

Chapter 3

Experimental Analysis



This chapter provides experimental information on water absorption in unsaturated porous media, with particular reference to vegetable fiber-reinforced polymer composites. Herein, important information related to fiber morphology, manufacturing, moisture absorption, and mechanical characterization tests in polymer composites reinforced by caroá, macambira and sisal fibers are presented in detail. Results of the fiber morphology, moisture gain kinetics, for different sample thickness and process temperature, and mechanical properties (tensile strength, elastic modulus, impact resistance and elongation) are shown and analyzed.

3.1 Background

In physical problems involving moisture absorption or desorption in composites, it is very important to determine the moisture content at any instant of the process. In general, mass transfer rate from or to the body depends on different factors such as temperature, filler content, initial moisture content, and nature and orientation of the reinforcement. Further, hydric, thermal and mechanical events (in a micro scale analysis) occurring in short fiber reinforced composites are very different from those verified for long fiber reinforced composites [1].

All of the factors mentioned above affect mechanical performance and properties and limit the field of application of vegetable reinforced polymer composites [2–9] and new researches in this area are required. Several of the papers reported in the literature are directed to the problem of moisture absorption by composites reinforced by plant fibers and their influence on the properties of the material such as those by Marcovich et al. [10], Thwe and Liao [11], Mulinari [12], Sreekala et al. [13], Espert et al. [14], Angrizani et al. [15], Fernandes [16], Vieira [17], Jayamol et al. [18], Cavalcanti et al. [19], Santos [20], Sanchez et al. [21], Tita et al. [22], Rao et al. [23],

Sensarzadeh and Amiri [24], Idriss et al. [25], Soni and Soni [26], Srihari et al. [27], Pavlidou and Papaspyrides [28], Pegoretti and Penati [29], Kumosa et al. [30], and many others.

As a complement to the theme discussed in this book, and due to the importance of accurately describing the water sorption phenomena in vegetable fiber-reinforced polymer composites, experimental studies about water sorption by unsaturated polyester composites reinforced with short fibers of macambira (*Bromelia Laciniosa*), caroá (*Neoglaziovia Variegata*), and sisal (*Agave sisalana*) fibers are presented here.

3.2 Experimental Setup

Nóbrega [8] and Nóbrega et al. [31] conducted several experiments on the water absorption of caroá fiber-reinforced unsaturated polyester composites. Similarly Cruz et al. [32] conducted experiments with macambira fiber-reinforced unsaturated polyester composites, while Santos [20] conducted sorption experiments with sisal fiber-reinforced unsaturated polyester composites.

Macambira fibers (Fig. 3.1) are extracted from the macambira plant, which belongs to the family Bromeliaceae. The plant used in the study, was obtained from the Cariri region of the State of Paraíba State in Brazil and its chemical composition was determined to be: α -cellulose (58.72%), hemicellulose (19.37%), lignin (12.62%), moisture (8.94%), and others [33].

Caroá fibers (Fig. 3.2) are extracted from the caroá plant, which also belongs to the family Bromeliaceae. The plant employed in the study reported here was obtained at a farm in the town of Pocinhos in the State of Paraíba State in Brazil. Caroá leaves provide long fibers, of great resistance and durability. The chemical composition of the caroá fiber is as follows: cellulose (35.5%), hemicellulose (17.9%) and lignin (30.1%) and others [8, 34].



Fig. 3.1 Macambira (*Bromelia Laciniosa*). **a** Plant and **b** fibers

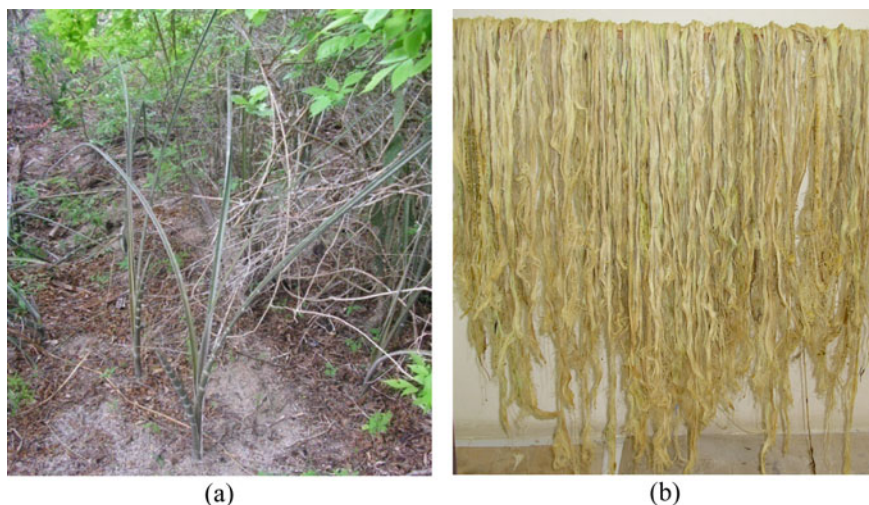


Fig. 3.2 Caroá (*Neoglaziovia Variegata*). **a** Plant and **b** fibers

Sisal fibers (Fig. 3.3) are extracted from the Sisal plant which belongs to the family Agavaceae. The plant obtained in the study reported here came from a farm in the town of Pocinhos in the State of Paraíba in Brazil. Sisal leaves provide long fibers, of great resistance and durability. The chemical composition of the sisal fiber is as follows: cellulose (65.8%), hemicellulose (12.0%) and lignin (9.9%) and others [6, 20, 34].

The fibers of caroá and macambira were washed with running water, allowed to air dry at room temperature for at least 72 h before being combed and cut up to 5 cm.

