Moisture Transport Process in Vegetable Fiber Composites: Theory and Analysis for Technological Applications

L. H. Carvalho, E. L. Canedo, S. R. Farias Neto, A. G. Barbosa de Lima and C. J. Silva

Abstract This chapter provides theoretical and experimental information about water absorption in unsaturated polyester polymer composites reinforced with vegetable fibers. The use of raw materials from renewable sources, such as natural fibers, has shown great promise in a variety of engineering applications. Composites reinforced with natural fibers are sensitive to influences from environmental agents such as water and temperature. The organic nature of vegetable fibers is responsible for the higher moisture sensitivity of the mechanical properties of natural fiber reinforced composites when compared to synthetic fiber reinforced composites. Here, topics related to theory, experiments, mathematical modeling and numerical procedures, and technological applications for different natural fibers are presented and discussed in detail. Results of microscopy, water absorption kinetics, moisture content distribution, and area/volume relationships for unsaturated polyester composites reinforced with caroá and macambira vegetable fibers are shown and analyzed. The knowledge of moisture distribution allows the determination of areas that may show delamination problems (moisture

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induced degradation) due to the weakness of the fiber-matrix interface and consequently reduction in the composites mechanical properties.

Keywords Polymer composite · Vegetable fiber · Water sorption · Modeling · Simulation · Experimentation

1 Introduction

Composites are multiphase materials consisting of one or more discontinuous phases (reinforcing filler) embedded in a continuous phase (matrix) [1]. The matrix phase can be metallic, ceramic, or polymeric, while the reinforcement can be synthetic fiber (glass, carbon, aramid, etc.) or natural fiber (sisal, ramie, jute, cotton, kenaf, pineapple, etc.) or particles (clay, mica, tungsten carbide, titanium carbide, etc.).

Polymer matrices can be classified as thermosets (epoxy, polyester, phenolic, silicone, polyimide, etc.) or thermoplastics (polyethylene, polystyrene, nylons, polycarbonate, polyether-ether ketone, polyphenylene sulfide, etc.). These matrices are most widely used due to their moderate cost, easy processibility, good chemical resistance, and low density [1]. The reinforcing material is usually harder and stronger than the matrix. Thus, composite properties are strongly dependents of the properties of their constituent (matrix and reinforcement). The goal is to achieve the best properties for a given application. The shape, size, distribution, content and filler orientation as well as the matrix/filler interfacial bonding strongly affect the initial and long term properties (hydro, thermal, electrical and mechanical) of the composites whose performance can also be affected by environmental exposure resulting in volumetric variations (or swelling)

Due to their small cross-sectional dimensions, fibers and isometric particulate materials are seldom directly used in engineering applications. For these applications they are embedded in a polymer matrix, for example, in order to form fibrous composites, or ceramic particles are embedded in a polymer matrix to form particulate composites. In general, composite materials can be classified as fiber-reinforced composites (fibrous composites) and particle-reinforced composites (particulate composites). Figure 1 illustrates a scheme for the classification and types of composite materials.

Composite materials are used in aerospace, underwater, biomedical and transportation specific applications as well as in high-tech sporting equipment.

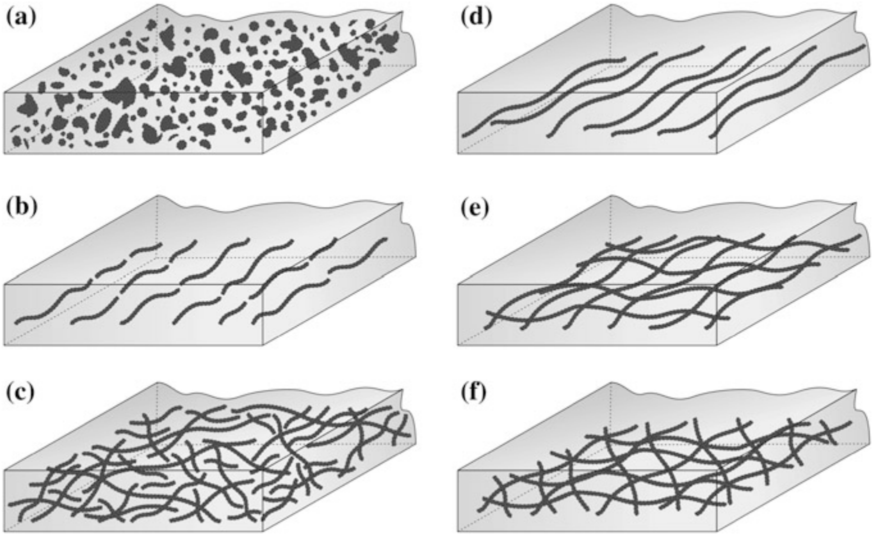


Fig. 1 Classification and types of composite materials. **a** Dispersed particle-reinforced, **b** discontinuous fiber-reinforced (aligned), **c** discontinuous fiber-reinforced (randomly oriented), **d** continuous fiber-reinforced (aligned), **e** continuous fiber-reinforced (aligned 0°–90° fiber orientation angle), and **f** continuous fiber-reinforced (multidirectional fiber orientation angle)

2 Fiber-Reinforced Composites: Biocomposites

2.1 Basic Concepts

Natural fibres are subdivided based on their origin, as coming from plants, animals or minerals. Plant fibres are composed of cellulose while animal fibres consist of proteins (hair, silk, and wool). Plant fibres are derived from renewable resources and are classified according to their origin as bast or soft fiber, leaf or hard fibers, seed, fruit, wood, cereal straw, and other grass fibers [2].

The major constituents of vegetable fibers are cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances. Plant fibers are also known as lignocellulosic fibers or biofibers and can be considered as naturally occurring composites of cellulose fibrils embedded in a lignin matrix [3]. The cellulose fibrils are aligned along the length of the fiber, which renders maximum tensile and flexural strengths, in addition to stiffness. Vegetable fibers can be used as reinforcing fillers for polymer composites and their reinforcing efficiency is related to the fiber nature and cellulose content and to its crystallinity. The structure, microfibrillar angle, cell dimensions, defects, and fiber chemical composition are important variables that determine the overall properties of the fibres [4]. Generally, tensile strength and Young's modulus of fibers increase with increasing cellulose content, while microfibrillar angle determines the stiffness of the fibers.

The fibers will be rigid, inflexible and have high tensile strength if the microfibrils are oriented parallel to the fiber axis and more ductile if the microfibrils have a spiral orientation with respect to the fiber axis [2].

