

# Experimental Investigation of Water Absorptions and Charpy Test of Epoxy Composite Immersed in Different Aqueous Medium



Pankaj Singh Chandel, Nitin Kumar Gupta, Yogesh Kumar Tyagi, and Kanishka Jha

**Abstract** In this research work, an experimentation has been performed to investigate the water absorption and effect of rupture energy of the epoxy composite immersed in different aqueous medium. An attempt has been made to assess the rate of moisture absorption in composite materials. The percentage of moisture absorption in the specimen is observed when they are placed in four different mediums, i.e., normal water, air, half-saturated salt solution, and fully saturated salt solution. In order to check the moisture absorbed, the specimen is taken out after fixed intervals and their weights have been measured. In the beginning, a rough linear increment was observed in the weight gained by the specimens immersed in aqueous mediums. After the initial results, the results obtained were depicting non-Fickian type of moisture absorption behavior. Factors, such as initial swelling of matrix, percentage void content, edge effect, and the mechanical adhesion between fibers and matrix, greatly influenced the behavior of moisture absorption in composite. The diffusivity of the moisture in the specimens kept in air is very low owing to which a negligible change occurs in the weight of specimen immersed in air for the total period of experimentation.

**Keywords** Moisture absorption · E-glass fiber composite · Pressure · Fickian model · Non-Fickian behavior · Deboning · Diffusivity

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## 1 Introduction

Composites are significantly used everywhere. We have been using composites since a long time. People used to make bricks from mud and many other materials with raw composites only. Now in present date, we make tennis racket, vehicle body and aircrafts, and many other things using composite materials only. Now a question arises what is composite? It is nothing but a multiphase material with distinct properties. Generally, they are made up of only two materials, one is the continuous phase (matrix) and the other is the discontinuous phase (reinforcement) glue to bind and reinforce fibers [1, 2].

Presently, when an engineer requires to work with materials, he/she prefers using a composite material over steel or other metals because composites are stronger, lighter, durable, much more resistant to extreme pressure and temperature, has biodegradability, and has low cost (particularly in natural fibers). These composites are used for making aircraft parts and also used in marine infrastructures, as it increases its superior strength and makes it more efficient than the older ones. The only goal of aeronautical industry to use a composite material in aircrafts is to achieve better structural properties along with lightweight, leading to improved fuel efficiency and performance from an aircraft. With the properties such as strength, lightweight, corrosion resistance, and better performance at elevated temperatures, these materials have become center of attraction in almost every field [3, 4].

With the help of composite materials, aeronautical/space engineering is upgrading day by day. In early 1970s a carbon fiber composite was introduced to engineers, which is nowadays used in making aircrafts, unlike alloy of different metals or a single metal as used earlier. High-performance composites are being increasingly used in aerospace and marine structural applications. For marine infrastructures and sustainability of materials in various moisture varying environment, glass fiber-reinforced composites are preferred better than others due to its high corrosion resistance and lightweight property, for example—in pipelines of desalination plants, turbine blades, ship building industry, water storage equipment's, boats, etc. Cost of installation is reportedly lower than that by some conventional materials like steel or aluminum [5, 6]. Hence, parts like turbine blades which are particularly immersed in water for a long period of time, moisture absorption plays a very important role, because it affects the strength of structure materials and its durability also.

Organic matrix composites are often subjected to moisture and temperature environments. These environmental effects can lead to composite degradation and consequent loss of mechanical properties. An essential feature of almost all combinations of weathering conditions is humidity. Hence, topics pertinent to the question of performance in the presence of moisture are of prime importance. These topics are commonly divided into two subjects. The first relates to factors which drive moisture into the composites, namely the penetration mechanisms. The second deals with the effects of the presence of water on the performance and durability of the composites. Several studies showed the important effects of humidity on the mechanical properties of composite materials and on their long-term behavior [7, 8]. In general,

moisture diffusion in composites relies on factors such as the volume fraction of fiber, void volume, additives, humidity, and temperature. Moisture diffusion in polymer composites is seen to be governed by the variables such as humidity, temperature, and void volume. There are three different mechanisms mainly used for moisture diffusion in polymer composite. The first one is diffusion of water inside the tiny gaps, between the polymer chains. The next one is capillary action into the gaps that flows to the interfaces between the fiber and the matrix. And the last one is transportation of the microcracks in the matrix, occurring due to the swelling of fibers [9, 10].

