



Comparative study of mechanical properties of polymeric composite materials with polyester matrix using natural and synthetic fibers as reinforcement

Raimundo Nonato Alves da Silva¹, Lucas Carvalho Cruz², Waldeir S. Dias³, Bruno Mello de Freitas⁴, Solenise Pinto R. Kimura⁵, Guilherme S. Moreira⁶, José Costa de Macedo Neto⁷, Vanderléia Castro Bezerra⁸, Ronaldo da Silva Cruz⁹

Resumo

Estudo comparativo das propriedades mecânicas de materiais compósitos poliméricos com matriz de poliéster utilizando fibras naturais e sintéticas como reforço. O uso de fibras naturais como reforço em matrizes poliméricas se justifica na questão socioambiental, com o aproveitamento integral da fibra, como incentivo à produção vegetal na região, na geração de emprego e renda para a população local e no investimento de novos materiais naturais da região amazônica para uso comercial e industrial. O objetivo é o estudo de um material compósito com matriz de poliéster utilizando fibras naturais e sintéticas (contínuas) como reforço. Juta e fibra de vidro foram utilizadas e para a moldagem foi realizado o método da gravidade. A taxa de armadura foi determinada pela densidade com aproximadamente 5% de fibra, tendo em conta os parâmetros de fabricação de materiais compósitos em geral. O teste mecânico de tração dos corpos de prova seguiu a norma ASTM D638-01 numa máquina universal de testes INSTRON. Os resultados mostraram que o compósito híbrido (juta / fibra de vidro) comparado ao não reforçado obteve um potencial de 36% e 45% a mais em relação ao teste de tração e alongamento médio, respectivamente, referente ao comportamento entre a armadura e a matriz, analisado através do Microscópio Eletrônico de Varredura (MEV).

Palavras-Chave: Compósitos híbridos, fibra natural, fibra de vidro, fibra de juta, fibras amazônicas.

Abstract

The use of natural fibers as reinforcement in polymeric matrices is justified on the socio-environmental issue, with the full use of fiber, as an incentive to the vegetal production in the region, in the creation of employment and income for the local population and in the investment of new natural materials from the Amazon region for commercial and industrial use. The goal of this project is the study of a polyester matrix composite material using natural and synthetic (continuous) fibers as reinforcement. Jute and fiberglass were used and for the molding the gravity method was performed. The reinforcement ratio was determined by density with approximately 5% of fiber, taking into account the manufacturing parameters for composite materials in general. The mechanical test of traction of the specimens followed the standard ASTM D638-01 on an INSTRON universal testing machine. The results showed that the hybrid composite (jute / fiberglass) compared to non-reinforced obtained a potential of 36% and 45% more relating to tensile stress and average elongation respectively, referring to behavior between the reinforcement and the matrix, analyzed through the Scanning Electron Microscope (SEM) techniques.

Key-words: Hybrid composites, natural fiber, fiberglass, jute fiber, Amazon fibers.

¹ Professor Assistente, Dept. Engenharia de Materiais, EST/UEA, Manaus, Amazonas, Brasil; rmasilva@uea.edu.br

² Bolsista, Dept. de Engenharia Materiais, EST/UEA, Manaus, Amazonas, Brasil; lucascruz99@gmail.com

³ Bolsista, Dept. de Engenharia Materiais, EST/UEA, Manaus, Amazonas, Brasil; wsd.mec@uea.edu.br

⁴ Professor Assistente, Dept. de Engenharia de Materiais, EST/UEA, Manaus, Amazonas, Brasil; bfreitas@uea.edu.br

⁵ Profa Adjunta, Dept. Engenharia de Materiais, EST/UEA, Manaus, Amazonas, Brasil; solenisekimura@yahoo.com.br

⁶ Bolsista, Dept. de Engenharia de Materiais, EST/UEA, Manaus, Amazonas, Brasil; gsm.mec@uea.edu.br

⁷ Professor Adjunto, Dept. Engenharia de Materiais, EST/UEA, Manaus, Amazonas, Brasil; jotacostaneto@gmail.com