More recently, environmental concerns as well as very stringent regulations and standards have prompted the industries to research and use more eco-friendly and/or sustainable processing methods or products. In order to achieve this goal, available manufacturing processes are modified or adapted to comply with these regulations, alternative technologies are developed or else renewable biomaterials and biofibers are used to generate composite materials. Broadly defined, biocomposites are composite materials made from natural/bio fibers; the polymer matrix may be petroleum or naturally derived (biopolymer), and the former may be biodegradable or not. Biocomposites derived from plant fibers and biopolymers are likely to be more eco-friendly and such composites are termed as green composites. While synthetic matrix/plant fiber composites are not as ecofriendly, they are still more environmental friendly than an all-synthetic material.

Vegetable fibers are biodegradable, sustainable and renewable, carbon dioxide neutral and have a positive environmental impact, as they are nontoxic and nonabrasive to mixing and molding equipment, leading to significant production cost reductions. Thus, plant fibers are a choice material for biocomposite production due to their worldwide availability, economical production, safer handling and working conditions compared to synthetic reinforcements, good set of mechanical properties, low cost per volume, low density and high relative strength and stiffness, which results in composites with specific strength and stiffness comparable to that of glass reinforced ones. Their main disadvantages for polymer composite applications are related to their high moisture sorption, relatively low processing temperatures ($\leq$200 °C), low resistance to microorganisms and poor fiber/polymer adhesion to most polymer matrices, which ends up by compromising the composite's immediate and long term mechanical performance [2].

Since composite materials are widely used as a technological solution for the development of new products, environmental and economic concerns have pointed out to the use of vegetable fibers instead of synthetic ones (such as glass or carbon) as polymer reinforcements. The development of biocomposites is economically sound and allow for the development of environmental friendly materials with high value-added [5, 6].

According to Zhang et al. [5], more recently, several researches have pointed to the use of plant fiber with biodegradable polymers based on food, and feed such as starch plastics, soybean plastics and cellulosic plastics for producing fully biodegradable composites. As a result of intense research, reinforced-fiber polymer composites now have acceptable properties and thus being used in a large variety of industrial applications that were dominated by synthetic fibers, such as automobile and packaging industries, structural applications with modest demand on strength reliability, and lightweight construction. As construction material these composites can be applied in decking and railing products. Besides, biocomposites can be used for biomedical and orthopedic applications (bioengineering).

2.2 Performance of Fiber Composites

Despite their attractiveness, natural fiber reinforced polymer matrix composites are very sensitive to influences from external environmental agents such as water (in the liquid or vapor phases), relative humidity, temperature, radiation (X-rays, β and γ rays, ultraviolet), chemical agents or a combination between them [1, 6, 7]. They are also sensitive to internal factors such as fiber volume fraction and orientation. Moisture in any form is deleterious to polymer composites, especially to those reinforced by natural fibers. Vegetable fibers are hydrophilic and fiber moisture not only acts a plasticizer but makes polymer impregnation more difficult, causing weak adhesion on the polymer matrix-fiber interface, which leads to internal tensions, porosity and premature failure of the system [8–10]. Biocomposites display lower mechanical properties than synthetic fiber-reinforced composites as water sorption adversely affects the performance, physical and mechanical integrity of the composites. Thus, knowing the effect of moisture on the composite properties is fundamental for outdoor applications. The incompatibilities between natural fiber reinforced composites and water may be diminished by surface modification (chemical treatment) of the fiber or the matrix. In general it is the fiber, not the matrix, that is treated [11].

Several studies show that the mechanical properties of vegetable fiber reinforced composites significantly improve at high fiber content [12–14]. However, when fibrous polymer composites absorb moisture they suffer swelling, plasticizing, dissolving, leaching and/or hydrolyzing, resulting in discoloration, embrittlement, lower resistance to heat and weathering and lower mechanical properties. The amount of water absorbed by a sample varies as a function of its composition, dimensions, void fraction (available free volume), temperature, surface area, surface protection, and exposure time. The effects of moisture and temperature of composites on several performance parameters, such as tensile and shear strengths, elastic moduli, fatigue behavior, creep, rupture stress, response to dynamic impact, and electrical resistance, has been investigated [1]. Due to their importance, several works on the water sorption kinetics of vegetable fiber reinforced polymer composites are reported in the literature [15, 16].

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, the 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, the hydric, thermal and mechanical events (in a microscale analysis) occurring in short fiber reinforced composites are very different than those verified for a long fiber reinforced composite [17].

All the factors cited before limit the field of application of vegetable reinforced polymer composites [18–25] and new research in this area is required. For this reason, several works on the absorption kinetics of water and/or moisture in composite systems presenting analytical and/or numerical solutions are reported in the literature. However, all these works consider the diffusion through the solid to be one-dimensional [26–33]. Yao and Ziegmann [31] related the water absorption behavior of three types of glass-fiber reinforced plastic (GRP) pipes. A model was developed from Fick's law to predict water diffusion curves of the GRP pipe specimens. It was found that only one GPR pipe specimens exhibited near-Fickian behavior and the other two behaved like a two-stage diffusion process because of the abnormal non-Fickian process. Srihari et al. [30] concluded in their studies that the composite (glass/epoxy) and neat resin casting specimens immersed in distilled water and artificial seawater exhibited Fickian behavior. Najafi et al. [32] studied water absorption of wood plastic composites. They concluded that water absorption in these composites follows the kinetics of a Fickian diffusion process. Czél and Czigány [34] in their study of water absorption of glass fiber/polyester composite pipes concluded that the Fickian diffusion model did not fit the absorption curves, but a special explicit, asymptotically correct function based on the Lucas-Washburn equation was found to approximate the water uptake process.

In some cases, in order to take into consideration three-dimensional problems, some researchers use analytical solutions applicable for long times which greatly simplify the problem [35–37]. Chateauminois et al. [35] studied the water diffusion in solids with parallelepiped shape using the short term solution of the one-dimensional Fick's law. For longer times, the water absorption kinetics was computed using the three-dimensional solution of Fick's law. Pavan et al. [36] in their studies on moisture diffusion behavior of glass/epoxy composites found that the diffusion parameters obtained from the experiment conducted up to saturation limits agreed well with the data computed. Bao and Yee [37] showed in their studies of moisture absorption based on long term absorption data that the fast Fickian diffusion is followed by a slow gradual increase in weight gain. This two-step diffusion behavior can be successfully described by the proposed two-stage diffusion model. In this two-stage model, the first and second stage of diffusion are assumed to be diffusion and relaxation-controlled, respectively.

