IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 4 Issue 6, June 2017
ISSN (Online) 2348 – 7968 | Impact Factor (2016) – 5.264
www.ijiset.com

Effect of Cenosphere on Moisture Absorption Performance of Woven Hybrid Jute-Glass Epoxy Composites

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Abstract

This paper depicts the investigation of moisture absorption behaviour and kinetics of hybrid composites consisting of jute-glass fiber as reinforcement, epoxy as matrix and cenosphere as particulate filler at different environmental conditions.Hand lay-up technique is used to fabricate the composites made from different doses of cenosphere filler content (5, 10, 15 and 20 Wt.%). Moisture absorption tests were conducted by immersing composite specimens in two different environmental conditions such as distilled and saline water at room temperature for a period up to 4 weeks. Moisture absorption curves, characteristic parameter D(diffusion coefficient) and M_m(maximum moisture content) were also determined. The water absorption of cenosphere filled hybrid jute-glass fibre epoxy composites was found to follow fickian behaviour. The flexural and tensile strength were found to increase with the increase in filler percentage in different environment.The surface morphologies of composites were also analyzed by means of Scanning Electron Microscopy (SEM).

Keywords: *Composite fabrics, Hybrid composite, Cenosphere, Moisture absorption, Thickness swelling, SEM*

1. Introduction

The more effort to environmental awareness today motivates researchers towards the study of natural fibre-reinforced composite as a substitute for conventional materials. The natural fibre as reinforcement in composite is a cost-effective option with advantages like high specific strength, availability, biodegradability as compared to synthetic fiber and hence they have the potential to be used as structural materials [1]. The jute fiber has high aspect ratio,high strength to weight ratio and low in energy conversion and has good insulation properties[2]. Among all the natural fibre as reinforcing materials, it is relatively inexpensive and commercially available in required form. However, its mechanical and physical properties are highly inconsistent and depend on geographic origin, climatic growth conditions and processing techniques [3]. But being hydrophilic in nature, the jute fibre leads to weak interfacial bonding between fibres and matrix which in turn deteriorates its mechanical properties [4-5] and causes dimensional instability [6]. The rate of moisture absorption of composites depends on resistance of the fibres to water absorption, reaction between moisture with the matrix, chemical composition and microstructure of polymer matrix. Moisture penetration into composite materials occurs by diffusion of water molecules inside micro-gaps between polymer chains, capillary transport into gaps and flaws at interfaces between fibres and polymer. Generally, based on these mechanisms, diffusion behaviour of composites can be classified as fickian, non-fickian, anomalous, or an intermediate behaviour between fickian and non-fickian [7,8].Coupling agents, fillers, compatibilizers or other chemical modifications are used to improve the moisture resistance of composites and are reported by several authors[9-14].

Hybrid composite consists of two or more reinforcements that are compatible with each other so that the properties of one are reimbursed by the other. Hybridization of natural fibre with different synthetic fibres like carbon, aramid, glass etc. can improve its moisture resistance[15-20].The addition of third phase in the hybrid composite plays an important role in determining the properties of the composite[21,22].The fillers are added to composites to reduce the material cost, improve mechanical properties and processability.For that, a proper material design is required for making composite with good performance and cost effective. Cenosphere shown in figure 1(a) is a hollow microsphere produced during phase transformation and thermochemical reaction during combustion of coal in thermal power plan [23]. They are mainly composed of mixtures of oxides such as SiO_2,Al_2O_3 and Fe_2O_3 as indicated by the EDX analysis as in figure 1(b).Investigation on mechanical properties of the cenosphere filled polymer composites are reported by several authors [24-26].

