INVESTIGATIONS ON THE HYGROSCOPIC BEHAVIOR ON CHEMICALLY GLUED COMPOSITE LAMINATES

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Abstract

The main goal of this research is to investigate how hygroscopic performance influences chemically glued composite materials, which were manufactured using both hand layup and vacuum bag techniques. The study involved employing numerical techniques to measure density and conducting a hygroscopic test following the ASTM D570 standard, all with the aim of identifying the most promising composite candidates. The results unveiled that the L3 and L4 laminated composites, composed of hemp-carbon and hemp-aramid combinations, demonstrated the lowest density and water absorption percentage compared to other composites. This favourable outcome was attributed to the reduced number of voids resulting from the vacuum production process. These principal findings indicate the potential for improved mechanical properties in these materials. The study's findings are of great value to designers and engineers, providing crucial insights and data for evaluating the material's stability and making necessary adjustments in the design or manufacturing process. Understanding the hygroscopic behaviour and its impact on composite materials is crucial for ensuring their long-term performance and durability.

Keywords: Composite, Jute, Hemp, Basalt, Carbon, E-glass, Density, Hygroscopic test.

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1. Introduction

Water molecules have the ability to penetrate a composite network, leading to modifications in its structural characteristics. According to Marom [1], in the short term, water increases the (mode I) fracture toughness, but over the long term, it causes a decrease. Springer and Shen [2, 3] observed that as the moisture content increases, the ultimate tensile strength and elastic moduli of 90-degree laminates decrease. This reduction can be significant, ranging from 50 to 90 percent. Moisture diffusion into composites adversely affects the fiber-matrix interfacial bonding [4, 5], resulting in a decrease in the glass transition temperature, swelling, plasticizing, hydrolyzing, and sometimes microcracking the matrix [6, 7]. To accurately predict the long-term behaviour of composites, it is essential to have the capability to forecast water diffusion and its effects on resin properties. This knowledge is crucial to understanding how water can influence the performance and durability of composite materials over time. The uptake of moisture in materials is typically quantified through weight gain, while the process of water diffusion is commonly characterized using Fick's law [8].In 1975, Shen [8] investigated the absorption and desorption of water in composite materials based on Fick's law. They derived expressions for moisture distribution and moisture content as a function of time for one-dimensional composite materials. The analytical solution proposed by him has gained widespread acceptance and finds support from numerous experimental data, effectively describing the water diffusion behaviour in composites. However, certain composites exhibit water absorption behaviour that deviates significantly from the Fickian model. Understanding such non-Fickian mechanisms has proven challenging due to the complexities of absorption behaviour and variations in experimental data. In an effort to address this issue, several methods and computing codes have been introduced to analyse non-Fickian moisture content data and assess diffusivity and moisture profiles across the thickness of laminates [9-11].

Present investigations involved conducting density measurements and performing a water absorption test following the ASTM D570 standard [12], with the objective of identifying the most promising composite candidates.

2. Materials And Fabrication Methods

In the present work, epoxy resin was selected as the matrix material for developing the required composites. Bi-directional woven Natural and synthetic fabrics were selected as reinforcement materials. These resins are simple linear polymers that are produced by condensing epichlorhydrin with bisphenol. These materials are used in the temperature range of 50–1000 °C since they have a relatively high viscosity. These resins are odorless, colorless, and non-toxic. In this work, Lapox L12 resin was used. Table 1 gives the properties of Lapox L12 epoxy resin. The curing agent utilized in the current fabrication is K6 hardener. Hardener was stirred with epoxy in a ratio of 10:1 for proper strengthening.

Table 1 Properties of Lapox L12 Resin

Property	Min. Value (S.I.)	Max. Value (S.I.)	Unit (S.I.)
Tensile Strength	50	60	MPa
Compressive Strength	110	120	MPa
Flexural Strength	130	150	MPa
Impact Strength	17	20	KJ/m^2
Modulus of Elasticity	4400	4600	MPa

2.1 Reinforcement Materials

In the present work, synthetic and natural fabric mats were used as reinforcements. Synthetic fibers were applied as extreme top layers and extreme bottom layers as they give good finishing to the composite, but they are expensive. Natural fibers were utilized as intermediate layers to obtain the required thickness, and they are less expensive. Four types of synthetic fiber mats were used for this investigation and are as follows: i. E-Glass fiber; ii. Basalt fiber; iii. Aramid fiber; iv. Carbon fiber. Two types of natural fabric mats were considered to develop the composites and are as follows: i. Hemp fiber; ii. Jute fiber

2.2 Laminate Configuration

In the present investigation, eight types of laminated composites were developed. The arrangement of stacks in each type of laminate is shown in Figs. 1 (a) and (b).

