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Study of saturated and unsaturated permeability in natural fiber fabrics

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ABSTRACT

The main goal of this work is to understand how the main processing variables are affected when glass fibers are replaced by natural fibers in reinforced plastics. In this publication, a jute fabric was characterized in terms of its saturated and unsaturated permeability. It was found that fluid absorption and swelling are mechanisms present in natural fibers that reduce both permeabilities. Fluid absorption removes fluid from the main stream as it travels through the reinforcement, acting as a sink component and thus decreasing flow velocity during the unsaturated flow. Also, the saturation of the natural fibers cause swelling, reducing the porosity and increasing flow resistance during saturated flow.

1. Introduction

In the last years, governmental regulations about carbon dioxide emissions and recyclability of the materials have produced an increase in the use of natural fiber composites both in the automotive and construction industries. Many studies of sustainability and life cycle assessments have demonstrated the environmental advantages of these materials [1–5]. But one of the keys of its success is the possibility of using the well-studied glass fiber composites processing techniques, like RTM, VARTM or SCRIMP. Therefore, it is crucial to understand how the main processing variables are affected when glass fibers are replaced by natural fibers, which have different structure, different fabric architecture and different chemical interactions with the resins. One of those variables is the fabric permeability, which is the key parameter that governs the flow in the fiber bed, together with the fluid viscosity. Fabrics permeability is especially important in low pressure injection techniques like VARTM or vacuum infusion where void formation and injection time can be increased dramatically when the permeability decreases.

The processing of natural fiber composites by Liquid Composite Molding (LCM) techniques has been studied by several authors in the last years [6–10]. Most of the works have focused on the determination of the physical and mechanical properties of the composites obtained but little research have been conducted studying the injection process itself and the effect of using natural fibers on the processing variables. Richardson et al. studied the mold filling process in RTM with non-woven hemp and phenolic resin. Fiber washing and edge flow were the main problems founded while injecting with these materials. O'Donnell et al. [11] developed composites panels of soy oil-based resin and different natural fibers (flax, hemp and cellulose mats and recycled paper). They determined the permeability of the reinforcements and, except for the case of the recycled paper, the obtained values was high enough for infusing by Vacuum assisted RTM (VARTM). Umer et al. [12] characterized the permeability and compaction behavior of wood fiber mats obtained by different manufacturing techniques. The permeability values that they obtained depended on the test fluid used for the experiments. When using glucose syrup the permeability was lower than when using mineral oil. This behavior is due to the swelling of the fibers exposed to the water-base solution that reduces the size of the open flow paths. Recently, Liu and Dai [13] studied the impregnation of a jute fiber mat by a thermoplastic resin. They found for natural fabrics permeabilities an order of magnitude higher than the obtained for glass fiber mat. Due to the high viscosity melt used in their work, they did not observe impregnation inside fiber bundles.

Even though some permeability values have been reported for natural reinforcements, a detailed insight on their flow behavior is still required. In addition, the results obtained for one kind of fiber and fabric architecture are difficult to compare with that obtained for other fibers. Therefore, it is very important is to identify the main mechanisms present in natural fibers impregnation. One key aspect that has been studied by several authors in glass fibers is the difference between saturated and unsaturated permeability. The investigations made on this topic are not consistent, and a wide variety of results have been reported. Kim et al. [14] and Diallo et al. [15] found that the saturated permeability

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was always lower than the unsaturated permeability, while other authors obtained opposite results [16–18]. Also, results have been reported showing that the saturated and unsaturated permeabilities were almost equal [19]. These discrepancies are usually attributed to experimental issues that could modify the saturated and unsaturated permeability ratio, such as mold deflection, capillary effect, microscopic flow, fiber channeling, and air bubbles [18]. Pillai and Advani [20] have studied in detail the unsaturated flow in woven fibers preforms, taking into account the delayed impregnation of fiber tows through the use of a sink function in the equation of continuity for the macro flow. Micro flow can also occur through the micro pores generated during the stacking and compression of the layers of reinforcement. Unlike synthetic fibers, natural fibers absorb fluid, acting as a sink. The fluid absorption consumes fluid from the main stream as it travels through the reinforcement, increasing flow resistance during the unsaturated flow. Furthermore, saturation of natural fibers can cause swelling [21], and it could reduce the porosity and increase flow resistance during saturated flow.

