Effect of Moisture Absorption on the Mechanical Properties of Ceramic Filled Jute/Epoxy Hybrid Composites

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Abstract. The present work emphasizes on the mechanical properties such as micro-hardness, flexural and impact strength of jute fiber and Al₂O₃ filler based polymer composites at dry and wet conditions. Composite samples reinforced with different wt.% of fibers and filler were prepared by hand lay-up technique. To improve the mechanical properties, jute fiber was hybridized with Al₂O₃ filler. The maximum flexural strength of 72.94 MPa and impact strength of 1.902 J is obtained for composites with 30 wt.% fiber content and 10 wt.% of filler content. The hardness of composite increases with increase in fiber and filler loading i.e 40 wt.% fiber content and 10 wt.% of filler content. The maximum hardness value is obtained 29.9 Hv. The effect of water absorption on mechanical properties of jute reinforced hybrid polymer composites is also investigated. To determine the influence of water absorption on the mechanical properties, specimens were immersed in distilled water for 10 days before testing. For reference purpose, dry specimens were tested. It is observed that the rate of water absorption depends on the fiber content as well as filler content. All the mechanical properties of composites are decreased after water absorption. Scanning electron microscopy (SEM) is used to characterise the microstructure and failure mechanisms of dry and wet jute fiber reinforced polymer composites.

Keywords: Jute fiber, Water absorption, Flexural strength, Impact strength, Scanning electron microscope.

1. Introduction
Recently, natural fiber composites pay attention for making environmental suitable engineering materials. The main advantages of natural plant fibers over synthetic fibers are acceptable as good specific strengths and modulus, cost-effective, low density and bio-degradability [1, 2]. The use of natural fibers reduces the weight by 10% and reduces the required energy to generate 80% while the cost of this component is 5% less than the comparable component fiber reinforced glass. Natural plant fiber-reinforced polymeric composites; also have some drawbacks such as the unsuitability between the hydrophilic natural fibers and hydrophobic thermoplastic and thermoset matrices. However, requiring the suitable use of physical and chemical treatments to enhance the adhesion between fiber and the matrix [3]. It is necessary that the composites retain their mechanical properties with less degradation in the water or moisture environments. Jute fibers are produced in large quantities, but jute easily absorbs water. The hydrophilic nature of the jute fibers makes the composites to water
absorption sensitive, which in turn reduces the interfacial bonding between the fibers and the resin and also plasticizes the resin matrix. Poor processing to induce voids leading to the water intake. Furthermore, the mechanical characteristics of composites can be improved by the inclusion of inorganic fillers to the matrix which aimed the cost reduction, improvement in performance of polymer composites and some cases improve process ability. Several researchers have studied that addition of ceramic fillers in polymer composite enhances the mechanical properties of the resulted composite [4, 5]. Alumina oxide (Al₂O₃) commonly referred to as alumina, is the most cost effective and broadly used material in the family of engineering ceramics and has the potential to be used as filler in various polymer matrices. With an excellent arrangement of properties and a reasonable price, it is no wonder that fine grain technical grade alumina has a very wide range of applications. It is essential that the composites retain their mechanical properties with less degradation in the water or moisture environments. Also, it is believed from the water environment that water molecules will enter quickly the interphase of the composites between the fiber and the resin because of the capillarity. Moisture absorption leads to the degradation of fiber-matrix interface region creating poor stress transfer efficiencies resulting in the reduction of mechanical and dimensional properties. Therefore, the present study investigates the effect of Al₂O₃ filler on the water absorption behaviour of jute fiber-reinforced epoxy composite with a different percentage. However, according to the best knowledge of authors, no research was reported about the effect of water absorption on the mechanical properties of jute/Al₂O₃ reinforced hybrid polymer composites. Comparative analyses of composites were studied before and after water absorption.

2. Materials and methods

In the current study woven jute fiber and Al₂O₃ is taken as reinforcement and epoxy is taken as matrix material. The jute fiber is collected from local sources and the Al₂O₃ is obtained from NICE Ltd India in a range of 80-100 micron. The epoxy resin and the corresponding hardener HY-951 is brought from Ciba Geigy India Ltd.

