# Studies on Water Absorption of Bamboo-Epoxy Composites: Effect of Silane Treatment of Mercerized Bamboo

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**ABSTRACT:** The effect of silane treatment on the water absorption properties of bamboo matting reinforced epoxy composites were investigated. Experiments using  $\gamma$ -aminopropyltriethoxy silane, 3-trimethoxysilylpropylmethacrylate, vinyltris(2-methoxyethoxy)silane, bis[3-(triethoxysilyl) propyl]tetrasulfide, 3-aminopropyltrimethoxysilane, and *n*-octyltrimethoxysilane were carried out to improve the water resistant property of the bamboo fiber composites. Water absorption in the composites was studied by long term immersion and 2 h boiling in distilled water. The process of absorption of water was found to follow the kinetics and mechanism described by Fick's theory. Alkali treatment results in reduction of water absorption from 41 to 26%. Further reduction is observed with silane treatment. Water absorption varies between 21 and 24%, minimum being for aminopropyltriethoxysilane treated composite. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 115: 1846–1852, 2010

**Key words:** biofibers; composites; modification; thermoset; diffusion

# **INTRODUCTION**

The use of biofibers, as opposed to synthetic fibers, has been well recognized due to the positive environmental benefits with respect to ultimate disposability and raw material utilization. In addition, it has properties like low cost, lightweight, high specific strength, and free from health hazard. The presence of hydroxyl and other polar groups in the natural fibers would lead to the weak interfacial bonding between fibers and the relatively hydrophobic polymers.

One of the important factors for obtaining good fiber reinforcement in the composite is the bonding strength between natural fiber and polymer matrix. Due to the presence of pendant hydroxyl and polar groups in various constituents of fiber, moisture absorption of fiber is very high, which leads to poor interfacial bonding with the hydrophobic matrix polymers. The hydrophilic natural fibers absorb a large amount of water in the composite leading to failure by delamination. Adequate adhesion across the interface can be achieved at desirable levels by better wetting and chemical bonding between fiber and matrix. To make good use of biofiber reinforcement in composites, fiber surface treatment must be carried out to obtain an enhanced interface between hydrophilic bamboo fiber and hydrophobic polymer matrices. Such treatments will decrease the moisture absorption and hydrophilic character of fibers. Surface modification is therefore necessary to obtain better performance of the resulting composite.<sup>1</sup>

Silane interfacial coupling agents widely used on glass fiber to form stable covalent bonds with both the mineral fiber surface and the resin are potentially suitable for use on cellulosic fibers.<sup>2</sup> The silane coupling agent is believed to improve the surface functionality of the bamboo fibers and subsequently enabled bamboo fibers bond chemically to the rubber matrix.<sup>3</sup>

The bamboo fibers were mercerized before silanization because the surface of untreated fiber is richer in lignin and waxes than the surface of alkali treated fiber, the hydrolyzed silane has to diffuse first between the crevices of the fibers in order to be able to interact with the cellulosic hydroxyl groups. The surface of alkali treated fiber is rougher than the surface of untreated fiber, thereby resulting in larger effective area of contact.4 Treatment of mercerized sisal in silane results in improved moisture resistance in both unbonded and bonded fibers.<sup>2</sup> Treatment of the cellulose fibers with alkali brings about the process of swelling and dissolution. This enhances the accessibility of the cellulosic hydroxyl groups for a subsequent reaction.<sup>5</sup> Valadez et al.<sup>4</sup> pointed that neither the modification of the fiber topography using the alkaline solution nor the silane treatment by themselves result in a noticeable increase in the