The aim of this work is to achieve knowledge on mechanisms related to natural fibers impregnation in order to understand the resin flow behavior through natural fiber preforms, and improve the quality and performance of their composites. The relation between permeability and porosity of jute bidirectional fabrics is obtained, by using the Vacuum Assisted Resin Infusion (VARI) process. Both saturated and unsaturated permeability are obtained from the infusion tests. The Carman–Kozeny model is used in order to get an analytical relation between permeability and porosity. Furthermore, a brief analysis on jute fibers water absorption and its effect on permeability values is performed. Jute fabrics are coated with polyhydroxybutyrate (PHB) in order to reduce the fiber fluid absorption. PHB (a type of polyhydroxyalkanoate) is a biodegradable thermoplastic polymer with a high hydrophobic character. Permeability tests results obtained with jute and PHB treated jute fabrics are compared in order to study the fluid absorption effect on the permeability values. Also, the swelling degree of the treated and untreated fibers is determined and related to the saturated–unsaturated permeability ratio.

2. Experimental

2.1. Raw materials

Commercial bidirectional woven jute fabrics (Castanhal Textil, Brasil; surface density = 0.0300 g/cm^2) have been used in this study. The fabrics were washed with a 2% V/V distilled water and detergent solution, to remove contaminants and normalize the fabrics conditions for all the injections.

2.2. Treatment of PHB on natural fibers

In order to study the fluid absorption effect on permeability measurements, two different treatments were performed to make fibers less hydrophilic. The first treatment (treatment A = TA) consisted of wetting out the jute fabric with a 2% polyhydroxybutyrate (PHB, Biocycle-Brasil) in chloroform solution [22]. After wetting, the fabrics were air dried leading to surface density values of 0.033 g/cm^2. The second one (treatment B = TB) consisted of immerging the fabric into a container with the same solution, and leaving the fibers immersed until the solvent completely evaporates. In this case PHB content obtained was much higher than with TA, and surface density reached 0.046 g/cm^2. Also, bidirectional glass fabrics were used for comparative purposes (surface density = 0.02 g/cm^2).

2.3. Flow experiments

The test fluid used for the flow experiments was a 22% V/V water/glycerin solution, leading to viscosity values near 0.150 Pa s. Few drops of red colorant were used to improve the flow front visibility. For the swelling tests, besides glycerin, two thermosetting resins were used: a commercial vinylester (Derakane 411-350, from Ashland) and a phenolic resin synthesized in our laboratory (molar ratio: 1.3; solid content: 53.67%) [23].

Unidirectional injection experiments were performed in a rectangular metallic mold (500 mm × 100 mm) with an acrylic lid. The depth of the mold cavity used for each injection was set in order to obtain the desired values of porosity. In order to avoid mold deflection during the infiltration tests, a 3 cm thick lid was used. The uniformity of the cavity depth was checked by putting clay in different parts of the cavity and conducting one injection. The depth of the mold cavity was estimated by measuring the thickness of the clay. Two injections were conducted for each porosity and type of fabric.

The viscosity of the fluid used was measured before every infusion by means of a Brookfield DV–II + cone and plate viscometer. A vacuum pump was used to force the fluid flow though the mold cavity. The pressure gradient achieved was measured with a vacuometer, located at the outlet line of the mold.

2.4. Data analysis

In this study, Darcy's Law for unidirectional flow was used to estimate the permeability. To validate the use of Darcy's Law, it is assumed that the pore volume of the portion of the preform behind the fluid front is fully saturated with fluid, which allows the use of the quasi-steady-state assumption. Unsaturated permeability can be obtained using the following equation:

$$K_{\text{unsat}} = \frac{(\Phi \cdot m \cdot \mu)}{2\Delta P} \tag{1}$$

where K_{unsat} is the unsaturated permeability (m^2), m (m^2/s) is the slope of the curve x^2 (square of the flow front position) vs. time, μ is the fluid viscosity (Pa s) and ΔP (Pa) is the pressure drop along the fiber bed. The relation between the flow front position and the injection time was obtained by recording the infusion process with a camera mounted on top of the transparent flow cell.

