

## Moisture Diffusion Studies on Silk Reinforced Composites

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### Abstract

Nowadays, the natural fibre reinforced composites particularly silk fibre gained interest because of its low impact to the environment, high strength, light weight etc. Textile reinforced composites are preferred to metals in medical devices and implantable materials the major drawback with this composite is its absorption of moisture due to the presence of the hydrophilic group. During exposure to humidity and temperature, the properties of the composites are prone to degrade affecting the overall objective. Water tends to penetrate in to the composite structure affecting the material properties of the composite structure. Based on this, the present study focuses on the moisture diffusion behavior of the woven silk epoxy resin composite. The woven silk fabric was produced using weaving machine and the composite was made using the simple hand lay-up technique. The moisture diffusion behavior study was carried out by immersing the sample in the water. Composites has been studied at different intervals of time namely 5 days, 10 days, 15 days, 20 days and 25 days. The effect of water at various intervals of time on the mechanical properties such as tensile, flexural and impact were also carried out. Moisture diffusion increases at different time intervals. The rate of diffusion significantly changes with respect to time. The tensile and flexural modulus of immersed sample is less compared to control sample. The mechanical properties such as tensile and flexural properties of the composites decreases with the increase in the immersion time because of the increase in the absorption of moisture with time leads to the matrix cracking and de-bonding of the composite.

**Keywords:** Silk composite, Compression moulding, Silk fabric, Moisture, Diffusion

### INTRODUCTION

Nowadays, the composite materials constitute the significant proportion of the engineered materials market, ranging from everyday products to sophisticated niche applications. A composite is a heterogeneous combination of two or more materials differing in their form or composition on a macro scale. By combining different materials in an integral composite material it is possible to satisfy the requirement of the user. Such composite material system results in a performance unattainable by the individual constituents and they offer an advantage of a flexible design. Using the composite technology, the material can be made as per the specifications of an optimum design, which makes it used in an aerospace structure, an automobile, a boat or an electric

motor to meet the need. Composites, the wonderful material with light weight, high strength and sufficient amount of stiffness properties have come a long way in replacing the conventional materials like metals, woods etc.

There are three commonly accepted types of composites are available. They are fibrous composites, structural composites and particulate composites. Among the various types of composites fibrous composites have been more prominent than the other types of composites because most of the materials are stronger and stiffer in their fibrous forms than in the any other forms. In recent years, the natural fibres such as banana, coir, sisal, jute etc as reinforcement occupies the major role because of its low cost, easy availability and environmental friendliness. These natural fibre reinforced composites find their application in consumer goods, low cost housing and other civil structures. Among the various other natural fibres, silk is also appears as a right candidate for composites because possible to achieve sufficient amount of tensile strength, specific modulus and stiffness with its minimum mass per area and thickness. Therefore investigation of silk based composites and its characteristics with the consideration of its thickness and mass per unit area will give inside to produce special composites for different application in the different environment.

Composites encompass both the properties of the reinforcement and the matrix and therefore the property of the composite depends not only on the reinforcement but also on the surrounding matrix in which it is embedded. In contrast, these composites are very sensitive to the atmosphere or the environment in which it is present and absorbs the moisture from the atmosphere irrespective of the knitted, braided or woven performs. In particular, the natural fibres absorb more moisture when compared to the other high performance synthetic fibres such as glass. On absorption of the moisture the weight of the composites changes as the consequence of the water absorption for a prolonged period of time. This change in weight of the composite is closely related to the degradation of the mechanical properties. This reduction in the mechanical properties is mainly due to the weakening of the interface adhesion. The moisture absorption of the composites are influenced by the various internal and external factors such as the fibre orientation, fibre volume fraction, relative humidity and temperature. Many of the studies has been carried out for stability towards moist environment. Silk is also one of the special composite it has fine thickness and low mass per unit area. Hence silk based composites and its properties with respect to different moist environment are one of the essential requirements for being utilize in the moist environment.