For a more general formulation, Cavalcanti et al. [21] and Cavalcanti [38] report a three-dimensional mathematical modeling approach to describe moisture absorption in an unsaturated polyester composite reinforced by a hybrid jute/glass fabric. Predicted results on the average moisture constant inside the material during the whole process were shown and compared with experimental data, in order to validate the model and to obtain the diffusion coefficient. According to the authors a good agreement was obtained.

Due to the importance of accurately describing the water sorption phenomena in vegetable fiber reinforced polymer composites, the present work deals with the development and study of water sorption kinetics of unsaturated polyester composites reinforced with short fibers of macambira (*Bromelia Laciniosa*) and caroá (*Neoglaziovia Variegata*) fibers. Herein, experimental and theoretical (three-dimensional) studies about water absorption as a function of sample dimensions (rectangular parallelepiped shape) and temperature was conducted.

3 Water Diffusion in Polymer Composites

3.1 Experimental Study

Nóbrega [24] and Nóbrega et al. [39] conducted several experiments on the water absorption of caroá fiber reinforced unsaturated polyester composites, while Cruz et al. [40] conducted similar experiments with macambira fiber reinforced unsaturated polyester composites. The unsaturated polyester was cured with 1 % MEK (methyl ethyl ketone).

The macambira fibers (Fig. 2) were obtained from the Cariri region of Paraíba State (Brazil) while the caroá fibers (Fig. 3) were obtained at a farm in Pocinhos, Paraíba State (Brazil). The fibers 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.

Composites with varying fiber content were compression molded. A fiber mat was produced by randomly distributing a pre-determined amount of fibers in steel molds (220 × 180 × 3 mm and 220 × 180 × 6 mm) and compressing with two ton for 5 min at room temperature. Following the mats were removed from the mold for further use. 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 to remove the composite plate from the mold. The composite plates obtained were post-cured in an oven at 50 °C for 48 h. Tests for mechanical properties were conducted according to ASTM standards D-3039 for tensile and D-256 for impact tests. Composites samples of 20 × 20 mm were cut-off from these plates, and their edges sealed with resin prior to the water absorption test (to avoid water transport by capillarity) and dried in an air-circulating oven at 105 °C (to constant weight or dry mass).

The water absorption experiments were carried out according to the following procedure. Firstly, the pre-dried composites samples were immersed fully into a

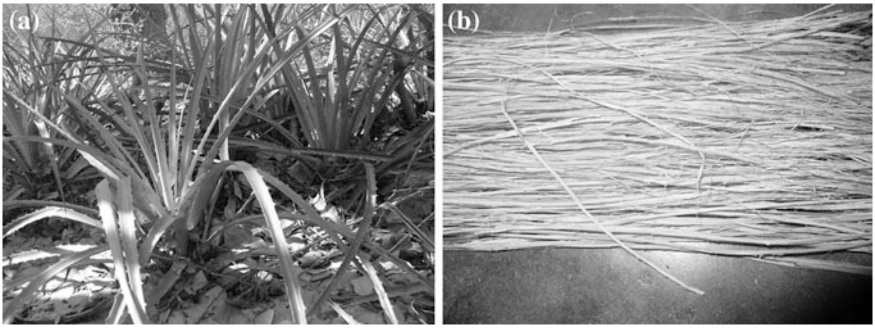


Fig. 2 Macambira (*Bromelia Laciniosa*). **a** Plant and **b** fibers

Fig. 3 Caroá (*Neoglaziovia Variegata*). **a** Plant and **b** fibers

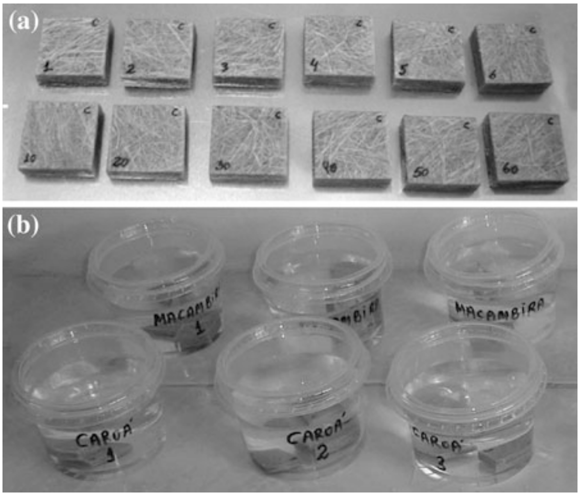


Fig. 4 **a** Caroá composite samples and **b** Macambira and caroá composites samples in water bath

water baths (Fig. 4) kept at 25, 50 and 70 °C. At regular intervals the samples were removed from the water bath, wiped with tissue paper to remove surface water and immediately weighted to ±1 mg (uncertainty ±0.001 g). Following, the samples were re-immersed in the water bath to continue the sorption process until the equilibrium condition is reached. Each measurement took less than 1 min, so water evaporation at the surface was insignificant.

The results of absorbed moisture were presented as mass of absorbed water by unit dry composites mass. The moisture content was computed as follows:

$$\bar{M}(t) = \frac{W_t - W_0}{W_0} \times 100\% \quad (1)$$

where W_0 and W_t represent the dry weight of the composites samples ($t = 0$) and the wet weight 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 result in lower equilibrium moisture content.

3.2 Theoretical Study

Transport phenomena in porous media represents an important research area related to heat and mass transfer, and fluid fields. Mass and heat transport through unsaturated matrices, as with heat and mass diffusion/convection through any heterogeneous media, depends on the structure of the matrix and the physical properties of each phase (fluid and solid).

One of the most difficult aspects of the analysis is structural modeling. A theoretical treatment is based upon the traditional method and start with a potential per unit time balance in a differential control volume with arbitrary shape in macroscopic scale (i.e. porous material is represented as a fictitious continuum).

By assuming local equilibrium between solid and fluid, the mass transfer phenomenon in porous media can be modeled by the macroscopic conservation equation in a short form, as follows:

$$\frac{\partial}{\partial t}(\lambda\Phi) + \nabla \cdot (\lambda\vec{v}\Phi) = \nabla \cdot (\Gamma^{\Phi}\nabla\Phi) + S^{\Phi} \quad (2)$$

In the Eq. (2), we have $\lambda = \rho$; $\Phi = M$ and $\Gamma^{\Phi} = \rho$ D, where ρ, M, and D correspond to density, moisture content, and effective mass diffusivity, respectively, and t is the time. In this same equation, $\vec{v}$ is the velocity vector and S^{Φ} is a source term.

