IRIET Volume: 08 Issue: 12 | Dec 2021

# Investigation of Water Absorption and Diffusion Behaviour of Luffa-**Epoxy Polymer Composites**

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**Abstract** - Study of water absorption behaviour of the natural fibre reinforced composites is important to decide their potential application as composites are prolonged exposure to water, sun, wind, and rain. The water absorption, thickness swelling, kinetics and diffusion behaviour of NaOH treated luffa fibre reinforced epoxy composites were studied. Composites were fabricated with different fiber shape and fiber volume and. It was revealed that the composites followed Fickian diffusion behaviour and the lowest value of percentage of thickness swelling, diffusion, sorption and permeability coefficient of composites were 3.5%, 1.2396x10<sup>-10</sup> mm<sup>2</sup>/s, 1.0768, and 6.3096x10<sup>-11</sup> mm<sup>2</sup>/s respectively. Field emission scanning electron microscopy was carried out to study the interfacial bonding between the luffa and epoxy, presence of pores and voids in the composites.

## Key Words: Natural fiber, Water absorption, Thickness swelling, Diffusion coefficient, SEM analysis

# **1.INTRODUCTION**

The environmental regulations and global concern have enforced research for developing new materials that are environmentally friendly and an alternative to nonrenewable material such as glass fiber, aramid, carbon fiber etc. Natural fibers are used as a potential substitute to synthetic fiber in polymer composites, due to their low density, environment friendliness, low cost, natural abundance, biodegradability, damping, and insulation characteristics. However, natural fiber reinforced composites have some demerits like the incompatibility between the fiber and thermoplastics/thermosetting polymers. Since natural fibers have hydrophilic behaviour, they have weak interface bonding with a hydrophobic polymer. Poor interface bonding leads the composites to have lower mechanical properties. Hence it is important to enhance the interfacial bonding between the hydrophilic fiber and hydrophobic polymer either by physical or chemical treatment of fiber. The surface treatment of cellulosic fiber helps to improve the mechanical, thermal properties and the characteristics of fiber [1-4].

The luffa fibre consisted of 66% of cellulose, 8-22% of hemi cellulose, 10-22% of lignin, 0.4% of ashes, and 3.2% extractives [5, 6] and these values depend on different factors like plant origin, soil, nature, weather condition and growth period. Natural fibers possess rich cellulose content

and crystallinity and shows better mechanical properties, moreover, the high cellulose, and hemicellulose are the main factors for the susceptibility to moisture absorption [2]. The high lignin content of natural fiber is responsible for thermal stability and lower water absorption, since it has a hydrophobic nature [7]. Actually, coir fiber possesses the lowest value of moisture diffusion among the cellulose fiber sisal and luffa, due to their higher lignin content. Water absorption behavior of the composites followed the kinetics and diffusion mechanism as described by Fick's [8] theory. The three different diffusion mechanisms are, case I (Fickian diffusion): diffusion of water molecules get into the polymer chain through the micro gap. The diffusion rate is very less and maintained independently of time, Case II: water molecules enter at the interface between the fiber and matrix through the crack and flaw due to the capillary action, which are formed due to the incomplete wetting of fiber with a matrix. It is characterized by the formation of the boundary between the swollen outer part and the glassy core of the polymer, Case III: The capillary transportation of water molecules gets into the natural fiber and developed fiber swelling, resulting in development of microcrack in the matrix during the process [8, 9].

The effect of moisture absorption of jute fiber-HDPE composite was investigated by [10]. The result revealed that the rate of moisture absorption increases with an increase of jute fiber since an increase of free -OH groups in high cellulose jute fiber. They observed the maximum percentage of moisture absorption as 1.2%, 2.5%, and 3.2% by different composites in distilled water at room temperature. Lee et al. [4] have fabricated the kenaf fiber reinforced nonbiodegradable polypropylene and bio-degraded poly(Butylene succinate) PBS with an addition of maleic anhydride-grafted(MAg) compatibilizer composite. They observed and concluded that the maximum percentage of moisture absorption was 18% for KF/PP and 6% for KF/PBS. So the moisture absorption of KF/PBS composite is significantly lower than that of KF/PP composite, since the water molecules penetrate through PP composites more quickly through voids presented in the interface.