⁸ Engenheira Ambiental, CETAM, Manaus, Amazonas, Brasil; vanderleiacb3@hotmail.com

⁹ Professor Assistente, UFRR, Boa Vista, Roraima, Brasil; rscfisio@gmail.com

1. Introduction

The composite materials are composed of two or more types of materials, in which the compatibility between their constituent phases, that is, matrix and reinforcement, is taken into account. The composite materials give designers considerable flexibility in the design of structures, as it allows the development of materials for specific applications (FREIRE J, 2005). The interest in natural fiber-reinforced polymer composite materials is rapidly growing both in terms of their industrial applications and fundamental research. They are renewable, cheap, completely or partially recyclable, and biodegradable (PUJARI; RAMAKRISHNA; SURESH KUMAR, 2014). The polymeric matrix composite materials can be manufactured by joining thermoset or thermoplastic matrices with various types of reinforcements, for example: fiberglass, carbon fibers, boron fibers or even vegetal fibers (TINÔ, 2010). Meanwhile, several natural fibers are being used in the polymeric matrix production process in order to obtain better mechanical characteristics in the final product. Research has also been carried out to evaluate the properties of polymeric composites reinforced with vegetal fibers. The results obtained so far are quite encouraging, since these fibers present mechanical resistance (GUPTA; SRIVASTAVA, 2016), comparable to fibers that have been studied for a long time (BLEDZKI; GASSAN, 1999). In addition, composites reinforced with 20% in weight by natural fibers shredded in polyurethane and polypropylene matrix show a substantial increase in their rigidity (MOTHÉ; ARAUJO, 2004). More recently, it has been shown that the microstructure of each fiber consists of a filament beam and may help in the adhesion of polymeric resin (MONTEIRO et al., 2006). Researchers often improve the mechanical properties through changing the microstructure (ELANCHEZHIAN et al., 2018). The fiber/matrix compatibility in relation to the results should indicate that all the

treatments performed could increase the compatibility between the fiber and the matrix, intensifying the interactions in the interfacial region. This higher fiber-matrix adhesion should be the main responsible for the better performance of fiber-containing composites. The alkaline treatment (NaOH) promotes wettability and better fiber-matrix adhesion, allowing efficient transfer of tension between the matrix and the fibers, removes the impurities and promotes defibrillation, increasing the effective surface area, consequently, provides better fiber-matrix adhesion and an increase in mechanical properties (KALIA; KAITH; KAUR, 2009; ROSA et al., 2009).

2. Materials and methods

2.1 Materials

Jute fiber (*Corchorus capsularis*) from the city of Manacapuru / AM was supplied by Brasjuta da Amazônia S/A after maceration process. Pool Poste de Fibra Company made available the unsaturated orthophthalic polyester resin, continuous fiberglass and methyl ethyl ketone peroxide reaction initiator supplied respectively by Reichhold do Brasil, Jushi Group Co. and Polinox do Brasil.

2.2 Organophilic Treatment

The organophilic treatment was used to obtain efficient fiber/matrix adhesion, which consists of the mercerization process (NaOH) and alkaline peroxide (H_2O_2) (ROSA et al., 2009). In Figure 1, we show the initial scheme for the organophilic treatment in the natural fiber. The fibers were cut into lengths of 6mm and 10mm. bleaching (alkaline peroxide treatment). That is, an alkaline NaOH treatment was performed in 20 g of fiber to 40 ml, and the material was stirred at 90 °C for one and a half hours, then vacuum filtration was performed, and washing in distilled water until pH has reached neutrality and oven drying at 50 °C. In Figure 2, it shows the natural fiber *in natura* and after the chemical treatment.