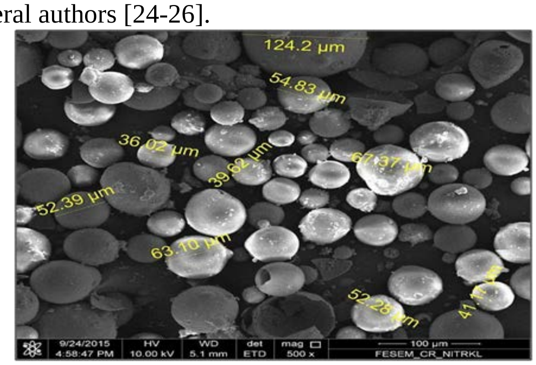

Fig. 1 (a) Cenosphere filler

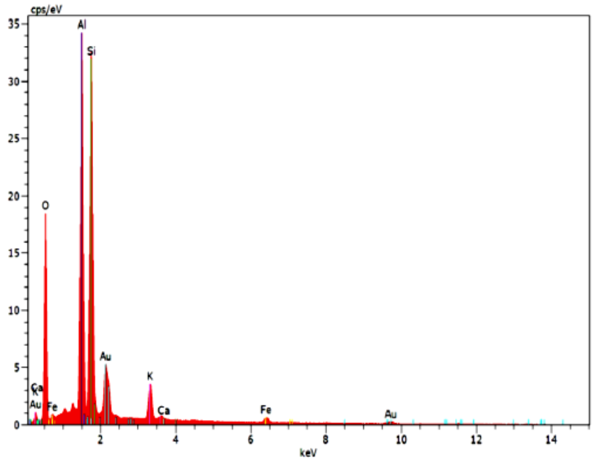

Fig. 1 (b) EDX analysis of cenosphere particle

The mechanical properties of woven hybrid jute-glass epoxy composite for different stacking sequences along with cenosphere addition are studied by the authors[27,28]. However, the study on the effect of cenosphere as a filler material on the moisture absorption performance of hybrid woven jute-glass epoxy composites has not been observed till to date.

Keeping this view, the present work focusses on the experimental effect of cenosphere on the moisture absorption performance of hybrid jute-glass epoxy composites. The jute fiber has been kept as (45^0-45^0) and that of glass as (0^0-90^0) orientation while making the hybrid composite.In addition, the thickness swelling, weight gain and diffusion kinetics behaviour of various weight percentages (5, 10, 15 and 20%) of cenosphere filled developed composites were also investigated.The fractured surface of composites were analyzed by means of scanning electron microscopy (SEM).

2. Materials and method

2.1 Raw materials

The following materials are used for preparation of filled hybrid composites to study the moisture absorption characterstics: jute fiber (woven), E-glass fiber, cenosphere, epoxies resin(LY-556), and hardener (HY-951).

2.2 Composite fabrication

Four groups of hybrid laminate composite samples of stacking sequence (GJJG) were prepared with varying weight percentage of cenosphere(5%,10%,15% and 20%) by usual hand lay-up technique.A wooden mold of dimension (150×60×5) mm was used for manufacturing the composite slab. For easy removal of the composite, teflon sheet and silicon spray are used which prevent adhesion between mold wall and composite sample. The composites with their designation, volume fraction of fiber and thicknesses calculated are presented in table 1.

Table 1: Laminate stacking sequences of cenosphere filled hybrid jute-glass epoxy composites

Symbol	Stacking sequence	Cenosphere Wt. (%)	Total fiber		Thickness (mm)
			Volume fraction %	Weight fraction %	
S1	GJJG	5	16.4	29.3	5
S2	GJJG	10	18.2	32.6	5
S3	GJJG	15	20.8	37.1	5
S4	GJJG	20	23.3	41.4	5

2.3 Moisture absorption and thickness swelling test

Water absorption and thickness swelling of the composites were performed as per ASTM D 570-98.Three test specimens were prepared from each stacking sequence of different cenosphere weight percentages having dimensions of length 140 mm and width 15mm.The weights of the composite samples were taken before submerged in two types of aqueous environments which are distilled water (pH=7) and saline water (pH=8) at room temperature. After immersion in different environments, the samples were removed at 24h and wiped with tissue paper to remove surface moisture. These samples were reweighed with an electronic balance having a resolution of 0.001 mg. During this process, moisture absorption takes place through the surface and the edges of the specimen resulting in weight gain. The above process was repeated at regular intervals of 24 h until an equilibrium value is reached. The percentage weight gain of the samples was measured by using the following relation.

$$\%M = \frac{W_t - W_0}{W_0} \times 100$$

The percentage weight gain of these samples was measured at different time intervals and the graph of moisture content versus immersion time is plotted. The thickness swelling is determined by the same procedure using the following relation.

$$\%\,T = \frac{T_t - T_0}{T_0} \times 100$$

Where T_t and T_0 are the composite thickness after and before immersion in water, respectively. The equilibrium moisture content (EMC) of the sample is the moisture content when the daily weight change of the sample was less than 0.01% and thus the equilibrium state was assumed to be reached and verified.

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3. Mechanical Testing

3.1 Tensile Testing

The tension test is generally performed on flat specimens.The standard test method used is as per ASTM D 3039-76. The tensile test was performed in universal testing machine INSTRON H10KS with a cross head speed of 2mm/min.The tensile stress is found out by dividing total load exerted on specimen by actual cross sectional area through which the force is applied. For each stacking sequence, five identical specimens were tested, and average result was obtained.

