

**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH
TECHNOLOGY****EXPERIMENTAL STUDY AND ANALYSIS OF MECHANICAL BEHAVIOR OF
HYGROTHERMALLY CONDITIONED GLASS/EPOXY COMPOSITES****Dr. Shivakumar S^{*1}, Nikhil R², Dr. Geetanjali V Patil³ & Ashok H R⁴**^{*1}Professor & Head, Department of Industrial & Production Engineering, KLS Gogte Institute of Technology, Belagavi.²Assistant Professor, Department of Mechanical Engineering, KLS Gogte Institute of Technology, Belagavi.³Professor, Department of Mechanical Engineering, BLDEA's CET, Vijayapur.⁴Assistant Professor, Department of Mechanical Engineering, Bahubali College of Engineering, Shravanabelagola.**ABSTRACT**

High strength to weight ratio, corrosion resistance, good chemical stability, easy processing, and high insulating properties enable composites as advanced materials for marine, aerospace, automotive, and other high end applications. It is also well known that there is a degradation of material property during its service life, as it is often subjected to environments with high temperature and humidity or having a sharp rise and fall of temperature. The absorption of moisture can be attributed largely to the affinity for moisture of specific functional groups of a highly polar nature in the cured resin. In this work, the specimens were tested for their UTS, FS and ILSS and studied degradation pattern of Carbon FRPs when exposed to alkaline solution.

Keywords: Alkaline solution, FRP, SEM, Degradation Studies**INTRODUCTION**

Fiber reinforced plastics (FRP) are increasingly used in civil and marine structures due to their corrosion resistance, light weight, high strength and durability. Epoxy-glass and carbon fiber reinforced composites have found extensive applications in naval structures [1–3]. FRPs are considered for use in cost sensitive primary structural components for marine and offshore application [4]. But, the main drawback of using these materials is their susceptibility to degradation initiated by moisture, temperature, and corrosive chemical environment. The characterization in physical and chemical changes in the resin and reinforcement under these conditions it is very important to assess their performance. [5]. FRP degradation is a change in the properties such as physical (color, size and shape), mechanical tensile strength of FRP and FRP based product under the influence of one or more environmental factors such as heat, light or chemicals like acids, alkalis and some salts. These changes are usually undesirable, such as cracking and chemical disintegration of products or, more rarely, desirable, as in biodegradation, or deliberately lowering the molecular weight of a polymer for recycling. The changes in properties are often termed "ageing". In a finished product such a change is to be prevented or delayed. The degradation can be useful for recycling/reusing the polymer waste to prevent or reduce environmental pollution. Degradation can also be induced deliberately to assist structure determination. Polymeric molecules are very large (on the molecular scale), and their unique and useful properties are mainly a result of their size. Any loss in chain length lowers tensile strength and is a primary cause of premature cracking. The objective of this paper is to explore the useful properties of a hand lay-up processed glass/epoxy FRP materials. The research work attempts to analyze the effect of saltwater and hygrothermal on mechanical properties.

EXPERIMENTAL STUDIES**Materials and fabrication**

The glass/epoxy composites fabricated by hand lay-up technique. The Epoxy-glass plain woven- fabric was supplied by vertex. The fabric had an average filament diameter of 12 μ m & aerial weight of 0.329 kg/m². The

[ICAMS-2017: March, 17]

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glass fiber was supplied as coated with an emulsion based sizing agent to promote good chemical adhesion with the resin matrix. The Epoxy resin was supplied by Ciba-Geigy, Araldite-LY 556 and Amine Hardener HY 951. Fiber glass composite was fabricated using wet hand lay-up process into flat panels measuring 250 mm x 250 mm with a thickness of 3mm. The volume fraction of glass fiber was 65% and the porosity content about 2%. The composites were cured at room temperature without elevated temperature post curing because most marine composite structures are cured under ambient conditions.

