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EFFECT OF WETTING AND DRYING CYCLES ON THE INTERFACE OF NATURAL FIBERS WITH A PORTLAND CEMENT BASED MATRIX

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Abstract
Several fiber treatments are applied to mitigate the high water absorption of vegetal fibers. Wetting and drying cycles are usually performed in the industry of paper and cellulose to reduce the volume variation of the natural fibers. This procedure stiffens the polymeric structure of the fiber-cells (process known as hornification) resulting in a higher dimensional stability. The aim of this study is to determine the effect of the hornification on the fiber-matrix interface of natural fibers and Portland cement matrices. For this purpose, 2, 5 and 10 cycles of wet and drying was applied on Sisal, Curaua and Jute fibers. Fiber pull-out tests were performed in embedment lengths of 25mm. Furthermore, the influence of the hornification in the fibers mechanical (under tensile loading) and microstructural (surface modifications of the fiber and changes in the fiber-cell structure) behavior were investigated. The results indicate changes on the tensile strength and strain capacity of the studied fibers, showing that morphology, physical aspects and chemical composition play an important role on the efficiency rate of hornification.

Keywords:
Sisal fiber; Curaua fiber; Jute fiber; interface; hornification.

1 INTRODUCTION

Vegetable fiber reinforced composites have the potentiality to become the ultimate green material option, minimizing the use of natural resources and overall lifetime impact. Due to its wide availability, especially in tropical countries, these fibers have a low cost and present the great advantage of being renewable. Regarding the drawbacks in the use of those fibers, treatments have been studied to reduce the impact of a low chemical bond and the high water absorption capacity which causes a volumetric instability. This volumetric instability results in a partial loss of the fiber-matrix physical contact damaging the interfacial transition zone.

There are several types of physical fiber treatments, and the most common are the chemical ones. These treatments can clean the fiber surface, modify their surface chemistry, lower the moisture uptake and increase the surface roughness [Ton-That 2007]. As natural fibers bear hydroxyl groups from cellulose and lignin, they are subject to chemical modification. The hydroxyl groups may be involved in the hydrogen bonding within the cellulose molecules thereby reducing the activity towards the matrix. Chemical modifications may activate these groups or can introduce new moieties that can effectively lead to chemical interlock with the matrix. Mercerization, isocyanate treatment, acrylation, permanganate treatment, acetylation, silane treatment and peroxide treatment with various coupling agents and other pretreatments of natural fibers have achieved various levels of success for improving fiber strength, fiber fitness and fiber-matrix bond [Altundal 2013].

A particular procedure to reduce the water absorption capacity of natural fibers and to improve the fiber-matrix bond have been pursued by applying cycles of wetting and drying in the fibers [Angrizani 2006; Claramunt 2010; Lopes 2010; Ferraz 2011; Ferreira 2012]. These treatments promoted a reduction in volumetric changes of pulps and fibers of natural origin as well as alterations in their mechanical properties. This is a result of repeated wetting and drying cycles which increase the degree of crosslink within the fiber microstructure. The application of the mentioned treatments have shown to be effective in promoting changes in the natural fiber morphologies as well as in

their physical and mechanical behavior [Ferreira 2012]. However, there still exists a lack of information on the use of such treatment to improve the interface of different natural fibers. The objective of the present research is to investigate the effect of cycles of wet and drying on the properties and fiber-matrix interface characteristics of sisal, curaua and jute fibers with a cement matrix free of calcium hydroxide. Tensile tests were carried out to study the effect of the treatments on the fiber mechanical properties. After the treatments, the fibers were analyzed through, SEM analysis. Pullout tests were carried out to investigate the potential benefits caused by the treatments in the interface bond.

2 EXPERIMENTAL PROGRAM

2.1 Material and processing

The sisal fibers used in the present study were obtained from the sisal plant cultivated in farms located in the Bahia state, Brazil. They were extracted from the sisal plant leaves in the form of long fiber bundles. The curaua fibers were provided by the company Pematec, located in the city of Santarém, in the state of Pará. The curauá fiber is produced in the Amazon region, and extracted from the leaf of the plant Ananas erectifolius by a process similar to the process used for sisal fiber. The jute fiber was obtainedfrom the Amazon region. It is extracted from the stem of the plant Corchorus capsularisby in a combination of processes which comprises the following steps: cutting, retting, shredding, drying, packing and classification [Silva 2009]. Regarding to the fiber microstructure it is formed by numerous individual fibers (fiber-cells).