3.2 Flexural Testing

Flexural tests were also conducted on the same machine for tensile testing in accordance with ASTM D2344-84.Specimens of 140mm length and 15mm wide were cut and loaded on three points bending with a recommended span to depth ratio of 16:1.The test was conducted using the load cell of 10kN at 2 mm/min rate of loading. The flexural stress in a three-point bending test is found out by using the equation below.

$$\sigma = \frac{3F_{max}L}{2bt^2}$$

F is the maximum load (N) at failure, L is the distance between the supports (mm), b and t are the width and thickness (mm) of the specimens, respectively. For each stacking sequence of different cenosphere weight percentages, five identical samples were tested, and the average result was obtained.

3.3 Fractography Studies

To investigate the effect of moisture absorption on the microstructure of cenosphere filled hybrid jute-glass epoxy composite of varying weight percentages (5, 10, 15 and 20%) and to know the fractured behavior of filled hybrid composites,few samples were subjected to scanning electron microscopy (SEM).The samples were examined by using Scanning Electron Microscope of model JEOL JSM-6480LV.

4. Results and Discussions

4.1 Effect of filler loading on moisture absorption

Figures 2 and 3 shows the moisture absorption of cenosphere filled hybrid jute-glass epoxy composites of different weight percentages (5%,10%,15% and 20%) for both distilled and saline water environment. With addition of 5 and 20 wt.% filler the maximum moisture absorption increases from 1.815% to 3.252% in distilled water and from 1.352% to 2.094% for saline water, respectively [29].Presence of filler particles in the matrix produces voids at the interface and increases the ability of water molecules for piercing the composite through capillary transport and causes disorder in the structural homogeneity of the material. As the filler loading increases, the weak interfacial bonding between the filler and matrix resulted in increased number of micro voids, causing more moisture absorption. From the figures, it is observed that the moisture absorption increases and reaches an equilibrium state after the initial take-off. The pattern for thickness swelling is similar to the weight gain in composite for both environmental conditions as shown in figures 4 and 5. The results showed that with addition of 5 wt.% and 20 wt.% filler to the developed glass-jute composite, the thickness swelling increases from 8.734% to 16.63% and from 2.886% to 6.9% in both saline and distilled water conditions respectively. This might have happened because of the increased number of micro voids caused by the larger amount of poorly bonded area between the hydrophilic filler and the hydrophobic polymer matrix. From the test it was confirmed that the long-term immersion in water causes the dimensional instability of the composites.

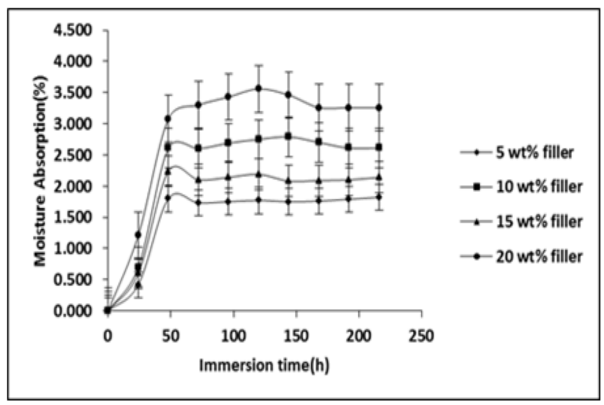

Fig. 2 Variation of moisture absorption of cenosphere filled hybrid jute-glass (GJJG) composites with immersion time for distilled water

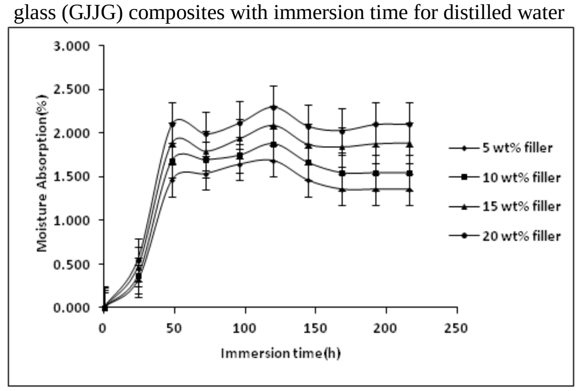

Fig. 3 Variation of moisture absorption of cenosphere filled hybrid jute-glass (GJJG) composites with immersion time for saline water