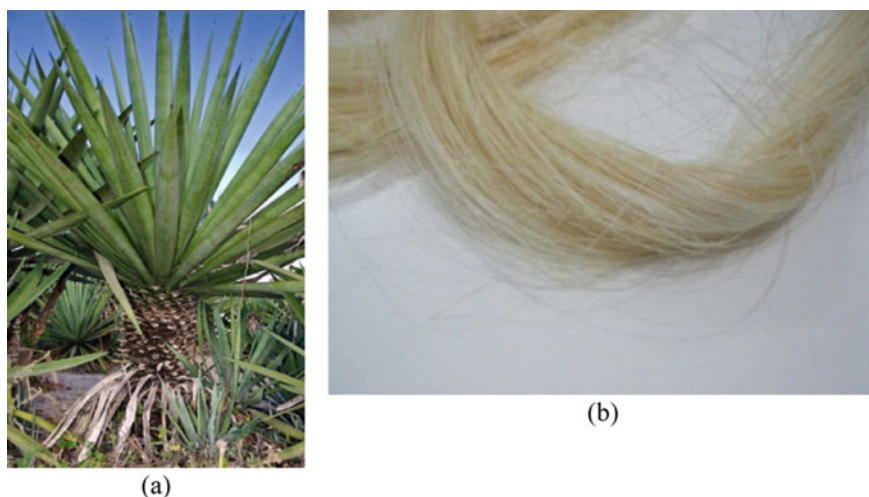


Fig. 3.3 Sisal (*Agave sisalana*). **a** Plant and **b** fibers

The sisal fibers were dried in an air circulation oven operating at 60 °C until constant weight and subsequently heated at 105 °C for 24 h, in order to obtain the dry fiber. The dried sisal fibers were cut in 35 mm lengths prior to use. Unsaturated polyester cured with 1% MEK (methyl ethyl ketone) was used as the matrix.

Composites with varying fiber contents were compression molded. Since the thermal degradation of natural fibers and thermoset or thermoplastic polymers occurs at upper temperature limits, composite samples were made at low temperature. A fiber mat was produced by randomly distributing a pre-determined amount of fibers in steel molds ($220 \times 180 \times 3$ mm and $220 \times 180 \times 6$ mm) and compressed with 2 ton for 5 min at room temperature. The mats thus produced were removed from the mold for further use as reinforcement. An appropriate quantity of polyester resin was mixed with the catalyst and a small amount was poured onto the mold. The fiber mats were placed in the steel mold, impregnated with more resin and the mold was closed (8 ton).

The system was allowed to cure under pressure for 4 h at room temperature before the composite plate was removed from the mold. The caroá and macabira reinforced composite plates obtained were post-cured in an air circulating oven operating at 50 °C for 48 h (Fig. 3.4), while sisal composite plates were post-cured in an air circulating oven operating at 60 °C for 48 h.

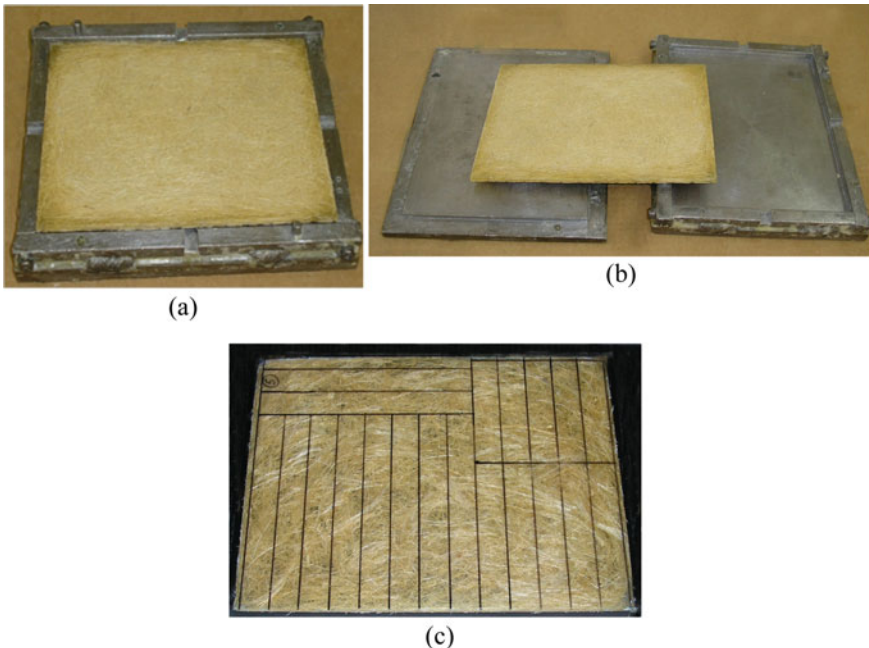


Fig. 3.4 Manufacturing of caroá fiber-reinforced polymer composite by hand lay up technique. **a** Fiber mat in the mold, **b** Demolding, and **c** cut samples

Tests for mechanical properties were conducted according to ASTM standards D-3039 and D-256 for tensile and impact tests (Fig. 3.5a–b), respectively. Composites samples of 20 × 20 mm were cut-off from the plates, and their edges were sealed with resin prior to the water absorption tests (to avoid water transport by capillarity) and dried in an air-circulating oven at 105 °C to constant weight or dry mass (Fig. 3.5c).

The water absorption experiments were carried out according to the following procedure. Firstly, the pre-dried composites samples were fully immersed in water baths (Fig. 3.6) kept at 25, 50 and 70 °C. At regular time intervals, the samples were removed from the water bath; pat-dried with a paper tissue to remove surface water

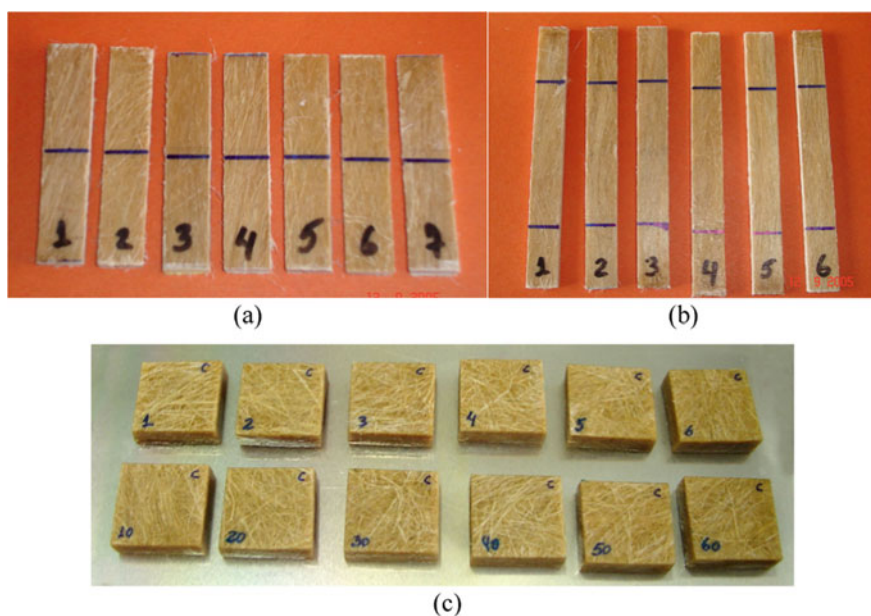


Fig. 3.5 Caroá fiber-reinforced polymer composite samples used at different test. **a** Impact, **b** Tensile and **c** Water sorption



Fig. 3.6 **a** Caroá and macambira composite specimens and **b** Macambira and caroá composite samples in water bath

and immediately weighted in a digital scale (uncertainty ± 0.001 g). The samples were re-immersed in the water bath and the procedure repeated so that the water sorption process continued until equilibrium was reached. Each measurement took less than 1 min, so water evaporation at the surface was deemed insignificant.