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tensile strength, however, the combination of both surface treatments result in much better results. The efficiency of the silane treatment was higher for the alkaline treated fiber than for the untreated one. A significant increase in the bond strength between the Woodstock and polymer was found for the sample, in which the Woodstock was treated in the solution of NaOH. Initial NaOH treatment followed with the coupling agent treatment showed the highest coupling strength.<sup>6</sup> With silane treatment better mechanical properties of composite materials were obtained.<sup>7–10</sup> The effect of fiber surface modification on water sorption behavior of oil palm empty fruit bunch fiber has been investigated.<sup>11</sup> Oil palm fiber modification by alkali treatment and silane treatment improves the overall mechanical performance of the fiber.<sup>12</sup> Composites prepared from fluorosilane treated fibers exhibited better mechanical properties among other silane treated composites.<sup>1</sup> Silane treatment leads to distinctly improved mechanical properties of jute-epoxy composite.<sup>13</sup> The fiber surface silanization results in better interfacial load transfer efficiency but do not seem to improve the wetting of the fiber.<sup>14</sup> The adhesion between industrial hemp fibers and unsaturated polyester (UPE) matrix was increased by treatment of hemp fiber surface with silane.<sup>15</sup> Silane improved the adhesion between the bamboo fiber and the rubber matrix.<sup>3</sup> The treatment of wood fibers with a methacrylate silane improves the dimensional stability of wood.<sup>16</sup> In contrast, a decrease of mechanical properties was observed for coir-UPE composites, after fiber modification with dichloromethylvinyl silane.17 The treatment of mercerized sisal fibers with aminosilanes before forming the sisal epoxy composites markedly improves composite moisture resistance.<sup>2</sup> The silane modified matrix showed the best overall mechanical performance, i.e., the best compromise among Young's modulus, tensile strength, deformation at break, and impact strength.<sup>18</sup> The improvement in mechanical properties was enhanced by the inclusion of the silane coupling agents.<sup>19</sup> Amino-silane coupling agent modified the jute-polycarbonate (PC) composite to improve the interfacial adhesion between jute fiber and PC.<sup>20</sup> Treatment of fibers with silane or titanate coupling agents do not result in significant changes in the mechanical properties of the composites formed there from.<sup>21</sup> The use of silane coupling agent in polyester resin concrete improves its mechanical properties due to improved adhesion between the binder and aggregates at the interface.<sup>22</sup>

Bamboo is a natural material, and is abundantly available natural resource in Asia and South America. It has been used traditionally as a structural material for fabrication of village houses. Based on its anatomical properties, ultra structure, and plant fracture mechanism bamboo establishes itself as a superior natural fiber among other known natural fibers (like jute, coir, sisal, straw, banana etc.).<sup>23-25</sup> Jain et al.<sup>26,27</sup> reported that bamboo has 60% cellulose with a considerably higher percentage of lignin ( $\sim$  32%), its microfibrillar angle being relatively small  $(2-10^{\circ})$ . These facts about bamboo support its high tensile strength. As a cheap and fast-grown resource with superior physical and mechanical properties compared to most wood species, bamboo offers great potential as an alternative to wood. Although, there are many works and views on the effects of silane treatment of various natural fibers, there is hardly any information about silane treated bamboo fiber reinforced polymer (thermosetting/thermoplastic) composite. In this article, we have studied the effect of silane treatments of bamboo fiber on the water absorption behavior of bamboo-epoxy composites. Most of the natural fiber reinforced polymer composites are used for interior applications owing to the hydrophilic characteristic of the natural fiber. This study, therefore, attempts to address this important characteristic of the bamboo fiber to aid their increasing utilization in outdoor applications.

### Kinetics of water absorption

The poor resistance of the fibers to water absorption can have undesirable effects on the mechanical properties and the dimensional stability of the composites.<sup>5–10</sup> Therefore, it is important to study the water absorption behavior in detail, in order to estimate the consequences that the water absorbed may have and also how this water uptake can be minimized in some way. Moisture penetration into composite materials is conducted by three different mechanisms and they include. (i) diffusion of water molecules inside the microgaps between polymer chains; (ii) capillary transport of water molecules into the gaps and flaws at the interface between fibers and the polymer due to the incomplete wettability and impregnation; and (iii) transport of water molecules by micro cracks in the matrix, formed during the compounding process.<sup>28–30</sup> Though all three mechanisms are active, the overall effect can be modeled conveniently considering the diffusion mechanism.