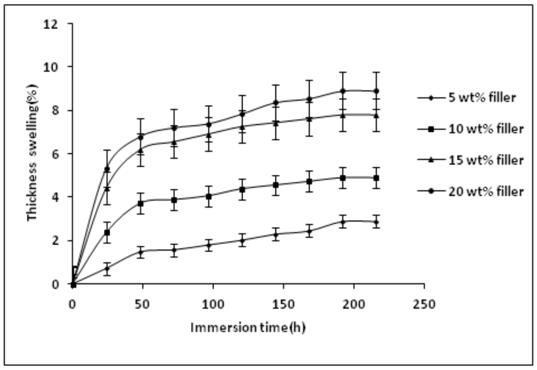

Fig. 4 Thickness swelling (%) of cenosphere filled hybrid jute-glass (GJJG) composites with immersion time for distilled water

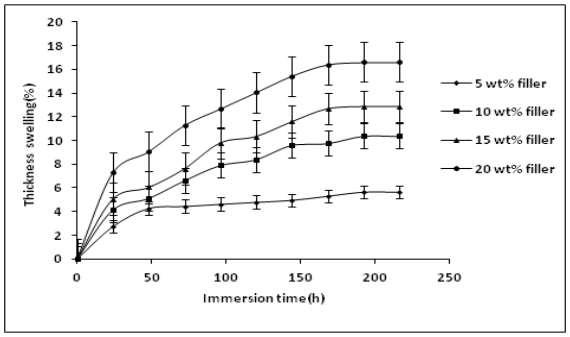

Fig. 5 Thickness swelling (%) of cenosphere filled hybrid jute-glass (GJJG) composites with immersion time for saline water

The EMC of the composites for different filler loading (5%,10%,15% and 20%) is shown in figure 6.It is observed that the EMC values reduces with the addition of cenosphere to the composites.The EMC value is more in distilled water as compared to saline water.

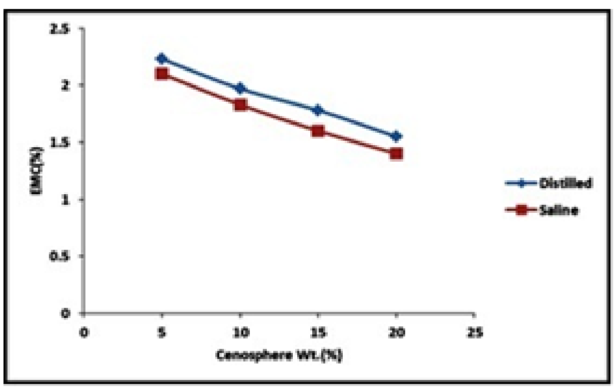

Fig. 6 Variation of (EMC) vs. cenosphere filler loading of hybrid jute-glass (GJJG) epoxy composites at different environmental conditions.EMC: Equlibrium moisture content

4.2 Moisture absorption kinetics

In order to investigate the type of diffusion mechanism, the moisture absorption datas have been fitted to the following relations [30]

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

Where
M_t: Moisture absorption at time t.
M_m: Moisture absorption at the saturation point.
k and n: Constants

The value of n indicates the type of transport mechanism and k indicates the interaction between the sample and water in addition to its structural characteristics of the polymer network. Figure 7 shows the fitting curve of log (M_t/M_m) as a function of log (t) for cenosphere filled hybrid jute-glass(GJJG) epoxy composites to determine the value of n and k.They are determined by linear regression analysis and the values are given in table 2 for different amounts(5,10,15 and 20%) of cenosphere filled glass-jute(GJJG) epoxy composites.For a Fickian diffusion mechanism, n has a value of 0.5.When n=1,the mechanism is non-fickian and when it lies between 0.5 and 1,the diffusion is anomalous[31-32].If the value of coefficient n gets smaller than 0.5,then moisture absorption behaviour follows the Fickian diffusion process[33].The value of n falls below 0.5 indicating the transport mechanism as fickian for all composite types as given in table 2.Similarly the value of k tends to be less for the cenosphere filled jute-glass (GJJG) composites, confirming the reduction in moisture consumption in saline water as compared to distilled environment.

Table 2: Diffusion case selection parameter of cenosphere filled hybrid jute-glass (GJJG) epoxy comosite

Environment	(%) of cenosphere filled (GJJG) Composite	n	k	k(h)²
Distilled water	5	0.434	0.116	6.770
	10	0.469	0.095	4.223
	15	0.468	0.096	3.372
	20	0.530	0.069	3.741
Saline water	5	0.433	0.112	4.766
	10	0.418	0.120	5.812
	15	0.374	0.145	0.972
	20	0.477	0.088	4.532