For the best analysis, consider a dry porous media (for example, a polymer composite) of complex geometry, at low temperature, which is suddenly immersed in a flowing or stationary, heated, saturated fluid (for example, water) at a different temperature. When this dry porous media is being heated by the fluid surrounding it, heat penetrates into the solid (as the result of a temperature difference) and moisture migrates into the solid by diffusion from the surface.

Mass diffusion is a phenomenon by which matter is transported from one region in space to another due to random molecular motions [41]. Heat and mass transfer creates temperature and moisture gradients inside the solid, which, in general, depends on the internal and external conditions of the porous media. For simplicity, several researchers report a model that assumes that water migrates at very low velocity from the external surrounding medium to the center of the solid only by liquid diffusion (diffusive effects are greater than convective effects). Gravity, capillarity, and other effects are neglected.

In situations where the solid density may be considered constant and without mass generation inside the solid, the general differential equation that describes the mass diffusion phenomenon (for example, water absorption) can be written as follows:

$$\frac{\partial M}{\partial t} = \nabla \cdot (D \nabla M) \tag{3}$$

where D is the diffusion coefficient and M is the moisture content in a dry basis. Equation (3) is called Fick's second law of diffusion.

Despite of the extended use of the Fick's second law to study diffusion process, it fails to address the complex structure of the composite due to different water absorption capacity of the polymer matrix and the natural fiber.

Based on the hypotheses reported above, the mass transfer in a solid of parallelepiped shape, of dimensions $2R_1 \times 2R_2 \times 2R_3$ (Fig. 5), the mass conservation equation written in Cartesian coordinates for the three-dimensional case, is given as follows:

$$\frac{\partial M}{\partial t} = \frac{\partial}{\partial x}\left(D\frac{\partial M}{\partial x}\right) + \frac{\partial}{\partial y}\left(D\frac{\partial M}{\partial y}\right) + \frac{\partial}{\partial z}\left(D\frac{\partial M}{\partial z}\right) \tag{4}$$

For a well-posed mathematical model it is necessary to specify the initial and boundary conditions. Because the symmetry of the problem, we will consider only 1/8 of the volume of the solid. In this chapter, the initial, symmetry and boundary conditions are as follows:

(a) Initial condition:

$$M(x, y, z, t = 0) = M_o, \quad 0 < x < R_1, \quad 0 < y < R_2, \quad 0 < z < R_3 \tag{5}$$

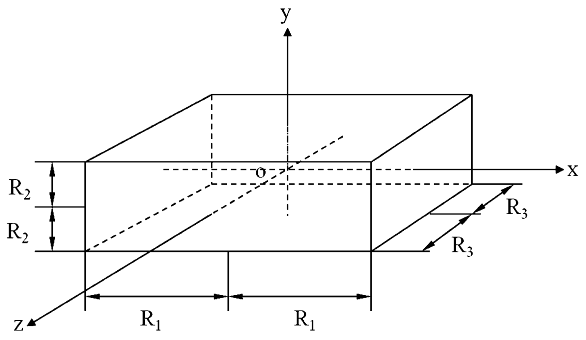


Fig. 5 Geometrical configuration of the physical problem

(b) Symmetry conditions:

$$\frac{\partial M(x=0,y,z,t)}{\partial x}=\frac{\partial M(x,y=0,z,t)}{\partial y}=\frac{\partial M(x,y,z=0,t)}{\partial x}=0,\ t>0 \tag{6}$$

(c) Boundary conditions:

$$M(x=R_1,y,z,t)=M(x,y=R_2,z,t)=M(x,y,z=R_3,t)=M_e,\ t>0 \tag{7}$$

The average moisture content within the solid may be computed as:

$$\overline{M}(t)=\frac{1}{V}\int\limits_V M(x,y,z,t)dV \tag{8}$$

where V is the volume of the porous solid.

In general, the diffusion coefficient has been considered constant or a function of the average moisture content or temperature, or yet as a combination between of moisture content and temperature. Then, for example, we may write:

$$D=D_oExp(c\bar{M}) \tag{9}$$

$$D=D_1Exp\left(\frac{d}{T}\right) \tag{10}$$

where D_o, D_1, c and d are constants, obtained from experimental data.

To solve the diffusion partial differential equation (Eq. 1) in conjunction with appropriate initial and boundary conditions different analytical (for example, separation of variables and Galerkin-based integral method) and numerical (for example, finite-difference, finite-element, boundary-element, and finite-volume) techniques may be used.

Based on a review of the technical literature, it was noted that some works solve analytically the diffusion problems with prescribed boundary conditions and constant thermo-physical properties. Using separation of variable technique, the analytical solution of the mass diffusion equation applied to a parallelepiped will be [42, 43]:

$$\begin{aligned}\frac{M(x,y,z,t)-M_e}{M_o-M_e}=&\sum_{n=1}^{\infty}\sum_{m=1}^{\infty}\sum_{k=1}^{\infty}A_nA_mA_k\times Cos(\beta_nx)\times Cos(\beta_my)\\&\times Cos(\beta_kz)\times Exp[-(\beta_n^2+\beta_m^2+\beta_k^2)Dt]\end{aligned} \tag{11}$$

where

$$A_n=\frac{2Sen(\beta_nR_1)}{\beta_nR_1} \tag{12}$$

$$A_m=\frac{2Sen(\beta_mR_2)}{\beta_mR_2} \tag{13}$$

$$A_k = \frac{2Sen(\beta_k R_3)}{\beta_k R_3} \tag{14}$$

Using Eq. (11) we obtain the moisture content distribution inside the porous media at different times. In this equation, the coefficients β_n, βm and $\beta_k$are the so-called eigenvalues. They are obtained as follows:

$$Cos(\beta_n R_1) = 0 \tag{15}$$

$$Cos(\beta_m R_2) = 0 \tag{16}$$

$$Cos(\beta_k R_3) = 0 \tag{17}$$

The average moisture content can be calculated by the following equation:

$$\frac{\bar{M}(t) - M_e}{M_o - M_e} = \sum_{n=1}^{\infty}\sum_{m=1}^{\infty}\sum_{k=1}^{\infty} B_n B_m B_k . e^{-[\beta_n^2 + \beta_m^2 + \beta_k^2]Dt} \tag{18}$$

where

$$B_n = \frac{2}{(\beta_n R_1)^2} \tag{19}$$

$$B_m = \frac{2}{(\beta_m R_2)^2} \tag{20}$$

$$B_k = \frac{2}{(\beta_k R_3)^2} \tag{21}$$

It may be difficult to obtain the analytical solution of a partial differential equation with a high level of complexity, for example, when variable thermo-physical properties are used. In this case a numerical solution may be used as an appropriate alternative technique. We notice that the finite-volume method has been used to discretize the governing equations. Figure 6 represents the differential volume of the physical domain (Fig. 5), where the nodal points (W, E, N, S, F, T), the dimensions of the control volume (Δx, Δy, Δz) and the distances between nodal points (δx, δy, δz) are presented.