The results of absorbed moisture were reported as mass of absorbed water per unit of dry composites mass. Thus, moisture content was computed as follows:

$$\bar{M}(t) = \frac{m_t - m_0}{m_0} \times 100\% \quad (3.1)$$

where m_0 and m_t represent the dry mass of the composites samples ($t = 0$) and the wet mass at any specific time t , respectively.

Saturation (equilibrium) condition was assumed when the daily weight gain of the composite samples was less than 0.1%. We notice that complete immersion of composite samples in water bath constitutes the most severe physical situation; exposure to humid air results in lower equilibrium moisture content.

3.3 Experimental Results Analysis

3.3.1 Morphology Characterization of the Fibers

The microstructure of vegetable fibers is complex, and dependent on each kind of the fiber. In this research some single caroá, macambira and sisal fiber samples (untreated) were analyzed by scanning electron microscopy (SEM), in order to characterize their morphology. Figures 3.7, 3.8 and 3.9 present micrographs (transversal section) of macambira, caroá and sisal fibers in the natural state, respectively.

Analysis of these figures shows that the arrangement is similar to other natural fibers, with a spongy aspect and thin, compacted cellular arrangement (regular fibrillar arrangement). In some places is verified the presence of small amount of organic material at the fiber surface, probably a residue left when the fibers were extracted from the leaves of the each plant.

Images obtained from SEM also showed rougher surfaces and many void spaces, which implies that macambira, caroá and sisal fibers could be adequate to be used as reinforcement in composite materials; this surface condition allows good adhesion between fibers and polymer matrix. However, it is important to state that, after treatment or mechanical characterization, the fibers achieve some degradation, and impurities existing at the surface are minimized. Furthermore, since cellulose is the major constituent with a crystalline structure, it is the main contributor towards the mechanical properties.

From the viewpoint of water absorption, cellulose fibers are difficult to dissolve because of their high crystallinity, but, they tend to retain liquids in the interfibrillar space [35].

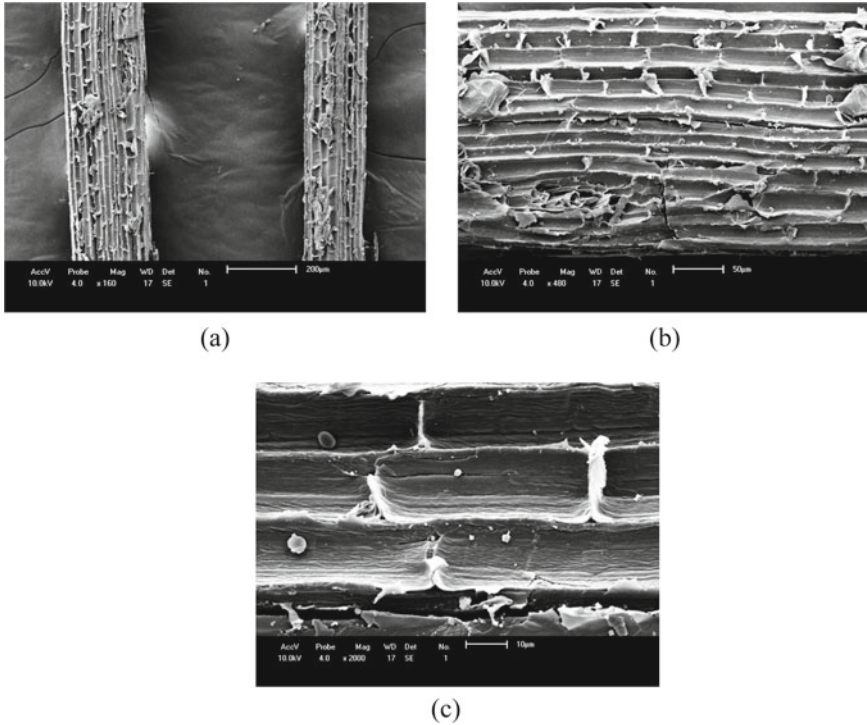


Fig. 3.7 SEM micrographs for untreated macambira fiber: **a** 160 \times , **b** 480 \times and **c** 2000 \times

3.3.2 Water Absorption Kinetics

Water sorption curves of unsaturated polyester/macambira composites, unsaturated polyester/caroá composites and unsaturated polyester/sisal composites are illustrated in Figs. 3.10 and 3.11. Results are reported for different dimensions of the samples (3 mm and 6 mm thickness) and water bath temperatures (25, 50 and 70 °C) as function of exposure (immersion) time. Upon analyzing of these figures it can be seen that the general shape of the curves is similar to those of others natural fiber-reinforced polymer matrix composites.

For all investigated composites the moisture content increases monotonically with water immersion time until it reaches a maximum value, the so-called equilibrium moisture content (saturation or hygroscopic condition). This behavior strongly indicates that the vegetable fibers were uniformly distributed in the matrix, as reported in the literature [2]. Figures 3.10, 3.11 and 3.12 also show that water sorption for all composites increase with the area/volume ratio and temperature (Table 3.1). Sample size effect was more pronounced at the lower temperature.