The individual fiber-cells are linked together by means of the middle lamella. More information on the studied fibers microstructure can be found in the authors previous works [Toledo Filho 2009; Ferreira 2012].

The used matrix presented a mix design of 1:0.5:0.4 (binder: sand: water/binder ratio) by weight. The binder was composed by 30% of Portland cement CP-32 F II, 30% of metakaolin and 40% of fly ash. This ratio of metakaolin and fly ash aims to guarantee the durability of the fiber once a matrix free of calcium hydroxide is obtained, as shown in the previous research [Toledo Filho 2009]. The fly ash also ensures greater workability to the matrix that, within the context of high-performance composites, is a desirable property as it ensures a better homogenization of the vegetable fibers [Ferreira 2012].

The used sand was processed to obtain a maximum diameter of 840μm. The used superplasticizer was the Glenium 51 (type PA) with solids content of 31%. A viscosity modifier Rheomac UW 410, (manufactured by BASF), at a dosage of 0.8 kg/m^3 was also used in order to avoid segregation and bleeding during molding. The matrix used in this study showed a flow table spread value of 450 mm according to the brazilian standard NBR 13276/05 [NBR 13276] and compressive strength at 28 days of 31 MPa, according to NBR 7215/96 [NBR 7215].

The mixtures were produced in a room with controlled temperature (21 ± 1°C) using a mixer with capacity of 5 dm^3. The mixing procedure is described as follow. All dry components were homogenized in the mixer. The water and superplasticizer were added and mixed for 2 minutes at a speed of 125 RPM. After that, the process was stopped during 30 seconds to remove the material retained in the mixer. Then, the mixing procedure continued for 2 minutes at 220 RPM and, finally for a further 5 minutes at 450 RPM.

To mold the pullout specimens a special mold was developed. After filling the mold with the matrix, the top cap was fixed and the fiber stretched slightly for alignment. The mortar was placed in plastic bags before being placed in the mold as to facilitate the casting process. Embedment lengths of 25mm were studied. After 24 hours, the specimens were demolded and placed in a fog room (HR% ≥ 95%) to moist curing for 7 days for the pullout test (proceeding followed according [Ferreira 2012].

2.2 Fiber treatment

The detailed description of the used treatment is described as follow. The natural fibers were placed in a container with water (T = 22 C) during three hours before it reached its maximum water absorption capacity. The drying process was carried out in a furnace at a temperature of 80°C. The furnace used is equipped with an electronic temperature control and connected to a scale, with a precision of 0.01g to record the mass loss during the drying process.

The furnace was programmed to reach 80°C at a heating rate of 1°C/min and to maintain this temperature for 16 hours. After 16 hours of drying, the furnace was cooled down to the temperature of 22°C in order to avoid possible thermal shock to the fibers. This procedure was repeated ten times. More details can be obtained elsewhere [Ferreira 2012].

3 MECHANICAL AND ANALYTICAL TESTS

3.1 Thermogravimetrical analysis (TGA)

TGA was performed in natural and treated fibers. The tests were performed in a SDT Q600 simultaneous TGA/DTA/DSC from TA Instruments. Samples weighing 10mg were submitted to a heating rate of 10°C/min until reaching 1000°C in a platinum crucible using 100 ml/min of nitrogen as the purge gas.

3.2 Scanning electron microscopy (SEM)

The fiber's microstructure and interface with the matrix was investigated using an SEM Hitachi TM3000. The microscope was operated under an accelerating voltage of 15kV.

A pre-coating with a thin layer of approximately 20 nm of gold was done to make the fiber conductive and suitable for analysis. In order to measure the fiber's cross-sectional area, for each single fiber used in the pullout and tensile test, an adjacent piece of the fiber (immediately next to the one tested) was kept for future measurement and morphology characterization using the SEM. The obtained images were post-processed using ImageJ, a Java-based image processing program.

3.3 Single fiber pullout tests

The pullout tests were performed in an electromechanical testing machine Shimadzu AG-X with a load cell of 1kN. The tests were carried out using a displacement rate of 0.1 mm/min. The samples were fixed in the machine grips through a system with hinged-fixed boundary conditions as shown in Fig. 1.