Assuming a fully implicit formulation, where all terms are estimated in t + Δt, Eq. (4) was integrated in the control volume of Fig. 4 which corresponds to the internal points of the domain, and also in time. As a result, Eq. (4) was discretized by a finite-volume method using practice B (nodal points in the center of control-volume) [44, 45] resulting in the following linear form:

$$A_P M_P^* = A_E M_E^* + A_W M_W^* + A_N M_N^* + A_S M_S^* + A_T M_T^* + A_F M_F^* + B \tag{22}$$

where

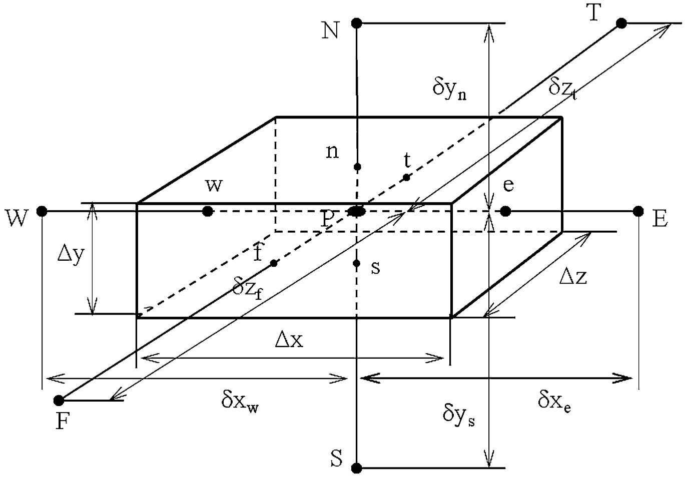


Fig. 6 Control volume used in this work

$$M^* = \frac{M - M_e}{M_o - M_e} \tag{23}$$

$$A_E = \frac{D_e \Delta y \Delta z}{\delta x_e} \tag{24}$$

$$A_W = \frac{D_w \Delta y \Delta z}{\delta x_w} \tag{25}$$

$$A_N = \frac{D_n \Delta z \Delta x}{\delta y_n} \tag{26}$$

$$A_S = \frac{D_s \Delta z \Delta x}{\delta y_s} \tag{27}$$

$$A_T = \frac{D_t \Delta x \Delta y}{\delta z_t} \tag{28}$$

$$A_F = \frac{D_f \Delta x \Delta y}{\delta z_f} \tag{29}$$

$$A_P^o = \frac{\Delta x \Delta y \Delta z}{\Delta t} \tag{30}$$

$$B = A_P^o M_p^{*o} \tag{31}$$

$$A_P = A_E + A_W + A_N + A_S + A_T + A_F + A_P^o \tag{32}$$

The diffusion coefficient (D) at the interface of the control-volume was calculated according to the literature [44]. The set of algebraic linear equations obtained from Eq. (22) (when applied for all nodal points) was solved interactively using the Gauss–Seidel method. The following convergence criterion was used:

$$|M^{*n-1} - M^{*n}| \leq 10^{-8} \tag{33}$$

where n represents the nth iteration in each time.

In practical applications, because of the large variety of materials and operating conditions, the diffusion coefficient is unknown. In general, this parameter has been found by varying it to minimize the sum of the squared deviations between the actual and predicted data. The relative deviation between the experimental and calculated values (relative residual, ERMQ) and the variance (S^2) are defined as follows [46]:

$$ERMQ = \sum_{i=1}^{m} (\bar{M}^{*}_{i,Num} - \bar{M}^{*}_{i,Exp})^2 \tag{34}$$

$$S^2 = \frac{ERMQ}{(m-1)} \tag{35}$$

where m is the number of experimental points. The smallest values of ERMQ and S^2 has been used as criteria to obtain the best value of the diffusion coefficient.

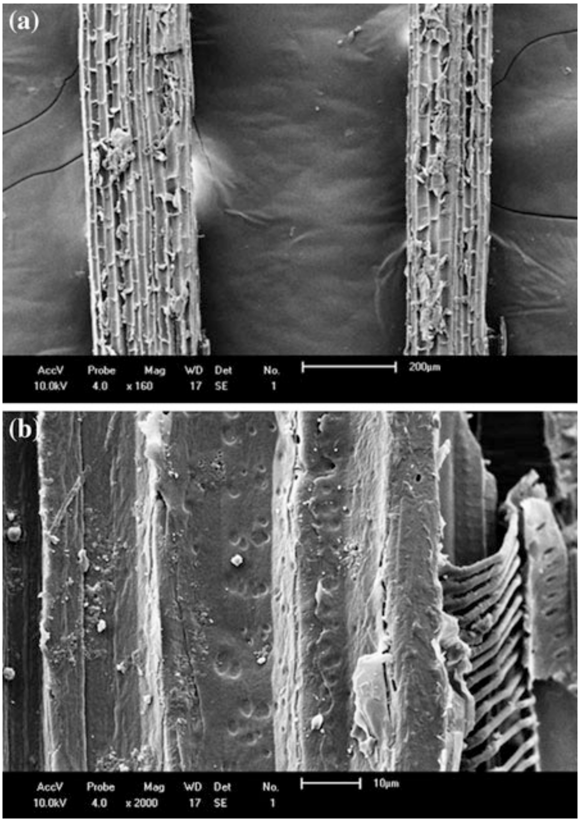
3.3 Results Analysis

3.3.1 Morphology Characterization

In this research some fiber samples (untreated) were analyzed by scanning electron microscopy (SEM) in order to characterize the morphology of the fiber. Figures 7 and 8 present micrographs of macambira and caroá fibers in the natural state, respectively. It is verified that the arrangement is similar to other natural fibers, with spongier aspect and a thin, compacted cellular arrangement. Images obtained from SEM also showed a rougher surface, which implies that macambira fibers could be an adequate to reinforce composite materials, due to a good adhesion between fibers and matrix. After treatment or mechanical characterization the fiber suffers some degradation. Besides, since cellulose is the principal 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 interfibrilar space [12].