2.1. Composite fabrication

The fabrications of the composite slab were carried out by conventional hand layup technique. The low-temperature curing epoxy resin and corresponding hardener were mixed in a ratio of 10:1 by weight as recommended by the manufacturer. Composites with four different fiber loading (10, 20, 30 and 40 wt.%) with three different filler loading (0, 5 and 10 wt.%) were fabricated and subjected to post-curing at room temperature for 24 h. The detail designation and composition of composites are given in Table 1.

| Composites | Compositions               |
|------------|---------------------------|
| J1         | Fiber (10%)+Filler(0%)+Epoxy(90%) |
| J2         | Fiber (20%)+Filler(0%)+Epoxy(80%) |
| J3         | Fiber (30%)+Filler(0%)+Epoxy(70%) |
| J4         | Fiber (40%)+Filler(0%)+Epoxy(60%) |
| J5         | Fiber (10%)+Filler(5%)+Epoxy(85%) |
| J6         | Fiber (20%)+Filler(5%)+Epoxy(75%) |
| J7         | Fiber (30%)+Filler(5%)+Epoxy(65%) |
| J8         | Fiber (40%)+Filler(5%)+Epoxy(55%) |
| J9         | Fiber (10%)+Filler(10%)+Epoxy(80%) |
| J10        | Fiber (20%)+Filler(10%)+Epoxy(70%) |
| J11        | Fiber (30%)+Filler(10%)+Epoxy(60%) |
| J12        | Fiber (40%)+Filler(10%)+Epoxy(50%) |
2.2. Water Absorption
Moisture absorption studies were performed according to ASTM D 570-98 standard test method for moisture absorption of plastics. The samples were taken out periodically and after wiping out the water from the surface of the sample weighted immediately using a precise balance machine to find out the content of water absorbed. The specimens were weighed regularly at 24, 48, 72, 96, 120, 144, 168, 192, 216, 240, 264 hours. The moisture absorption was calculated by the weight difference. The weight gain in percentage of the samples was measured at regular time intervals of time by using the following equation:

\[
\text{Water absorption (\%) } = \frac{W_n - W_d}{W_d}
\]  

(1)

Where \( W_n \) is the weight of composite samples after immersion and \( W_d \) is the weight of composite samples before immersion.

2.3. Mechanical testing
The flexural strength of a composite is determined by the maximum tensile stress that it can withstand during bending before getting the breaking point. The flexural strength measured in three-point bend test to understand the flexural behaviour of composites using the universal testing machine (Instron 1195). The test is conducted as per ASTM standard D2344-84.

\[
F.S = \frac{3PL}{2bh^2}
\]  

(2)

Where \( P \) represent maximum load, \( b \) width of specimen, \( t \) thickness of specimen and \( L \) span length of the specimen.

Impact is a single point test that measures the resistance of material to impact from a fluctuation pendulum. This test can be used as a fast and easy quality control check to determine if a material meets specific impact properties or to compare materials for general toughness. For composite specimens low velocity instrumented impact tests are carried out. The tests are done as per ASTM-D 256 using an impact tester.

Leitz micro-hardness tester is used to found out micro-hardness measurement. A diamond indenter, in the form of a right pyramid with a quadrangular base and an angle 136° between opposite faces, is hard-pressed into the material under a load \( F \). The two diagonals \( X \) and \( Y \) of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean \( L \) is calculated. In the current study, the load considered \( F = 24.54 \text{ N} \) and Vickers hardness number is calculated using the following equation.

\[
H_v = 0.1889 \frac{F}{L^2} \quad \text{and} \quad L = \frac{X + Y}{2}
\]  

(3)

Where \( F \) is the applied load \( (N) \), \( L \) is the diagonal of square impression (mm), \( X \) is the horizontal length (mm) and \( Y \) is the vertical length (mm).