In general, diffusion behavior in glassy polymers can be classified according to the relative mobility of the penetrant and of the polymer segments. With this, there are three different categories of diffusion behavior.<sup>30</sup> Case I, or Fickian diffusion, in which the rate of diffusion is much less than that of the polymer segment mobility. The equilibrium inside the polymer is rapidly reached and it is maintained with independence of time. Case II (and Super Case II), in which penetrant mobility is much greater than other relaxation processes. This diffusion is characterized by the development of a boundary between the swollen outer part and the inner glassy core of the polymer. The boundary advances at a constant velocity and the core diminishes in size until an equilibrium penetrant concentration is reached in the whole polymer. Non-Fickian or anomalous diffusion occurs when the penetrant mobility and the polymer segment relaxation are comparable. It is then, an intermediate behavior between Case I and Case II diffusion. These three cases of diffusion can be distinguished theoretically by the shape of the sorption curve represented by

$$M_t/M_s = kt^n \tag{1}$$

where,  $M_t$  is the moisture content at time t;  $M_s$  is the moisture content at the equilibrium; *k* and *n* are constants. k is a constant characteristic of the sample, which indicates the interaction between the sample and water and n indicates the mechanism of sorption. The values of n and k were determined by linear regression analysis. The value of coefficient n shows different behavior between cases; for Fickian diffusion n = 0.5; while for Case II n = 1 (and for Super Case II n > 1). For anomalous diffusion, n shows an intermediate value (0.5 < n < 1). Moisture absorption in natural fiber reinforced plastics usually follows Case I Fickian behavior, so, further attention will be focused on its study. As mentioned earlier, apart from diffusion, two other minor mechanisms are active in moisture exposure of composite materials. The capillary mechanism involves the flow of water molecules into the interface between fibers and matrix. It is particularly important when the interfacial adhesion is weak and when the debonding of the fibers and the matrix has started. On the other hand, transport by microcracks includes the flow and storage of water in the cracks, pores, or small channels in the composite structure. These imperfections can be originated during the processing of the material or due to environmental and service effects.

In most cases, moisture absorption processes often follow the prediction of Fick's law, where the mass of water absorbed increases linearly with square root of time, and then gradually slows until equilibrium plateau. The diffusion coefficient, *D*, can be determined from:

$$Mt/Ms = (4/h)(D/\pi)^{1/2}t^{1/2}$$
 (2)

where h is the sample thickness.

# MATERIALS AND METHODS

Orthogonal bamboo strip mats were acquired from the local market (New Delhi, India). The cross-section of the strip used in the mats is  $4.25 \times 0.5$  mm<sup>2</sup>.

Bamboo mats cut to size  $300 \times 200 \text{ mm}^2$  were cleaned with washing powder and washed in running water thoroughly to remove dust and other deposits from the surface. They were left in the open to dry out for 4 h. Commercial grade cycloaliphatic epoxy resin (CY-230, Ciba Geigy (Chennai, India) product, epoxy equivalent 4.1–4.5 eq/kg), a pale yellow viscous liquid, and Aradur HY-951 hardener was used. The hardener was an aliphatic polyamine (triethylenetetramine). Epoxy resin was heated in an oven at 80°C for 10 min to remove the moisture and air bubbles. After cooling the resin to room temperature, hardener, 10% by weight was added, stirred to mix thoroughly, and subsequently applied on the bamboo mats piled one over another upto seven layers. These were placed in between two Perspex sheets, 300  $\times$ 200 mm<sup>2</sup>, and pressed in the hydraulic press under a pressure of 170 KN for 24 h. After this, the composite laminates were cured at 80°C for 4 h in an oven. The volume percent of bamboo mats was  $64 \pm 1\%$  in the composites. This was maintained all throughout.

Cleaned and dried mats were dipped in 5% NaOH solution for 30 min at room temperature. These mats were thoroughly washed in distilled water and then neutralized in 2% HCl solution. A litmus paper test was carried out to check the neutrality.<sup>31</sup>

Silanes used for treatment were  $\gamma$ -aminopropyltriethoxysilane (H<sub>2</sub>NCH<sub>3</sub>H<sub>6</sub>—Si(OC<sub>2</sub>H<sub>5</sub>)<sub>3</sub>), 3-trimethoxysilylpropyl methacrylate (H<sub>2</sub>C=C(CH<sub>3</sub>)CO<sub>2</sub> (CH<sub>2</sub>)<sub>3</sub>Si(OCH<sub>3</sub>)<sub>3</sub>), Vinyltris(2-methoxyethoxy)silane ((CH<sub>3</sub>OCH<sub>2</sub>CH<sub>2</sub>O)<sub>3</sub>SiCH=CH<sub>2</sub>), bis[3-(triethoxysilyl) propyl]tetrasulfide (C<sub>18</sub>H<sub>42</sub>O<sub>6</sub>S<sub>4</sub>Si<sub>2</sub>), 3-aminopropyltrimethoxysilane (H<sub>2</sub>NCH<sub>2</sub>H<sub>4</sub>NHC<sub>3</sub>H<sub>6</sub>—Si(OCH<sub>3</sub>)<sub>3</sub>) (Acros Organics, Geel, Belgium), and *n*-octyltrimethoxysilane (C<sub>8</sub>H<sub>17</sub>—Si(OCH<sub>3</sub>)<sub>3</sub>) (Alfa Aesar, Heysham, Lancs, UK). The methods of treatment were described by the authors in previous article.<sup>32</sup> The nomenclature used for different composites is given in Table I.