The laminates with chopped strand mat as a skin layers have a higher diffusion coefficient than those with woven roving as skin layers of glass reinforced composite and diffusion coefficient increases with the increase in the number of chopped strand mat layers in the laminates<sup>1-10</sup>. The moisture content of the glass epoxy composites increases with the increase in the relative humidity and the diffusion coefficient value decreases marginally with an increase in the relative humidity<sup>15-20</sup>. The good correlation of Fickian diffusion behavior in polymer composites containing impermeable fibres<sup>33-37</sup>. The moisture absorption of the silk and the glass composites varies with the various factors such as fibre volume fraction, fibre orientation angle, relative humidity and temperature<sup>41-44</sup>. The sisal composites exhibit higher moisture absorption than the jute composites and the jute composites exhibit good correlation than the sisal composites<sup>44-46</sup>. Composites comprising both impermeable and permeable fibre composites show good correlations with the analytical Fickian diffusion plot<sup>51-55</sup>. One of the most important issues of polymeric composites is their degradation behavior upon exposure to environmental conditions such as humidity and temperature<sup>1-3</sup>. Typical epoxy formulation can absorb several weight percent of water, seriously degrading the physical properties of the resin Fibrous composites are susceptible to heat and moisture when they are used in harsh and changing environmental conditions. Moisture transport in polymer systems is related to the availability of molecular- sized holes in the polymer structure and the polymer-water affinity. The mechanical response of materials is sensitive to the rate of loading.

In addition the moisture absorption behavior of the silk composites are affected by the various factors such as relative humidity, temperature, orientation angle, number of lay ups etc., so, it is essential in studying the moisture diffusion behavior and the effect of the above said various factors on the moisture absorption behavior of the composites. In addition the mechanical properties of both the matrix and the silk component degrade with the prolonged exposure to the environment during their service time. Hence it is important to study the effect of the environmental conditions such as moisture and relative humidity on the mechanical properties of the silk composites. On the whole silk is also a right candidate for composites because possible to achieve sufficient amount of tensile strength, specific modulus and stiffness with its minimum mass per unit area. Hence, the attempts were made for production of silk based composite and assess the suitability of different environments.

The objectives of the present work are

- To study the moisture diffusion on the woven silk epoxy resin composites
- To characterize the woven silk epoxy resin composites through various tests such as tensile test and flexural test by varying the different intervals due to the mobility of the absorbed water in the composite that leads to the degradation of the adhesion strength in the composite.

## MATERIALS AND METHODS

The present study is focused on the moisture diffusion studies on the woven silk epoxy resin composites and the effect of moisture on the mechanical properties of the woven silk epoxy resin composites. The woven fabric obtained by weaving process was then made into a composite. The samples were immersed at various intervals of time and also to study the effects of mechanical properties of the composites. The particulars of the materials used and the various experimental procedures adopted in the study are described in this chapter. Commercial silk filament having the following particulars is used in the study.

### Fabric specification

Ends/inch	:	63
Picks/inch	:	53
Warp count	:	147s
Weft count	:	147
Fabric cover factor	:	9.2254
Fabric weight	:	19.4 g/m <sup>2</sup>

### Resin specification

Epoxy resin	:	Araldite LY556
Hardener	:	HY951
Curing	:	Room Temperature
Density of epoxy resin	:	1.6g/cm <sup>3</sup>

### Composite Preparation

The composites were prepared by means of simple hand lay up technique. Initially, the working surfaces were coated with wax for easy removal of the laminates from the moulds. The mould was prepared in square shape of 250mm length, 250mm width and 5mm thickness. Before placing the reinforced material in the mould a wax coating was applied over the mould, followed by 5 to 10 min of time was allowed for drying through sunlight. A thin polyethylene sheet was wrapped over wax coated surface to avoid sticking of reinforced material in the mould while separating the composites from the mould.

### Preparation of epoxy and hardener

The matrix that is used to fabricate the fibre specimen is epoxy LY 556 of density 1.6 g/cm<sup>3</sup> and hardener HY 951 of density 1.0 – 1.05 g/cm<sup>3</sup>. The epoxy and hardener was mixed with weight ratio 10:1.

Some of the test methods are shown below

**Moisture Studies**

The laminates were cut to a dimension of 25.4 mm X 76.4 mm X 1 mm 8 lay up sequence. The dry specimens were kept immersed in the distilled water maintained at room temperature. The weight gain of the specimens was carefully monitored by weighing the samples periodically. Precautions were taken to remove the moisture from the specimen surfaces by wiping them off each time before weighing. The percentage weight gain was calculated by means of using the formula,

$$M\% = \frac{W_2 - W_1}{W_1} \times 100$$

where,

M% = moisture absorption percentage

W<sub>2</sub> = weight of the wet specimen

W<sub>1</sub> = weight of the bone-dry specimen

The specimens were cut as per the ASTM standards specifications of the test method and kept immersed in the distilled water for various time intervals of 5 days, 10 days, 15 days, 20 days and 25 days.