3.4 Direct tensile tests

The tensile tests were performed in an electromechanical testing machine Shimadzu AG-X

with a load cell of 1kN. The tests were performed in 15 of each natural fiber using a displacement rate of 0.1 mm/min. The fibers with a gage length of 50 mm were glued to a paper template for better alignment in the machine and for a better griping with the upper and lower jaws in accordance with [ASTM C1557].

Fig. 1: Pullout and tensile test: (a) overall view of the testing machine showing in (b) the pullout test setup and in (c) the tensile test setup.

4 RESULTS AND DISCUSSION

The chemical composition and cellulose crystallinity of the used natural fibers are shown in Figure 2 and Table 1. More information on the natural fibers microstructure and mechanical properties can be found in the authors previous work [Silva 2009. Ferreira 2012].

The degree of cellulose crystallinity is one of the most important crystalline structure parameters. The rigidity of cellulose fibers increases and their flexibility decreases with increasing the ratio of crystalline to amorphous regions [Gümüskaya 2003]. Curaua present a higher crystallinity index followed by jute. Sisal present lower values, as observed by [Poletto 2014].

Table 1: Summary of average values of the chemical composition and cellulose cristalinity (Crl) of sisal, curaua and jute fibers.

Fiber	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Crl (%)
Curaua	58.8	23.8	14.7	91%
Sisal	60.5	25.7	12.1	80%
Juta	74.4	15	8.4	87.9%

Typical stress-strain curves of untreated (UN) and treated (TR) fibers are shown in Figure 3. Results of individual treatments values and averages are presented in Table 2.

The mechanical and physical properties of the fibers are mainly influenced by their composition, mainly cellulose, hemicellulose and lignin [Poletto 2012; Ornaghi 2014]. As an example, higher tensile strength and higher thermal stability are obtained for fibers that contain more crystalline cellulose [Gümüskaya 2003]. Hemicellulose is one of the fiber components responsible for the initial thermal degradation behavior and is also associated with the moisture content [Ornaghi 2014]. Therefore, fibers containing high hemicellulose content should absorb more moisture and degrade at a lower temperature. In addition, higher quantities of extractives may promote fiber degradation at low temperatures [Poletto 2012]. Thus, the degradation characteristics of natural fibers may be estimated based on their chemical composition.

The tensile strength of the sisal fiber increased after treatment. Not only increased by 5% but also was followed by a lower data scattering. It was observed a reduction on the stiffness of 16% and a higher strain capacity.

For the Curauá fibers, the hornification treatment resulted in an increase of tensile strength and stiffiness by about 50% and 35%, respectively, keeping the same strain capacity.

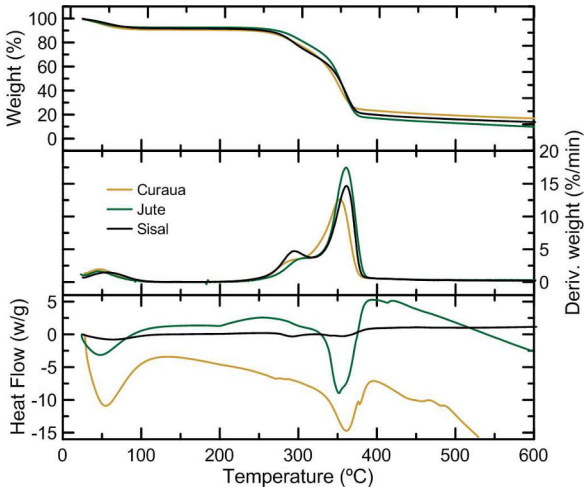

Fig. 2: TG/DTG/DSC thermograms for Natural fibers (Curaua, Sisal and Jute).

Cycles of wetting and drying performed on jute fibers resulted in a small increase in the average strength and a moderate reduction in the Young's modulus.