Moisture observation test

After fabrication the composites were cured at room temperature for several weeks before being immersed in salt water. The edges of the panels were sealed with the matrix resin and after wet hand lay-up both types of laminates were pressed in hot press at room temperature to reduce the void contents. In cold soaking test the specimens were soaked continuously for one week, i.e., 100 hours. The weight and thickness of the specimens were measured before and after the soaking using electronic balance of accuracy 0.1 mg and digital Vernier calipers of accuracy 0.01mm to determine their rate of water absorption and thickness swelling. In long duration soaking test the specimens were soaked continuously for ten weeks i.e. 1000 hours. The rate of water absorption and thickness swelling were determined.

In soaking in boiling condition tests, the specimens were soaked continuously in boiling water (100 °C) for 20 hours. The same procedure was adopted for determining the rate of water absorption and thickness swelling. The top faces of the specimens were exposed to a 6500-watt xenon arc light source, which closely approximates natural sunlight spectrum in the visible and UV regions in an enclosed chamber at 40 to 43 °C. One face of each specimen was covered with aluminum foil so only one face of each specimen was exposed to UV light. The test contains exposure to UV radiation alternated with a spray of distilled water for 18 minutes followed by 102 minutes of UV light exposure without water spray and exposure time expressed as hours of exposure to light and at the end of 200 hours the specimens were weighed and their thickness measured.

Strength degradation due to Hygrothermal exposure

For hygrothermal condition the specimens of dimensions 120×12×3 mm (flexural test; ASTM D 790), 24×6×3 mm (inter-laminar shear strength test: ASTM D2344) and 220×19×3 mm (tensile test: ASTM D 638) were kept in the environmental chamber at 95% RH and 50°C, 60°C, and 70°C, respectively. The tests were conducted in a maximum duration of 170 days. The specifications of the chamber are: temperature: 20°C to 200°C, humidity: 0-95% RH with provision for salt spray. The specimens were taken at regular intervals, wiped dry to remove the surface moisture and then weighed by an electronic balance to monitor the mass change behavior or moisture diffusion. The percentage mass change (M) of the composite panel was computed.

RESULTS AND DISCUSSIONS

Moisture observation test

The rate of change of weight and the thickness swelling of the specimens due to water absorption in short duration soaking test is shown in Fig. 1(a). The rate of water picked up by the specimens more in the case of epoxy compared with vinyl ester based FRPs. The thickness swelling is very small in case of glass fiber / vinyl ester specimens and the highest swelling observed in epoxy based FRPs specimens. Fig. 1(b) shows the changes in rate of water absorption and thickness swelling of the specimens in case of long duration soaking. The rate of water absorption is lower in case of long duration soaking than that observed in short duration soaking irrespective of the specimen type.