**Tensile Test**

Tensile Tests were carried out using Instron (4301) model. The dimension of the specimens was 250mm X 25.4mm X 1 mm composites. Test on the specimen having the gauge length of 115mm are carried out at the cross head speed of 5 mm/min.

$$\text{Tensile strength} = \frac{\text{load} \times 9.81}{bd}$$

$$\text{Tensile modulus} = \frac{P_2 - P_1}{E_2 - E_1} \times \frac{\text{gauge length}}{\text{area}} \times 9.81$$

Where,

b = width of the specimen (mm)

d = thickness of the specimen (mm)

P<sub>1</sub>, P<sub>2</sub>, E<sub>1</sub> and E<sub>2</sub> = points obtained from the Figure 1.

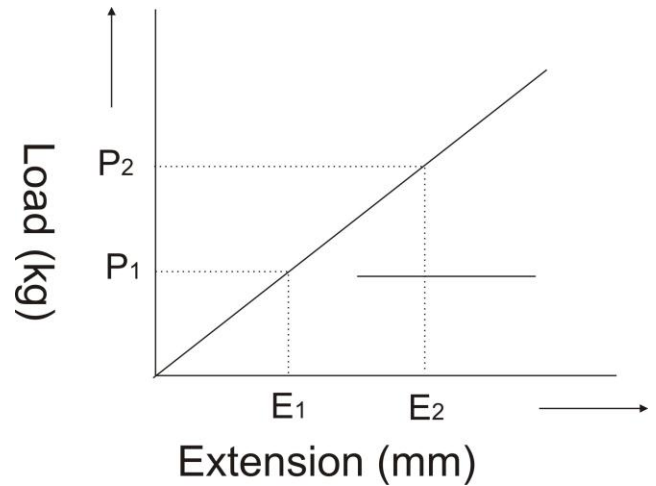


Figure 1. Load Extension curve

**Flexural Test**

Flexural tests can be carried out on three-point bending load using Instron (4301) model. The flexural test is initiated by applying the load perpendicular to the fibre direction. The dimension of the specimens was 80mm X 10mm X 1 mm for 8 lay sequences.

$$\text{Flexural strength} = \frac{3}{2} \times \frac{W \times L}{bd^2} \times 9.81$$

$$\text{Flexural modulus} = \frac{\text{load}}{\text{deflection}} \times \frac{L^3}{4bd^3} \times 9.81$$

where,

W = breaking load (kg)

L = span length (mm)

b = width (mm)

d = thickness

**RESULTS AND DISCUSSION**

**Silk reinforced composite**

Silk reinforcement layer is the right choice for special application which needs composite with less than one mm of thickness with sufficient amount of mechanical properties. Thickness of composite is only 0.65 mm even increase the number of silk reinforcement layers up to 8 or 10. Therefore, silk is also considered as a reinforcement layer for production of composite for special application.

Hence, study was carried out for production of silk reinforced composite and its properties at moist environment. This study will gives insight for understanding the suitability of silk composite for different applications like implant making protective layer in medical field etc

### Moisture sorption of silk reinforced composites after immersion

The moisture sorption of woven silk reinforced composite is shown in Fig 2. In general, absorption of water molecules gradually increases with increase in immersion time. Even though rate of absorption will significantly changes from beginning of the immersion process. Initial absorption of moisture result in reduction of internal structural cohesion in the composite. This in turn leads to affects the mechanical properties of the composites.