Saturated permeability was calculated by measuring the fluid volumetric flow rate, once the reinforcement was fully saturated (Eq. (2)). A standard flow meter connected at the output line of the mold was used for this purpose and a plot of volume vs. time could be obtained.

$$K_{\text{sat}} = \frac{(Q \cdot \mu \cdot \Delta L)}{(A \cdot \Delta P)} \tag{2}$$

where K_{sat} is saturated permeability (m^2), Q the volumetric flow rate (m^3/s), ΔL the preform length (m) and A the mold cavity transverse area (m^2). The Carman–Kozeny model was used to establish a relationship between permeability and porosity. This relationship was developed to predict the behavior of a flow passing through a porous medium, and it was deduced by taking the medium as an arrangement of parallel tubes of any cross section [24]. The model has the following expression:

$$K = \frac{d_f^2}{k} \frac{\phi^3}{(1-\phi)^2} \tag{3}$$

where ϕ is the porosity, d_f the fiber diameter and k is the so-called Kozeny constant. Unfortunately, for many types of performs the assumptions behind the Carman–Kozeny model are not justified [25], and this equation is not able to properly fit permeability

experimental values. The prediction can be improved by modifying Eq. (3) as follows:

$$K = \frac{\phi^{n+1}}{C(1-\phi)^n} \tag{4}$$

where n and C are empirical parameters. This expression is often known as the modified Carman–Kozeny model. The use of n exponent other than 2, is not based on a flow mechanism and the model can be taken as a empirical model which fitted the experimental data [26,27].

2.5. Humidity absorption and swelling tests

Humidity absorption tests were performed in a humidity chamber, with controlled relative humidity of 65.1%. The samples were weighed using an analytical scale and the time of each weight measure was controlled with a chronometer. Samples of jute, J-TA, J-TB and bidirectional glass fabric were dried under vacuum at 90 °C for 24 h, before performing the humidity absorption test.

Swelling degree was evaluated for treated and untreated jute fibers by optical microscopy. Single fibers were put with the test fluid (glycerin + water) between glass slides, and the change in fibers diameter was measured by taking pictures at different times. The pictures were analyzed using image processing software.

In order to analyze the PHB treatment effect on fiber morphology and structure, images of the fibers were obtained by Scanning Electron Microscopy (SEM JEOL JSM-6460 LV). Sample preparation for this technique consisted in coating the fibers with a thin layer of gold.

The specimen weight and time were collected. In order to become independent of the weight of the specimen tested, the relative humidity absorption was calculated with the following equation:

$$A_r(t) = \frac{[W(t) - W_0]}{W_0} \tag{5}$$

where $A_r(t)$ is the relative humidity absorption of the specimen at each time, $W(t)$ the specimen weight at each time and W_0 the initial specimen weight.

3. Results and discussion

3.1. Humidity absorption and swelling tests

The results of the humidity absorption tests are shown in Fig. 1. Even though tests of absorption of liquid by immersion would be more representative of the real infusion process, the interpretation of the results in this kind of tests is difficult since part of liquid is retained by the inter-fiber capillaries instead of been absorbed by the fibers itself. Humidity absorption tests are less representative but suitable for estimating the effect of the treatments on the fluid absorption of jute fibers and the difference with synthetic fibers. It can be seen in Fig. 1 that glass fabric showed almost no humidity absorption. On the other hand, jute fabrics reached sorption values of 25%, and the saturation was reached very fast. The sorption equilibrium of jute filaments is in general attained within a few minutes [28]. Jute fiber is notorious for its hygroscopic nature, reaching values of 36% of moisture regain at 100% RH [29]. As seen in the plot, PHB treatments were effective for reducing fiber water absorption. These results are in agreement with the obtained by Cyras et al. [22] for cellulose paper and PHB. Treatment B had reduced the relative humidity absorption value to 5% being more effective than treatment A, which leaded to values of 7.5% of water absorption. This difference was expected because in treatment B the resulting PHB coating is thicker and more uniform, since during