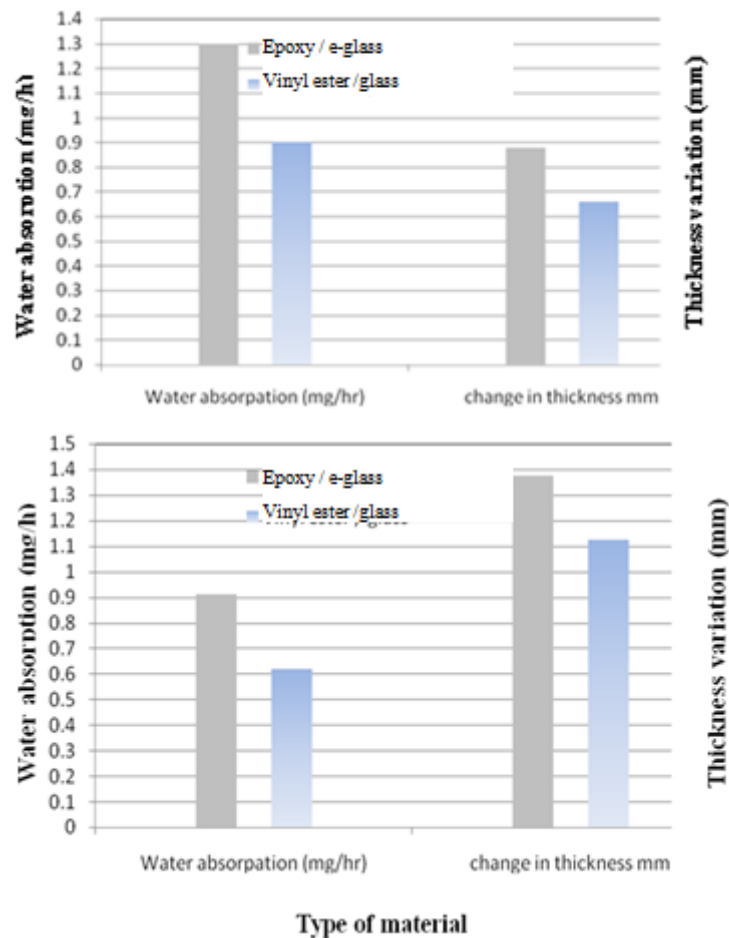


Fig. 1 a) Short duration soaking tests (100 hrs) b) long duration soaking test (1000 hrs)-water absorption and thickness variation of epoxy and vinyl ester composites

Although thickness swelling is on the higher side in long duration swelling the rate of thickness swelling is on the lower side compared to short duration soaking. Delamination is observed in epoxy / glass specimens only after the long duration soaking tests.

Fig. 2(a) shows the results of soaking under boiling conditions. The highest and lowest values of the rate of water absorption and thickness swelling were observed for epoxy / glass and vinyl ester / glass specimens respectively. Delamination was observed in all the specimens. The working temperature of epoxy specimens is known to be around 60 °C. Since the test is conducted at 100 °C in boiling condition with water circulation, interfacial strength between epoxy plies or epoxy and fiber reduces, leading to delamination.

Fig. 2(b) shows the rate of water absorption and thickness swelling due to UV and water spraying. The experimental values are highest in case of epoxy / glass and lowest in case of vinyl ester / glass specimens. UV rays more effectively create free radicals in these materials, whereas glass is inert since it has no C or H atoms, which evidence the experimental values obtained for the two types of specimens. In a typical fiber reinforced laminate, the system is generally modeled as having three phases: the fiber phase, the resin phase and resin-fiber interface. Because of high level of moisture absorption, poor wettability and insufficient adhesion between glass fiber and epoxy, these specimens suffer from debonding after the moisture exposures which also causes cracking between the fiber and resin, leading to swelling and deformation. On the other hand, in case of vinyl ester / glass specimens the interface region serves as an adhesion layer binding the resin to the glass and is formed by a coupling agent coated on the glass fibers which improves the wettability and bond strength between glass fiber and matrix hence it can be observed that there is no crack between the fibers. It is well known that epoxy is not

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stable to UV radiation. With a large number of tertiary carbon sites, epoxy is very sensitive to free radical formation and decomposition by chain scission resulting in the formation of lower molecular weight polymers on the surface. Hence, in case of epoxy specimens exposed to UV radiation, the color shifts from white to yellow.