During the research, mainly our focus was on the absorption of the moisture by the random E-glass fiber composite when they are maintained at two distinct pressures and kept in varying salt concentrated aqueous media. These media are categorized as air, normal water and two different types of salt solution according to the concentration of salt. The percentage of moisture gain is described by the weight gain at different time instants. Durability nature of the composite is predicted by the diffusion of water and its impacts on the resin properties.

## 2 Materials

In this research work, epoxy resin has been selected as a matrix and the random E-glass fiber were used as a reinforcement. The mixture of Araldite AY 105-Epoxy resin and HY951 Hardener were used prepare the matrix. Both the materials were mixed in the weight ratio of 10:1. All the chemical materials were procured from an Indian manufacturer Huntsman Ltd. Table 1 presents the physical properties of the Araldite AY 105 Epoxy received from the company manual. Similarly, the random E-glass has been procured from an Indian local market. Table 2 presents the physical properties of the random E-glass fiber that were used in the fabrication process.

**Table 1** Properties of epoxy AY 105

Properties	Color (visual)	Specific gravity	Viscosity (Pa-s)
Araldite AY 105	Clear liquid	1:1	6–8.5

**Table 2** Properties of random E-glass fiber

Properties	Density (g/cc)	Modulus (GPa)	Tensile strength (GPa)	Elongation (%)
E-glass fiber	2.57	73.5	3.5	4.8

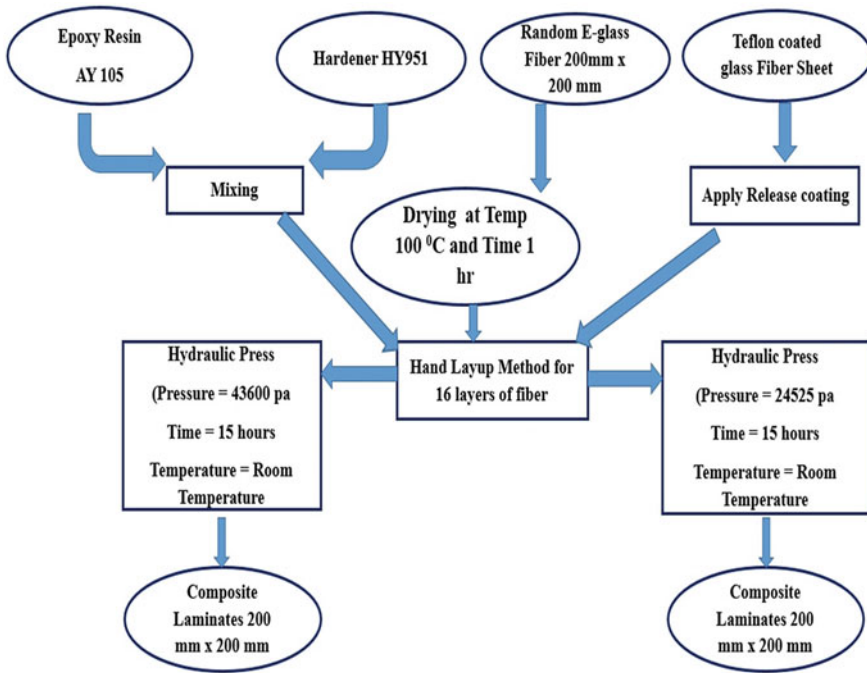


Fig. 1 Fabrication procedure of the laminated test sample with specification

## 2.1 Fabrication Procedure of Test Specimens

Two samples were made each having 16 layers of E-glass fiber composite fabricated by the conventional hand lay-up technique. The dimensions of the samples were taken as  $150 \times 150 \times 4.7 \text{ mm}^3$  and  $100 \times 100 \times 6.85 \text{ mm}^3$  under pressure of 43,600 Pa and 24,525 Pa, respectively, at room temperature ( $\sim 20^\circ\text{C}$ ) for 15 h. Figure 1 represents the fabrication procedure at various condition. And Fig. 2 represents the schematic representation of the hydraulic press.

## 2.2 Procedure of Moisture Absorption Testing and Evaluation

The water absorption tests were conducted in distilled water and based on *ASTM D57011*. For each laminate, five test specimens measuring  $25 \times 76 \text{ mm}$  were used [11]. The differences in these pressures are to ensure the distinct percentage of void content in the corresponding samples of composite (Fig. 3).