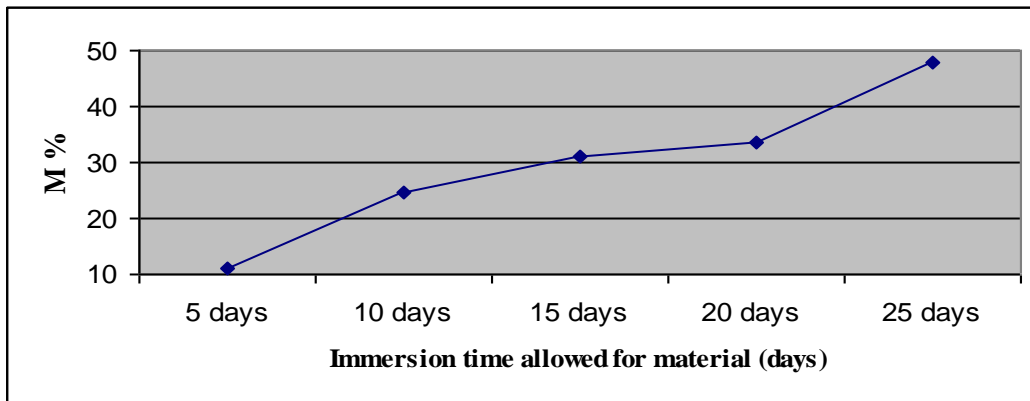


Figure 2. Moisture sorption curve of silk reinforced composite

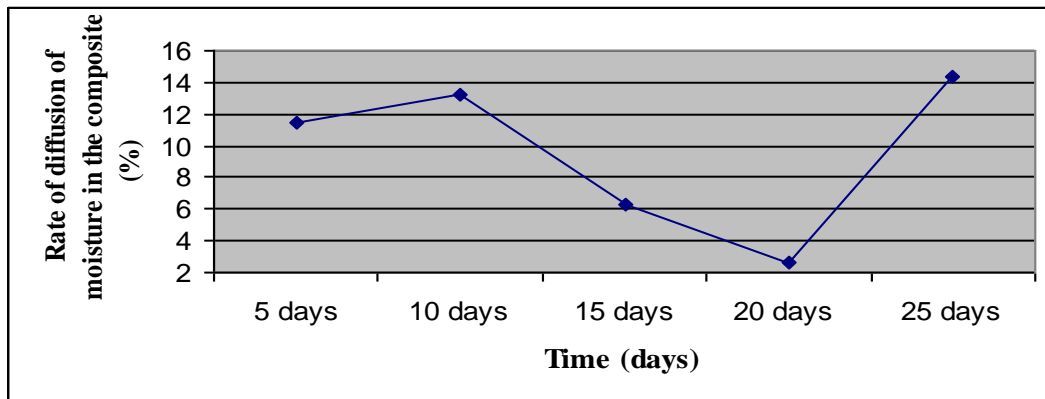
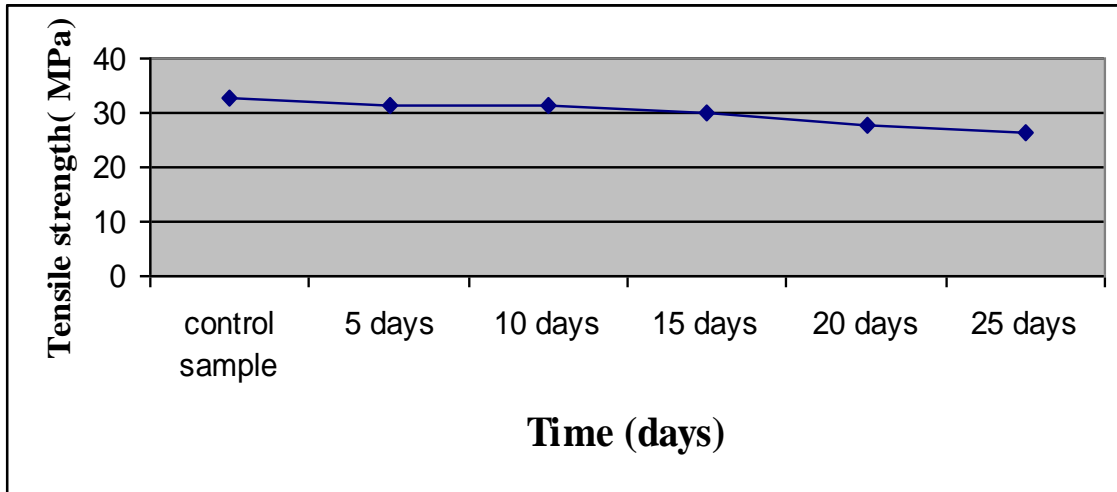