Strength degradation due to saltwater exposure

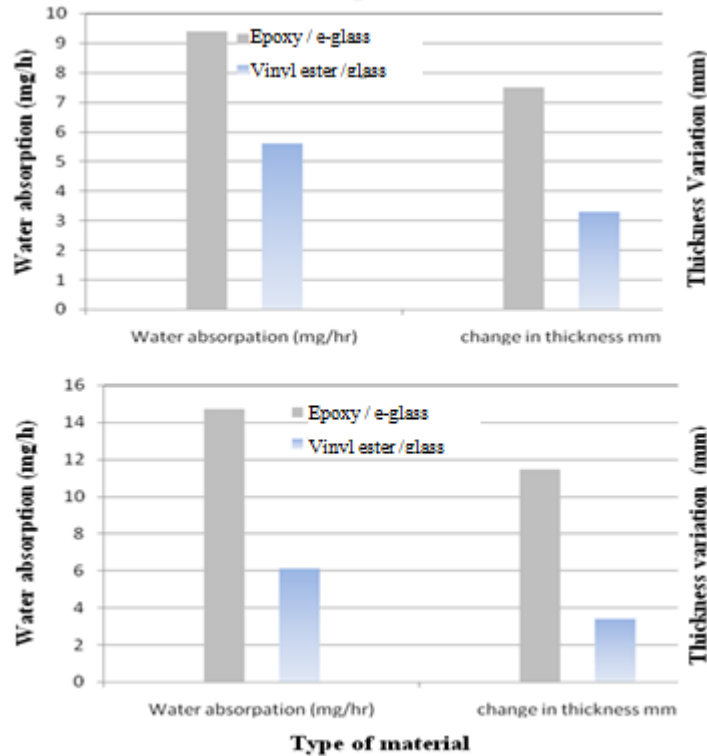


Fig. 2 a) Soaking in boiling conditions (20 hrs) b) UV rays with water spraying (200 hr)-water absorption and thickness variation of epoxy and vinyl ester composites