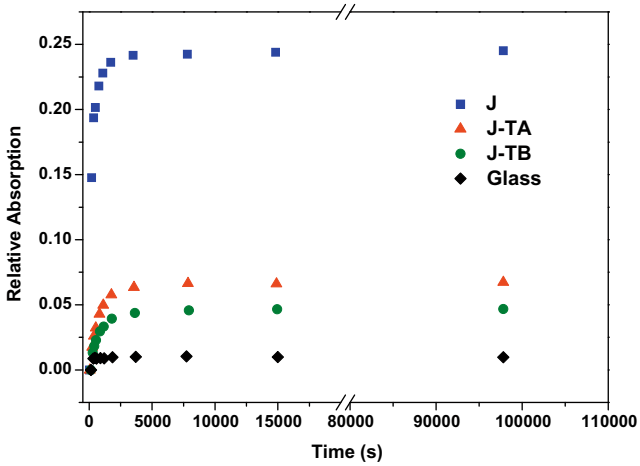

Fig. 1. Humidity absorption tests results.

the treatment all the polymer dissolved in the chloroform is deposited in the fibers.

Regarding the swelling tests, Fig. 2 shows the change in fibers diameter as a function of time in different fluids. Pictures of the extreme portion of an untreated (a) and a treated (b) jute fiber can be seen in Fig. 3. When immersed in a glycerin + water solution, untreated jute showed an increase in diameter of 22%. This value is lower than that obtained by Siddiqur Rahman et al. [21] (29%) but the difference is expected since the author used pure water for the experiments and the fluid used in this work has only 22% v/v of water in glycerin. Glycerol is a polar molecule but not to the extent of water, and the swelling power of the solution is lower than pure water. Swelling of jute fibers has been studied by several authors because it is an important mechanism for crimp formation [28,30,31]. The highly hygroscopic components of the fibers like cellulose and specially hemicellulose are responsible for this phenomenon and chemical treatments like bleaching or mercerization can increase the swelling degree by increasing the proportion of these components in the fibers and facilitating the access of the polar molecules to its cellular structure. Fig. 2 shows that the treatment with PHB was effective for reducing the swelling degree by isolating the fibers from the fluid. It is important to remark that the swelling took place in a period of time similar to the time needed to conduct a permeability determination test. Within the

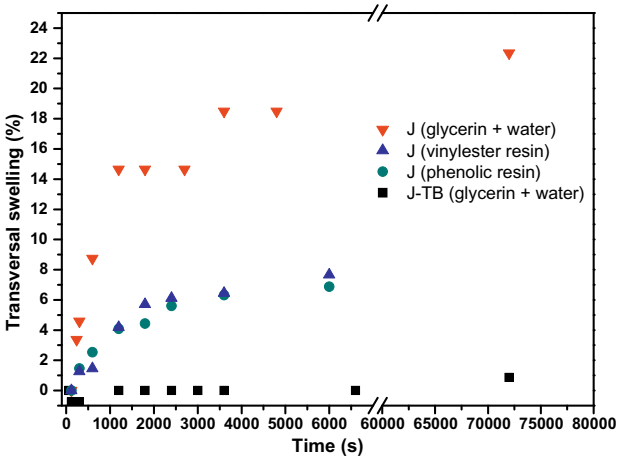

Fig. 2. Change in fibers diameter as a function of time. The fibers were immersed in the fluid used for the injections (glycerin + water solution) and in thermosetting resins for comparison purposes.

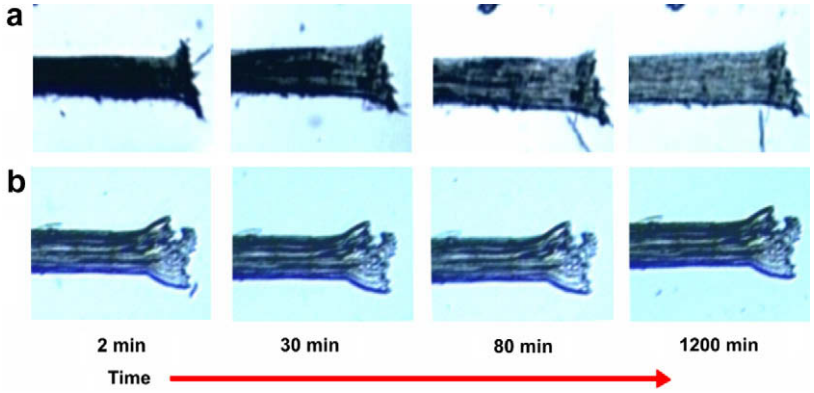

Fig. 3. Pictures of the extreme portion of jute fibers during the swelling tests. (a) Untreated jute (J). (b) Treated jute (J-TB).