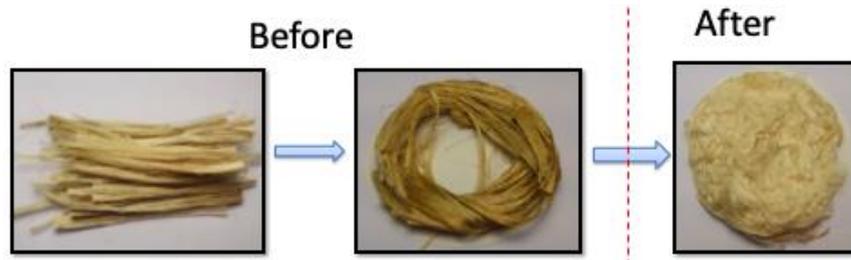


Figura 1. Natural fiber jute in mercerization process (NaOH) and alkaline peroxide (H_2O_2)

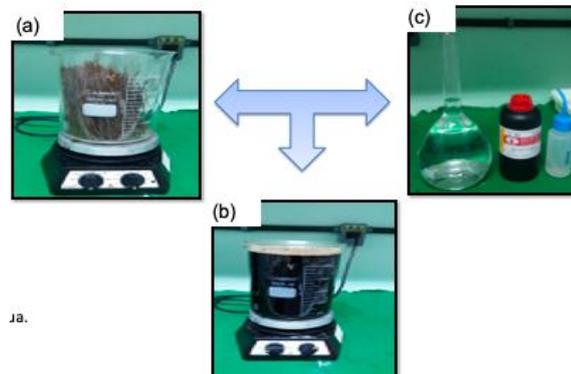


Figura 2: (a) Natural fiber, (b) Fiber in alkaline peroxide solution and (c) Chemical product

The jute fiber was bleached with the NaOH + H₂O₂ solution. Treatment with alkaline peroxide (H₂O₂), was due to the need to leave the organophilic fiber to improve the adhesion to the matrix. 10g of fiber was added to the hydrogen peroxide solution, then the sample was stirred at 55°C for one and a half hours, then cooled to room temperature and vacuum filtered, followed by washing with distilled water to neutral pH, finally oven drying at 50 °C until a constant mass is obtained (weighing every 10 minutes).

2.3 Sample Preparation

A mold made of standard ASTM 638-01 with 4 holes was used to perform the conformation of the specimens by the gravity method. Twelve (12) samples were made using the tripled method, three (3) samples for each compositional variation: non-reinforced, jute, fiberglass and hybrid (jute/fiberglass, 50%/50%).

The proportion of 5% of reinforcement per density was determined for all samples using a profile projector because of the non-regular surface of the jute fiber. In order to obtain dimensional characterization, (BARBOSA, 2011) the diameter was randomly measured at 3 (three) points along the length of 30 fibrils, at each position a second measurement was obtained by rotating each fibril 90°. Statistical analysis was performed in order to obtain the value of the reinforcement ratio through density, by the Equation (1):

$$\rho = \frac{P}{V} \quad (1)$$

Being the density “ ρ ” (g/cm³), “P” is the mass of the specimen (g) and “V” is the volume of the specimen. The obtained samples were submitted to the mechanical tensile test in an Instron machine with load cell of 5 kN and test speed of 1 mm/min. After the mechanical tests, the characterization of the fracture in the samples with reinforcement of jute fiber and hybrid was made in a scanning electron microscope (SEM). Figure 3 shows the mold with the dimensions of ASTM D636 (I).

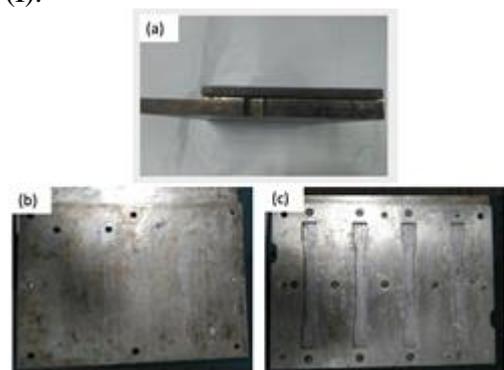


Figura 3: Mold parts for composites (a) closed mold; (b) Top side; (c) Bottom side. According to ASTM D636 (I) rules.

The mold was constructed of SAE 1045 steel and positioned in a hydraulic press. This kind of mold allows the appliance of pressure directly to the composite eliminating the formation of bubbles contributing in the curing process of the resin. It also allowed to position the fibers well directed and fixed in such a way that they maintain the position without movement during the curing process of the resin. No mold release was used for this product as the mold enables easy removal.

