Acid resistance of flax braided reinforced epoxy composite tubes

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Flax braided reinforced epoxy composites have been prepared by vacuum assisted direct resin injection moulding method. The prepared composites are then tested for flexural strength and flexural modulus. The effect of sulphuric acid concentration and immersion time on the diffusion behavior of the composite tubes has been studied. The result indicates that the sulphuric acid has a significant effect on the diffusion coefficient of the composites. With the increase in acid concentration, the diffusion coefficient values decrease initially and later increase at higher concentration. The flexural strength of the composites is found to be lower for water immersed samples as compared to that for the control samples.

Keywords: Braided composite, Circular braided fabric, Flax fibre, Flexural strength

1 Introduction

Textile structural composites are nowadays finding interesting applications right from low end applications such as furniture and tables to high end applications such as golf clubs, tennis rackets, aircraft and missiles as they offer high degree of anisotropy and formability. Moreover, the fibre reinforced plastics (FRP) has been in prominence in past few years because of their light weight and high strength. Fibres such as glass, carbon, boron and kevlar have been predominantly used as reinforcement in FRPs. Natural fibre, such as jute, coir, banana, flax, sisal and pineapple, are nowadays gaining importance as reinforcement in FRPs. Natural fibres like flax, jute and sisal have specific strength and specific modulus, similar to that of glass fibres.

One of the important methods to prepare the preform using natural fibres is braiding, which is an efficient method of arranging fibres. The advantage of braiding is that fibre orientation and placement can be precisely controlled. A braid is a textile structure that is produced by interlacing of three or more strands together and is very suitable for composite industry as the process has the ability to form different geometric shapes, ranging from thin walled tubes to solid shaped beams. Braided composites offer two basic mechanical characteristics such as effective delamination resistance and good formability. The good formability of braiding eliminates the need for cutting fibres to join, splice or overlap and associated local strength loss.

Generally, glass fibre is used in composite industry because of strength and chemical resistance but nowadays natural fibre based composites are replacing glass fibre based composites due to their low density, easy availability and liberation of low toxic content on decomposition. Among all the natural fibres, flax fibre has higher strength and stiffness and lower density as compared to that of glass fibre, thereby offering scope for higher specific modulus and specific strength. But moisture and chemicals (such as acids) are the two most important factors that affect the performance of the natural fibre reinforced composite in service. These media can attack the fibre matrix interface, thereby causing substantial loss in the composite strength.

In the present study, flax braided fabrics have been used as reinforcement and the composites are prepared with epoxy resin as a matrix. The flexural strength and flexural modulus of the prepared composites are tested by immersing the specimen in water and sulphuric acid.

2 Materials and Methods

2.1 Braided Flax Reinforcement

Flax yarn of 44 linen counts was used for braiding. The braided preform was prepared in a conventional maypole braiding machine with 32 spindles. The braiding angle of the specimen was kept at 45°.

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2.2 Preparation of Hollow Tube

A mandrel with size equal to the inner diameter of the braided composite tube was produced. The mandrel was coated with paraffin wax along with mould releasing agent for smooth release of the composites. The braided fabric reinforcements were then cut to desired dimension and inserted into the mandrel. The fabric along with the mandrel was placed in the tubular vacuum chamber.

Epoxy resin (HY 951) along with hardener (XY 54) in ratio of 2:1 was prepared and then injected in to vacuum chamber by vacuum assisted resin injection method as shown in Fig. 1 (ref. 11). The composite was cured at room temperature. The volume fraction and void fraction of the composites were 28% and 5% respectively.

2.3 Preparation of Test Specimen and Exposure to Acid and Water

The braided fabric reinforced composites were immersed separately in distilled water and sulphuric acid (at various concentrations) in a container. The test specimens were withdrawn periodically and then weighed carefully after wiping them dry. Two way analysis of variance (ANOVA) test was carried out to test the significance of the effect of the concentration and immersion time on the percentage weight gain of the prepared composite. The absorption of water and sulphuric acid has been calculated using the following relationship:

\[
M(t) = \frac{W_t - W_o}{W_o} \times 100 \quad \ldots (1)
\]

where \(M(t)\) is the percentage of weight gain; \(W_t\), the weight measured after immersion of specimen; and \(W_o\), the weight measured before immersion of specimen. The percentage of weight gain was then plotted against \(\sqrt{t}\) to generate the moisture absorption curves necessary to calculate the composite diffusion coefficient. Each data point in the plot represents an average of three specimens. Diffusion coefficient \(D_c\) is calculated from the slope of moisture content vs the square root of time plot by using the following formula:

\[
D_c = \pi \left( \frac{h}{4M_m} \right)^2 \frac{M_2 - M_1}{(\sqrt{t_2} - \sqrt{t_1})^2} \quad \ldots (2)
\]

where \(M_m\) is the maximum moisture content; \(M_t\), the moisture content at time \(t_1\); \(M_2\), the moisture content time \(t_2\); and \(h\), the specimen thickness.

