Journal of Computers, Mechanical and Management

Journal homepage: www.jcmm.co.in

Volume 1 Issue 1

Influence of Seawater Absorption on the Hardness of Glass Fiber/Polyester Composite

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Abstract

In the marine industry, glass fibers are commonly used to reinforce polyesters for ship hulls, submarine components, and other marine structures. Isophthalic polyesters are a feasible alternative due to their superior mechanical qualities and added end-of-life scenarios compared to orthophthalic polyesters. However, like other fiber composite systems, glass fiber reinforced polymer (GFRP) composites are also water sensitive. Here, GFRP composites of three different thicknesses are aged under three different immersion periods in seawater (20, 40 and 60 days). All samples are evaluated for hardness following aging. Significant emphasis is placed on the presence of calcium carbonate, over which increases in moisture content irrevocably reduce the composite's hardness. Compared to untreated material, the hardness of 6, 8 and 10 mm composites decreased by 25.64, 10.92 and 4.63% after the 60-day aging period. This drop is mostly the result of microstructure evolution manifesting as an increase in porosity. Consequently, fiber deterioration, fiber cracks, and degradation of polymer-fiber bonding emerge in the composite, decreasing hardness.

Keywords: Degradation; Hydrolysis; Polyester Composites; Hardness; Material Chemistry

1 Introduction

Components and structures functioning in the marine environment are subjected to high stresses caused by wind, waves, and tides. In addition, they must contend with hostile and harsh environmental circumstances throughout their existence, as they are placed in the splash zone if not submerged in saltwater. The application of polymer composites in maritime systems has been the subject of significant research over the past few decades, showing the potential benefits of replacing numerous components, including ship hulls, domes and non-pressure hull decking for submarines, propeller blades, wind turbine blades, and tidal turbine blades, to name a few [1–3]. To be precise, it is since the mid-1980s that the usage of composite materials in marine constructions has expanded dramatically, and the global industry anticipates an annual growth rate of 5.8% as an alternative to galvanic corrosion-prone classical materials [4, 5]. However, fiber reinforced polymer (FRP) composite materials are sensitive to moisture and temperature, and prolonged exposure to such a harsh environment degrades the material's mechanical characteristics. The seawater environment tends to chemically alter the composition of FRP composites [6–8]. Therefore, to anticipate the service life of structures and the functional stability of the composites, it is vital to understand the robustness and life cycle of composites in terms of their mechanical, chemical, and thermal properties [9]. In other words, it is crucial to assess the degradation of mechanical properties caused by exposure to seawater for marine composites [9]. Hardness, a mechanical attribute of the material, is its ability to resist deformation and is determined by a standard test that measures the surface resistance to indentation [10–12].

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Received: 24 August 2022; **Accepted:** 04 September 2022; **Published:** 30 October 2022

DOI: 10.57159/gadl.jcmm.1.1.23003.

Hardness not only affects the tribological performance of a material [13] but also is an important factor to be considered while machining, in particular drilling, as the drill tool life is found to be a strong function of it [14, 15]. A mechanical connection is required between FRP composites and other metal or composite structures. As a crucial final manufacturing stage for composite laminates, drilling is widely employed to facilitate riveted and bolted joints for attaching the composite structure to other components [16]. The most prominent concerns in FRP composite drilling are delamination and hole surface roughness. It has been discovered that tool wears greatly contributes to both problems [17–20]. From the literature reviewed, hardness is determined to be an important mechanical property to be investigated as it holds significance in machining materials, particularly the drilling of composites. It is also learned that seawater exposure has adverse effects on the FRP composites, leading to an alteration in the material chemistry resulting in material degradation. Though several researchers have been focusing on investigating the various mechanical properties of the marine FRP composites, there is no document that exclusively discusses the investigation of seawater immersion effect on the FRP composite hardness. Thus, filling the literature gap and emphasizing the non-addressed but important mechanical property, this study aims to investigate seawater's effect on the hardness of marine FRP composites of varying thicknesses. The present study not only experimentally indicates the effect but also discusses the root cause concerning the chemistry behind it at the microstructural level using SEM and EDX analysis.

2 Materials and Methods

2.1 Composite composition and fabrication

The glass fiber reinforced polymer (GFRP) composite material used in this investigation consisted of chopped stranded mats containing discontinuous E-glass fibers with a density of 1.56 gcm-2. The matrix phase is composed of thermoset polymer resins with a density of 2.56 g/cm2, which are unsaturated (isophthalic) polyesters. Methyl-ethyl-ketone peroxide (MEKPO) is used as a hardener, also known as a curing agent or catalyst, to enhance the crosslinking of unsaturated polyesters (curing) [21, 22]. The composites produced contained 33.4 wt.% of fibers. When creating composites, a hardener-to-resin ratio of 0.0012:1 and a matrix-to-fiber ratio of 2:0 was considered. For manufacturing GFRP composites, the manual open-mold hand-layup approach [23] was adopted. Before applying the mold release agent, the plywood mold was thoroughly cleaned. As a release agent for the mold, wax and polyvinyl acetate was utilized [24, 25]. The liquid polyester was used to create the gel coating, followed by applying the first layer of the fiber mat. The matrix consisting of the polyester-hardener mixture was then brushed over it, and the second layer of glass fiber was deposited on top. The matrix was distributed evenly throughout the composite using a hand roller. Thus, the described layer-by-layer hand-layup technique was employed to manufacture 6, 8, and 10 mm thick GFRP composites.