Table 3.1 summarizes the saturation data for all tested composites and for neat polyester. The data indicates that water absorption for the composites is higher than

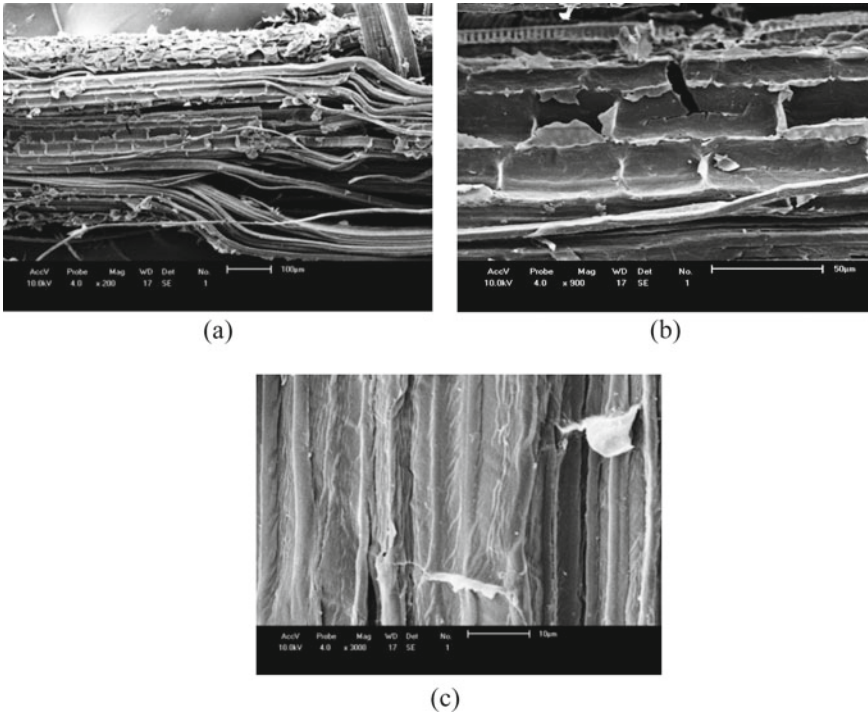


Fig. 3.8 SEM micrographs for untreated caroá fiber: **a** 200 \times , **b** 900 \times and **c** 3000 \times

for the matrix. Neat polyester shows a very low percentage of absorbed water. While the water sorption at the equilibrium condition for the unsaturated polyester is about 1%, for the composites it ranged from 14.04 to 18.09%. It was verified that the weight gain of pure polyester was almost insignificant, indicating minimal degradation. These results are consistent with studies reported in the literature for similar systems [2, 8, 10, 13, 36]. Some authors attribute the increase in the water sorption by polymer composites reinforced with vegetable fiber not only to the hydrophilic nature and the permeability of this type of reinforcement but also to the sample surface area exposed to water. Capillarity effects and the interfacial area between fiber and matrix may also contribute to water pick-up by these composites.

Results indicate that the water uptake of the composite sample immersed in a water bath at 70 °C was faster than under the other (25 and 50 °C) experimental conditions. This behavior is attributed to the increased water mobility within the solid at higher temperature. It is believed that higher water temperatures lead to thermal dilation of the composites and to increased composite porosity, which would, in turn, cause a faster moisture migration. Temperature activates the water diffusion process inside the sample, and sorption rate increases with the increases in temperature (thermo-activation).

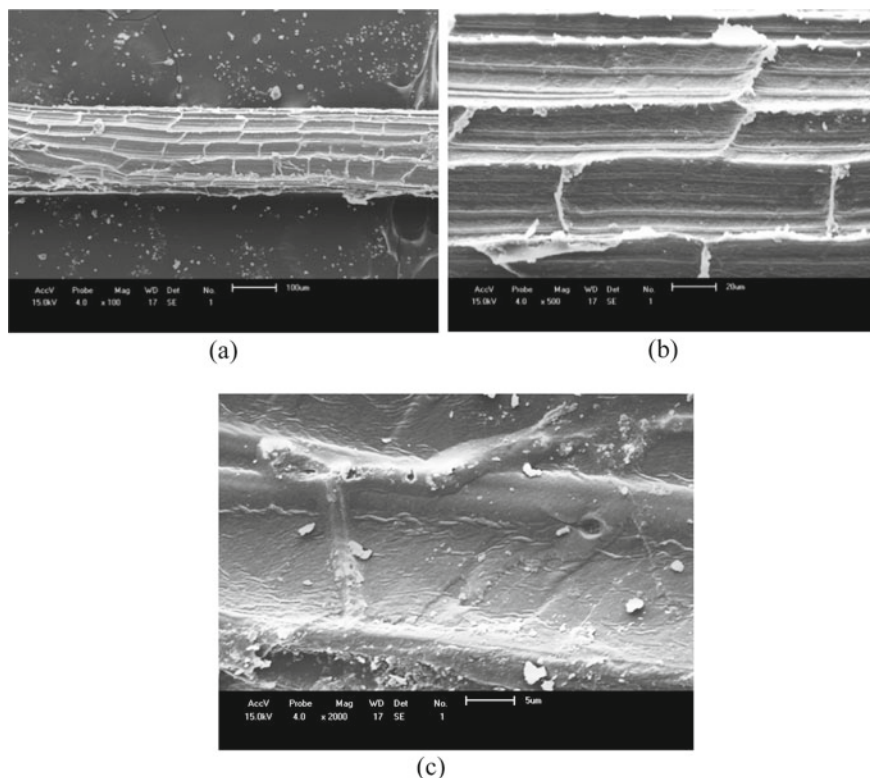


Fig. 3.9 SEM micrographs for untreated sisal fiber: **a** 100×, **b** 500× and **c** 2000×

These results are consistent with those reported in similar systems for unsaturated polyester/caroá composites [8, 31], unsaturated polyester/jute composites [36] and unsaturated polypropylene/sisal composites [35]. Other aspects were not analyzed, such as fiber size and orientation (events that occur in long fiber reinforced composites are different those observed in short fiber reinforced composites) [1], and water migration by capillarity into micro cracks inside the solid, mainly in the fiber-matrix interface where adhesion plays an important role. Reading reported works on these topics is strongly recommended.