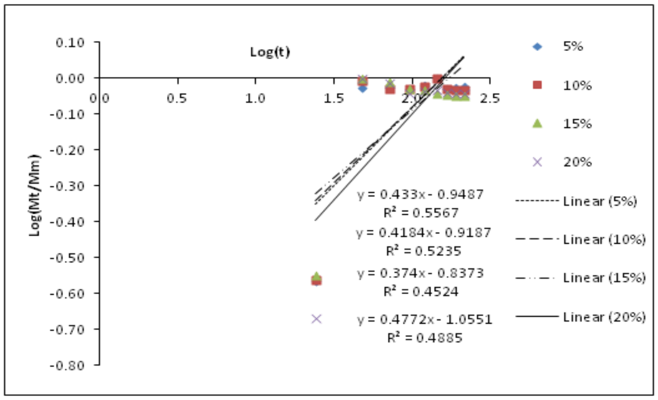

Fig. 7 Diffusion curve fitting for cenosphere filled hybrid jute-glass(GJJG) epoxy composites

The diffusion coefficient (D) is one of the important parameters of fick's model and shows the ability of water molecules to penetrate inside the composite structures. The values of D can be obtained from the initial slope of the plot of (M_t/M_m) against time $\sqrt{t}$ using the following equation.

$$D_x = \pi\left[\frac{h}{4M_m}\right]^2\left[\frac{M_2 - M_1}{\sqrt{t_2} - \sqrt{t_1}}\right]^2$$

Where
M_m: Equilibrium moisture content.
h: Thickness of the sample.
t_1 and t_2: Selected time points in the initial linear portion of the curve.
M_1 and M_2: Moisture content at time t_1 and t_2

The diffusivity value of filled hybrid jute-glass (GJJG) epoxy composite is given in table-3.

Table 3: Diffusivity of cenosphere filled hybrid jute-glass (GJJG) epoxy composite

Environment	**(%) Filler of Cenosphere**	**EMC (%)**	**Diffusivity (D_x) x 10^{-6} (mm^2/sec)**
Distilled water	5	3.26	1.6224
	10	2.14	3.1579
	15	2.61	2.4813
	20	1.82	3.8899
Saline water	5	2.118	2.4621
	10	2.091	2.0346
	15	1.880	2.7091
	20	1.680	2.8846

The maximum diffusivity value is found for 20wt.% cenosphere filled (GJJG) composite as compared to 5wt.% in both environmental conditions. The results for diffusion coefficients of developed hybrid composites were close to the reported value of literatures [34-35].The direct comparison of the diffusion coefficient obtained from this work with previous is difficult due to variation in filler content, manufacturing methods and test conditions.With better adhesion between matrix and filler,the velocity of the diffusion process decreases due to fewer gaps in interfacial region and presence of more hydrophilic groups as OH, that are blocked by the filler content. The presence of glass fiber at outer layers of composites also shows lower diffusion coefficient. It is possible due to lower hydrophilicity of the glass fiber as compared to jute fiber. In case of non-fickian transport, observed at high temperature, there is no cracking in the initial stage of the moisture absorption in the material and fick's law is obeyed. When cracking occurs, the experimental data deviates from the fickian behaviour, resulting in increasing water intake. According to Fujita [36], the non-fickian absorption process is one of the most general absorption features of glassy polymers. It is generally acquired that the penetrate diffuses rapidly into the polymer, accompanied by reversible elastic swelling of the matrix. The stress developed is then slowly relieved by molecular relaxation process such that the chemical potential of the absorbed water is decreased, leading to further absorption as reported [37].

4.3 Effect of moisture absorption on tensile and flexural properties

From figures 8 and 9, it is observed that flexural strength increases in 20wt.% cenosphere filled hybrid jute-glass (GJJG) epoxy composite and tensile strength is higher in 10wt.% cenosphere filled composite in distilled water as the immersion time increases.The results indicate that the material has practised some forms of physical damage or chemical degradation. The deterioration of bonding between fibre and matrix is also the reason for the decreased composite strength. The orientation of fiber also produces lower mechanical properties. This fibre mix-up creates resin rich areas, which can accord to the formation of voids and porosity. Voids and porosity can act as stress concentrator leading to failure of composite samples. The decrease of flexural strength after immersion can also be related to the weak fibre matrix interface due to moisture absorption [37].