In general, the hornification process modifies the polymeric structure of the fiber-cells resulting in changes on tensile strength and strain. According to [Zugernmaier 2008], the hydroxyl groups (-OH) in the cellulose, hemicelluloses and lignin build a large amount of hydrogen bonds between the macromolecules in the plant fiber cell wall. Subjecting the plant fibers to humidity causes those bonds to break. The hydroxyl groups then form new hydrogen bonds with the water molecules when at wetting condition, which induce swelling. It is needed a high energy to brake those bonds, therefore the cycles applied to the fiber increase the number of hydrogen bonds, structural changes in the fibers wall and an increase in the number of –OH bonds. The cycles also can modify another functional group as the COOH-groups. The increase of this particular group results in an increase of the hydrophilicity of lignin. Carboxyl groups are able to connect to other functional groups by hydrogen bonds which may result in increased lignin macromolecule netting [Gümüskaya 2003]. That promotes a better interaction between lignin and cellulose and hemicellulose, resulting in a stronger fiber, with a higher crystallinity, stiffness and strain capacity. In fact the application of 10 cycles of treatment reflects a tendency on that direction even though not all results showed an improvement in the fiber mechanical properties. The reason is probably due to the effects of fatigue stress applied on fibers during the wet and dry process.

According to [Santos 2012], the excessive number of cycles may promote a decrease on the sisal fiber tensile strength. For treatment with 20 cycles, the

hornification promoted a higher reduction on tensile strength of sisal fibers (336 to 234 MPa).

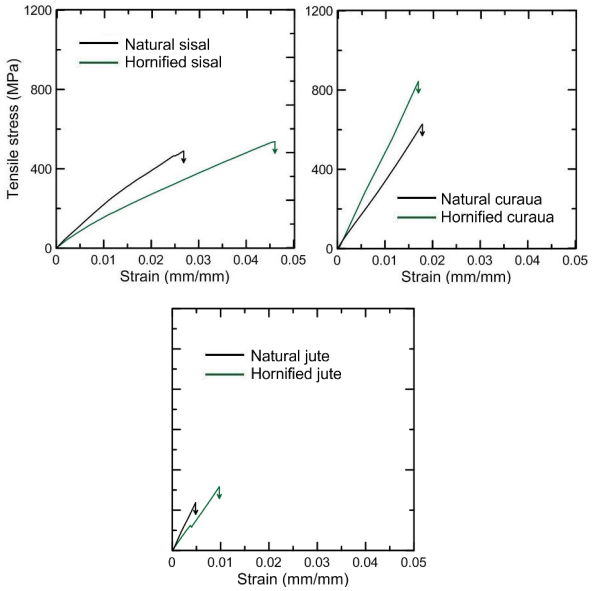

Fig. 3: Influence of hornification (after 10 cycles of wetting and drying) on the natural fibers tensile behavior: Sisal (a); Curaua (b) and Jute (c)

Tensile tests with fibers submitted to different cycles of wetting and drying has been performed. After 2 and 5 cycles itwas observed a distinctbehavior for each fiber (Figure 3).

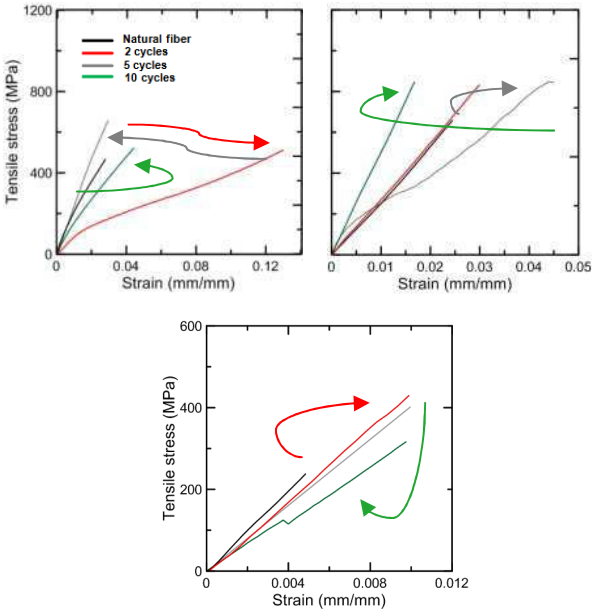

Fig. 3: Influence of cycles of wet and drying on the sisal, curaua and jute fibers tensile behavio: Sisal (a); Curaua (b) and Jute (c)

Apparently, after 2 cycles the curaua and jute fiber show an increase on tensile strength. The Sisal fiber loses stiffness increasing its strain capacity. At the cycle number 5 the sisal fiber increases its tensile strength and stiffness. Curaua has the opposite behavior, with a decrease in stiffness, a higher strain capacityand different curvebehavior in comparison to the previous cycles.