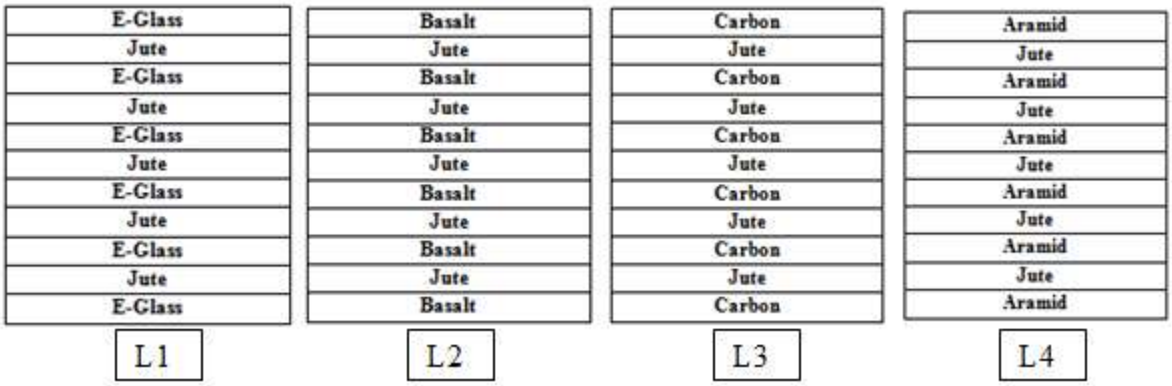

Fig.1 (a) Arrangement of fiber mats with jute as natural fiber

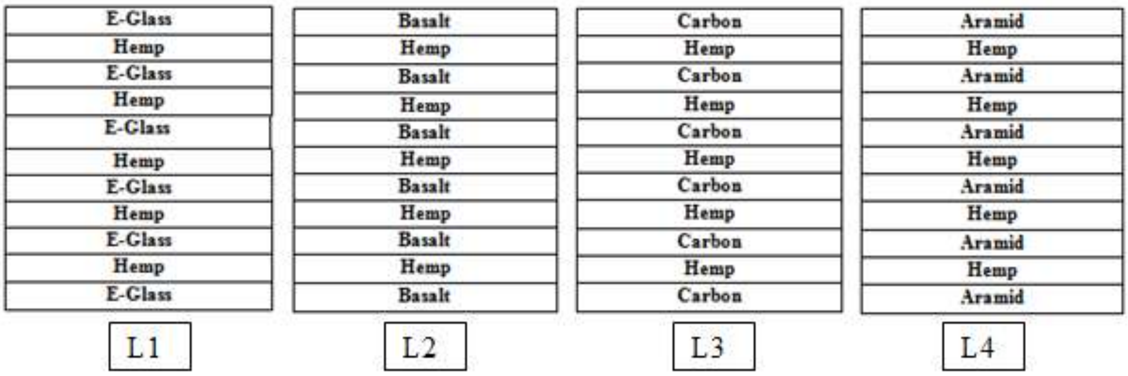

Fig.1 (b) Arrangement of fiber mats with hemp as natural fiber

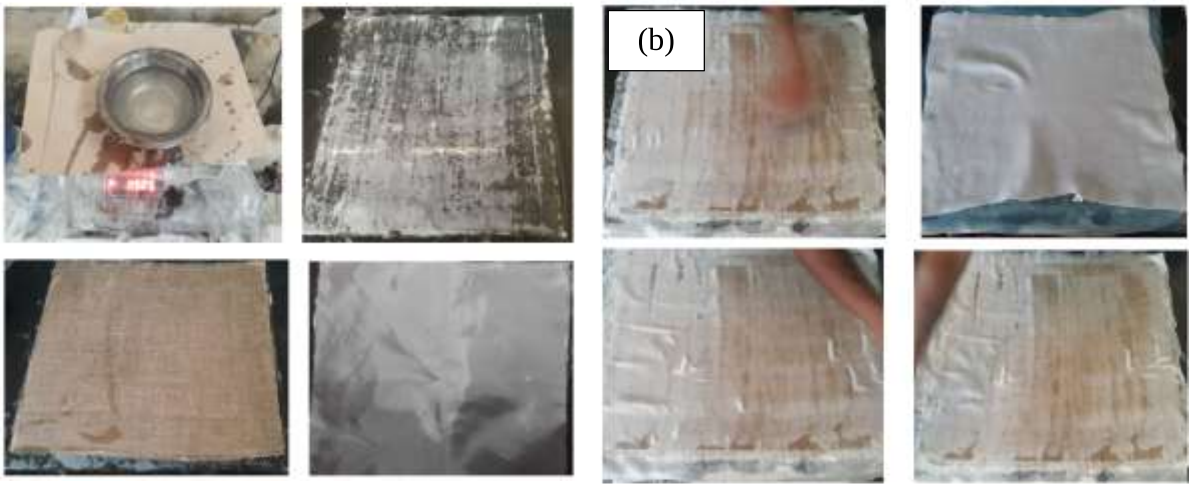

Fig.2.3 (a) Hand layup (b) Vacuum bagging composite laminate fabrication steps

2.3 Density Test

Laminate specimens of definite dimension were made ready for this purpose. The test specimen's mass was computed using a high-accuracy digital balance. Then the volume of the specimen was calculated by applying mathematical relations (see Equation 1). Three trials of experiments were conducted on each laminated composite, and the mean value of density was recorded. Mass and volume are directly proportional to the quantity of material present; they are extensive properties of the material, but density is an intrinsic property that gives how much mass is packed into a given three-dimensional space. Typically, densities are measured in g/cm^3.

$$\rho = \frac{m}{v} \quad \text{........ Eqn.1}$$

2.4 Water Diffusion Test

The ASTM D570 standard [12] was followed for the water diffusion test. For conducting the test, the preliminary mass of the laminated composite, tensile, and flexural test specimens was first noted. Then the test samples were tied in a different coloured rubber band for easy identification of hand layup and vacuum-bagged specimens with jute or hemp, a natural fiber. After this, the test samples were immersed in a bottle containing sea water. The sample laminates were kept in a submerged state for 100 days. After 100 days, the sample laminates were removed from the bottle, and the final mass of the same was recorded. The percentage (%) of water absorbed was calculated using the empirical relations given in equation 2. Three trials of tests were conducted on each type of specimen, and the average value was tabulated. The water absorption experimental setup is shown in Fig. 2.