3.3.3 Mechanical Properties

As already mentioned in the previous sections, the mechanical performance of the composites depends on the properties of both reinforcement and matrix, and the adhesion between them after manufacturing. Figure 3.13 show the results of tensile strength of the caroá fiber-reinforced polymer composites (in dry condition) as a

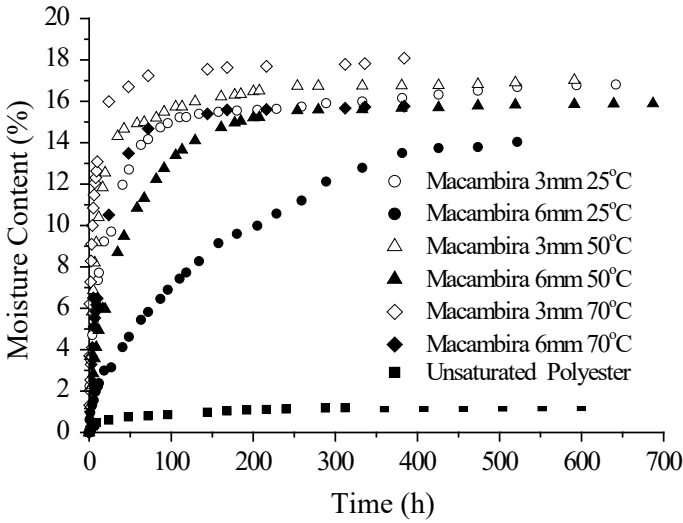


Fig. 3.10 Water sorption kinetics of macambira fiber-reinforced unsaturated polyester composites (30% w/w fiber content)

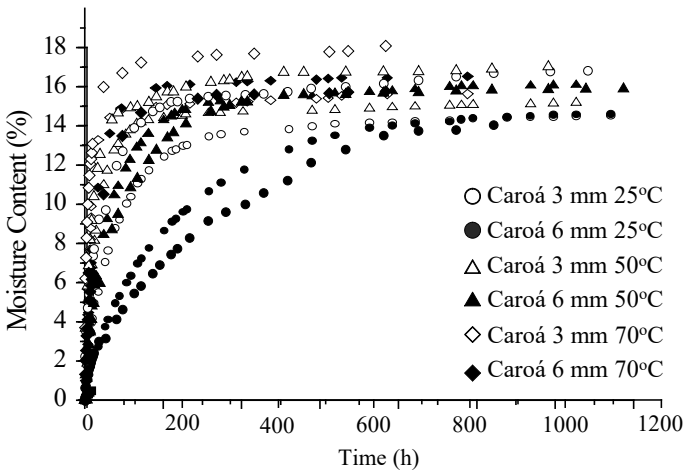


Fig. 3.11 Water sorption kinetics of caroá fiber-reinforced unsaturated polyester composites (30% w/w fiber content)

function of fiber content. Analysis of this figure, shows that the tensile strength of the composite is lower than that of the matrix for loadings up to 25% by weight for caroá fibers. This behavior can be attributed to inefficient loading, so the fibers act as defects (stress concentrators) and effectively weaken the matrix, resulting in a composite with lower mechanical strength. At low fiber content, the matrix is not sufficiently fixed

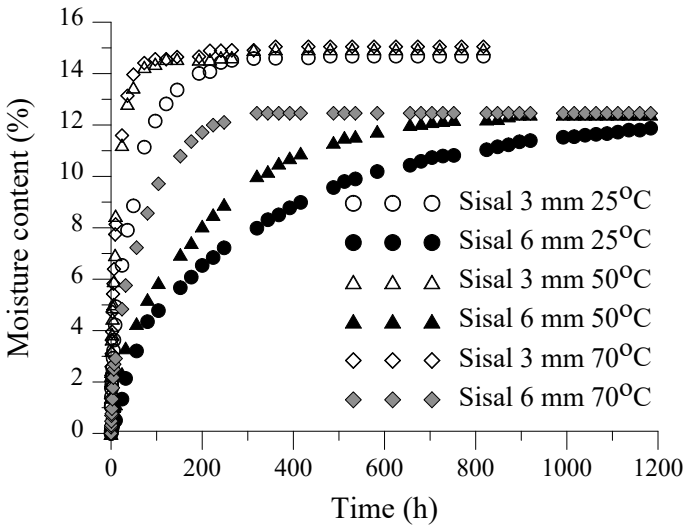


Fig. 3.12 Water sorption kinetics of sisal fiber-reinforced unsaturated polyester composites (46.6% w/w fiber content)

(poor adhesion) and high deformations imposed on it lead to a break of the matrix-fiber bond. As the loading increases, stresses are more uniformly distributed and the composite’s strength increases. Obviously this behavior is dependent on the critical volume of the fiber into the matrix.

At loadings above 40% by weight, the tensile strength of the composites decreases with a further increase in fiber content. This behavior associated with fiber agglomeration and poor impregnation, which leads to an increase in their effective diameter, a decrease in their aspect ratio and to void formation.

Figure 3.14 illustrates the effect of the fiber content on the elastic modulus of the caroá fiber-reinforced polymer composite composites (in dry condition). The results show that fiber incorporation increases the stiffness of the composite. This behavior was expected, since the strength and Young’s modulus of the fibers used are higher than the matrix. In addition, fiber addition minimizes the movement of polymer chains resulting in an increase in the elasticity modulus of the composite with fiber content.

Figure 3.15 displays the impact resistance of the caroá fiber-reinforced polymer composite as a function of fiber content (in dry condition). Upon analyzing this figure, it can be verified that impact strength increases with fiber content at all loading levels for the type of fiber tested. Based on the results, we state that the caroá fiber is able to diverge crack propagation and delay breakage, thus increasing impact strength. It is worth noticing that impact properties are not as dependent on fiber/matrix adhesion as the tensile strength and, in fact, this mechanical property often is improved for physical situations with looser interfaces.

Table 3.1 Moisture content and geometric data for the water sorption in vegetable fiber reinforced composites (30% w/w fiber contents)

Sample	Thickness (mm)	T _w (°C)	t (h)	M _t (%) (d. b.) ($t \rightarrow \infty$)	Area/Volume (mm ² /mm ³) ($t = 0$)
Polyester matrix	3.00	25	600	1.26	866.67
Macambira composite	3.00	25	642	16.81	866.67
	6.00	25	594	14.04	533.33
	3.00	50	687	17.02	866.67
	6.00	50	687	15.89	533.33
	3.00	70	384	18.09	866.67
	6.00	70	648	16.18	533.33
Caroá composite	3.00	25	1100	14.49	866.67
	6.00	25	1623	14.81	533.33
	3.00	50	1022	15.16	866.67
	6.00	50	1022	16.08	533.33
	3.00	70	796	15.61	866.67
	6.00	70	796	16.52	533.33
Sisal composite	3.00	25	817	14.68	533.33
	6.00	25	1328	12.27	866.67
	3.00	50	817	14.96	533.33
	6.00	50	848	12.42	866.67
	3.00	70	817	15.05	533.33
	6.00	70	848	12.46	866.67

Figure 3.16 shows the elongation at break of the caroá fiber-reinforced polyester composite as function of fiber content (in dry condition). It can be observed that elongation at break increases with fiber incorporation the incorporation of fiber increases the properties of elongation at fiber contents above 23%. This behavior is attributed to the greater stiffness of the fiber, thus, increasing the stiffness of the composite.