Fig. 7 SEM micrographs for untreated macambira fiber: **a** 160× and **b** 2000×



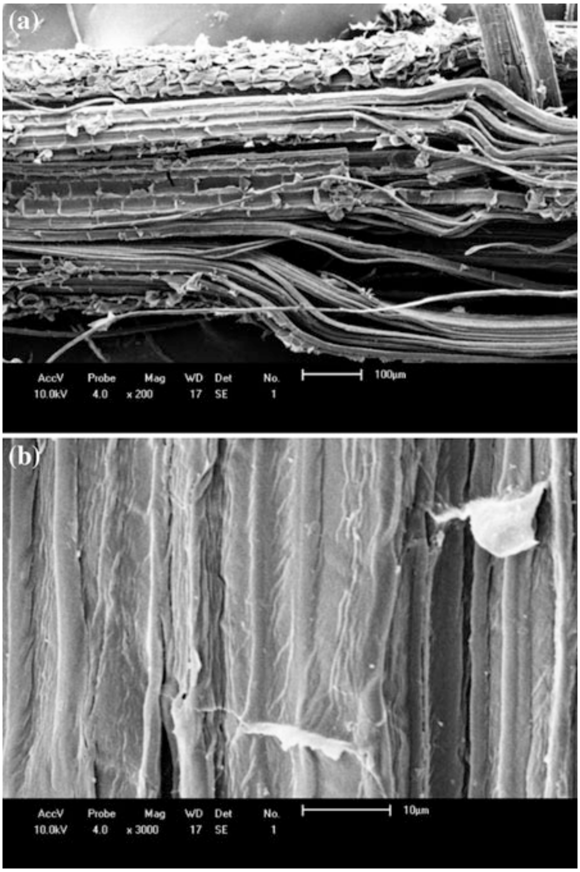
3.3.2 Water Absorption Kinetics

Water sorption curves for different dimensions (3 and 6 mm thickness) and temperatures as functions of water exposure (immersion) time for unsaturated polyester/macambira composites with 30 % w/w fiber contents. 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 condition). This behavior strongly indicates that the macambira fibers were uniformly distributed in the matrix, in accordance to work reported in the literature [18].

The data indicates that water absorption for the composites is higher than for the matrix. Pure polyester shows a very low percentage of absorbed water. While the water sorption in equilibrium for the unsaturated polyester is about 1 %, for the composites it was about 14 %. The weight loss of pure polyester was almost insignificant, indicating minimal degradation. These results are consistent with

Fig. 8 SEM micrographs for untreated caroá fiber: **a** 200× and **b** 3000×



studies reported in the literature for similar systems [16, 18, 24, 38, 47]. Some authors attribute the increase in the water sorption by polymer composites reinforced with vegetable fiber to the hydrophilic nature and the permeability of this type of reinforcement and also to the sample surface area exposed to water. Capillarity effects and the interfacial area between fiber and matrix may contribute too.

Figure 9 also shows that water sorption for all composites increase with the area/volume ratio and temperature (Table 1). The effect of the size was more pronounced at the lower temperature.

The results indicate that, as expected, the water uptake of the composite sample immersed in a water bath at 70 °C was faster than under the other (25 °C 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).

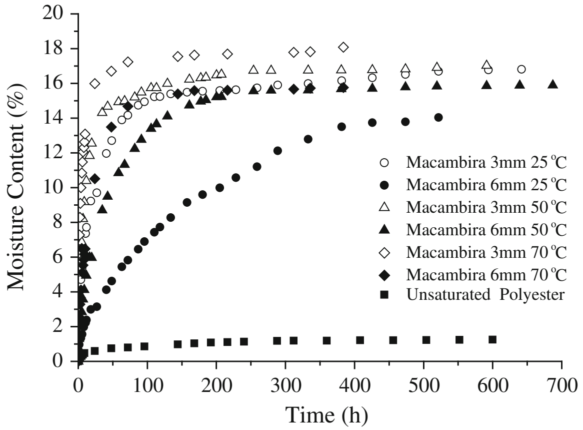


Fig. 9 Effect of sample dimensions and bath temperature in the water sorption of Macambira fiber reinforced unsaturated polyester composites

Table 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)	M_t (%) (d. b.) ($t \rightarrow \infty$)	Area/volume (mm^2/mm^3) ($t = 0$)
Polyester matrix	3.00	25	1.26	866.67
Macambira composites	3.00	25	16.81	866.67
	6.00	25	14.04	533.33
	3.00	50	17.02	866.67
	6.00	50	15.89	533.33
	3.00	70	18.09	866.67
	6.00	70	16.18	533.33
Caroá composites	3.00	25	14.49	866.67
	6.00	25	14.81	533.33
	3.00	50	15.16	866.67
	6.00	50	16.08	533.33
	3.00	70	15.61	866.67
	6.00	70	16.52	533.33

These results are consistent with those reported in similar systems for unsaturated polyester/caroá composites [24, 39], unsaturated polyester/jute composites [38] and unsaturated polypropylene/sisal composites [12]. Other aspects were not analyzed, such as size and orientation of fiber (events that occurs in long fiber reinforced composites are different those observed in short fiber reinforced composites) [17], and water migration by capillarity in micro cracks inside the solid, mainly in the fiber-matrix interface where adhesion is of fundamental importance. Research in these topics is strongly recommended.

Numerical results of average moisture content of the composites under investigation were compared with the experimental data. To obtain the numerical

Fig. 10 Comparison between predicted and experimental dimensionless mean moisture content during the water absorption in caroá fiber reinforced unsaturated polyester composites, at 25 °C