2.4 Flexural Strength Determination

The flexural strength of the specimens was determined according to ASTM D 790 standard. The span length of the test specimen was 100mm and the cross-head speed was kept at 2mm/min. Two way analysis of variance (ANOVA) test was carried out to study the significance of the effect of acid concentration and immersion time on the flexural strength and flexural modulus of the prepared composite. The flexural strength of the specimen was calculated using the following formula:

\[
R_f = \frac{P_{Max} \times L \times C}{4I_x} \quad \ldots (3)
\]

where \(R_f\) is the bending strength (MPa); \(P_{Max}\), the maximal load (N); \(L\), the distance between supports (mm); \(C\), the \(D/2\); \(D_s\), the outer diameter of pipe (mm); \(I_x\), the section modulus (mm\(^4\)).

Section modulus \(I_x\) was calculated using the following formula:

\[
I_x = \frac{\pi}{64} \left( D_s^4 - d_u^4 \right) \quad \ldots (4)
\]

where \(D_s\) is the outer diameter of pipe (mm); and \(d_u\), the inner diameter of pipe (mm).
Modulus of elasticity \( (E_f) \) was calculated using the following formula:

\[
E_f = \frac{PL^3}{48\delta I}
\]

(5)

where \( E_f \) is the bending modulus (MPa); \( P \), the load force (N); \( L \), the distance between supports (mm); \( I \), the moment of inertia (mm\(^4\)); and \( \delta \), the deflection (m\(^4\)).

3 Result and Discussion

In the present study, hollow tube to be used for fluid transmission has been prepared and its performance against corrosive environment such as sulphuric acid is discussed hereunder.

3.1 Effect of Acid and its Diffusion into Composites

Figure 2 shows the effect of acid concentration on weight gain of the composites. Both acid concentration and immersion time show significant effect on the moisture absorption. Moreover, it can be observed from the figure that with the increase in duration of immersion in acid and water, the amount of water absorbed by the composite increases up to a certain period and gets saturated after a time period of thirty days. It is interesting to note that the increase in weight gain of composites is higher for water immersed samples as compared to that for acid soaked samples (Fig. 2). This may be due to the easier penetration of water molecules inside the composite through capillary action as compared to acid which may lead to etching of matrix and reinforcement. The mechanism of diffusion of water and other molecules in epoxy matrix system has been well explained by other researchers\(^ {14} \). The degradation of resin system involves the formation of hydrophilic layer followed by reorientation of hydrophilic group inside the resin system in a cyclic manner. Moreover, the degradation of epoxy matrix composite involves a complex process of thermodynamic action and electrostatic energy, as suggested by other researchers\(^ {14} \).

3.2 Fickian Plot and Calculation of Diffusion Coefficient

For the concentration independent moisture diffusion process following the Fickian second law, the general solution is given by the following relationship\(^ {12} \):

\[
M - M_i = 1 - \frac{4}{\pi} \sum_{j=0}^{\infty} \frac{1}{(2j+1)} \times \sin \left( \frac{(2j+1)\pi x}{h} \right) \exp \left( -\frac{(2j+1)^2 \pi^2 D_c t}{h^2} \right)
\]

(6)

where \( M \) is the moisture gained by the composite at any time \( t \); \( M_m \), the maximum moisture gained by composite; \( D_c \), the diffusion coefficient; \( T \), the time duration; and \( h \), the thickness of laminate.

For longer period of exposure Eq (6) can be approximated by the first term in the series\(^ {12} \), as shown below:

\[
\frac{M}{M_m} = 1 - \frac{8}{\pi^2} \exp \left( -\frac{\pi^2 D_c t}{h^2} \right) \text{ for } \left( \frac{D_c t}{h^2} \right) < 0.05
\]

(7)

On the other hand, for short period, an approximation can be obtained from an alternative solution, as given below\(^ {12} \):

\[
\frac{M}{M_m} = \frac{4}{\pi} \left( \frac{D_c t}{h^2} \right)^{1/2} \text{ for } \left( \frac{D_c t}{h^2} \right) < 0.05
\]

(8)

Thus, the initial increase in moisture content is proportional to \( (t/h^2) \). The applicability of Eq (8) becomes apparent if the moisture content is plotted as a function of square root of time (\( \sqrt{t} \)).