Results and discussion
The results obtained from the experimental study can be divided into two parts. The first part reflects water absorption behaviour of jute/\( \text{Al}_2\text{O}_3 \) polymer composites and the second evaluates the effects of water absorption at room temperature on the mechanical properties.
3.1. Water absorption behaviour
The effect of fiber loading on the water absorption behaviour of jute-epoxy composites filled with Al$_2$O$_3$ particulate filler with an increase in immersion time is shown in Figure 1. It is evident from the figure that the rate of water absorption of the composites increases with increase in immersion time. After a certain value of water absorption a saturation point will reached where no more water absorption takes place. It has been reported by the researchers that the water absorption rate increases with the increase in fiber loading [6]. The reason may be due to the fact that as the content of natural fiber increases in the composite, the number of free OH groups of cellulose increases and therefore, the moisture absorption increases. These free OH groups come in contact with moisture and form hydrogen bonding and results in weight gain in the composites. From Figure 1, it is also observed that J12 composite (i.e. with 40 wt.% fiber loading and 10 wt.% filler content) has maximum water absorption. It is due to the presence of a large amount of fibers and voids which absorbs more water in the composites. In comparison to all the composites fabricated, composite J1 (having 10 wt.% fiber loading and 0 wt.% filler loading) exhibits minimum water absorption rate due to the presence of less fiber and voids in the composite.

![Figure 1. Water absorption behaviour of jute/Al$_2$O$_3$ composites](image)

The main disadvantage in the use of natural fibers as reinforcement is that the fibers are more sensitive to water which will increase the dimension of the composites and also will reduce the mechanical properties [7, 8]. Basically, moisture absorption of the composites would lead to swelling and degradation at the fiber-matrix interface which may result in poor stress transfer resulting in poor mechanical properties and dimensional stability [9].

3.2. Effect of water absorption on mechanical properties
The effect of water absorption on the mechanical properties of jute/Al$_2$O$_3$ polymer composites was investigated after placing specimens in water at room temperature and comparing them with samples of the same composites kept in dry conditions.
3.2.1. Flexural strength
The effect of fiber and filler content on the flexural strength of dry jute/Al$_2$O$_3$ polymer composites is presented in Figure 2. In the dry condition, flexural strength increased as fiber content increased at 30 wt.% of fiber loading and 10 wt.% of filler loading. The flexural strength of jute/Al$_2$O$_3$ Polymer increased from 48.1 to 72.94 MPa due to the addition of Al$_2$O$_3$. This enhancement in flexural strength of jute/Al$_2$O$_3$ Polymer composites is may be due to the ability of natural fiber to resist bending forces and good stress transfer from the matrix resulting in improve strength properties [10].

![Figure 2. Flexural strength of jute/Al$_2$O$_3$ polymer composites in dry and wet conditions](image)

The effect of water absorption on flexural strength of jute/Al$_2$O$_3$ polymer composites is also presented in Figure 2. It can be seen that the flexural strength of composites decreased after water absorption. Wet composites characteristics become decrease as compared to dry composites. This may due to the fact that the immersion of the composite samples in water affects the interfacial adhesion between fiber and matrix and makes de-bonding, leading to a decrease in mechanical properties of composite. When the fiber matrix interface was accessible to moisture in the environment the jute fibers expanded. Development of shear stress at the interface led to the ultimate de-bonding of the fibers, delamination and damage of structural integrity [11].

3.2.2. Impact strength
Impact strength is an important property that provides a sign of overall material toughness. Impact strength of fiber/filler-reinforced polymer is governed by the matrix–fiber/filler interfacial bonding, and the properties of both matrix and fiber/filler. After composites endure a sudden load, the impact energy is dissipated by the combination of fiber/filler pull-outs, fiber rupture and matrix deformation.
Generally impact strength increases as fiber content increases in fiber-reinforced polymer composites because of the increase in fiber pull out and fiber breakage [13].