% of water or moisture absorbed=

$$[(m_f - m_i)/m_i] \times 100 \text{.......... Eqn. 2}$$

Where m_i=Initial mass of specimen, kg

m_f= Final mass of specimen, kg

Fig. 2 Moisture absorption experimental setup

3. Result And Discussion

3.1 Density test results

The laminated composites developed by hand layup and vacuum bagging techniques were subjected to a density test. The mass density of the hybrid composites developed was computed from the fundamental relations in Equation 1. The weight of the manufactured composite material was recorded using the digital weighing machine, and the volume was found by measuring dimensions like length, width, and thickness of the sample. Three trials of the test were conducted on each sample, and the mean density is tabulated as indicated in Table 2. Fig. 3 gives the comparison of density for hand layup and vacuum bagging composites. It is noticed that the values of mass density are the maximum for vacuum-bagged composites. It is mainly because of the greater compactness of the composite due to the application of vacuum pressure. Also, the mass density values are lower for carbon fiber composites and aramid fiber composites.

The dimensional stability of fiber-reinforced laminated composites depends on density. In fiber-reinforced composites, the density has been classified as Low (Up to 500 g/cm^3), medium (up to 800 g/cm^3), and high density (above 800 g/cm^3). Laminated composites, which have a low mass density, are capable of holding more moisture and water as compared with high-density composite materials. Laminated composites with low density have more spaces, voids, and porosities. In the case of reinforced hybrid composite materials where more than two different materials are mixed together, the dimensional stability of the composites depends on the fiber and material loading. It was noticed that reinforcement materials like Jute or hemp have a greater tendency to absorb moisture and water.