#### **RESULTS AND DISCUSSIONS**

## Water absorption studies

Water absorption studies were performed following the ASTM D570-98 method. The samples were taken out periodically and weighed immediately, after wiping out the water from the surface of the sample, to find out the amount of water absorbed. The amount of water absorbed in the composite was calculated by the weight difference between the sample exposed to water and the dried samples.

The optimum alkali treatment is both a very effective surface modification and also a low cost surface treatment of natural fibers. The reaction of sodium hydroxide with natural fiber is thought to occur as shown:

TABLE I	
Materials Analyzed	

Sample	Specification		
BE	Bamboo-epoxy composite		
BE5A	Bamboo-epoxy composite with 5% NaOH treated bamboo.		
BASE1	5% NaOH and Aminopropyl triethoxy silane treated bamboo-epoxy composite		
BASE2	5% NaOH and 3-trimethoxysilyl propyl meth- acrylate treated bamboo-epoxy composite		
BASE3	5% NaOH and Vinyltris(2-methoxyethoxy)si- lane treated bamboo-epoxy composite		
BASE4	5% NaOH and Bis[3-(triethoxysilyl)Propyl]te- trasulphide treated bamboo-epoxy composite		
BASE5	5% NaOH and Aminopropyltrimethoxy silane treated bamboo-epoxy composite		
BASE6	5% NaOH and <i>n</i> -Octyltrimethoxysilane treated bamboo-epoxy composite		

 $Fiber - OH + NaOH \rightarrow Fiber - O^{-}Na^{+}$ 

+ H<sub>2</sub>O + surface impurities

This treatment removes a certain amount of hemicellulose, lignin, wax, and oils covering the external surface of the fiber cell wall. The maximum improvement compared with untreated bamboo composite is at 5% of NaOH treatment.<sup>31</sup> The mercerization of natural fibers generates changes of the surface energy (in all probability to a more polar behavior) and surface topography (rougher surface with excavated fibrils). The more polar fiber surface improves the connection to the coupling agent's polar part and the rougher surface topography furthers the wettability and form fit of the matrix material and thus causes a better fiber–matrix adhesion.<sup>33</sup>

Silane coupling agents are silicon-based chemicals that contain two types of reactivity – inorganic and organic – in the same molecule. A typical general structure is

#### (RO)3SiCH2CH2CH2 - X,

where RO is a hydrolyzable group, such as methoxy, ethoxy, or acetoxy, and X is an organofunctional group, such as amino, methacryloxy, epoxy, etc. A silane coupling agent will act as an interface between an inorganic substrate (such as glass, metal, or mineral) and an organic material (such as an organic polymer, coating, or adhesive) to bond, or couple, the two dissimilar materials.

When organic polymers are reinforced with glass fibers or minerals, the interface, or interphase region, between the polymer and the inorganic substrate is involved in a complex interplay of physical and chemical factors. These factors are related to adhesion, physical strength and coefficient of expansion, concentration gradients, and retention of product properties. A very destructive force affecting adhesion is migration of water to the hydrophilic surface of the inorganic reinforcement. Water attacks the interface, destroying the bond between the polymer and reinforcement, but a "true" coupling agent creates a water resistant bond at the interface between the inorganic and organic materials. Silane coupling agents have the unique chemical and physical properties not only to enhance bond strength but also, more importantly, to prevent debonding at the interface during composite aging and use. The coupling agent provides a stable bond between two otherwise poorly bonding surfaces.<sup>34</sup>