The effect of water absorption on the fabricated sample was investigated through the *ASTM D57011* procedure. Specimens are prepared with the help of two samples

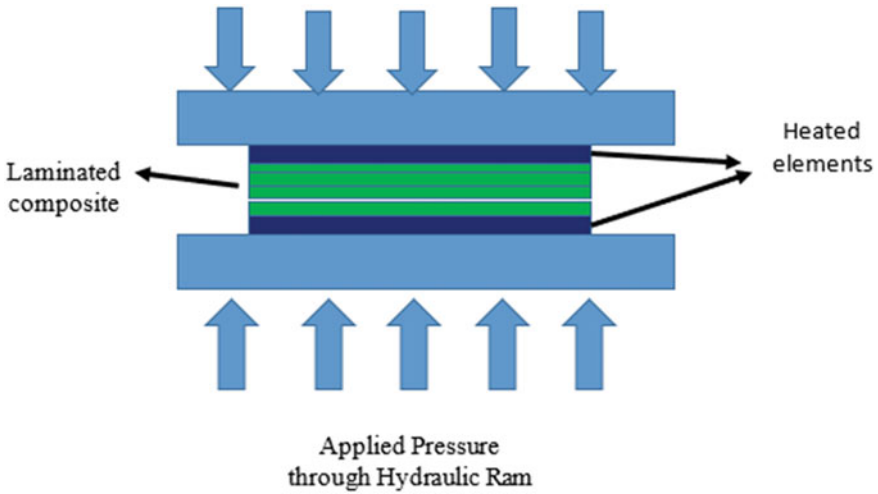


Fig. 2 Schematic representation of hydraulic press used for laminate fabrication

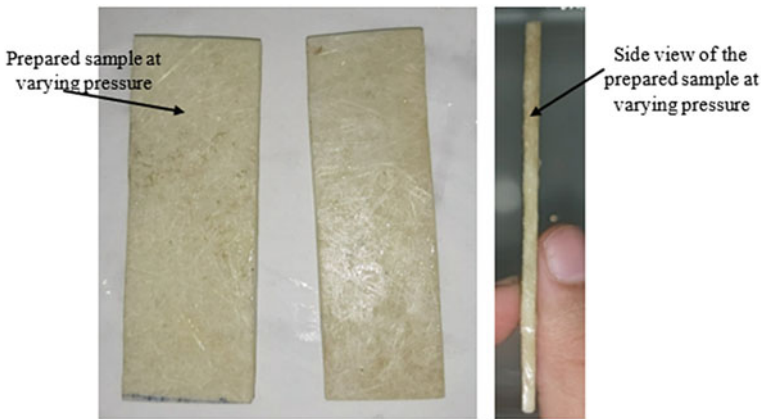


Fig. 3 Actual fabricated sample used for the experimentation work

and both of them are different from each other in terms of dimensions, as required by the experiment in testing. Since a lot of research has already been done on glass fiber-reinforced composite placed in sea water, hence in this project, specimens are submerged in water, fully saturated salt solution and half-saturated salt solution. The fully saturated salt solution and half-saturated salt solution at room temperature (ambient temperature) 20 °C contains 10.2 times (357 gm. of salt in 1 L of water) and 5.1 times (178.5 gm. of salt in 1 L of the salt) as present in sea water (35 g of salt in 1 L of water roughly equivalent to 35,000 ppm), respectively. These procedures

are followed only to evaluate the changes occurred on the composite by a highly concentrated brine solution [12–14].

Kinetics of moisture absorption is studied by placing the epoxy E-glass fiber laminate composite in these solutions. Moisture absorbed by the specimen is regularly checked by taking out the specimen after fixed interval of time. Before taking the weight measurements, water absorbent paper is used to wipe out the surface moisture from the specimen. Electronic weighing machine of 0.0001 gm accuracy is used to measure the weight of specimens.