After 10 cycles of treatment all fibers present a higher tensile stress, in comparison to thereference fibers. The main difference is observed on the stiffness. Sisal shows a reduction, while curaua increases this property and jute presents the same values observed before the treatment.

Typical load-slip curves of untreated and treated fibers are shown in Figure 5. Results of individual treatments values and averages are presented in Table 2.

Regarding to the pullout behavior, only the influence of 10 cycles of treatment was evaluated. The hornified sisal showed a higher adhesional and frictional bond when compared to the untreated fiber, whereas the other fibers showed a reduction in the bond. The increase in the sisal fiber-matrix bond process can be traced back to a packing of the fiber cells due to the formation of hydrogen-bond, reducing the water absorption and dimensional variation. Another reason for the better bonds can be related to the increasing of the superficial roughness after 10 cycles of wetting and drying. A direct consequence is the reduction of the lumen size, which consequently reduces the capacity of water absorption and dimensional change. The same reduction in lumen size and packing of the fiber cells was observed for curaua fibers. Even with an increasing on the tensile strength, the pullout behavior show a decrease on fiber-matrix bond. It is possible to correlate this result to a surface inactivation process. Lignin, for example, can block the formation of hydrogen bonds and prevent inter-fiber bonding. After the pullout test, the jute and curaua fibers were analyzed by SEM analysis. It was observed a fiber cell delamination, justifying the decrease instiffness as observed on Figure 4.

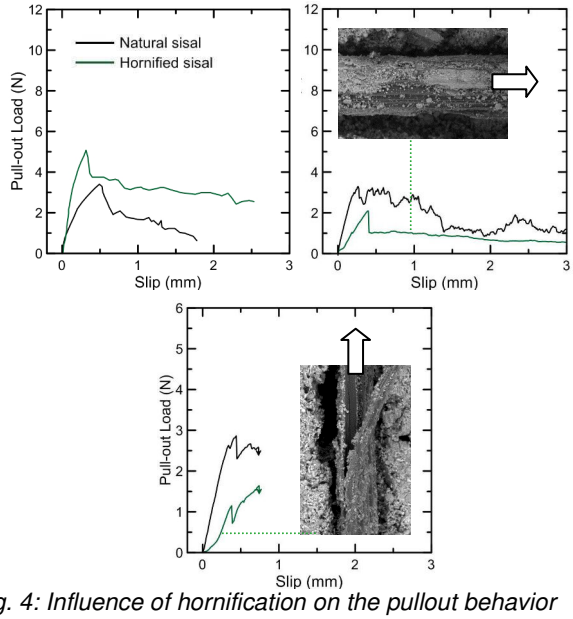

Fig. 4: Influence of hornification on the pullout behavior of natural fibers (Sisal [a], Curaua [b] and Jute [c]).

Observing Figure 5, it is possible to conclude that fibers present a different sensibility to water and to the numbers of wetting and drying cycles. Damage was observed on jute fibers, initiated on cycle number 4. Several changes on the curaua fiber-cells was observed, resulting on the closure of the lumen and in a reduction at the fiber cross section since the first wet and drying process. In the case of sisal, signals of failure due to swelling fatigue is observed only after 20 cycles, as reported in [Santos 2012].

Probably no chemical bonds exist between cellulose and hemicellulose, but mutual adhesion is provided by hydrogen bonds and van der Waals forces. [Dimitriun 2004].

In this case the treatment induces a larger occurrence of intermolecular hydrogen bonding. The cellulose molecules have a strong tendency to form intermolecular and intramolecular hydrogen bonds Cellulose and hemicellulose are non-covalently bonded (i.e. hydrogen bounded) to each other [Page 1976; Ishii 2001] , while lignin and hemicellulose are covalently bonded [Johnsson 1991].

The orientation of the hemicelluloses and lignin, which are amorphous polymers, may, particularly under moist conditions, play a dominant role for the mechanical properties [Salmén 1998].

Recently, however, it has been suggested that irreversible hydrogen bonding occurs in wood during drying, similar to that observed in pulp fibres, though to a lesser extent [Suchy 2010]. The presence of the ligno-hemicellulosic matrix in wood may restrict the formation of hydrogen bonds within the fibres [Duchesne 2001]. In addition to hydrogen bonding, cross-linking in the lignin complex might also occur due to application of heat during drying [Tjeerdsma 1998; Sivonen 2002; Weiland 2003; Nuopponen 2004].