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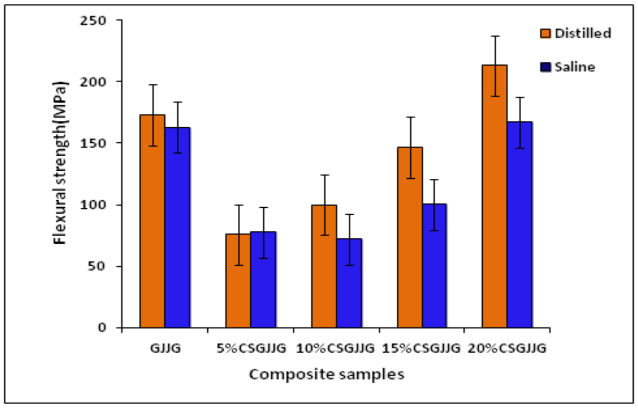


Fig. 8: Flexural strength of cenosphere filled hybrid jute-glass (GJJG) epoxy composites after exposure to different environmental conditions

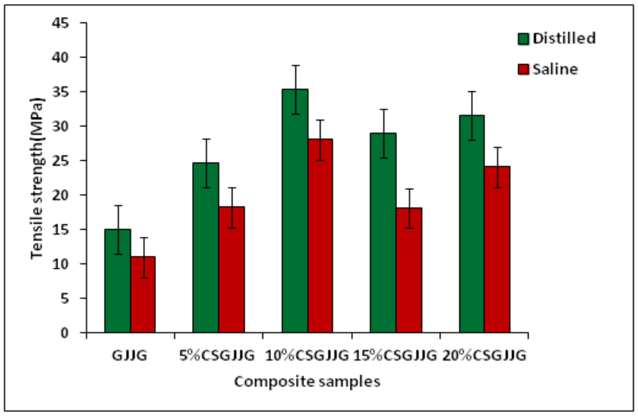


Fig. 9: Tensile strength of cenosphere filled hybrid jute-glass (GJJG) epoxy composites after exposure to different environmental conditions

5. SEM Analysis

Figures 10(a-d) presents the morphology of the tensile tested fractured surface of 20wt.% of cenosphere filled glass-jute(GJJG) epoxy composites exposed to both distilled and saline water environment at lower and higher magnification. Generally the mechanical properties of fiber reinforced composites depend on the nature of the polymer matrix, distribution and orientation of the reinforcing fibres with fillers and the nature of the fiber-matrix interfaces. Figures 10(a) presents the morphology of the fractured surface of cenosphere filled GJJG epoxy composites exposed to distilled water environment at lower magnification. From the figure the pull out of both jute and glass fiber is clearly visible. The same figure at higher magnification is shown in fig. 10(b). The pull out of fiber is clearly visible due to tensile load along with cavity formation. The immersion of cenosphere filled developed composites in water results in development of poor interfacial adhesion between fiber and matrix when subjected to tensile loading. This might have happened due to swelling of fiber which leads to breakage of jute fiber. However glass fibers are seen to be intact indicating no effect of swelling of glass fiber.

The fractured surface of the above composite exposed to saline water environment is shown in figure 10(c). Fiber breakage and swelling of fiber is observed for the composite under tensile loading. Small voids are being created in the fiber structure. This void gives space for water absorption due to which swelling of composites occurs that finally leads to deterioration of fiber strength. Figure 10(d) shows the same morphology of cenosphere filled hybrid glass-jute composite under saline water at higher magnification. Delamination and fiber pull out from the composite matrix surface is clearly visible in the figures. This is due to fact that when the fiber/matrix interface was subjected to moisture under saline water environment, the cellulose fibers got swelled. This resulted in the development of shear stress at the interface, which led to the ultimate debonding of the fibers, delamination and loss of structural integrity of filled hybrid composites.

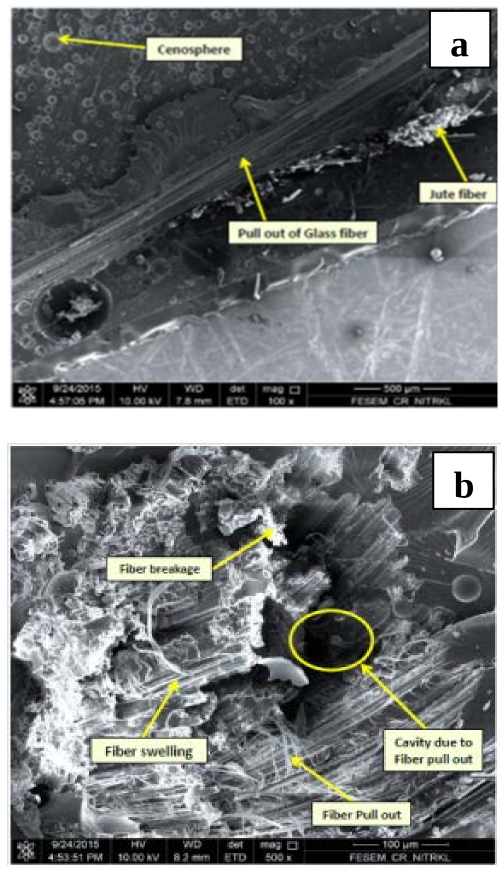


Fig. 10(a) and (b): SEM images of tensile fractured surfaces of 20wt.% cenosphere filled hybrid glass-jute (GJJG) epoxy composites in distilled water environment at lower (100x) and higher (500x) magnification