It is clear (Fig. 1) that the composites absorb water very rapidly at the initial stage, and later a saturation level was attained without any further increase in water absorption. In the case of untreated bamboo composite, higher water absorption 41%, may be due to poor wettability and weak adhesion between untreated bamboo and matrix, and defects such as voids. Both the initial rate of water absorption and the maximum water uptake increase as the void content increases.<sup>35</sup> The hydrophilicity, due to the presence of lignin and hemicellulose component, was responsible for the higher percentage of water uptake in untreated bamboo composite. Hemicelluloses are mainly responsible for the water uptake, although noncrystalline cellulose and lignin also play an important role in the process. The water swells the cell wall of the fiber until its saturation. Next, it goes on to occupy the void spaces of the fiber, and this free water does not cause further swelling. Alkali treatment of bamboo fibers and treatment of mercerized bamboo fibers with silane results in greater hydrophobicity and less water absorption by the composite; lower initial rate of water absorption and less



**Figure 1** Water absorption curve for composites. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

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water absorption at saturation. This is due to improved wettability and strong adhesion. Among the probable factors that may have led to this difference, the extraction of hemicellulose by the alkali treatment stands out because hemicellulose is considered to be mainly responsible for water uptake, given that it is more accessible than the crystalline regions of the cellulose.<sup>36</sup> Alkali treatment resulted in reduction of water absorption from 41 to 26%. Further reduction was observed with silane treatment. Water absorption varied between 21 and 24%, minimum being for BASE1. The improved moisture resistance caused by the application of the coupling agents can be explained by an improved fiber-matrix adhesion. The inactive hydroxyl groups become active by alkali treatment, which leads to better silane deposition and a strong chemical interaction between the fiber and matrix is possible. This will reduce the moisture sorption and is evident from the sorption curves.<sup>37</sup> It is believed that the coupling agent can immigrate into the alkali treated sisal fibers easily and its reaction with cellulose could lead to an interfacial interaction even stronger.<sup>9</sup> The coupling agent builds chemical bonds (silanol bonds) and hydrogen bonds, which reduce the moisture-caused fiber-matrix debonding.13 As observed by Valadez-Gonzalez et al.<sup>14</sup> the amount of silane adsorbed onto the alkali treated fibers is higher than that absorbed by the untreated fibers. This difference in the amount of adsorbed silane could be attributed to the fact that the alkali treated fibers have a larger amount of exposed cellulose on the surface, and the cellulosic hydroxyl groups have a better capability to interact with the hydrolyzed silane. Higher silane deposition reduces the amount of hydroxyl groups, which were free to bind moisture. Valadez et al.4 have pointed out that the main effect of washing a natural fiber with a mild alkaline solution is the removal of waxes, hemicelluloses and a partial removal of the lignin present on their surface. The leaching of these compounds enhances the availability of sites for the cellulose–silane interaction.

Figure 2 shows the water absorption by the composites after 2 h of boiling in distilled water. After alkali treatment water absorption is reduced from 14 to 9.9%. It is apparent that the NaOH treatment of bamboo reduced the water uptake of the composite system. As shown, chemical treatments can reduce the hydroxyl group in the cell wall of the natural fiber molecule, thus decreasing the water absorption of the composites. The result of water uptake however is in accordance with earlier studies.<sup>4,38</sup> Minimum percent of water is absorbed by BASE1 (6.61%) and BASE5 (6.85%) composites. This may be due to better interaction between the amino silanes in composites BASE1 and BASE5. Aminofunctional silanes work well with epoxy resins.<sup>39,40</sup> Presumably, the



Figure 2 Percent increase in water absorption after 2 h boiling.

amino groups of the adsorbed silane coupling agent react with the epoxy moieties of the resin, in a manner similar to the reaction between epoxy and an amine based hardener in the bulk phase.<sup>41</sup> Aminopropyltriethoxy silane works better for bambooepoxy composites than the aminopropyltrimethoxy silane as BASE1 has better resistance to water both in long term immersion and short term boiling in water. Silane treatment has also lead to less water absorption by BASE2, BASE3, and BASE4. This shows 3-trimethoxysilylpropyl methacrylate, Vinyltris(2-methoxyethoxy)silane, and Bis[3-(triethoxysilyl)propyl]tetrasulfide can work well with bamboo and epoxy matrix. Percent water absorbed by BASE6 is 14.24, well above that by BE5A.