Since moisture absorption affects the mechanical performance of fiber-reinforced polymer composites, results of the mechanical tests of sisal fiber-reinforced polymer composites are shown in Figs. 3.17, 3.18, 3.19 and 3.20, as reported by Santos [20] and Santos et al. [37]. These results were obtained with 3 and 6 mm thick samples. Saturation conditions at different water bath temperatures are cited in Table 3.1.

Analysis of these figures indicate that tensile strength, impact strength, Young's modulus and elongation at break of the composites decrease with the increasing water bath temperature. This behavior can probably be associated with the fact that an increase in water bath temperature provokes deterioration and dilation of the samples and, thus, increasing moisture rate and facilitating water migration to inside the composite and, thus leading to a decrease in mechanical properties. The effect of sample thickness also is evident.

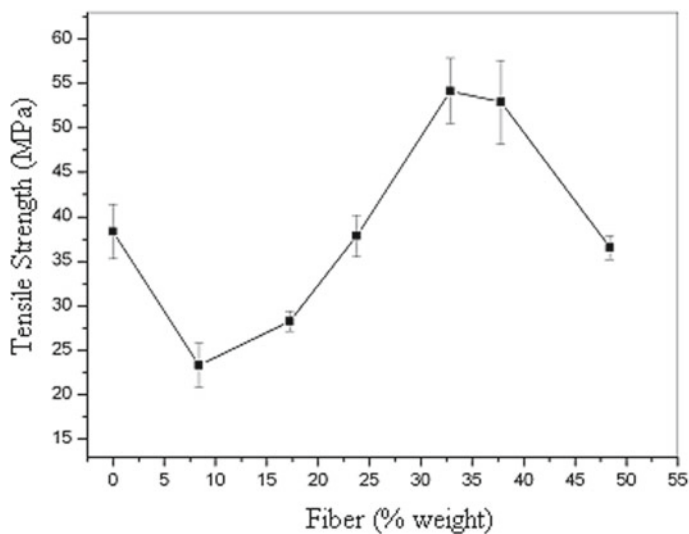


Fig. 3.13 Tensile strength of the caroá fiber-reinforced polymer composite as a function of the fiber content

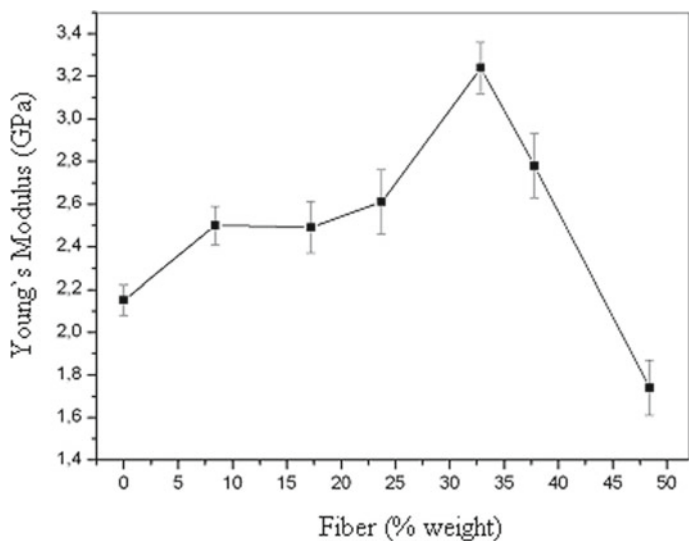


Fig. 3.14 Young's modulus of caroá fiber-reinforced polymer composite as a function of the fiber content

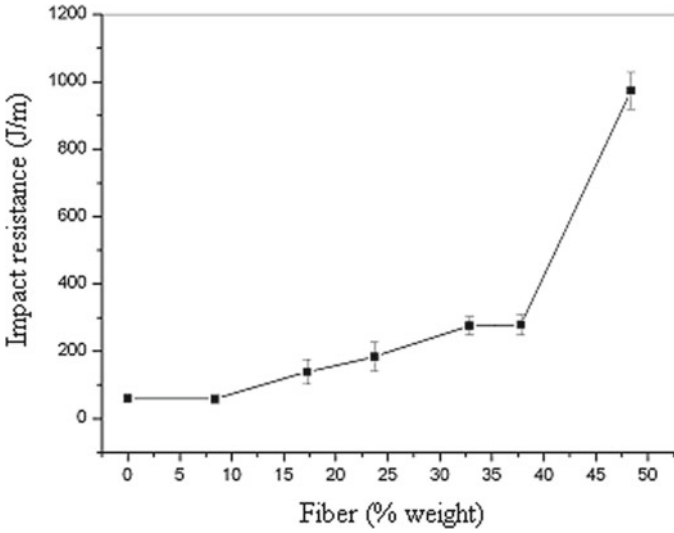


Fig. 3.15 Impact resistance of caroá fiber-reinforced polymer composite as a function of the fiber content

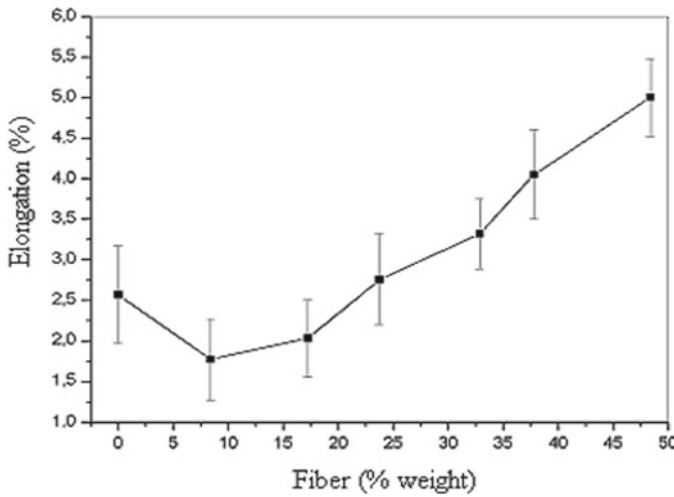


Fig. 3.16 Elongation at break of caroá fiber-reinforced polymer composite as a function of fiber content

Furthermore, it is important to notice that the decrease in mechanical property values of the composite are associated with the poor fiber-matrix interfacial adhesion caused by water absorption as previously discussed [14].