Moisture absorption behavior and mechanical properties of napier fiber reinforced polyester composites [11], luffa/epoxy [12,13], rice husk and woof flour/polyolefin bamboo/vinylester [15], and bio-composite [14], sugarpalm/plasticized sugar palm starch [16], luffa/kenaf/

PBS-co-lactate /starch blends [17] were studied and it was observed that the mechanical properties of the composites decrease with increase in fiber loading, since increase of fiber increases the water absorption and swelling of the composites. In moisture absorbed samples, the intermolecular hydrogen bonding among the cellulose molecule present in the fiber is reduced by the absorbed water molecule. This reaction reduces the interfacial adhesion between the fiber and matrix, resulting in decreased mechanical strength [10].

In this work, it is intended to decide the end use of luffa-epoxy composites against weathering, the moisture absorption behavior, thickness swelling, kinetics and diffusion behaviour for various volume fractions and dimension of fiber were studied. Fourier transform infrared spectroscopy (FTIR) study was carried out to understand the hydroscopic behavior of untreated and NaOH treated luffa fiber reinforced composites. Field emission scanning electron microscopy (FESEM) was employed to study the interfacial adhesion.

# **1.1 Experimental Materials**

Generally, thermoset polymers have excellent thermal stability and lower water absorption behavior compared to thermoplastic polymers. The araldite CY 205 (Diglycidyl ether of bisphenol-A) resin is used as a matrix to fabricate the composite with 1% of cured catalyst amine agent TETA (Triethylene tetraamine) HY 951 used as hardener.

# **1.2 Processing Methods**

Luffa fibers were immersed in a 2% NaOH solution and kept it in the solution for an hour at room temperature, and then cleaned thoroughly with water to remove the composition of NaOH sticking to them. Finally fibers were washed with distilled water and dried in sunlight for 14 to 20 days and then prepared to the required dimensions. The epoxy and hardener mixture prepared at the ratio of 10:1 was stirred thoroughly by a mechanical stirrer. In order to avoid air trapping, epoxy mixture was poured slowly over the stacked mat fibers. Then, the closed mould setup was placed under a pressure of 5MPa in the compression machine for 24 hours at room temperature for curing. Similarly, the short /particle fiber was added to the epoxyhardener mixture, stirred uniformly to disperse the short /particle fiber in the epoxy. Finally, the laminate was removed carefully from the mould and allowed to dry in the air for one day.

## 1.3 Fourier transforms infrared (FTIR) spectroscopy

Fourier transmission infrared spectrometry (FTIR), spectrometer (Bruker Alpha-T Spectrometer) is used to analyze a samples of untreated and treated luffa-epoxy composites. Transmission mode was set to IR rays of 400 to 4000cm<sup>-1</sup>. FTIR analysis is being used to study the presence

of various functional groups in the fibers structure which are used as reinforcement in the composites. The functional group presented in the untreated and treated luffa fibre reinforced composites were analyzed and shown in Fig. 1.



Fig. 1 FTIR spectra untreated and NaOH treated luffaepoxy composites

Hydrogen bonded -OH stretching vibration peak observed at 3751cm<sup>-1</sup> indicated the presence of carbohydrates (cellulose and hemicellulose) and lignin in the luffa. The intensity and broadness of –OH band was slightly reduced for treated luffa-epoxy composites due to the reduction/removal of hemicellulose. A small peak observed at 2962cm<sup>-1</sup> was attributed to the presence of C-H stretching vibration of methyl and methylene group of cellulose and lignin. Then the band at around 1582.62cm<sup>-1</sup> and 1500cm<sup>-1</sup> were responsible for carbonyl (C=O) group, which shows the reduction of hemicellulose presented in the luffa. The skeletal stretching vibration C-O-C observed at 1237.7cm<sup>-1</sup> was attributed to presence of ether, ester and phenol group which shows the reduction in the absorption of carboxyl group region attributed to the removal of hemicellulose and lignin. The other bands between 700cm<sup>-1</sup> to 1065cm<sup>-1</sup> have indicated the cellulose constituent of fibre [6, 18].