first 15 min, jute fibers reached a 15% of increase in diameter. These results show the importance of taking into account the chemical and physical interaction of the test fluid with the fibers when measuring permeability of natural reinforcements. Also, swelling tests were conducted with vinyl ester and phenolic resins in order to determine if the fluid absorption phenomenon is present with true resins. Vinylester was chosen because is commonly used in composite materials, while phenolics are water-based resins. Fig. 2 shows that with both resins there is and increment in the fibers diameter of about 6% after 30 min of immersion. Therefore, swelling of the fibers during the infusion process is not only a mechanism that arises in permeability determination with water-based model fluids, but also occurs in the infiltration of composite parts with true resins, especially due to the low molecular weight compounds of such resins. For phenolic resin, the swelling agent is mainly the water, but also formaldehyde and phenol could produce such effect. For vinyl ester resin, the styrene could be the responsible for the swelling. Mantanis et al. [32] studied the swelling of cellulose fibers with different organic liquids. They found certain degree of swelling for toluene (chemically similar than styrene, and also with low hydrogen bonding).

3.2. Permeability experiments

The relationship between the square of the flow front position and time for two injections with different experimental conditions (Table 1) can be observed in Fig. 4. From these plots, unsaturated permeability was calculated using Eq. (1). We observed neither fiber washing nor race tracking during the experiments and, as a consequence, the flow front remained straight during the injections. This is important to obtain reliable results with less dispersion.

Saturated and unsaturated permeability values as a function of the porosity are shown in Fig. 5. The Carman–Kozeny model (Eq. (4)) was used to fit the experimental data. The fitting parameters can be seen in Table 2. As expected, both saturated and unsaturated permeabilities increase when the fiber volume fraction decrease. Also, it was observed accordingly with other authors [16–18] that the permeability is higher when the reinforcement is fully saturated than during the filling process. This observation can be attributed to the delayed impregnation of the dense fiber tows, with respect to the macroscopic flow front, due to the difference in the local permeability of the inter-tow and intra-tow re-

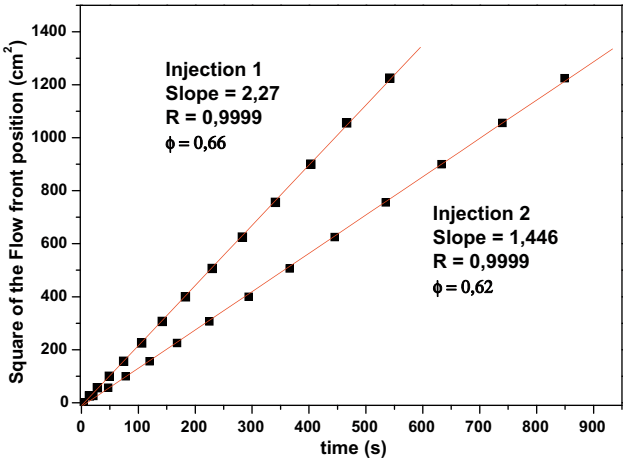

Fig. 4. Relationship between the square of the flow front position and time for two injections with different experimental conditions (Table 1).