2.3 FTIR spectrum analysis

The major functional groups presented in the natural fiber of jute were identified by using FTIR, as shown in Figure 4. The peaks observed at 3400 cm^{-1} corresponded to O–H stretching and O–H bending frequencies due to cellulose and water. The peak observed at 2920 cm^{-1} could be assigned to C–H from aliphatic group methyl and

methylene. The absorption peak observed at 1649 cm^{-1} corresponded to carbonyl group ($\text{C}=\text{O}$) stretching vibration of the alpha keto carboxylic acid in lignin and 1729 cm^{-1} in hemicellulose. The broad and strong absorption peak observed at 1655 cm^{-1} was attributed to $\text{C}=\text{O}$ stretching vibration of the acetyl group ($\text{CH}_3\text{C}=\text{O}$) present in lignin and hemicelluloses.

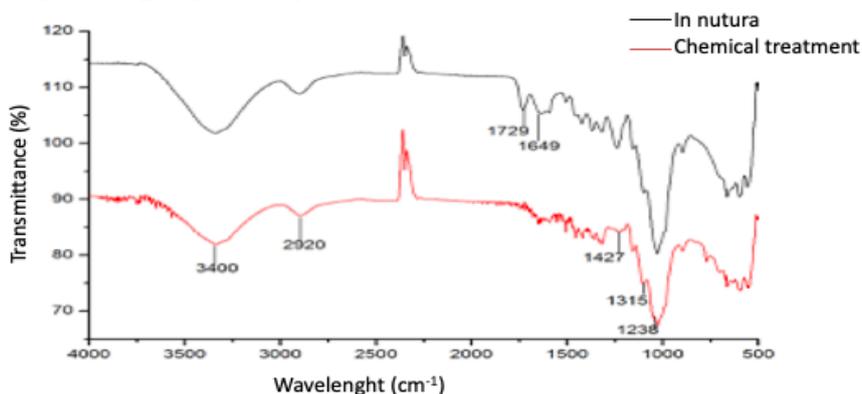


Figure 4. FTIR spectroscopy analysis of Jute

The peaks observed at 2507 cm^{-1} and 1238 cm^{-1} show bending vibration of the C–H and CH_3 groups present in aromatic rings of hemicellulose and lignin.

3. Results and Discussion

3.1 Mechanical properties

Through the data obtained in the tensile stress test, a graphical analysis was elaborated that allows us to identify, according to the type of reinforcement, the tensile stress applied in each case. In Figure 6 it is possible to identify that by increasing the proportion of fiberglass reinforcement in the composite, the material tends to behave mechanically more resistant to traction.

According to the result of an average tensile stress, a positive influence of the jute fiber as a reinforcing agent on the polymeric matrix is observed. The hybrid composite (jute/glass) showed superior mechanical strength, compared to reinforced jute fiber only. The presence of fiberglass as a composite constituent influenced the tensile stress, so much that the fiberglass reinforced material presented higher resistance in the comparative study. The excessive reinforcement of fiberglass has already been expected, due to its great application in several manufactured products that already presented high mechanical resistance due to the use of the material. Figs. 5 (a) and 5 (b), show the Instron machine ongoing test and some composite samples.

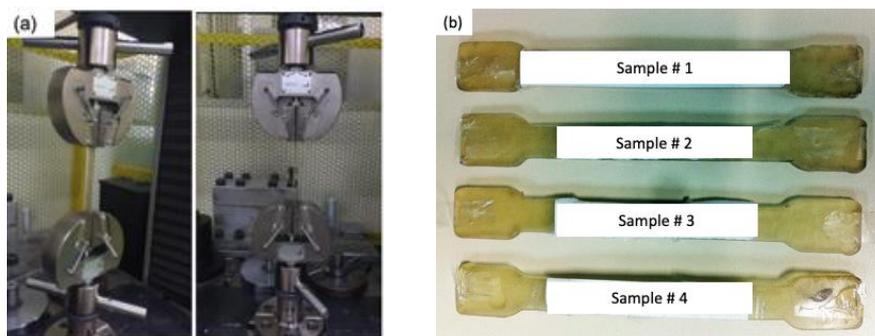


Figure 5 (a): Momento de realização dos ensaios de tração do compósito; and 5 (b): Composite samples