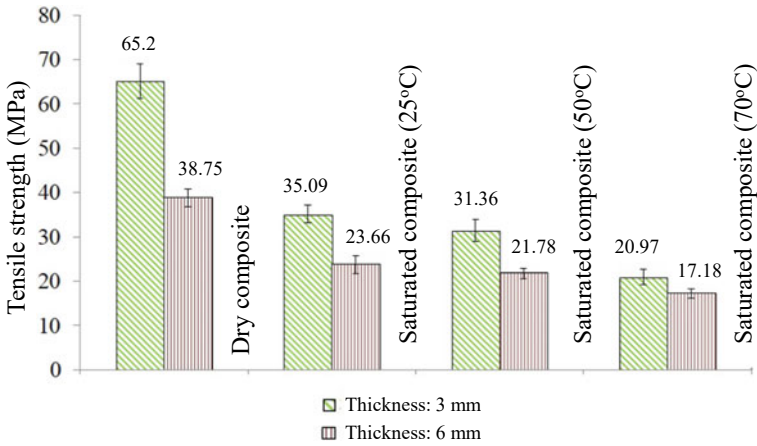


Fig. 3.17 Tensile strength of polyester composite (44.6% sisal fiber)

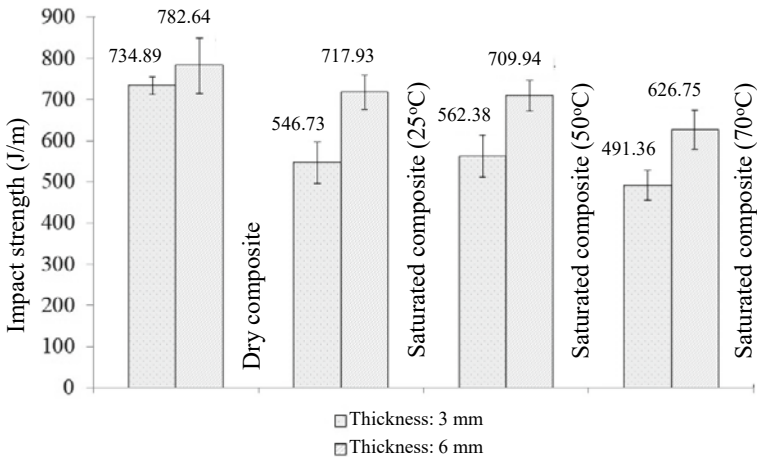


Fig. 3.18 Impact resistance of polyester composite (44.6% sisal fiber)

Different methods to improve fiber/matrix interfacial adhesion have been used in order to minimize this problem. In general, these methods are based on chemical treatment or by addition of coupling agent, as reported by Du et al. [38]. However, drying of vegetable fibers as a pre-treatment has been used as well [39]. Carvalho et al. [39] reported an experimental study about tensile properties of hybrid jute/cotton and sisal/cotton fabrics–polyester matrix composites. According to the authors, the amount of absorbed water on the composites was effectively reduced when the fabrics were pre-dried before their incorporation into the composites. Furthermore, it was verified that composites manufactured with pre-dried fabrics showed smaller strain at break, indicating a better fabric-matrix interaction.

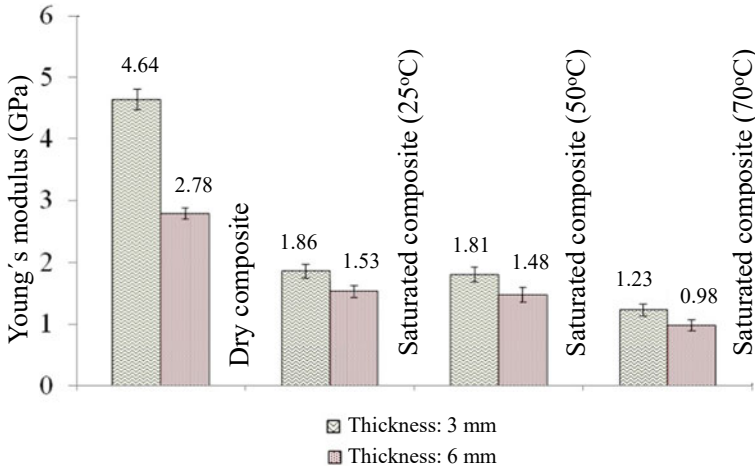


Fig. 3.19 Young's modulus of polyester composite (44.6% sisal fiber)

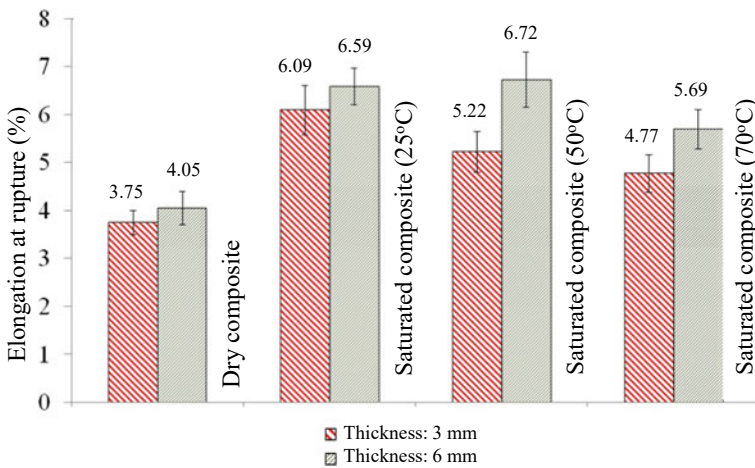


Fig. 3.20 Elongation at break (b) of polyester composite (44.6% sisal fiber)

Figures 3.21 and 3.22 illustrate the polyester-sisal samples with 3 mm and 6 mm thickness in the saturation condition at water bath temperature of 25 °C and 70 °C, respectively [20]. These figures show an increase in volume, as well as the deterioration of the composite samples, both due to the thermal effect and the presence of moisture. These effects are most severe at higher water bath temperature.