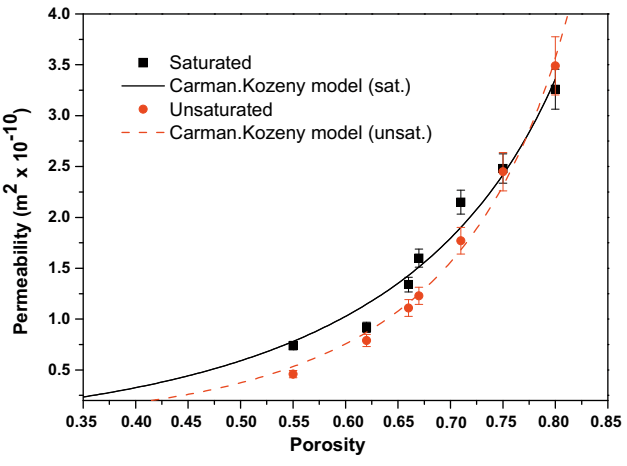

Fig. 5. Saturated and unsaturated permeability values as a function of the porosity.

Table 1
Experimental conditions for two permeability determination tests.

Variable	Injection 1	Injection 2
Preform length (ΔL)	0.38 m	0.38 m
Viscosity (μ)	0.136 Pa s	0.1585 Pa s
Cavity thickness	3.825×10^{-3} m	3.985×10^{-3} m
Width	0.095 m	0.095 m
Cross sectional area	3.633×10^{-4} m^2	3.785×10^{-4} m^2
Porosity (Φ)	0.66	0.62
Pressure (ΔP, vacuum)	680 mm Hg	670 mm Hg
Injection time	542 s	849 s

Table 2
Carman–Kozeny fitting parameters for the permeability–porosity curves of untreated jute fabric.

	K saturated	*K* unsaturated
n	0.9146	1.289
C	8.464E9	1.338E10

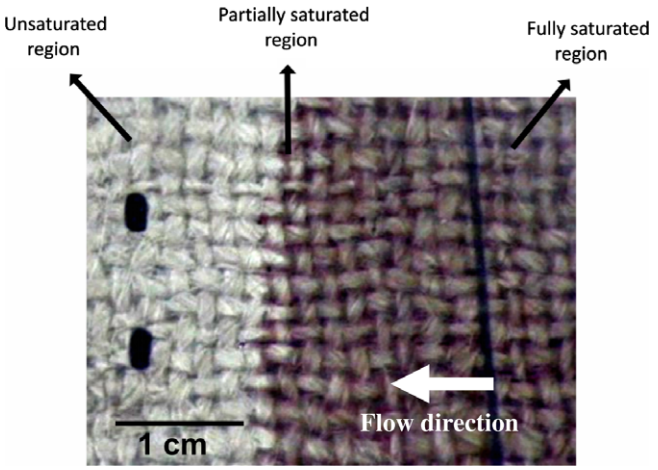

Fig. 6. Picture of the flow front during a permeability determination experiment.

Table 3
Permeability values of treated jute fabrics. Between brackets, the permeability values of untreated jute fabric extrapolated to the same porosity using the Carman–Kozeny model.

Sample	Porosity (%)	K_{unsat} ($m^2 \times 10^{-10}$)	K_{sat} ($m^2 \times 10^{-10}$)
J-TA	45	1.25 (0.263)	1.74 (0.446)
J-TB	40	1.54 (0.177)	1.86 (0.326)

gions [33]. Fiber tows acts as a sink, removing fluid from the main stream as it travels along the mold cavity, leading to lower permeability values. In other words, flow rate decrease when the micro pores are being filled with fluid. It appears that there is a dual scale phenomenon and it could be modeled according to Zhou et al. [34].