2.2 Seawater treatment and mass gain determination

Seawater was produced in the laboratory following ASTM D1141-98 Standard practice for the preparation of substitute ocean water [26, 27]. Seawater is slightly alkaline, with pH values between 8.0 and 8.5 [28]; hence the pH of the prepared seawater was maintained between 8.3 to 8.5. The chemicals used and their proportion for making 10 liters of seawater is as shown in Table 1.

Table 1: Chemical composition for preparing 10 liters of seawater as per ASTM D1141-98 standard.

Composition (chemical formula)	**Fraction (g/10 liters)**
Sodium chloride ($NaCl$)	245.34
Magnesium chloride ($MgCl_2{\cdot}6H2O$)	111.12
Anhydrous sodium sulfate (Na_2SO_4)	40.94
Anhydrous calcium chloride ($CaCl_2$)	11.58
Potassium chloride (KCl)	6.95
Sodium bicarbonate ($NaHCO_3$)	2.01
Potassium bromide (KBr)	1.00
Boric acid (H_3BO_3)	0.27
Strontium chloride ($SrCl_2{\cdot}6H_2O$)	0.42
Sodium fluoride (NaF)	0.03
0.1 N Sodium hydroxide ($NaOH$) solution	Added in quantity sufficient to get the pH level to 8.3 ±0.2

All chemicals utilized during the research were of research grade. Thus prepared, seawater was poured into three separate 10-liter-capacity containers. In addition, three different colors of buckets were used to distinguish between the specimen contained in each container, based on the material thicknesses. A precise digital weighing balance with a weight limit of 10 kg and an accuracy of 1 gram was used to weigh the aged GFRP composite slabs of every thickness at 20-day intervals up to 60 days to determine the water absorption by the material periodically. Before weighing, tissue paper was used to wipe the surface of the samples.

Using Eq. [1], the relative mass change m (%) was calculated, where mi and mf represent the initial and final mass of the material, respectively [29]. The weight variations of GFRP composites with thicknesses of 6, 8, and 10 mm were determined by weighing five replicates of each thickness.

$$m(\%) = \frac{m_f - m_i}{m_i} \times 100 \tag{1}$$

2.3 Hardness measurement

The ASTM D2583-07 standard test method for indentation hardness of rigid plastics [30] is used to perform Barcol hardness tests with the Barber Colman impresser. The hardness measurement characterizes the indentation strength of components as assessed by the indenter point's penetration depth. The test was conducted for untreated and treated composite samples. Five Barcol hardness measurements were taken for each GFRP slab of every thickness, and the average value was chosen for each specimen's final Barcol hardness measurement.

2.4 Material characterization

The material characterization was accomplished using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis. The SEM was employed to investigate the microstructural damage caused in all the composite samples of different thicknesses. The diameters for untreated and treated fibers were measured using the ImageJ software to check for degradation in terms of diameter reduction. The EDX analysis was used not only to check the probability of the presence of salts like potassium chloride [KCl], potassium bromide [KBr] and sodium chloride [NaCl] but also to determine the atomic weight percentage reduction in Silica [SiO_2]and other constituents of the E-glass fiber (magnesium and aluminum oxides) due to degradation in the seawater treated composites. The presence of calcium carbonate [$CaCO_3$] is also investigated using the EDX analysis. Zeiss Evo MA18 with Oxford EDS(X-act) equipment installed in the Central Instrumentation Facility (CIF), MIT, Manipal, India, was used to conduct both SEM and EDX analysis.

3 Results and Discussion

Figure 1 represents the hardness reduction (%) of 6, 8 and 10 mm thick GFRP composite at different immersion periods. The negative sign in Figure represents that reduction in the hardness value. A trendline is obtained for each represented graph considering a third-order polynomial equation.