For composites with 3 mm thickness, there was an increase in volume of 19.77%, 21.77% and 31.34% for temperatures 25 °C, 50 °C and 70 °C, respectively. For composites with a thickness of 6 mm, the increase in volume was 15.95%, 17.25% and 21.32% for the same temperature range. The increase in volume of the samples was

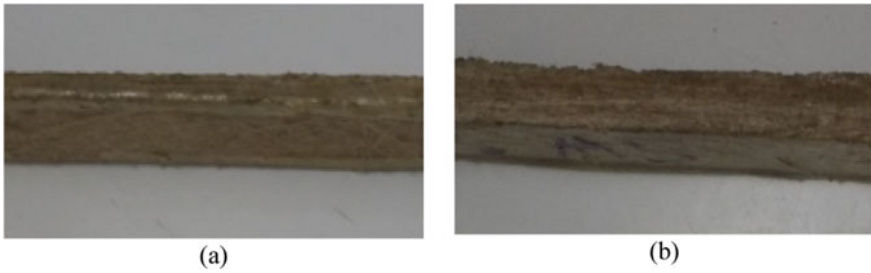


Fig. 3.21 Sisal fiber-reinforced polyester composite (44.6% sisal fiber) at saturation condition ($T = 25\text{ }^{\circ}\text{C}$). **a** 3 mm and **b** 6 mm thickness

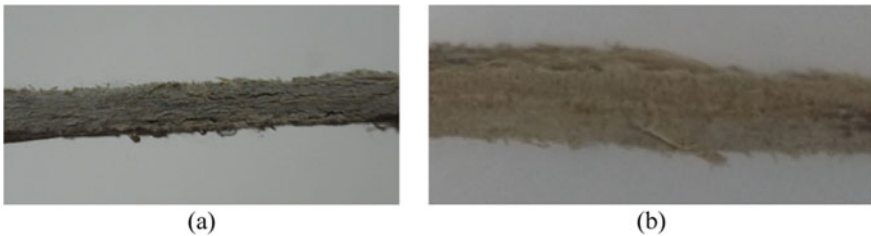


Fig. 3.22 Sisal fiber-reinforced polyester composite (44.6% sisal fiber) at saturation condition ($T = 70\text{ }^{\circ}\text{C}$). **a** 3 mm and **b** 6 mm thickness

greater than the maximum moisture content absorbed, which probably can be associated with the combined effect of the bath temperature and moisture that accelerates the sample deterioration.

With the aim to better identify the interactions between sisal fiber and polyester matrix (compatibility and dispersion), a SEM experimental study was carried out on tensile tested samples. Figures 3.23 and 3.24 illustrate the SEM micrographs of fractured surface of 3 mm and 6 mm thick untreated sisal fiber-reinforced polymer composite samples, respectively, in the saturation condition at water bath temperature $70\text{ }^{\circ}\text{C}$ [20]. It is possible to verify that, due to the random arrangement of the short fibers and severe experimental conditions, a moderate interfacial adhesion was obtained in the composite manufactured. The main failure mechanisms in the composite samples were debonding and fiber breakage, both due to the thermal effect and the presence of moisture, as highlighted in these figures. Other phenomena such as fiber splitting, brittle surface and pull-out were also verified.

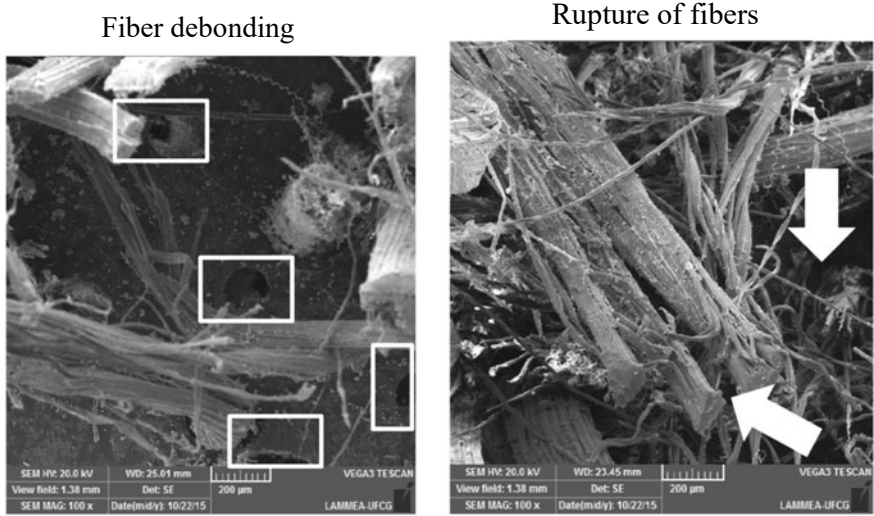


Fig. 3.23 SEM Micrographs (100X) of fractured surface of untreated sisal fiber-reinforced polyester composite (saturation condition at 70 °C and 3 mm thickness)

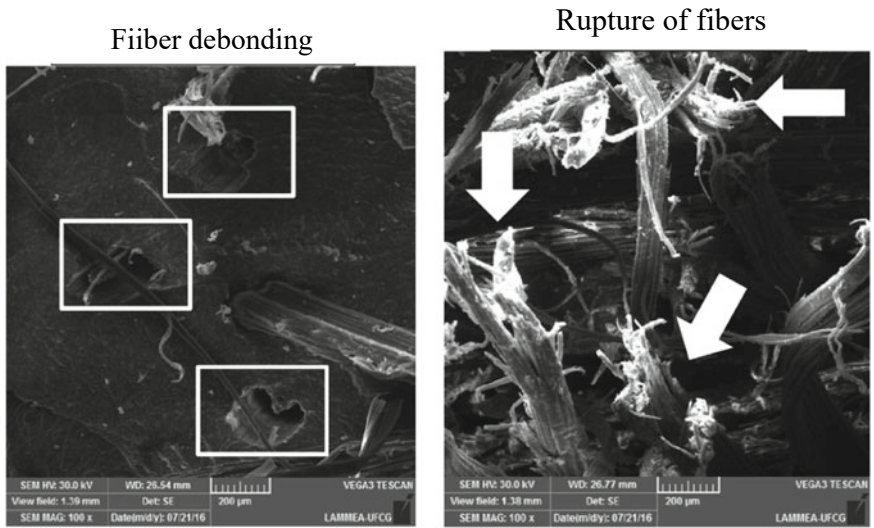


Fig. 3.24 SEM Micrographs (100X) of fractured surface of untreated sisal fiber-reinforced polyester composite (saturation condition at 70 °C and 6 mm thickness)

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