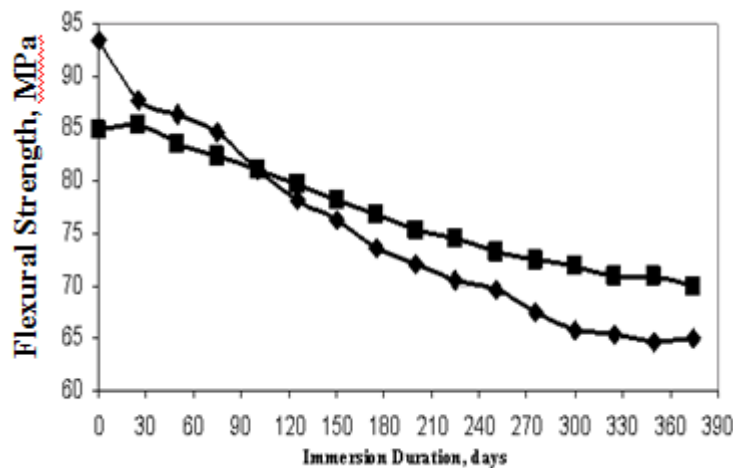


Fig. 3 Flexural strength degradation versus duration of saltwater exposure

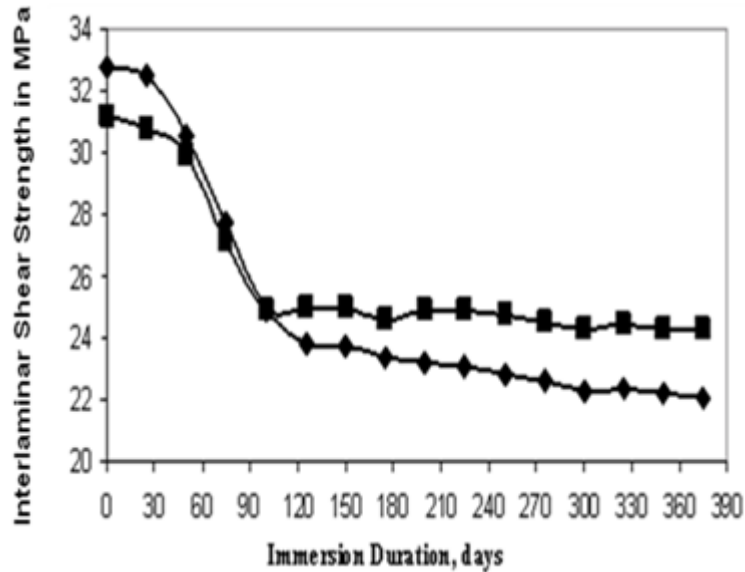


Fig. 4 ILSS degradation due to saltwater immersion

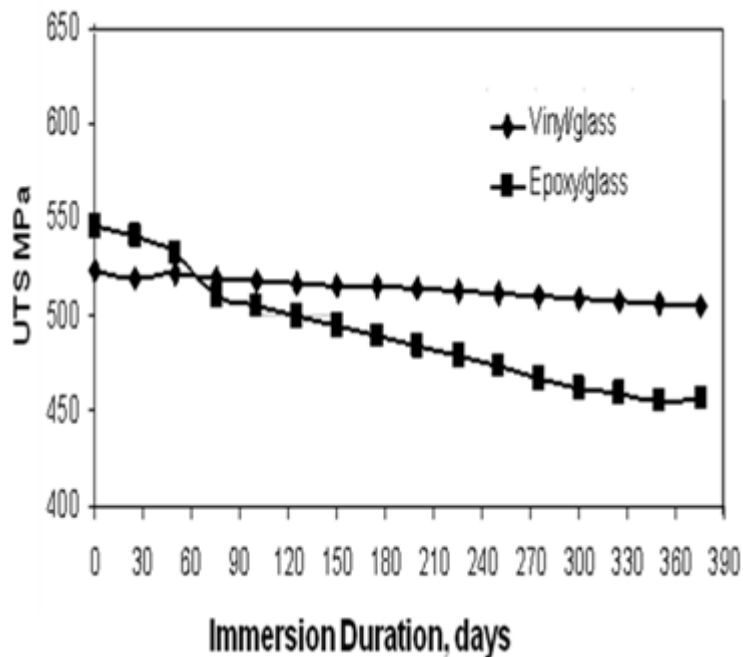


Fig. 5 UTS degradation versus immersion duration

Fig. 3 shows change in flexural strengths of epoxy/glass, vinyl ester/ glass FRP with respect to different salt water-exposure times. Though all the specimens showed drop in flexural strength with respect to immersion time because of moisture uptake, vinyl ester-glass specimens showed lower levels of degradation. Glass reinforced specimens the degradation was 24.75% for epoxy and 18.589% for vinylester. Vinyl ester composites showed higher strength than the epoxy, The epoxy based specimens showed the stability after 125 days. The trend in the interlaminar shear strength degradation behavior was observed to be very similar to flexural strength and it is shown in Fig. 4. ILSS is one of the important properties for composites, which determines the load sharing by the fibres, i.e. the interfacial strength. Vinyl ester /glass samples showed superior stability on ILSS degradation and the % drop was 7.882% and epoxy/glass showed a percentage drop of 10.567. Thus it concludes from the interlaminar shear strength properties that vinyl ester based specimens are superior to the other ones tested. Vinyl-ester based samples achieved its stability with in short period of time compared to

the other systems. Salt water in the resins could causes vibration and rotation on the cross links that may break the bonds. Surface changes in the e-glass fiber on saltwater immersion reduce the strength of the fiber by an extent also the reason for the degradation in the ILSS in the material reinforced with epoxy-glass fiber. A progression of change in tensile strength as a function of immersion time is shown in Fig. 5 for the specimens immersed in salt water.

It clearly shows that the degradation increases substantially with increase in immersion time. The rate of percentage degradation was observed to be higher in the initial stage of immersion up to 100 days for epoxy based specimens and it reduced thereafter but vinyl ester based samples reached its saturation even before 100 days. The UTS degradation was more in epoxy/glass- 41.078% and vinyl-ester/glass showed a drop of 32.954%. This results in a mismatch in the moisture-induced volumetric expansion at interfaces and this leads to the evolution of localized residual stress fields in the composites.