The gain in moisture content is calculated with the equation below:

$$\text{Absorbed Moisture(\%)} = [(mw - md) \times 100] / md \quad (1)$$

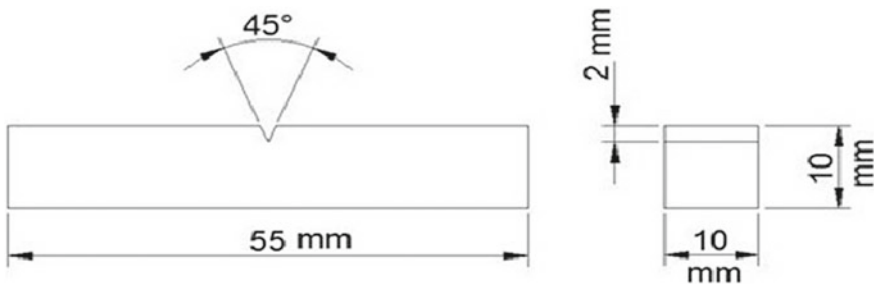
where  $mw$  is the wet weight of the specimen while  $md$  is the dry weight of the specimen.

### 2.3 Charpy Test Characterization on Fabricated Sample

To investigate the impact behavior on the developed sample, Charpy test has been performed. The standards procedure has been adopted as per the ASTM-23 of Charpy test. The specimen gets fabricated as per the above procedure (Fig. 1) followed by the finishing of the sample as per the required dimensions of the Charpy sample. Figure 4 represents the dimension sketch of the Charpy sample.

## 3 Results and Discussion

The results of the water absorptions and Charpy test have been analyzed and discussed.



**Fig. 4** Dimension sketch of the Charpy test sample

### 3.1 Water Absorption Test

To specify the result and objective of moisture absorption mechanism in the E-glass fiber laminated composites, respective graphs are plotted between percentages absorbed moisture (Y-axis) and square root of time in hours (X-Axis). The nature and reason behind the selection of such media has been already stated. Tables 3 and 4 represent the recorded data of the water absorption, respectively, for the sample prepared at 43,600 Pa and sample prepared at 24,525 Pa.

Figure 6 represents the graph between the water absorption with respect to the mean time at 43,600 Pa. Similarly, Fig. 7 represents the graphs between the water absorption with respect to the time of a composite sample fabricated at 24,525 Pa. The average data of the similar specimens is represented by each curve. Figure 5 represents the sample before the water absorption test and after the water absorption test for the effective visual.

The rate of moisture absorption varies in different samples in some composites, percentage moisture absorption roughly increases linearly with the increase in immersion time and gradually reaches the equilibrium after some time. The rate of moisture

**Table 3** At pressure 43,600 Pa and room temperature

Sr. no.	Square root of time in hours	% Moisture absorption			
		Specimen in air	Specimen in half-saturated salt solution	Specimen in normal water	Specimen in fully saturated salt solution
1	0	0.0	0.0	0.0	0.0
2	9.79	0.0	2.1	2.7	3.1
3	13.85	0.0	2.3	3.9	3.4
4	16.97	0.0	3.0	4.4	4.2
5	19.59	0.0	2.5	4.1	2.7
6	21.9	0.0	2.7	4.6	2.7

**Table 4** Pressure at 24,525 Pa and room temperature

S. no.	Square root of time in hours	% Moisture absorption			
		Specimen in air	Specimen in half-saturated salt solution	Specimen in normal water	Specimen in fully saturated salt solution
1	0	0.0	0.0	0.0	0.0
2	9.79	0.0	2.2	2.5	2.8
3	13.85	0.0	2.3	2.7	2.3
4	16.97	0.0	3.1	4.2	3.6
5	19.59	0.0	2.3	3.2	2.6
6	21.9	0.0	2.2	3.6	2.7