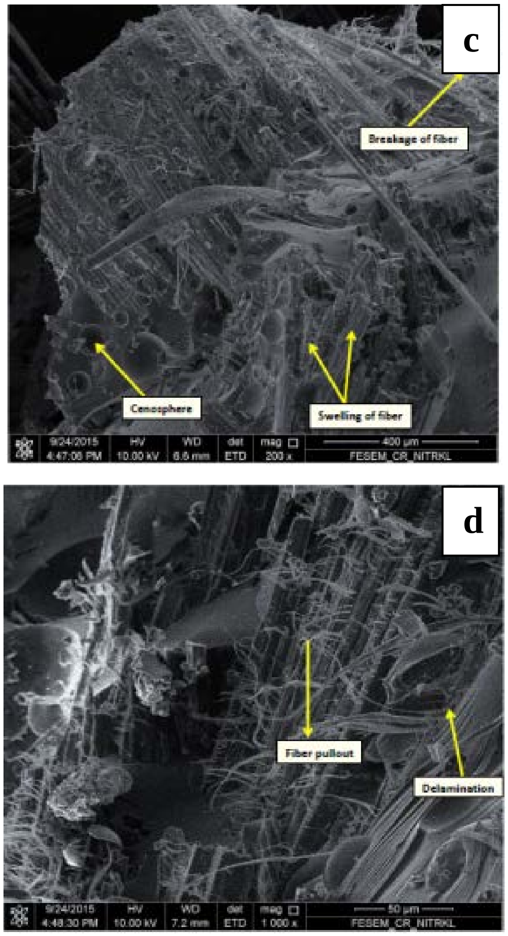
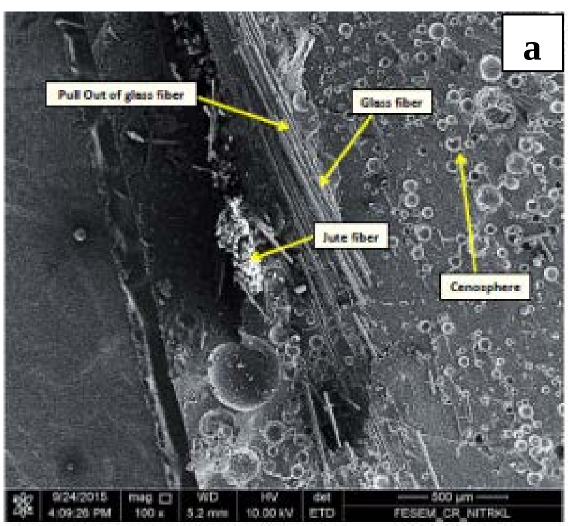


Fig. 10(c) and (d) SEM images of tensile fractured surfaces of 20wt.% cenosphere filled hybrid glass-jute (GJJG) epoxy composites in saline water environment at lower (200x) and higher (1000x) magnification



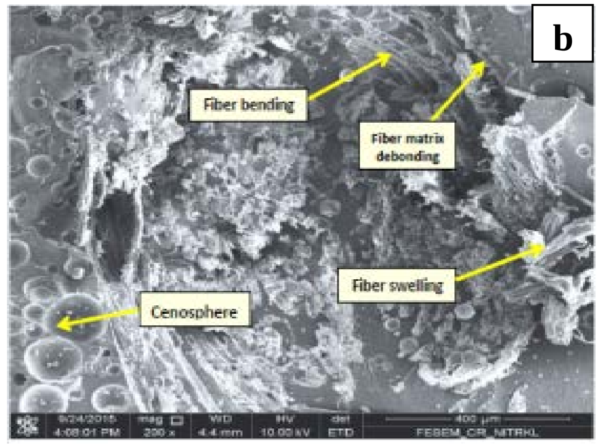


Fig. 11(a) and (b) SEM images of flexural fractured surfaces of 20wt.% cenosphere filled hybrid glass-jute(GJJG) epoxy composites in distilled water environment at lower (100x) and higher (200x) magnification