SEM provides good evidence of the binding between the resin and the fiber before and after the salt water immersion. The degradation in mechanical properties is due to the degradation in the fiber matrix interfacial bonding. The voids present in the matrix are the main reason for moisture diffusion. Fig. 6 shows the fracture section of (a) epoxy/glass after salt water immersion, (b) vinyl ester/glass with 170 days of salt water exposure. Fig. 6(a) shows a higher level of surface degradation and pitting, and also numerous bare debonded fibers, which substantiates the fact that the reduction in transverse strength is largely due to fiber/matrix interfacial degradation.

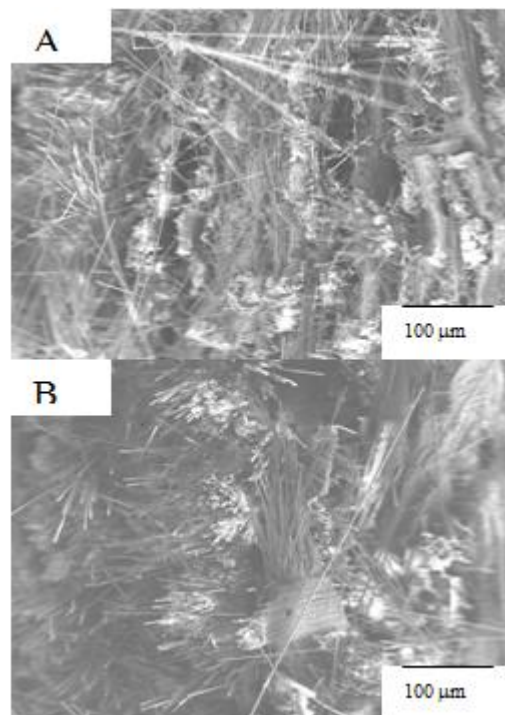


Fig. 6 Atypical SEM image of A) Glass/epoxy and B) Glass/vinyl ester FRP after immersion

Hygrothermic Durability Studies

Hygrothermal moisture absorption as a function of duration of exposure is presented in Fig. 7. Epoxy/glass absorbed the highest amount of moisture, i.e., 0.95 wt% at 70°C and 95% RH. The least was in the case of vinyl-ester/glass as 0.5 wt% at 70°C and 95% RH and 0.5 wt% at 50°C and 95% RH. The saturation in moisture uptake was observed by the asymptotic nature of the curves. Vinyl-ester/glass stabilized at much lower values of

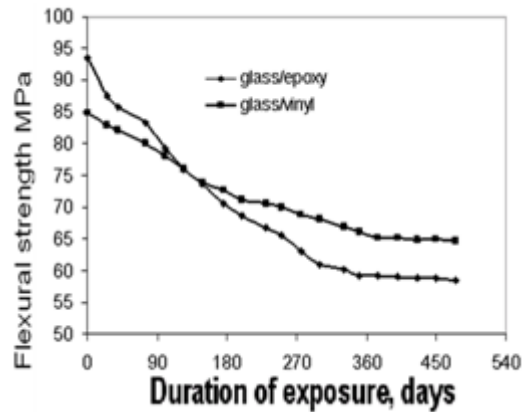


Fig. 7: A typical graph of flexural strength degradation of glass based PMCs due to hygrothermic exposure at 50C, 95% RH

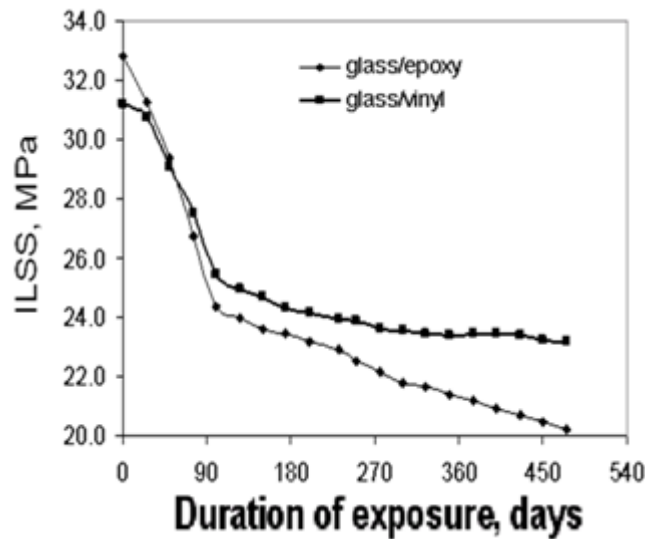


Fig. 8: A typical graph of ILSS degradation of glass based PMCs due to hygrothermic exposure at 50C, 95% RH