## 1.4 Water absorption test

Water absorption test was conducted as per ASME D570 to analyze the water absorption and thickness swelling behavior of the luffa-epoxy composites. Specimens were kept in an oven at 50° C to dry and then allowed to cool before weighing, and finally the dimensions of all the specimens were noted. A digital weighing machine with precision of 0.001g can be used to weight composites after every immersion. Similarly, a digital micrometer with precision of 0.001mm is used to measure thickness of the specimen. The weight of dry samples of the different composites were noted down, then immersed in de-ionized water at room temperature. They were taken out from the water after 24 hours of immersion and reweighed immediately and dimensions were measured spontaneously. They are then dried thoroughly and again immersed in water. Similarly all the specimens were weighed and measured regularly for 1, 2, 3, 4, 7, 8, 9, 10, 15, 20, 25, 30, 35, 40, 45 days until the weight of the wet sample reaches a repeatedly constant value [3]. The maximum percentage of water absorption, diffusion, sorption and permeability coefficient of the composites were calculated using the experimentally observed data.

#### **1.5 Field Emission Scanning Electron Microscope** *analysis*

The images of the fractured surface were observed by Field Emission Scanning Electron Microscope (FESEM) (Zeiss Supra 55) at predetermined magnification with an accelerator voltage of 3kV. This morphological examination is used to study the interface bonding between the fiber and matrix of composites. Small samples were cut off from the fractured specimens and coated with gold vanadium to avoid charging.

#### 2. Results and discussions

#### 2.1 Water absorption behaviour

Luffa-epoxy composites absorb water when immersed in water for long period; the intermolecular hydrogen bonding takes place between the luffa fiber cellulose molecule and the water molecule as shown in Fig. 2. The water absorption characteristics of composites were studied to predict the diffusion and permeability coefficient of the composites. The percentage of weight gain M(t) of the composite was calculated using equation(1) [15, 16, 19] for different time periods. where  $M_t$  is the mass of the sample at time t during immersion, and  $M_o$  is the initial mass of the dry sample before immersion.

$$M(t)(\%) = \frac{M_t - M_o}{M_o} \times 100$$
 (1)

Water absorption curves were plotted as weight gain in percentage against the square root of immersion time. Fig. 3 shows the water absorption curve for different composites as a function of fiber dimension and volume of fiber. The maximum percentage of moisture absorption was observed from Fig. 3 as 12% for MFR50 composite, and epoxy has moisture absorption of 4.6% as reported by [20]. Water absorption characteristics of luffa-epoxy composites are controlled by the luffa rather than by the epoxy. The cellulose constituent has the hydrophilic nature and thus increases the cellulose content at higher fiber volume of the composite [2, 21].



Fig. 2 Formation of hydrogen bonds between cellulose and water molecules.

From the Fig. 3, it could be observed that the moisture absorption pattern of these composites follows a fickian diffusion process since water absorption of all the composites increases with immersion time at the early stages. Later, they slow down and reach saturation condition [19] after a long time [4, 9, 22].



Fig. 3 Water absorption curve of treated MFR, SFR, and PFR luffa-epoxy composites.

#### 2.2Thickness swelling

The percentage of thickness swelling of the composite was calculated using equation (2) [9, 21, 23] for different time periods. Thickness swelling curves were plotted as the percentage of thickness swelling against the immersion time in sec.

$$TS(\%) = \frac{T_t - T_o}{T_o} \times 100$$
<sup>(2)</sup>

where  $T_t$  is the thickness of the sample at time t during immersion and T<sub>o</sub> is the initial thickness of the dry sample before immersion. Fig. 4 shows the percentage of thickness swelling for different composites as a function of fiber dimension and volume. It can also be observed from Fig. 4 that the thickness swelling is significantly influenced by fiber dimension than fiber volume. The maximum percentage of thickness swelling of PFR composites was noted as 3.2% at about 240hrs. As in the case of SFR composites, thickness swelling is takes place mostly within 100hrs, and the maximum percentage of swelling is 4.6% around 168hrs. Similarly, MFR composites reached maximum of swelling as 6.1% at around 168hrs. From this result, it could be clearly understood that the PFR composites have lower thickness swelling than SFR and MFR composites, but SFR and MFR composites absorbed water molecules steadily and reached optimum swelling.



Fig. 4 Thickness swelling behaviuor of treated MFR, SFR, and PFR luffa-epoxy composites



Fig. 5 Relation between water absorption and thickness of swelling for luffa-epoxy composites

The percentage of swelling depends on the % of water absorption by fiber, swelling of fiber, and water molecules entered. The relation between the water absorption and thickness swelling of composites helps to understand the thickness swelling mechanism as seen in Fig. 5. From this, it can be observed that the water absorption of PFR composites causes maximum thickness swelling as 3.2%. but, though the % of water absorption was not increased further, the thickness swelling increased upto the maximum value. It may be due to the effect of penetration of water molecules into the interface which is more than the effect of water absorption of particle fiber. Further, it is noticed that, the % of thickness swelling increased linearly with water absorption since the % of thickness swelling for SFR and MFR composites reached maximum value of water absorption at around 5.5% and 11.28% respectively. But beyond that % of water absorption, no change in the thickness of swelling was noticed.