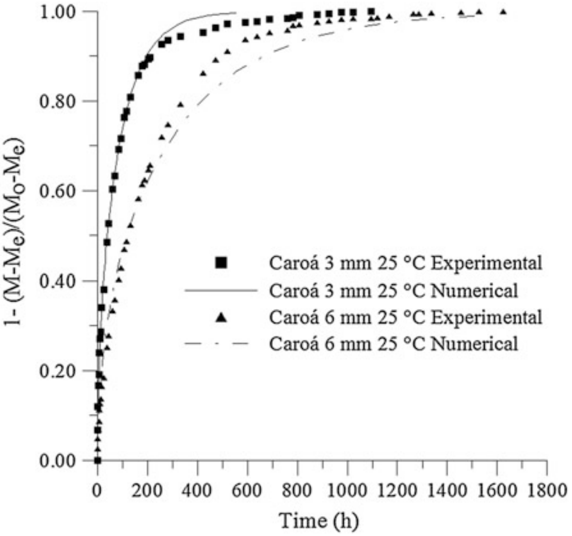
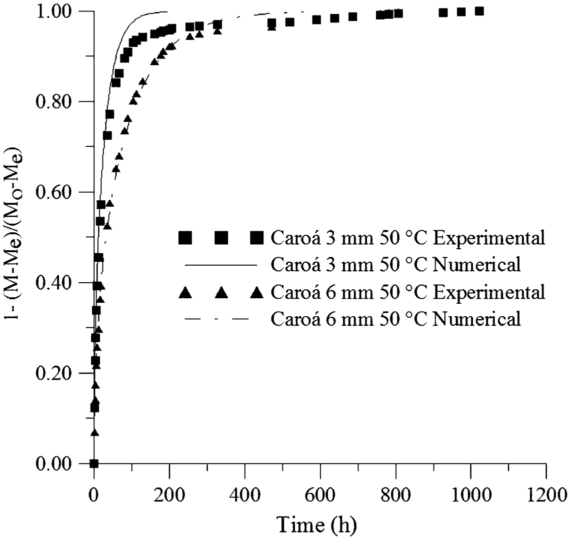


Fig. 11 Comparison between predicted and experimental dimensionless mean moisture content during the water absorption in caroá fiber reinforced unsaturated polyester composites, at 50 °C



results, a computational code using 20 × 20 × 20 nodal points and a time step of $\Delta t = 1$ s was developed. These parameters were obtained after a time step and mesh refining study [38, 48]. The numerical results obtained from a given mathematical model are strongly dependent on the boundary conditions, thermo-physical properties and geometry. A comparison between the experimental and numerical predicted average moisture content for the investigated composites, shown in Figs. 10, 11, 12, indicates that the model employed here is appropriate.

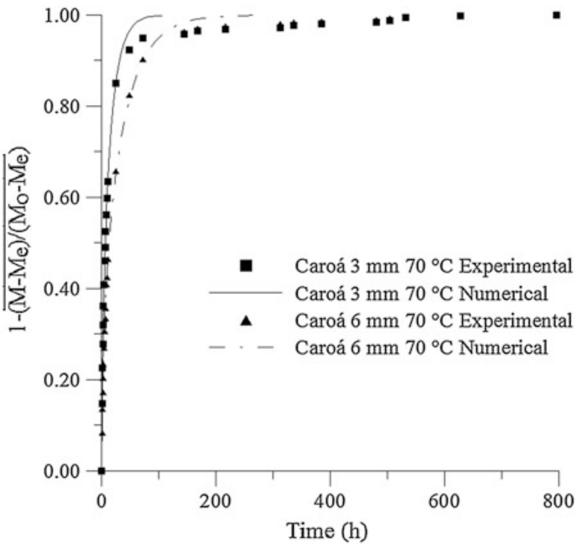


Fig. 12 Comparison between predicted and experimental dimensionless mean moisture content during the water absorption in caroá fiber reinforced unsaturated polyester composites, at 70 °C

A comparison between Figs. 9, 10, 11, and 12 indicates that the wetting process in the 3-mm thick caroá reinforced composite was faster than in the 6-mm thick composite. This behavior may be attributed to the increase in solid area/volume ratio and the high water affinity for the cellulosic material. The effect is more pronounced at lower temperatures. At high temperature the kinetics of water absorption is dominated by thermal effects, and the mass diffusion inside the material is strongly dependent on the temperature.

Moisture degrades the natural fiber reinforced composites, but this effect was not measured here. However, weight loss varying with immersion time and fiber content was verified for sisal fiber reinforced polypropylene composites properly dried before [18].

The mechanism of diffusion and structure/diffusivity relationships, have been little studied. Some authors have suggested that water diffusivity is due to morphology [49], local molecular mobility [50], free volume [51] or fiber distribution in polymer matrix [52].

According to [53] apparent water diffusivity decreases when hydrophilicity increases and it increase when temperature increases [25], showing that the fiber/polymer/water interactions strongly influence the diffusion rate within the solid. So, all factors mentioned above, including fiber concentration, thermal stability and shape, need to be studied to get a full understanding of the mechanism of moisture absorption.

3.3.3 Absorbed Water Distribution

The moisture content distribution in the 70 % unsaturated polyester/30 % caroá fiber composites in the plane $x = 5$ mm, at elapsed time of 20 h, is presented in

Fig. 13 Moisture content profiles inside the composite 30 % caroá fiber/70 % unsaturated polyester, at 25 °C (**a**), 50 °C (**b**) and 70 °C (**c**) for a thickness of 3 mm, in the plane $x = 5$ mm, elapsed time 20 h

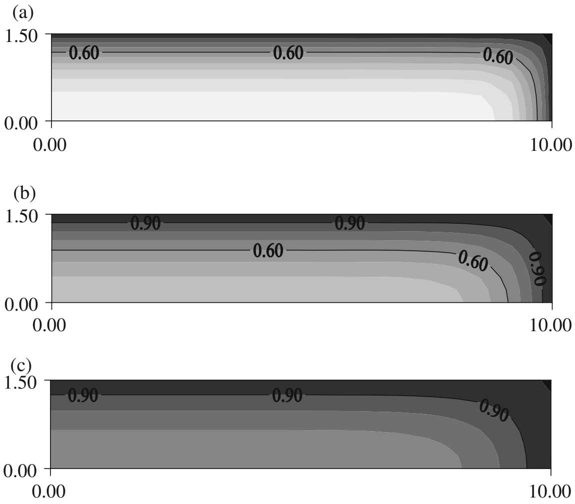
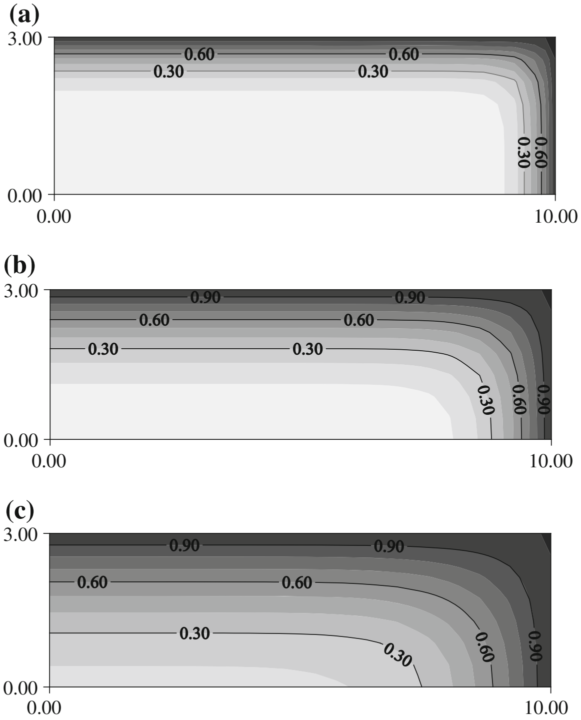


Fig. 14 Moisture content profiles inside the composite 30 % caroá fiber/70 % unsaturated polyester, at 25 °C (**a**), 50 °C (**b**) and 70 °C (**c**) for a thickness of 6 mm, in the plane $x = 5$ mm, elapsed time 20 h



Figs. 12 and 13, showing the existence of high moisture gradients next to the vertices of the solid. Figures 13 and 14 had shown the influence of temperature on the absorption of water. It can be observed that 20 h after the test had started, the moisture gradients at higher temperature is significantly higher for both thicknesses studied.