The density of laminated composites mainly depends on fiber and matrix physical properties. Usually, natural fibers have a higher density value compared to synthetic fibers. The density of the epoxy polymer is lower than the density of fibers. Hence, an increase in the percentage fraction of fibers in a composite may increase its density. Density plays a very important role in laminated composites. Generally, the composite weight increases with the increase in density. For most of the applications, low-weight composites are required. But, in this investigation, composites obtained from vacuum bagging have higher values of density. The main reason is the reduction in the number of voids because of the application of pressure. The density of L3 and L4 composites in vacuum bagging is almost the same. In the case of hemp as the natural fiber, for both L3 and L4.

Table 2 Density values of laminated composites

Natural fiber	**Laminate type**	**Density, g/cm^3**	
		Hand Layup	**Vacuum Bagging**
Jute	L1	1.56	1.88
	L2	1.25	1.37
	L3	1.25	1.09
	L4	1.04	1.19
Hemp	L1	1.39	1.46
	L2	1.25	1.4
	L3	1.2	1.2
	L4	1.1	1.19

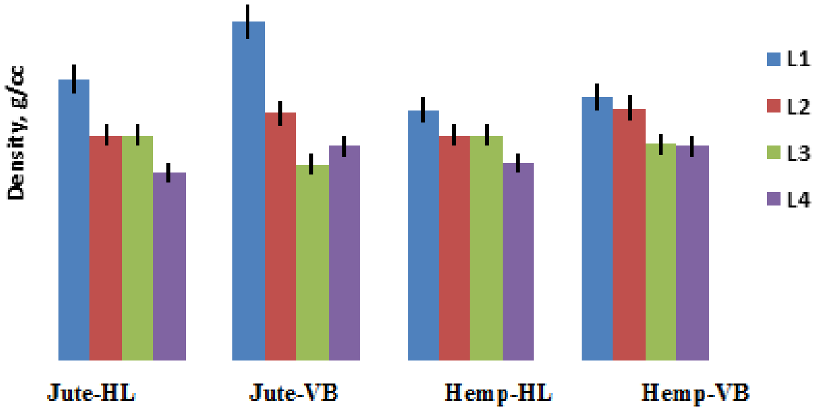

Fig. 3 Comparison of density of laminated composites

3.2 Water/Moisture absorption test results

The laminated composites developed in this work were subjected to a water absorption test by dipping composite samples in seawater for 100 days. The average and saturated percentages of water absorbed by each laminated composite sample are given in Table 3. Fig. 4 shows the graph of percentage water absorption of L1, L2, L3, and L4 composites with jute and hemp as natural fibers produced by hand layup and vacuum bagging techniques. By observing the figure, it is revealed that the percentage of water absorbed decreases with the type of synthetic fiber. That is, for hand layup and vacuum bagging techniques, the L1 composite has a maximum value of water absorbed percentage (23.97% and 21.17%), whereas L3 and L4 have lower and almost equal values of water absorption (16.23%, 18.17%, and 14.67%, 15.21%). L3 composite has a lower value for water absorption than others. Water absorption values are higher for jute composites in comparison to hemp composites. Again, it is noticed that water absorption is higher in the case of hand-layup composites compared to vacuum-bagged samples. It is possible that L3 composites might have better mechanical characteristics.

Usually, hand-layup composites are thicker than vacuum-bagged composites, and they possess more voids. This could be the primary reason for hand layup composites absorbing more water. Also, jute fibers tend to soak up more water compared to hemp fibers. Hence, composites hybridized with jute tend to soak up more water than hemp-hybrid composites. Water absorption decreases with the rise in fiber content [13]. It could be due to the matrix's increased affinity for water. As the composites have voids, pores, and interfacial bonding in a matrix, this can also increase the behaviour of the laminate towards water absorption. If fiber content in a composite decreases, there will be a rise in the above parameters, which results in more absorption of water by the composite. A change in the mass of the laminated composite sample has the least effect on water absorption. For a composite, the water absorption rate increases as the time of immersion increases. This increase will continue until the composite reaches saturation. In the present research work, enough time (100 days) was given for the water absorption test to attain a saturation state. Water absorption by the composites also increases with the temperature of the environment [14]. If the temperature of the water is high, then there will be an increase in the diffusion rate of water into the composite. This consequence is mainly due to the rapid movement of water molecules at higher water temperatures [15, 16]. In service applications, water absorption by the laminated composites causes severe effects on their life. Water absorption causes swelling of the composite due to the hygroscopic nature of the epoxy polymer.