Figure 3. Rate of diffusion of moisture in the composite

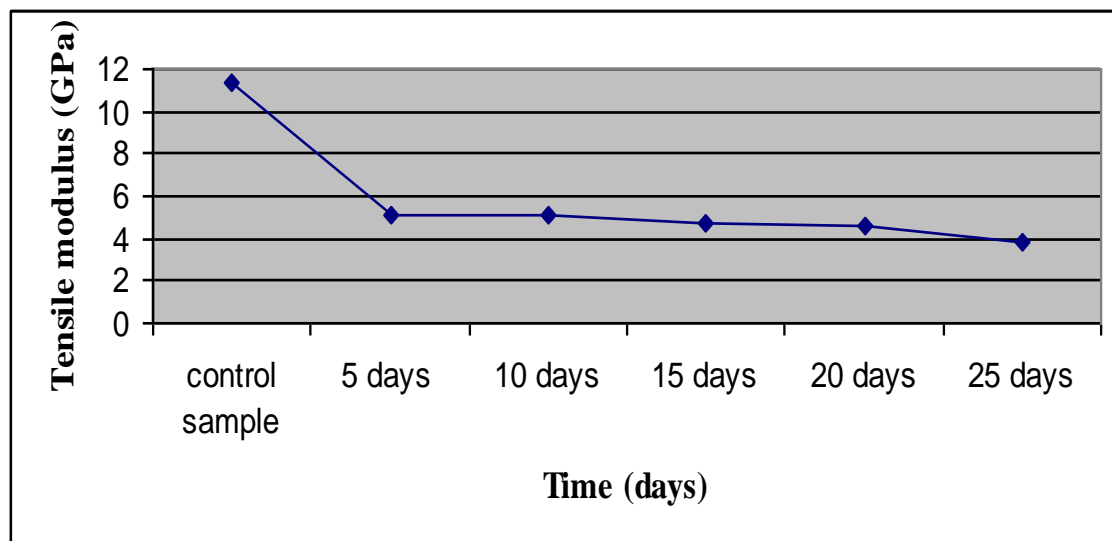
The rate of diffusion of moisture in the composite is shown in fig 3. It was observed that after 10 days the rate of diffusion of moisture decreases significantly. Therefore the changes in mechanical characteristics after immersion are very minimum as compared with composite which is allowed for 10 days immersion. After 20 days the rate of diffusion of moisture is significantly higher. This gives as insight that there may be a significant changes in structure will leads to remarkable changes in mechanical properties.

### Effect on immersion of tensile properties on silk reinforced composites

The tensile properties of woven silk reinforced composites is shown in figure 4 and figure 5. The impact of moisture absorption on mechanical properties of the composites has been carried out. The moisture diffusion doesn't have much influence on tensile strength but it significantly affects the tensile modulus of composite. This may be due to reduction of internal structure cohesion during absorption of water. Therefore absorption of water directly affects the modulus value. Eventhough rate of reduction of modulus with respect to immersion time is very minimum.



**Figure 4.** Variation of Tensile strength with different time intervals for control and immersed woven silk reinforced composite.



**Figure 5.** Variation of Tensile modulus with different time intervals for control and immersed woven silk reinforced composite.

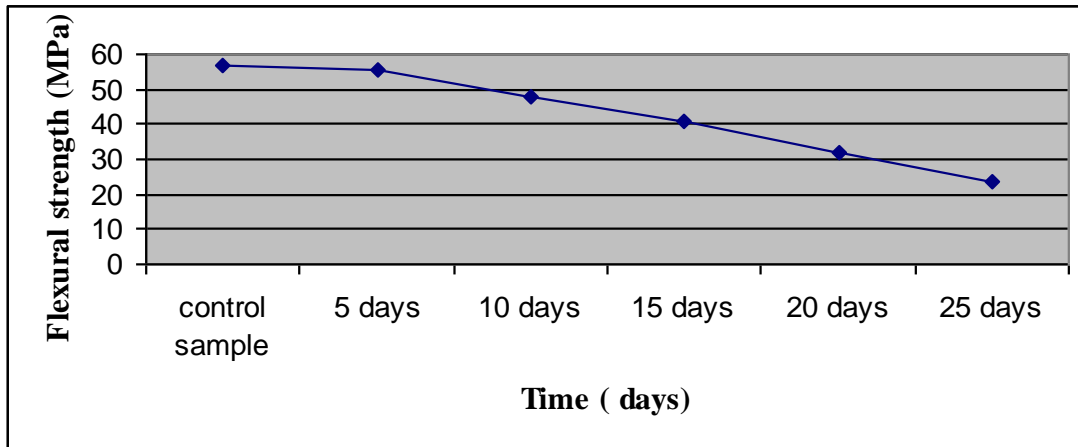
On the whole absorption of water have impact on tensile strength and tensile modulus of composite This is mainly due to the fact that the rate of diffusion of water into the composite depends on the environment in which it presents. Tensile modulus is decreasing. This is due to the penetration of water molecules into the composite structure leading to diffusion of polymer resin molecules. Hence de-bonding of matrix and reinforcement occurs at the interface of the composite leading to the reduction of load bearing capability.