The energy of hydrogen bonds and hydrogen bond distances was studied by [Poletto 2014]. Curaua and jute present a lower energy of hydrogen bonds values. This could be associated with a higher quantity of absorbed water in the structure of these fibers. Materials with higher quantities of lignin may contribute to form several intramolecular hydrogen bonds between neighbor phenolic groups in lignin, reducing the distances between the neighbor phenolic groups.

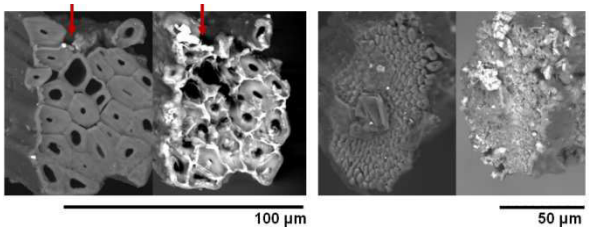

Fig. 5: Natural fibers cross-section morphology before (left) and after (right) hornification: Jute (a) and curaua fibers (b)

5 CONCLUSIONS

The work in hand investigated the effect of hornification treatment on natural fibers in their mechanical and fiber-matrix interface properties. The following conclusions can be drawn from the present research:

The sisal and curauá fibers tensile strength increased after 10 cycles of wetting and drying. This can be explained by the change in the cellulose cristalinity and the bonds created between different polymer chains in the microfibrils. On the other direction the jute fibers presented a small decrease on both the average strength and Young's modulus. A lower number of cycles (i.e. 2 and 5) results in a different behavior for the fibers tensile behavior. More investigation is needed in order to explain the involved mechanisms. After the hornification treatment the sisal fiber showed a higher adhesional and frictional bond when compared to the untreated fiber, whereas the other fibers showed a reduction in the bond. This can be explained due to a combination of increase in surface roughness and a packing of the fiber cells due to the formation of hydrogen-bond observed for the sisal fibers.

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Table 2: Results of tensile test of treated (10 cycles) and untreated natural fibers.

Fibers	**Treatments**	**Max Load (N)**	**Tensile Strength (MPa)**	**Strain capacity (mm/mm)**	**Young's modulus (GPa)**
Sisal	UN	10.28 (3.43)	447.20 (23.90)	0.03 (0.01)	20.16 (3.52)
	TR	15.66 (2.61)	470.25 (16.56)	0.05 (0.01)	17.60 (1.22)
Curauá	UN	2.54 (0.46)	632.14 (138)	0.021 (0.01)	38.1 (18.01)
	TR	3.62 (0.51)	815.12 (106)	0.019 (0.01)	81.4 (15.22)
Jute	UN	1.05 (0.37)	249.23 (89.02)	0.006 (0.002)	43.9 (12.31)
	TR	0.98 (0.35)	243.13 (43.24)	0.01 (0.001)	38.41 (9.21)

Table 3: Results of pullout test of treated (10 cycles) and untreated natural fibers.

Fibers	Treat.	Pad (N)	Dad (mm)	σad (MPa)	Pfr (N)	Dfr (mm)	σfr (MPa)	Stiffness (N/mm)
Sisal	UN	3.73 (1.02)	0.66 (0.19)	0.30 (0.08)	2.56 (0.72)	0.92 (0.25)	0.18 (0.04)	9.31 (4.39)
	TR	4.53 (1.07)	0.23 (0.11)	0.42 (0.08)	3.88 (0.77)	0.41 (0.16)	0.27 (0.03)	20.60 (6.37)
Curauá	UN	3.65 (1.21)	0.35 (0.19)	0.48 (0.22)	1.86 (0.95)	0.53 (0.21)	0.34 (0.19)	30.26 (0.36)
	TR	2.56 (1.07)	0.38 (0.10)	0,32 (0.18)	1.36 (1.44)	0.84 (0.22)	0.21 (0.09)	5.46 (0.12)
Jute	UN	2.35 (0.79)	0.63 (0.32)	0.86 (0.23)	0.62 (0.26)	1.46 (0.65)	0.67 (0.24)	23.76 (8.99)
	TR	1.73 (0.67)	0.78 (0.42)	0.42 (0.12)	-*	-*	-*	4.36 (0.23)

** Fiber fracture at pullout test*