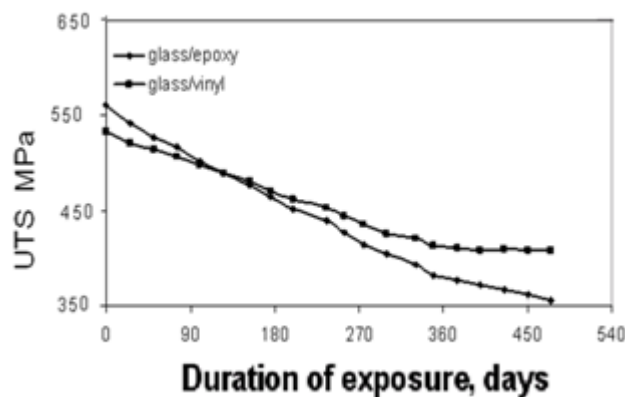


Fig.9: A typical graph of UTS degradation PMCs due to hygrothermic exposure at 50C, 95% RH

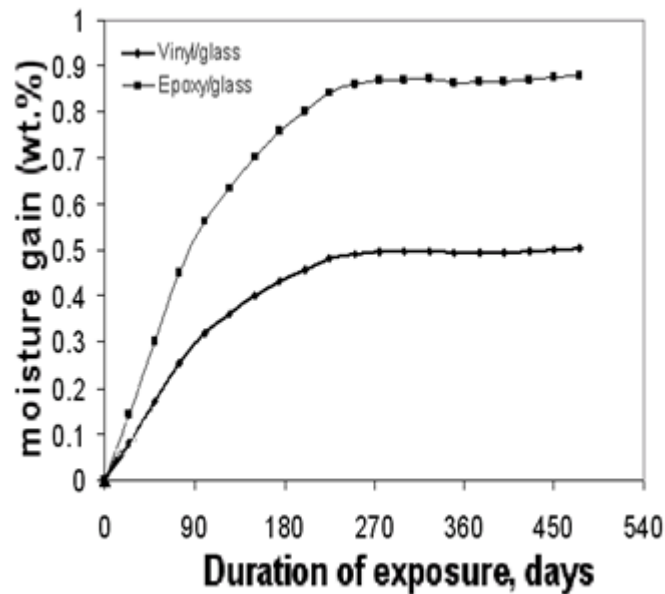


Fig. 10: A typical graph of % moisture absorption due to hygrothermic exposure of 50C, 95%RH

moisture uptake at all the three temperatures. Since the saturation levels of moisture uptake dictates the property of degradations in the materials employed for underwater applications, vinyl-ester based composites proved superior to epoxy based specimens.

All the composite materials showed degradation in flexural strength, UTS and ILSS due to hygrothermic conditioning (Figs. 8-10). Epoxy / glass showed greater degradation than vinyl-ester/glass in UTS, FS and ILSS. The degradation increased with increase in temperature. Water penetrates into the interface through the micro voids and micro cracks and the trapped water damages the interface region where it trapped. In longer duration of exposure, the water diffusion will be slowed because of the water saturation in the matrix phase. The property degradation in the post saturation period is considerably less than that of the pre-saturation period.

Fig. 11 provides evidence of the resin/fiber binding in fractured specimens. The degradation in mechanical properties is due to the degradation in fiber/matrix interface bonding. The voids present in the matrix are the main reasons for moisture diffusion. The micro cracks formed during the composite preparation or by the service condition can store the trapped water.