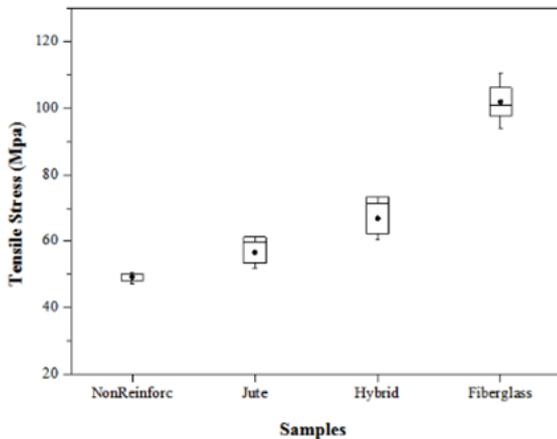


Figure 6: Graphic with tensile stress tests results in the samples.

The numerical values demonstrated in Table 1 indicate a 15% increase in tensile stress with the presence of jute fiber reinforcement and 36% with hybrid reinforcement (jute/glass). However, the fiberglass reinforced composite obtained a performance of 107%, while compared to non-reinforced composite. In relation to the average elongation, the hybrid composite provided better performance with an increase of 45% comparing to other reinforcements: jute and glass with 12% and 5% of increase respectively, so it is possible for it to be a material with great deformation potential.

Table 1: Average numeric values obtained by tensile stress tests.

Specimen (5% of reinforcement)	Ratio (%) jute/fiberglass	Tensile stress Avg (MPa)	Modulus Avg (GPa)	Elongation Avg (%)
Non-reinforced	(0/0)	49.26 ± 1.8	0.77 ± 0.77	7.79 ± 1.09
Jute	(100/0)	56.70 ± 6.50	1.24 ± 0.66	8.72 ± 0.60
Hybrid (jute/fiberglass)	(50/50)	66.90 ± 9.20	0.60 ± 0.76	11.28 ± 3.14
Fiberglass	(0/100)	101.96 ± 6.82	1.82 ± 0.08	8.19 ± 0.12

3.2 Fractographic Analysis

The fracture analysis was performed in two composite samples, one being reinforced with jute fiber and the other with hybrid reinforcement. In Figure 7, we show the images made with a scanning electron microscope (SEM).

(hybrid, 600x) c) jute fiber (hybrid, 300x), d) jute fiber amplified (jute, 1,50kx).

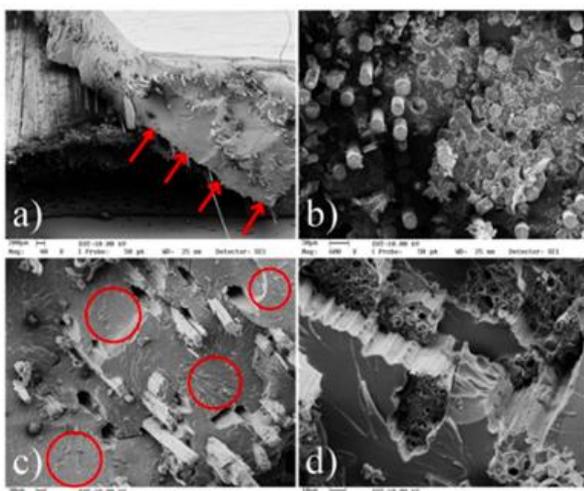


Figure 7: Fracture microscopy: a) general view of the fractured transversal section (hybrid 40x), b) fiberglass