Thus, the moisture absorption curve has been found to follow Eq (7), with an initial slope that can

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**Fig. 2—Effect of sulphuric acid concentration on flax braided composite tube**
conveniently be represented by Eq (8). It has also been shown that the plots represented by Eqs (6), (7) and (8) merged into a straight line during the initial part of the exposure period, indicating that the initial linear part of the moisture absorption curve could be conveniently used to evaluate the concentration independent diffusion coefficient for a practical analysis of the diffusion problems. With the above view, the Eq (7) also can be written as:

$$G = \frac{M}{M_m} = 1 - \frac{8}{\pi^2} \left( \frac{1}{\exp \left( \frac{D_c t}{h^2} \right) \pi^2} \right)$$ … (9)

$$G = \frac{M}{M_m} = 1 - \frac{8}{\pi^2} \left( \frac{1}{\exp \exp \left( \frac{D_c t}{h^2} \right) \pi^2} \right)$$ … (10)

From Eq (9), it can be noted that a plot between the dimensionless absorption parameter ($G$) and dimensionless parameter $\ln(D_c t/h^2)$ forms the analytical solution for the moisture absorption problem.

Then from the experimentally generated data, the two dimensionless parameters were calculated and plotted on the analytical curve. The extent of correlation between the two clearly shows the applicability of Fickian diffusion model to the composite system chosen$^{12}$. Fickian plot for flax braided composite tube in 35% v/v H$_2$SO$_4$ concentration is given in Fig. 3. Similar trend of Fickian plot has also been observed for other sulphuric acid concentrations (0%, 15% and 25% v/v). From the figure, it can be seen that the good correlation exists between the analytical curve and the experimental data, indicating good applicability of Fickian diffusion model to the composite specimen immersed in water and acid.

The comparison of $D_c$ values shows that as the acid concentration increases, the $D_c$ decreases (Fig. 3), thus indicating that the process of diffusion being sluggish as observed by other researchers in case of jute fibre reinforced epoxy system$^8$.

### 3.3 Effect of Acid Concentration on Flexural Strength

When the composite laminates are subjected to different kinds of forces, the behavior of tubes is bound to be different. When a laminate is subjected to a flexural load, it is subjected to both compression at the top and tension at the bottom. These result in competition between tensile, compressive and shear failure mechanism when the laminates are subjected to a bending load. In other words, the flexural behavior of the laminates depends on the fibre bundles bringing the fracture plane$^{15}$. The typical load elongation curve for the flexural test is given in Fig. 4. Figures 5 (a) and (b) show the SEM image for the flexural failure of flax braided reinforced composite tubes of control sample and after acid (35% H$_2$SO$_4$) immersion respectively.

Figures 6 (a) and (b) show the plot of flexural modulus and flexural strength of flax braided reinforced composites immersed at regular intervals of time. The ANOVA results obtained for two parameters show that both the acid concentration and

![Fig. 3—Fickian plot for flax braided composite tube in 35% H$_2$SO$_4$ concentration](image-url)

![Fig. 4—The typical load elongation curve for the flexural test](image-url)
immersion time has significant effect on the flexural modulus and flexural strength.

It is observed that the degradation due to water has a profound effect on flexural modulus and flexural strength of composites as compared to that of acid soaked samples. This is due to ease of diffusion of water molecules inside the composite, resulting in debonding of fibre and matrix compared to the acid soaked samples. A similar result has been observed for carbon, glass and jute composites by other researchers who attributed it to plasticization and viscoelastic stress relaxation.\(^\text{16}\)

### 4 Conclusion

4.1 The moisture absorption profile of the flax braided reinforced composites follows Fickian distribution.

4.2 The diffusion coefficient values of samples immersed in water are higher than that of samples immersed in acid, except at higher acid concentration (35%).

4.3 The increase in immersion period in both water and acid medium leads to decrease in flexural strength and flexural modulus.

4.4 The decrease in flexural modulus and the flexural strength is found to be higher for water immersed samples as compared to that for acid soaked samples.

### References