Hydraulic stresses are developed within the composite matrix, due to which catastrophic failure of the composite may take place. Evaluation of water-induced stresses is necessary to predict the life of laminated composites. In the material selection for the fabrication of laminate, the low or minimal value of water absorption plays an important role. To develop the composites for the expected levels of performance, the established type of resin, volatile content, percentage of flow, processing time, and temperature are to be considered. In mechanical service-oriented applications, laminated composites should have a minimum water absorption rate to ensure minimum dimensional changes. The lower value of the soak in water rate is also important for the stable chemical properties; otherwise, the composites may be attacked by acids or bases.

Above all, the above findings emphasize the need for careful consideration of moisture exposure and effective moisture management strategies during the design and fabrication of composite structures.

Natural fiber	**Laminate type**	**Hand Layup**			**Vacuum Bagging**		
		m_i g	**m_f g**	**% of water absorption**	**m_i g**	**m_f g**	**% of water absorption**
Jute	L1	16.23	20.12	23.97	14.23	17.24	21.17
	L2	22.13	26.78	21.01	15.23	18.21	19.54
	L3	22.31	25.93	16.23	14.56	16.70	14.67
	L4	14,25	16.84	18.17	16.78	19.33	15.21
Hemp	L1	20.56	24.47	19.02	14.12	16.64	17.87
	L2	16.34	19.22	17.62	14.98	17.37	15.94
	L3	18.52	21.14	14.14	15.24	17.25	13.21
	L4	10.42	12.10	16.8	15.57	17.71	13.68

Table 3 Percentage of water absorption of laminated composites

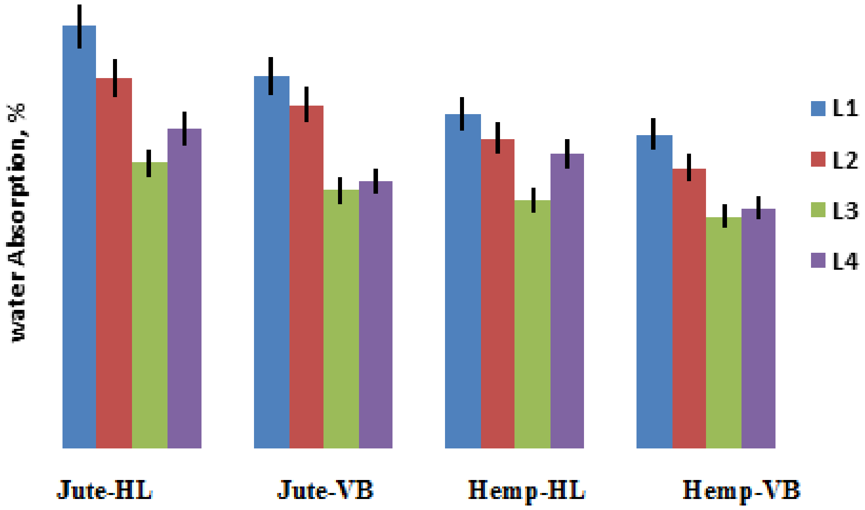

Fig. 4 Comparison of percentage of water absorption of laminated composites

4. Conclusion

In the present research work, sixteen types of fiber-reinforced hybrid laminated composites were developed, viz., four types of composites with jute as the natural fiber and E-glass, basalt, carbon, and aramid as synthetic fibers by hand layup technique, four types of composites with hemp as the natural fiber with the above synthetic fibers by hand layup technique, and the same number of composites by vacuum bagging technique. The composites were subjected to density tests and moisture absorption tests. Based on the results obtained from the investigations, the following conclusions can be drawn:

1. Density test results and analysis revealed that the density of L3 and L4 composites in both hand layup and vacuum bagging techniques was low compared to others. Again, in these composites, L3 and L4 composites with hemp-carbon and hemp-aramid fiber combinations have lesser densities. The composites should have a lower density and good mechanical properties to reduce the weight of a structure. In this context, the above composites can be implemented as structural components in the aircraft and automobile industries.

2. Moisture absorption investigations were accomplished on all types of composites developed. The percentage of water or moisture absorbed by the L3 (hemp-carbon) and L4 (hemp-aramid) composites, which were fabricated by the vacuum bagging technique, is less. Also, in the case of vacuum bag techniques, there are fewer voids as a result of vacuum production.

Conflict of Interest: The authors declare that there is no conflict of interests regarding the publication of this paper.

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