It is important to know the profiles and the moisture content inside the material in order to predict which areas are more sensitive to thermo-hygro-mechanical stresses, which may cause cracks and deformations, and consequently decrease the quality of the product [38, 48].

3.3.4 Mass Diffusion Coefficient Estimation

Tables 2 and 3 present the value of the diffusion coefficient obtained by the fit between numerical and experimental data. As expected, diffusion coefficients tend to decrease with increasing moisture content and to increase with increasing temperature for all samples. The use of mass diffusivity depending on moisture content led to numerical values that adjusted well to the experimental data, with less error than those obtained if constant diffusivity was assumed.

It is well known that diffusion processes in systems with two or more components depend on several factors such as volume fractions, nature, distribution and orientation of the fillers as well as on fiber/matrix interface characteristics [37, 47]. In general, the comparison with diffusion coefficients reported in the technical literature is very much difficult by the different models and calculation methods used, and by the different porosity, dimensions, temperature, compositions and

Table 2 Mass diffusion and variance estimated for all wetting experiments with caroá reinforced composites and pure polyester

Sample	$D_o \times 10^{+12}$ m^2/s	c	ERMQ	$S^2 \times 10^{+3}$	Time (h)
Pure polyester	2.8	−26.0	0.267	9.54	600
Caroá 3 mm	2.2	−0.4	0.045	1.12	1095
Caroá 6 mm	2.3	−0.1	0.093	1.94	1623
Caroá 3 mm	8.0	−0.4	0.086	2.39	1022
Caroá 6 mm	8.5	−0.1	0.022	0.61	1022
Caroá 3 mm	15.0	−0.4	0.035	1.35	796
Caroá 6 mm	21.0	−0.1	0.043	1.65	796

Table 3 Range of the mass diffusion coefficients estimated and initial and final moisture content

Sample	Fiber total content (%)/T (°C)	$D_{initial} \times 10^{+12}$– $D_{final} \times 10^{+12}$ (m^2/s)	Initial and final Moisture content (dry basis)
Pure polyester	0/28	2.80–2.31	0–0.01252
Caroá 3 mm	30/25	2.20–2.07	0–0.14488
Caroá 6 mm		2.30–2.26	0–0.14810
Caroá 3 mm	30/50	8.00–7.53	0–0.15162
Caroá 6 mm		8.50–8.36	0–0.16067
Caroá 3 mm	30/70	15.00–14.10	0–0.15609
Caroá 6 mm		21.00–20.65	0–0.16523

Table 4 Moisture diffusivity of composites for several temperatures and shapes

Composite sample (shape)	Time (h)	Dimension (mm)	T (°C)	$D \times 10^{+13}$ (m^2/s)	Reference
Carbon-glass fiber/epoxy (rod)	5300	66.5 × 9.53	40	1.42	[25]
Carbon-glass fiber/epoxy (rod)	5300	66.5 × 9.53	60	3.84	[25]
Carbon-glass fiber/epoxy (rod)	5300	66.5 × 9.53	90	27.00	[25]
Sisal fiber/polypropylene (rectangular)	166	25 × 10 × 2.50	28	0.36	[12]
Sisal fiber/polypropylene (rectangular)	166	25 × 10 × 2.50	50	0.56	[12]
Sisal fiber/polypropylene (rectangular)	166	25 × 10 × 2.50	28	0.69	[12]
Jute-glass fiber/polyester (rectangular)	1249	20 × 20 × 2.82	28	16.50	[55]
Jute fiber/polyester (rectangular)	500	20 × 20 × 2.30	28	18.00	[56]

physical and chemical structures of the material [38, 54]. However, by comparing the mass diffusivity values of the composites obtained in this study with others, reported in Table 4, it was observed reasonable concordance. As the diffusive model was used in all works reported in the Table 4, the difference between the values may be attributed mainly to the factors: composite variety; geometry assumptions; different equilibrium moisture content; composite physical structure and expansion effects by water absorption.

4 Concluding Remarks

In this chapter, water absorption in natural fiber reinforced polymer matrix composites has been explored. Interest in this type of physical problem is motivated by its importance in many practical applications. The use of raw materials from renewable sources, such as vegetable fibers, has been the subject of several studies. One major disadvantage of the use of vegetable fibers in polymer composites is their hydrophilic nature and weak interfacial adhesion to the polymer matrix.

The present work deals specifically with water absorption in unsaturated polyester composites reinforced with caroá and macambira natural fibers by theoretical and experimental techniques. A consistent three-dimensional transient mathematical model was proposed, and a numerical formalism based in finite-volume method was used to solve the governing equation. Experimental tests for water absorption were performed by immersing the samples in a bath of distilled water at different temperatures, and the water uptake was measured gravimetrically along the process. For both the theoretical and experimental analysis the effect of the composite dimensions and bath temperature were studied.

From the results of apparent weight gain of the composites we conclude that: the proposed mathematical model describes adequately the process of water diffusion inside the composites; water absorption by composites increases with fiber loading and it is higher than for the polymer matrix alone; the kinetics of water absorption is strongly influenced by temperature, the rate of absorption being higher at higher temperatures; higher rates of water diffusion were obtained at the first stages of sorption, and the sorption rate decreased at longer water immersion times; at lower temperature; sample thickness affects water absorption more than temperature; for higher temperature, the effects of the temperature in the water absorption were more relevant than those observed for changes in area/volume ratio of the sample; the regions in the neighborhood of the vertices of the solid presented the highest rates of mass transfer, being more susceptible to crack and deformation; the diffusion coefficient changes with the moisture content and temperature along the absorption process.

The information outlined in this chapter may help researchers and engineers in their study of the unknown features of related complex systems for technological applications in composites.

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