![Figure 3. Impact strength of jute/Al\textsubscript{2}O\textsubscript{3} polymer composites in dry and wet conditions](image)

It can be seen from Figure 3 that impact strength rapidly increased up to fiber content 30 wt.% and filler content 10 wt.% in dry composites. The presence of Al\textsubscript{2}O\textsubscript{3} in the matrix increases the ability of these composites to absorb impact energy. In dry conditions, the addition of Al\textsubscript{2}O\textsubscript{3} with fillings of 0, 5 and 10 wt.% and jute fiber with filling of 10, 20 and 30 wt.% increases the impact strength from 1.25 and 1.902 J respectively. After 30 wt.% of fiber loading and 10 wt.% of filler loading it decreases may be the matrix failure or deboning of fiber and filler loading.

Nevertheless, impact strength is unfavourably affected by water absorption. The decrease in impact properties after water immersion can be linked to the weak fiber–matrix interface, which resulted in a reduction of the mechanical properties and dimensional stability of composites [14].

3.2.3. Hardness

The effect of jute fiber and Al\textsubscript{2}O\textsubscript{3} filler contents on the hardness of the polymer composites is presented in Figure 4. The hardness of polymer composites reinforced with 0, 5 and 10 wt.% filler loading and 10, 20 and 30 wt.% of fiber loading increased from 21.4 to 29.9 Hv, respectively. This enhancement in hardness is caused by the distribution of the test load on the fibers, which reduced the penetration of the test ball on the surface of the composite material and accordingly improved the hardness of this material [15].
However, hardness is affected by water absorption, as shown in Figure 4. Hardness decreases in all cotton fiber-reinforced samples in wet condition, and is associated with the weakening of inter-face between the polymer matrix and the jute fiber caused by the water absorption. This type of decrease has been founded by other researchers working with natural fiber-based composites. As water absorption increased, the hardness of flax fiber-reinforced composites decreased, and found that the deformation depth increased for water-immersed specimens compared to dry ones, due to the hydrophilic nature of the fibers, and eventually led to the formation of a weak fiber–matrix interface [16].

3.2.4. Morphology study

Figure 5 a-c shows the micrographs of fractured specimen after flexural test in both dry and wet condition. SEM micrograph specifies the phenomenon of ‘pull-out’ occurred to a greater extent causing the failure of material. The image analysis also shows the formation of voids due to fiber pull-out, de-bonding, breakage. Overall, larger amount of broken fibers were observed if compared to the number of voids associated to the fiber pull-out. At some region weak fiber-matrix adhesion were observed, which can be attributed to low compatibility between hydrophilic nature of fiber and hydrophobic nature of matrix used.

Figure 5 (a) and (b) represents the wet fractured specimen, whereas Figure 5(c) shows the dry fractured specimen after the flexural test. It has been witnessed from the figure that more void created in wet type of fractured specimen. The fiber breakage, tearing of fibers, and pull-out were detected from fractographs. The voids and fiber entanglement were also found due to poor blending quality. These defects may act as crack-tips leading in matrix cracking portents or, in the second thing, resin
reach areas that leading failure of the composite bond. In the case of dry fractured test, fewer voids occur. Overall, it is witnessed that due to water absorption, the fiber strength is reduced and the swelling is more when compared to other composite, breakage of fiber is also observed along with fiber pull-out due to flexural loading.

Figure 5 (a-c). SEM of jute/Al₂O₃ polymer composites after flexural test in case of dry and wet condition

4. Conclusion
In the present investigation, the effect of moisture absorption characteristics and its effect on mechanical properties of jute fiber and Al₂O₃ filler reinforced epoxy composite were studied. The incorporation of jute fiber and Al₂O₃ filler significantly increases the mechanical properties (e.g. flexural strength, impact strength, micro-hardness) of the composite.

However, jute fibers are hydrophilic in nature and hence have a poor resistance to water absorption. The water absorption of jute-reinforced polymer composites at room temperature was found to increase with increasing fiber content. Introduction to moisture for an extended period causes a reduction in flexural strength, impact strength, and hardness. A reasonable clarification for this would be that bonding at the fiber–matrix interfaces is degraded as a result of water absorption. After water absorption, SEM images of the composites confirm that jute has a great tendency of swelling and absorb more moisture.
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