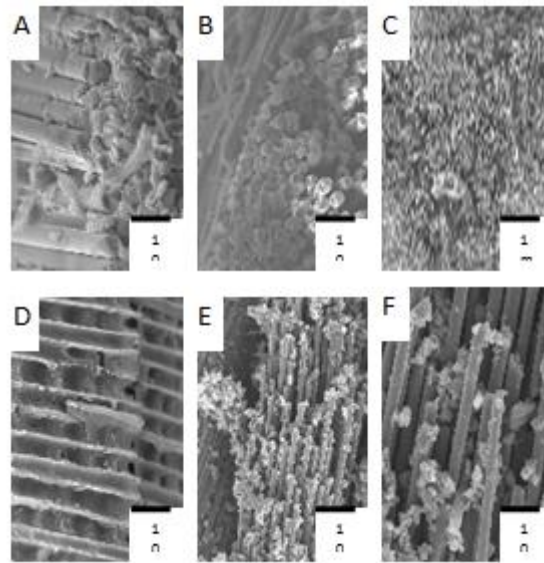


Fig. 11: A typical graph of fractured surface of glass based PMCs due to hygrothermic exposure (A, B & C 50, 60 & 70 °C of vinyl ester / glass and D, E & F 50, 60 & 70 °C of epoxy / glass composites)

The voids and micro cracks in the specimens can be traced using SEM images. Fiber breaking was the dominant failure mode in the specimens exposed to 50 °C, 60°C and 70°C for a period of 25 days and under these conditions only the specimens exposed to 70 °C showed slight matrix degradation. Fiber pull out was the main mode of failure in the specimens after 170 days of exposure at all the three temperatures. The effect of temperature is seen in terms of greater levels of matrix degradation and hence more fiber pullout as observed in 70 °C conditioned specimens.

All the three temperatures epoxy/glass showed greater degree of matrix degradation than that of other types of specimens. After 170 days of exposure, epoxy/glass showed greater levels of matrix degradation than that of vinyl-ester/glass. Thus, matrix degradation increased with temperature and duration of exposure in all the specimens.

CONCLUSION

In this work, behavior of glass reinforced polymer composites after salt water expose and hygrothermal ageing was studied experimentally. It was found that, in UV with moisture tests glass/ epoxy have both shown to be prone to degradation and hence degradation is severe in glass/ epoxy specimens whereas in glass/ vinyl ester specimens only glass has undergone degradation. Vinyl ester / glass showed least UTS degradation (12.9MPa), followed by Epoxy/glass (17.8 MPa). Among VE/glass and Epoxy/glass, the former proved superior in terms of lower UTS degradation being 12.9 % compared to that of the latter being 17.8 %, this is due to the moisture uptake levels in the two systems being 0.38 wt % and 0.63 wt % respectively. With respect to Flexural Strength degradation, VE / glass (47 MPa drop) proved superior to Epoxy / glass (67 MPa drop). Drop in ILSS was 26.5 MPa for VE / glass compared to 30.8 MPa for Epoxy / glass.

Scanning Electron Micrographs of saltwater exposed samples showed that moisture penetration along the fiber matrix interface can cause plasticization of the matrix and weakening of the interface, hence fiber pull out and debonding. Epoxy / glass specimens after long durations showed greater plasticization and fiber pull out compared to VE / glass. The moisture uptake in all the three test conditions showed that VE / glass specimens absorbed the least values of maximum moisture uptake, 0.42, 0.44 and 0.45 wt % at 50, 60 and 70 C. Increase in temperature caused higher level of diffusion and hence the onset of saturation showed advancement in the number of days of exposure to saturation. Consistently all the specimens showed increase in the moisture uptake with respect to increase in temperature. VE / glass proved superiority over epoxy / glass in terms of lower values of degradation in UTS, Flexural Strength and ILSS in all the three test conditions, 50, 60 and 70 C & 95 % RH..

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