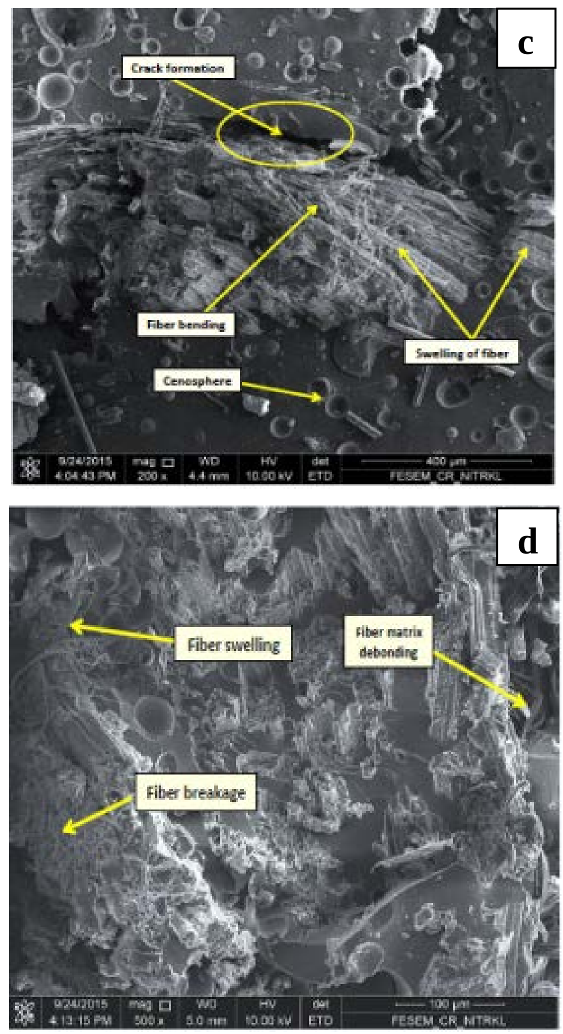


Fig. 11(c) and (d) SEM images of flexural fractured surfaces of 20wt. % cenosphere filled hybrid glass-jute (GJJG) epoxy composites in saline water environment at lower (200x) and higher (500x) magnification

Figures 11(a-d) shows the SEM micrograph of the bending fractured surface of 20wt.% cenosphere filled hybrid glass-jute(GJJG) epoxy composites exposed to both distilled and saline water environment at lower and higher magnification. Figures 11 (a) shows the pull out of glass fiber from the matrix surface along with jute fiber exposure during flexural failure under distilled water at lower magnification. The image analysis also shows the formation of voids due to the pulling out of the fiber. Figure 11 (b) shows the same micrograph at higher magnification. Fiber bending and swelling is also clearly seen in the fractured surface due to flexural load under distilled water.

When the cenosphere filled hybrid glass-jute (GJJG) composite is subjected to saline environment, crack formation takes place between fiber matrix interfaces along with bending of fiber as shown in figure 11(c).This might have happened due to the poor compatibility between fiber and the matrix surfaces. The same figure with higher magnification as shown in figure 11(d), the breakage of fiber along with fiber matrix debonding is clearly visible on the composite surface. Debonding between fiber and matrix and empty space between fibers and filler due to insufficient wetting are also clearly visible. This reveals that at higher filler loading poor fiber wetting occurs due to insufficient matrix material results in lower strength and modulus. This has also been seen in the experimental results shown in figures (8-9).This debonding at fiber-matrix interface is mainly responsible for degradation in mechanical properties. The decrease in mechanical properties with increase in moisture content is due to the formation of hydrogen bond between the water molecules and cellulose fiber leading to dimensional variation of composites products and poor interfacial bonding between the fiber and matrix.

6. Conclusion

From the experimental study of moisture absorption of various weight percentages (5,10,15 and 20%) of cenosphere filled woven hybrid jute-glass epoxy composites in two different environmental conditions (distilled and saline water), the following conclusions are made.

1. The maximum weight gain and thickness swelling increases in 20wt.% and decreases in 5wt.% cenosphere filled (GJJG) composite in both environmental conditions.It is due to the biodegradable filler particles generating voids at the interface increasing water uptake in the composite through capillary transport.
2. The moisture absorption trend is found to follow fickian behaviour and is predictable over period. The highest values of diffusion coefficient (D) and equilibrium moisture content (EMC) values were recorded for specimens immersed in distilled water as compared to saline water.
3. Exposure to moisture results in significant drops in tensile and flexural properties. The presence of porosity and degradation of fiber-matrix interface can be imputed to fiber-fiber interaction and proposed to have strong influence on flexural properties of composites for long time exposure in the hygrothermal environment.
4. SEM morphology confirms the fiber swelling, fiber pull out and poor interfacial bonding between fiber and matrix leading to variation in mechanical properties of composites.

Acknowledgments

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.I specially thankful to tribology laboratory of Mechanical Engineering department of Nit Rourkela for providing experimental facility in concerned research work.

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