisal laminate, respectively. So, the use of laminates to reinforce structures is viable once these laminates present considerable strength to his application.

5. ACKNOWLEDGEMENTS

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