Water absorption in polymers is often analyzed in terms of Fickian diffusion, which requires that the results are plotted as weight gain (as a percentage of dry weight) versus square root of time.<sup>35</sup> As shown in Figure 1, where percentage moisture absorption increases steadily with  $t^{1/2}$  in the initial stage and then tends to level off following the saturation point, indicating a Fickian mode of diffusion.

The analysis of the diffusion mechanism and kinetics were performed based on the Fick's theory. It is considered that the change of weight gain for all samples is a typical Fickian diffusion behavior. The mechanism of water uptake and hence the study of the kinetic parameters n and k, the data were analyzed by adjusting the experimental values to the following equation, which is derived from eq. (1).

$$\log(Mt/Ms) = \log(k) + n\log(t) \tag{3}$$

Figure 3(a,b) show examples of the fitting of the experimental data to eq. (3). The values of the parameters n and k obtained from the fitting curves of



**Figure 3** (a) Diffusion curve fitting plots for BE5A to determine constants n and k, (b) Diffusion curve fitting plots for BASE1 to determine constants n and k.

the water absorption of the composites are summarized in Table II. As the value of the parameter napproach to 0.5 the composites show a tendency to approach Fickian behavior. An increase in the kvalue is observed for some silane treated composites, indicating increase in the moisture interaction with the composite material.

The diffusion coefficient (*D*) is the most important parameter of the Fick's model, which shows the ability of the water molecules to penetrate inside the composites, was calculated using the following equation from the initial slope of the plot of Mt/Mm against (time)<sup>1/2</sup>:<sup>42</sup>

TABLE II Moisture Sorption Constants and Diffusion Coefficients of Composites

	1			1	
Sample	п	k	$D\times 10^{-12} \text{ m}^2/\text{s}$		
BE	0.4538	0.0438	11.36		
BE5A	0.3742	0.0749	14.01		
BASE1	0.3537	0.0846	14.59		
BASE2	0.3658	0.0775	15.62		
BASE3	0.3592	0.0821	26.27		
BASE4	0.3694	0.0772	18.70		
BASE5	0.3500	0.0884	24.66		
BASE6	0.3073	0.1194	21.81		



**Figure 4** Diffusion curve fitting plots for composites for diffusion coefficient. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

$$Mt/Ms = (4/h)(D/\pi)^{1/2}t^{1/2}$$

Figure 4 shows the diffusion curve fitting plots for composites for diffusion coefficient. The values of *D* are also summarized in Table II. Due to the hydrophilic character of natural fibers, the inclusion of water molecules inside the composite material is favored as demonstrated by the kinetics of the diffusion processes. The values obtained for diffusion coefficients are in agreement with the range of values reported by other authors.<sup>28,43–45</sup> According to these reports the values for diffusion coefficient for natural fiber reinforced composites fall in the order of  $10^{-12}$ – $10^{-13}$  m<sup>2</sup>/s.

#### CONCLUSION

Alkali treatment results in decrease in water absorption by the composite. With silane treatment further reduction is observed. The decrease in water absorption can be explained by the improved fiber-matrix adhesion, resulting from the alkali and silane treatment. Alkali and silane treatment of bamboo fiber results in greater hydrophobicity and less water absorption by the composites. Aminofunctional silanes work well with epoxy resins. Aminopropyltriethoxysilane works better for bamboo-epoxy composites than the aminopropyltrimethoxysilane. The best results are obtained for alkali and Aminopropyltriethoxysilane treated bamboo-epoxy composite. It has better resistance to water both in long term immersion and short term boiling in water. The effectiveness of silanes for water resistant on the bamboo-epoxy composites is in the order aminopropyltriethoxysilane > aminopropyltrimethoxysilane >

3-trimethoxysilyl propyl methacrylate > bis[3-(triethoxysilyl)propyl]tetrasulphide > *n*-octyltrimethoxysilane > vinyltris(2-methoxyethoxy)silane. BASE3 and BASE6 composites have higher water absorption than that of alkali composite for 2 h boiling. All the composites indicate a Fickian mode of diffusion and a tendency to approach Fickian behavior. The values obtained for diffusion coefficients are in agreement with the range of values reported, in the order of  $10^{-12}$ - $10^{-13}$  m<sup>2</sup>/s.

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