#### 2.3 Diffusion behaviour

The capillarity mechanism describes the flow of water molecules through the interface bonding between the fiber and matrix, and diffusion through the bulk matrix. Fick's theory has been applied to analyze the performance of diffusion mechanism and kinetics of water molecules [8]. The three different diffusion mechanisms are, in first mechanism (Fickian diffusion), diffusion of water molecules get into the polymer chain through the micro gap. The diffusion rate is very less and maintained independently of time. In the case of second mechanism, the capillary transportation of water molecules enters at the interface between the fiber and matrix through the crack and flaw, which are formed due to the incomplete wetting of fiber with a matrix. It is characterized by the formation of the boundary between the swollen outer part and the glassy core of the polymer. The third mechanism is the capillary transportation of water molecules that gets into the natural fiber and develops fiber swelling. This mechanism can be differentiated based on the shape of the water absorption curve, and represented by the eq(3) [18, 21, 22, 24].

$$\frac{M_t}{M_m} = kt^n \tag{3}$$

where  $M_t$  and  $M_m$  are the moisture content at time t and saturated condition respectively, and t is the duration of specimen immersed in water. k and n are constant. k value shows the interaction between the composite and water molecules, whereas, value of n shows the mode of diffusion occurred like If value of n is 0.5 means follows case I Fickian diffusion, while n is equal to one means case II and values of n lie in between 0.5 to 1 means follows case III. The values of constant n(slope) and k(intercept) can be predicted from the slope and intercept of log plot of  $M_t/M_m$  versus time curve using the eq(4) [24, 25].

$$\log\left(\frac{M_t}{M_m}\right) = \log(k) + n\log(t) \tag{4}$$



Fig. 6 Curve fitting plots for all composites to obtain the value of constant n and k

Curve fitting can be plotted using the water absorption experimental data. Fig. 6 shows the curve fitting plots for all composites to predict the constant n and k values [18]. From the result of linear curve fitting, a better relationship has been observed as denoted by  $R^2$ . The diffusion coefficient (D) of the composites were obtained by using the eq(5) [4, 22] and the values are tabulated in table 1.

$$D = \pi \left[\frac{kh}{4M_m}\right]^2$$

where  $M_m$  is the maximum water absorption at the saturated condition, K is the initial slope, which is measured from water absorption curve (Fig. 2) and h is a thickness of the specimen. The diffusion coefficient behaviour describes the capacity of the water molecules to move into the polymer. The permeability of water molecules throughout the composites depend on the sorption of water by the fiber. The sorption coefficient can be calculated by the following eq(6) [9, 21, 22, 25]

(5)

(6)

$$S = \frac{M_m}{M_i}$$

where  $M_m$  and  $M_i$  are the mass of moisture content taken up at saturated condition and mass of the initial specimen respectively. Permeability depends on the product of the diffusion and sorption coefficient, which can be calculated by using the relation (7) [21, 22]

$$P = D \times S \tag{7}$$

The values of sorption and permeability coefficient are also summarized in the table 1. In PFR wet composite, the value of n is found to be lower than 0.5, which means diffusion process deviated from fickian diffusion, and follows other mechanisms as a result of fiber swelling, interface weakening, and microcracking. This behavior has also been reported by other researchers [25] but diffusion and permeability of water molecules get into the composites are significantly low. The values of n for 40 SFR, 50SFR and all MFR composites are closer to 0.5 and hence it clearly shows that these composites approach Fickian diffusion behaviour

Table -1: Water absorption at equilibrium (M<sub>m</sub>)

Kinetics of Water absorption					
Specimen	$M_{\rm m}$ in %	n	k	R <sup>2</sup>	
PFR 30	0.444	0.209	0.292	0.924	
PFR 40	0.674	0.164	0.228	0.924	
PFR 50	0.773	0.114	0.161	0.924	
SFR 30	7.680	0.909	1.349	0.982	
SFR 40	8.135	0.661	0.989	0.965	
SFR 50	8.647	0.652	0.965	0.95	
MFR 30	9.896	0.485	0.670	0.924	
MFR 40	10.52	0.532	0.766	0.933	
MFR 50	12.96	0.451	0.630	0.867	