It was observed that the saturation gradient mentioned above exists in a narrow region next to the macroscopic flow front, so the quasi-steady-state assumption should hold valid (Fig. 6). Another possible explanation to the difference observed between saturated and unsaturated permeability, is the fluid absorption of the reinforcements. Jute fabrics absorb great amounts of liquid, as was shown in the absorption test. Absorption acts as another sink component, removing more liquid from the main fluid stream and retarding the fluid flow, thus the flow resistance increase and permeability values drop. When the mold is completely filled, and the reinforcement is fully impregnated and saturated with fluid (no more micro pores impregnation or fluid absorption takes place) its sink nature vanishes, and flow rate increases (for a given perform length). Thus, saturated permeability results were higher than unsaturated permeability. Also, other authors [34,35] found that bulk permeability increases with the saturation of the reinforcement. An interesting observation is that at high porosity values the difference mentioned decreases and almost disappears, due to the low fiber volume fraction. As discussed before, in natural fibers, unsaturated permeability is affected by fluid absorption and saturated permeability is affected by the swelled fiber (a consequence of fluid absorption), both effects leading to a decrease in the value of the permeability. This statement is confirmed by the results obtained from permeability tests performed on PHB treated jute (J-TA and J-TB) samples shown in Table 3. Between brackets are the permeability values of untreated jute that were extrapolated using the Carman–Kozeny model. Such low porosities are difficult to get due the low packing ability of plant fibers assemblies that lead to very high clamping forces. For the two treated fabrics, the permeability was higher than for untreated fabrics. What is more, fabrics with treatment B (more PHB) showed higher permeability for lower porosity. As seen before, the PHB coated the fibers reducing fluid absorption and therefore increasing unsaturated permeability. This procedure also reduces fibers swelling, increasing saturated permeability. Unfortunately, the treatment also produces some changes in the fabrics architecture that could affect the permeability values. Intra-tow region, that is responsible for the microflow, is expected to be more compact due to the presence of PHB (Fig. 7), that decreases the available space for the flow, decreasing the local permeability. But PHB also sticks the fibers of the bundles together reducing the flow resistance in the inter-tow region (macroflow), increasing the permeability value in that region. From the balance of this two opposite effects is expected an increase in the global permeability due to the higher influence of the macroflow on that parameter. Nevertheless, this mechanism cannot itself explain such an increment in fiber permeability (more than 5–9 times for TA and TB, respectively). Reduction in the sink effect by the PHB is then an important mechanism that contributes to the increase in fabrics permeability.

4. Conclusions

The relation between the porosity and the permeability was found for bidirectional Jute reinforcement fabrics. As expected, both saturated and unsaturated permeabilities increase as porosity does. Also, it was observed that saturated permeability values are higher than unsaturated permeability values, but this difference tends to vanish for porosities above 75%, due to the low fiber volume fraction.

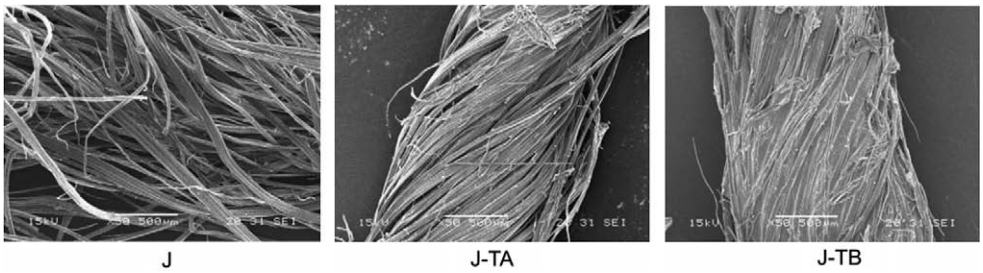

Fig. 7. Lateral view of different untreated and treated jute fibers obtained Scanning Electron Microscopy. The first picture (untreated) shows individual fibers because the bundle broke up during the handling needed to prepare the samples for SEM microscopy.

Also, the fluid absorption of Jute fibers and its effect on the permeability was analyzed. It was found that Jute reinforcements absorb great amount of fluid during the infusion process, when compared to glass fiber reinforcements. The fluid absorption affects the permeability value of the preform, because it removes fluid from the main stream as it travels through the reinforcement, acting as a sink component and thus decreasing flow velocity during the unsaturated flow. Furthermore, saturation of natural fibers can cause swelling, thus reducing the porosity and increasing flow resistance during saturated flow. So both saturated and unsaturated permeability values of the Jute preform are reduced because of the fluid absorption, and controlling this phenomenon it is possible to control fabrics permeability. Moreover, fiber swelling is still present in the infusion of composite parts performed with commercial resins, as was demonstrated by means of swelling tests with vinylester and phenolic resins. Therefore, flow simulation for predicting resin flow trough natural fiber preforms should take into account the chemical and physical interaction of the test fluids and resins with the fibers. As a future work this questions will be studied deeply with other resins formulation.

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