The mechanical properties such as tensile properties of the composites decreases with the increase in the immersion time because of the increase in the absorption of moisture with time leads to the matrix cracking and debonding of the

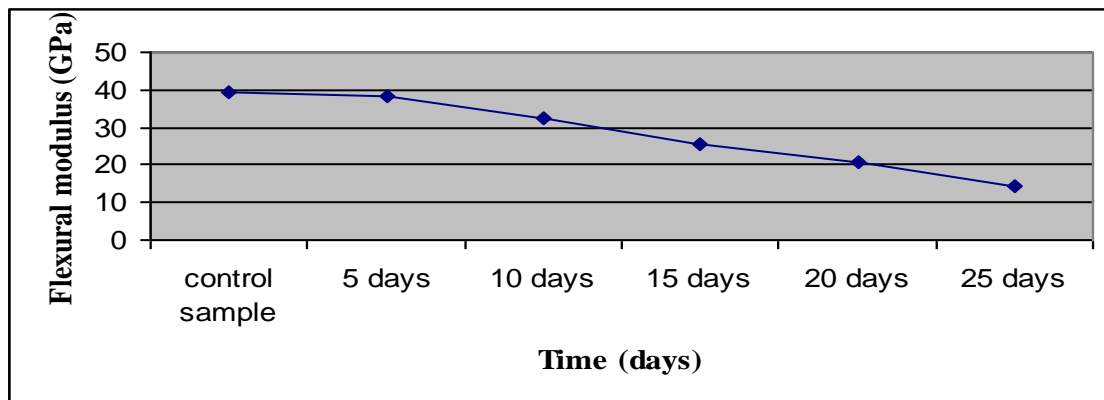
composite. The reduction in the mechanical properties such as tensile property gets reduced with the increase in the time of immersion. This is because the interaction between the composite and the water molecules get reduced as the composite reaches the relative stabilization with time.

#### **Effect on immersion of flexural properties of composites**

The flexural strength values of control and water immersed samples of woven silk reinforced composites are shown in fig 6 and fig 7. The impact of moisture diffusion on mechanical properties of the composites has been carried out.



**Figure 6.** Variation of Flexural strength for different time intervals of control and immersed woven silk composite



**Figure 7.** Variation of Flexural modulus for different time intervals of control and immersed woven silk composite.

On the whole the impact of moisture or the diffusion of moisture in the composites is very clear in the case of flexural strength and modulus. The structural changes is directly reflected in the result of flexural test because the enhanced flexural strength the composite is depend on characteristics of matrix component because very fine silk layer is used as a reinforcement in the composite. The mechanical properties such as flexural properties of the composites decreases with the increase in the immersion time because of the increase in the absorption of moisture with time leads to the matrix cracking and debonding of the composite.

The reduction in the mechanical properties such as flexural properties gets reduced with the increase in the time of immersion. This is because the interaction between the composite and the water molecules get reduced as the composite reaches the relative stabilization with time.

## CONCLUSION

Moisture diffusion studies carried out on the woven silk reinforced composite clearly show that the durability of composite systems under mechanical loading is greatly complicated by the occurrence of several interacting physico-

chemical and mechanical degradation mechanisms. Compared to other natural fibres silk fibre has low mass per unit area and low thickness. Water alters the properties of the polymeric matrix and degrades depending on the different time intervals. The effect of water on the mechanical properties and durability of polymer matrix composites will decide their feasibility for use in composite structures.

Based on the studies conducting following conclusions are arrived at

- Degummed silk layer is not suitable for making composites, because the fabric shrinkage due to removal of sericin gum.
- Moisture diffusion increases at different time intervals. The rate of diffusion is significantly changes with respect to time.
- Immersions of composite samples for 25 days result in remarkable change in mechanical properties.
- The percentage reduction of tensile and flexural strength of immersed sample is high compared to control sample.

- The reduction in the mechanical properties such as tensile and flexural gets reduced with the increase in the time of immersion. This in turn reduces the interaction between the composite and the water molecules get reduced as the composite reaches the relative stabilization with time.
- The tensile and flexural modulus of immersed sample is less compared to control sample. The mechanical properties such as tensile and flexural properties of the composites decreases with the increase in the immersion time because of the increase in the absorption of moisture with time leads to the matrix cracking and debonding of the composite.

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