It could be observed from the table 2 that the values of D, S and P are significantly changed with fiber volume and fiber dimension reinforced in the composites. eg., the value of D, S, and P increases from  $4.22417 \times 10^{-8}$  to  $4.4635 \times 10^{-8}$ , 0.09896 to 0.12960, and  $4.18041 \times 10^{-9}$  to  $5.78471 \times 10^{-9}$  respectively for the addition of mat fiber from  $0.3V_f$  to  $0.5V_f$ . These values for MFR composites show lower value compared with PFR composites due to their three dimensional array of mat fiber [5].

Transport coefficient						
Specimen	Diffusion coefficient (D) (mm²/s)	Sorption coefficient (S) (g/g)	Permeability coefficient (P) (mm2/s)			
PFR 30	1.935E-07	0.00444	8.588E-10			
PFR 40	2.413E-07	0.00674	1.626E-09			
PFR 50	2.545E-07	0.00773	1.968E-09			
SFR 30	9.707E-09	0.07680	7.455E-10			
SFR 40	2.009E-08	0.08135	1.634E-09			
SFR 50	2.262E-08	0.08647	1.956E-09			
MFR 30	4.224E-08	0.09896	4.180E-09			
MFR 40	4.210E-08	0.10526	4.431E-09			
MFR 50	4.463E-08	0.12960	5.784E-09			

Table -2: Diffusion coefficient (D) of luffa-epoxy composites

The maximum percentage of moisture absorption and the diffusion coefficient increase steadily from 30% to 50% of fiber due to their high cellulose content [19]. These values are discussed in some literature [4, 18, 26] for other natural fiber reinforced polymer composites. In this study, maximum of 50% of fibre is used and hence it affects the D value. As a result of swelling of the fibre, microcracks are formed in the composites that facilitate to absorb more moisture that penetrates the interface and develops swelling stress and these lead to damage of composites





Fig- 7: FESEM images of tensile fracture surface for treated luffa-epoxy composite. (a) Fracture surface of dry MFR composite (b) fracture surface of wet MFR composite, (c) fracture surface of treated dry SFR composite, (d) fracture surface of treated wet SFR composite

## 2.4 Morphology studies

Morphology studies by field emission scanning electron microscope was carried out for both dry and wet fiber reinforced composites to study the interface adhesion between the luffa fiber and epoxy, presence of voids and microcracks in the composites. FESEM images of dry and wet samples are shown in Fig. 7. It can be observed from Fig. 7a, and c, that there are less/no fiber pullout, no damaged fiber and also that epoxy layers are attached to the fiber surface which reveals the presence of better interface adhesion between the luffa and epoxy in dry composites. Whereas in case of wet composites, water molecules can be diffused by capillarity action into the interface bonding, the absorbed water molecules are formed as hydrogen bond that reduces the interaction between luffa and epoxy [19]. Due to the effect of water absorption fiber gets damaged, it is confirmed by the presence of fiber break or pull out and microcracks (Fig. 7b, and c). Due to the differential swelling of fiber and matrix induced the matrix microcrack and mechanical damage. These types of damages have been reported by an author [25].

# **3. CONCLUSIONS**

The water absorption and thickness swelling behaviour of luffa-epoxy composites were studied by the immersion of the specimens in water at room temperature as a function of fibre volume and fibre dimension and the following conclusion was arrived.

 It was revealed that SFR and MFR composites follow Fickian diffusion behaviour and the lowest value of D, S and P of the composites are 9.70732x10<sup>-8</sup> mm<sup>2</sup>/s, 0.07680, and 7.45555x10<sup>-10</sup> mm<sup>2</sup>/s respectively, due to the strong interface bonding strength between the fibre and matrix. Hence the strength of composites was not affected greatly by water absorption, finally

It can be concluded that the exposure of the composites to damp places, the mechanical properties will not be affected negatively. Hence the have the capacity to be used in interiors, damp places, and outdoor

The authors gratefully acknowledge DST, New Delhi for providing financial support to carry out this research work under DST – TDT scheme (DST File No. DST/TDT/LCCT-03/2017 (G)).

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