The cross-section analysis in Figure 7 (a) points out that at the moment of rupture, the applied force was prolonged in the region of the matrix between the fibers, promoting an inefficient distribution of the reinforcing agents. The fiberglass in Figure 7 (b) showed a fragile behavior because its transversal section was flat. Figure 7 (c) shows extensive regions without reinforcement, however, the fact that jute fiber has a larger surface area in relation to fiberglass, makes the distribution in the polymeric matrix difficult. In the composite sample reinforced only with jute fiber, Figure 7 (d) proves that the organophilic treatment applied on the jute fiber was efficient, since the fiber/matrix interfacial adhesion is visibly perceptible. While still analyzing the jute fiber, it is possible to notice that it is an aggregation of vascular microfibrils that promotes the appearance of microfissures responsible for the physical potential of deformation also found in other studies (FONSECA et al., 2013).



The preparation of the samples caused imperfection in the distribution among the fibers, favoring the propagation of cracks between the reinforcements in the hybrid composite. However, this crack propagation also refers to the proportion of fibers in the composite, so that the presence of more fibrous reinforcement would inhibit such propagation. The difference in the crack surface region between the glass and jute fibers is noticeable, one being smooth and the other irregular, respectively, this technical feature directly influences the mechanical properties so

that the jute demonstrates ductility potential while the fiberglass feature is fragile so that the joining of the two fibers creates a material with high tensile stress and at the same time boosts the elongation feature that only the fiberglass did not provide to the composite.

The goal of this project was to study mechanical and physical behavior using the traction and microstructure tests, besides evaluating the correlation with the behavior of the continuous alignment of fibers, Table 2 provides some results of the studies with hybrid composites.

Table 2: Comparative between different types in fibrous reinforcements in some hybrid composites.

Source	Fibers	Type	Tensile Stress Avg (MPa)	Modulus Avg (GPa)	Elongation Avg (%)
Costa, 2012	Sisal/fiberglass	Short fibers	33.75 ± 2.21	-	-
Carvalho, 2006	Sisal/fiberglass	Woven	19.0 ± 4.2	1.50 ± 0.31	10.4 ± 2.2
Singh, 1995	Sisal/fiberglass	Laminates	65.20	-	11.30
This Work	Jute/fiberglass	Continuous	66.90 ± 9.20	0.60 ± 0.76	11.28 ± 3.14

Fonte: (COSTA, 2012), (CARVALHO; CAVALCANTI, 2006) e (SINGH; GUPTA; VERMA, 1995).

The sisal fiber has a similar chemical composition as the jute fiber, in a way that it is possible to have an evaluation in the obtained data. Even though the composites made in form of overlapping or laminated woven already have efficiency in tensile stress resistance, the continuous alignment is favorable according with the obtained results. Regarding to the particulate composite, it has a superficial contact area larger than presented in this work, favoring the propagation of cracks in the polymeric matrix.

4. Conclusion

The fiberglass reinforced composite has better mechanical tensile properties compared to the reinforced composite with only natural fiber, but the use of natural jute fiber as the reinforcing agent in this composite provides an increase in the average elongation in addition to a considerable tensile stress. Jute fiber is shown to be a viable material for addition in the polyester/fiberglass composite in which its mechanical tensile properties are not very high. Moreover, jute fiber enhanced the elongation condition of the material, giving the composite a new condition, regarding applications that require a greater elongation of the composite with only fiberglass reinforcement. The continuous alignment had a positive influence in the tensile stress resistance increasing the reinforcement in the direction where the force is applied in the tensile stress tests, however a

possible increase in proportion of reinforcement could influence the result, filling the gaps identified in the fractography, decreasing crack propagation.

Acknowledgments

We would like to thank the coordination of LabMatPro - Laboratory of Materials and Processing for the support in carrying out this study.

Divulgação

Este artigo é inédito e não está sendo considerado para qualquer outra publicação. O(s) autor(es) e revisores não relataram qualquer conflito de interesse durante a sua avaliação. Logo, a revista *Scientia- Amazonia* detém os direitos autorais, tem a aprovação e a permissão dos autores para divulgação, deste artigo, por meio eletrônico.