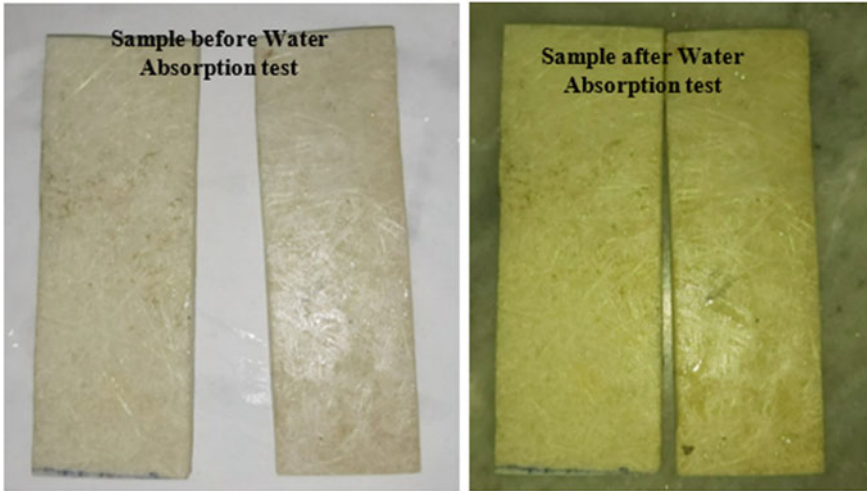


Fig. 5 Sample before water absorption test and after absorption test

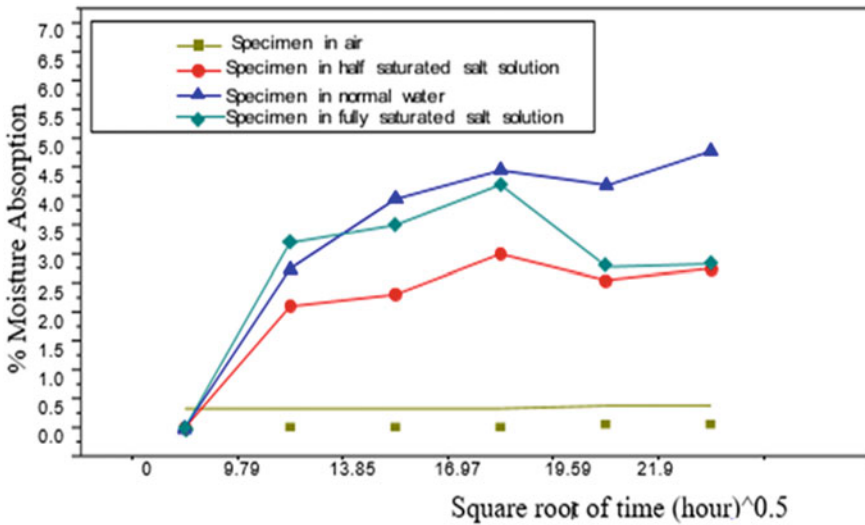


Fig. 6 Moisture absorption curve for sample fabricated at pressure of 43,600 Pa and room temperature

absorption assumes a high value initially followed by a comparatively slower rate moisture absorption as the time passes. This initial increase in moisture content in the composite is commanded by the maximum concentration difference between the composite and medium surrounding the composite. At a particular point, the



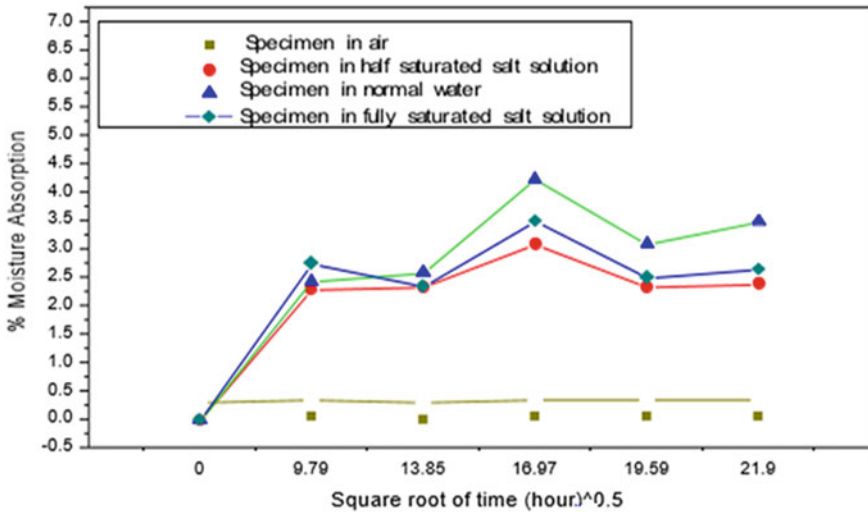


Fig. 7 Moisture absorption curve for sample fabricated at pressure of 24,525 Pa and room temperature

maximum weight is gained by the composite and further negligible moisture absorption or desorption takes place. This kind of behavior marks the presence of the Fickian model based on the Fick’s law.

From the theoretical analysis aspects, the time to reach 99% of the maximum moisture content (saturation point) is governed by two important parameters: the maximum moisture content  $M_m$  and one-dimensional diffusivity ( $D$ ). These two parameters are easier to calculate for the moisture diffusion which follows the Fickian model.