References

- BARBOSA, A. **Características estruturais e propriedades de compósitos poliméricos reforçados com fibras de Buriti**. 2011. Tese em Ciência dos Materiais, Universidade Estadual do Norte Fluminense ...
- BLEDZKI, A.; GASSAN, J. Composites reinforced with cellulose based fibres. **Progress in polymer science**, v. 24, n. 2, p. 221-274, 1999.



Engenharia

[https://doi.org/10.1016/S0079-6700\(98\)00018-5](https://doi.org/10.1016/S0079-6700(98)00018-5), 1999.

CARVALHO, L. H.; CAVALCANTI, W. S. Propriedades mecânicas de tração de compósitos poliéster/tecidos híbridos sisal/vidro. **Polímeros: Ciência e tecnologia**, v. 16, n. 1, 2006.

COSTA, D. **Caracterização de materiais compósitos de matriz poliéster e fibras de bambu, sisal e vidro e híbridos bambu/sisal, bambu/vidro e sisal/vidro. Belém: 2012, 106f.** 2012. Dissertação (Mestrado em Engenharia: Materiais e processos de Fabricação ...

ELANCHEZHIAN, C. et al. Review on mechanical properties of natural fiber composites. **Materials Today: Proceedings**, v. 5, n. 1, p. 1785-1790, 2018.

<https://doi.org/10.1016/j.matpr.2017.11.276>, 2018.

FONSECA, A. et al. Properties of an Amazonian vegetable fiber as a potential reinforcing material. **Industrial Crops and Products**, v. 47, p. 43-50, 2013.

<https://doi.org/10.1016/j.indcrop.2013.02.033>, 2013.

FREIRE J, R. C. S. **Fadiga de Alto Ciclo em Compósitos de PRFV. Modelagem por RNAs e Prevenção de Falha. 2005. 171 f.** 2005. Tese (Doutorado)-Universidade Federal do Rio Grande do Norte, Natal

GUPTA, M.; SRIVASTAVA, R. Mechanical properties of hybrid fibers-reinforced polymer composite: A review. **Polymer-Plastics Technology and Engineering**, v. 55, n. 6, p. 626-642, 2016. <https://doi.org/10.1080/03602559.2015.1098694>, 2016.

KALIA, S.; KAITH, B.; KAUR, I. Pretreatments of natural fibers and their application as reinforcing

material in polymer composites—a review. **Polymer Engineering & Science**, v. 49, n. 7, p. 1253-1272, 2009.

<https://doi.org/10.1002/pen.21328> 2009.

MONTEIRO, S. N. et al. Comportamento mecânico e características estruturais de compósitos poliméricos reforçados com fibras contínuas e alinhadas de curauá. **Revista Matéria**, v. 11, n. 3, p. 197-203, 2006.

MOTHÉ, C. G.; ARAUJO, C. R. D. Caracterização térmica e mecânica de compósitos de poliuretano com fibras de curauá. **Polímeros: Ciência e Tecnologia**, v. 14, n. 4, 2004.

PUJARI, S.; RAMAKRISHNA, A.; SURESH KUMAR, M. Comparison of jute and banana fiber composites: A review. **International Journal of Current Engineering and Technology**, v. 2, n. 2, p. 121-126, 2014. DOI: <http://dx.doi.org/10.14741/ijcet/spl.2.2014.22>, 2014.

ROSA, M. F. et al. Effect of fiber treatments on tensile and thermal properties of starch/ethylene vinyl alcohol copolymers/coir biocomposites. **Bioresource technology**, v. 100, n. 21, p. 5196-5202, 2009. <https://doi.org/10.1016/j.biortech.2009.03.085>, 2009.

SINGH, B.; GUPTA, M.; VERMA, A. Mechanical behaviour of particulate hybrid composite laminates as potential building materials. **Construction and Building Materials**, v. 9, n. 1, p. 39-44, 1995. [https://doi.org/10.1016/0950-0618\(95\)92859-F](https://doi.org/10.1016/0950-0618(95)92859-F), 1995.

TINÔ, S. **Descontinuidade na seção transversal em laminados compósitos poliméricos: efeitos e propriedades.** 2010. Universidade Federal do Rio Grande do Norte