Shen and Springer have given the following equation which estimates the moisture content of the material during both adsorption and desorption:

$$M = M_i + G(M - M_i) \tag{2}$$

$M_i$  is the initial moisture content of the material,  $M_m$  is the maximum moisture content which can be attained under a given environmental conditions, and  $G$  is a time-dependent parameter.

$$G = 1 - \frac{8}{\pi^2} \sum_{j=0}^{\infty} \frac{\exp[-(2j + 1)^2 \pi^2 (\frac{D_x t}{h^2})]}{(2j + 1)^2}$$

The above Eq. (3) can be approximated as:

$$1 - \exp \left[ -7.3 \left( \frac{Dx'}{s^2} \right)^{0.75} \right] \quad (3)$$

For a material exposed to two sides under environmental condition,  $S = h =$  thickness.

Insulating one side of the same material,  $S = 2h$ .

$D_x$  is the diffusivity of the material in the direction normal to the surface.

The time required by a material to attain at least 99.9% of its maximum possible moisture content is:

$$t_m = \frac{0.67 S^2}{D_x} \quad (4)$$

Graph plotted indicates that the moisture absorbed by E-glass fiber composite is not in accordance with Fickian model. There exists some amount of absorption, desorption, and equilibrium not attained in both the graphs. Such non-Fickian behavior is also reported in past and theoretical calculation of diffusivity and time required for attaining maximum moisture content cannot be estimated by Shen and Springer equation. This has made the prediction of diffusivity comparatively difficult. Cracks on surface, debonding between fiber and viscoelastic behavior in the polymer might result in such variations. Moreover, factors such as curing pressure, binding of salt and water molecules to the resin structure, void content in the resin and the composite might be among some of the other reasons also.

With the help of both graphs, we can comprehend the moisture absorption capabilities of specimen when they are placed in four different aqueous mediums, i.e., normal water, air, half-saturated salt solution, and fully saturated salt solution. From Fig. 4, we can see that the moisture content in air is negligible and we verified it, when the increase in weight of the specimen is found to be very less as in comparison with other solutions.

Due to higher concentration difference, the rate of flow across the boundary for first 4 days, the moisture absorption in specimen immersed in fully saturated salt solution is greater than the moisture absorbed by the specimen in normal water. In case of half-saturated solution, the graph depicts completely different result, and the moisture absorption in the former (43,600 Pa) is less than that of the latter (24,525 Pa) one, even if the concentration difference in the former is comparatively more. This is due to the ingress of normal water into the epoxy polymer is quite more feasible in comparison with saline water that contains dissolved salts. A concentration-driven osmotic pressure causing moisture ingress into the polymeric composite body is less of a problem in saline solutions than in pure water. Here, the ingress of normal water into the composite over-powers the concentration-driven moisture absorption. For fully saturated specimen, moisture absorption dominates.

After first 96 h of immersion, due to the presence of large salt molecules in those salt water solutions, the curve for the specimen in normal water overtakes the other two curves, i.e., the half-saturated and fully saturated salt solutions, in relation to

percentage moisture absorption which slows the diffusion process into the matrix of the composite materials, resulting in lower absorption kinetic parameters.

The decrease in the concentration difference is due to the decrease in the slope in the second stage of the weight measurement. However, the curves of the specimens in aqueous medium between the third and fourth stage of the weight measurements show a decreasing trend as each of the three curves attains the maximum moisture content around that particular point of time which is due to inertia of the movement of water in the composite which creates the reverse concentration difference causing reverse osmosis.

Moreover, there is deposition of bulky salt components elements on the surface of composite body intervening with the diffusion processes between the last and second last point of weight measurement, after that there is a gradual increase in the percentage moisture absorption observed following a simple absorption trend. Moisture absorption in these specimens can still attain a maximum value of moisture absorption as the time passes in months or years.

In Fig. 7, curve corresponds to the specimens fabricated at the pressure of the 24,525 Pa and shows trends similar to Fig. 6 curves. After the first 4 days of immersion for specimen in fully saturated salt solution, it is seen that desorption occurs. It could be due to the bonding between the water and the resin, matrix cracking, plasticization behavior of matrix, etc., in fact it is difficult to explain the causes behind such kind of behavior.

This can cause stronger adhesion between the fiber and the matrix which overpowers theoretically estimated percentage void content, and second reason underpinning this nature of curves can be the generation of internal cracking in the former situation due to which such behavior is being noticed. During machining, there were great chances of development of internal cracking in the former situation due to which internal cracking can be noticed.

The half-saturated solution shows more moisture absorption percentage in samples fabricated at 24,525 Pa than samples fabricated at the pressure of 43,600 Pa up to the third stage of weight measurement, and the percentage moisture absorption in the sample fabricated at 43,600 Pa becomes more due to initial swelling of matrix, dominated by the percentage void content, edge effect, and more moisture absorption taking place in the sample at 24,525 Pa while after the time instant of third stage of weight measurement, the mechanical adhesion between fibers and matrix dominates. Therefore, the swelling of the matrix in the case of half-saturated salt solution is much slower than the fully saturated salt solution. This may be due to that there was a distinct amount of swelling in the matrix of both the samples. It may happen because of more swelling in matrix of specimens maintained at 43,600 Pa than the specimens fabricated at 24,525 Pa. The gain in the weight at different time instants depicts the percentage moisture gain in the E-glass fiber composite. There is inapproachable equilibrium till the entire test and non-Fickian model was followed in the E-glass fiber epoxy composite during moisture absorption. Absorption of water continually rises initially in all the samples. And some inequality has been occurred at the interface such as breakdown of the chemical bonding between the glass fiber and the pattern at the interface. Therefore, there is a need to obtain more estimated data



**Fig. 8** i 99% saturated absorption, ii after an ample time. (saturated solution (43,600 pa), and iii after an ample time. (saturated solution (24,525 pa)

that can help us in the interpretation of more effects. It was also demonstrable that the flow of moisture absorption in the E-glass fiber epoxy composite can be significantly affected even by minor difference in the recovering phenomena (pressure in this case). Figure 8 represents the visual view of the test procedure.

### 3.2 Charpy Test Results

Table 5 shows the results of the all sample prepared at varying pressure condition of the hydraulics press. The effect of the varying pressure while fabricating sample was recorded. The results show the clear difference between the samples prepared at different pressure. The sample made up at high pressure shown the higher value of the rupture energy whereas the sample prepared at lower pressure shows the less amount of rupture taken to fracture. The sample prepared at 43,600 Pa shows 8.80% of changes in the rupture energy, similarly the sample prepared at 24,525 Pa shows 4.86% changes.

**Table 5** Charpy test results for both the samples prepared at 43,600 Pa and 24,525 Pa

	Specimen fabrication pressure	Specimen condition	Result (Joules)	% Change
Charpy test	43,600 Pa	Specimen in air	58.16	
		Specimen in half-saturated water for square root of 16.97 h	53.04	8.80
		Specimen in full saturated water for square root of 16.97 h	50.07	13.91
	24,525 Pa	Specimen in air	45.25	
		Specimen in half-saturated water for square root of 16.97 h	43.05	4.86
		Specimen in full saturated water for square root of 16.97 h	42.6	5.86

## 4 Conclusion

Following conclusion has been drawn from the experimentation.

1. The percentage absorption of moisture when it is in air for any time duration is always 0 at 43,600 Pa and 24,525 Pa.
2. If the square root time ranges from 0 to 9.79 percentage absorption at 43,600 Pa start increasing in half-saturated solution, normal water and in fully saturated solution but at 24,525 Pa after a specific time period there is variation in absorption it increases some time and sometimes decreases specifically in half-saturated solution and normal water.
3. The percentage of moisture absorption in samples fabricated at 43,600 Pa and immersed in normal water and fully saturated salt solution is more than the samples fabricated at 24,525 Pa for the entire phase of weight measurements. Now, when more time passes there is a small increase in absorption by 9.52% in half-saturated solution, but there is a large increase in absorption at this time in normal water it increased by 44.44% and a small increase in fully saturated solution, i.e., 9.68%.
4. In fully saturated solution after a time period percentage absorption becomes constant at both the pressures. The percentage void content in the latter case is approximately twice the percentage void content in the former case (43,600 Pa). It indicates that the sample fabricated at the pressure of 24,525 Pa (in the latter case) should have more moisture content.
5. Charpy results reveal that the pressure plays a critical role while fabricating the sample, as the pressure increases the sample particles get closed to each other and make strong bonding in between.

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