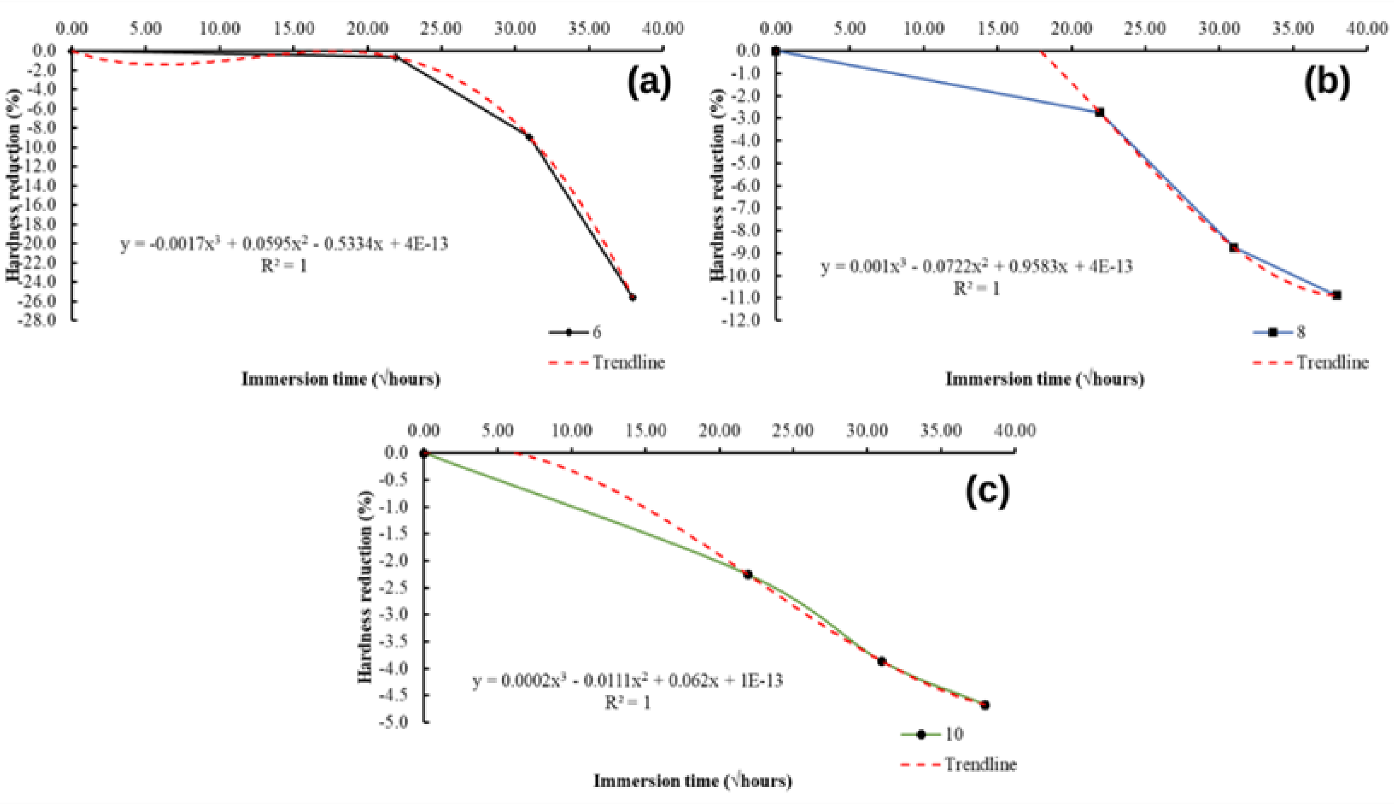

Figure 1: Results of hardness reduction for **(a)** 6mm; **(b)** 8 mm; **(c)** 10 mm GFRP composites.

The developed equation for 6, 8 and 10 mm GFRP composites are given by Eqs. [2], Eq. [3] and Eq. [4], respectively. The term y_i in the equations represents the hardness reduction (%) value for i^{th} thickness, and x represents the immersion period (square root of hours). All the developed equations possess high prediction accuracy (R^2 = 1) within the experimented limits. The average standard deviations are recorded as 0.531, 0.601 and 0.515 for 6, 8 and 10 mm GFRP composites.

$$y_6 = -0.0017x^3 + 0.0595x^2 - 0.5334x - 4e^{-13} \tag{2}$$

$$y_8 = 0.001x^3 - 0.0722x^2 + 0.9583x - 4e^{-13} \tag{3}$$

$$y_{10} = 0.00025x^3 - 0.0111x^2 - 0.062x - 1e^{-13} \tag{4}$$

The average Barcol hardness was recorded as 43.3, 46.7 and 49.7 BHN for 6, 8 and 10 mm thick fabricated untreated GFRP samples, respectively. A continuous drop was later observed in the hardness value irrespective of the thickness of the GFRP composites, and the decrement continued with the increase in the immersion period. Table 2 details the average hardness value for 6, 8 and 10 mm GFRP considering the three selected immersion times.

Table 2: Hardness test results for 6, 8 and 10 mm seawater treated GFRP composites.

Immersion time (days)	**Composite thickness (mm)**	**BHN of untreated specimen**	**BHN of seawater treated specimen**	**Reduction(%)**
20 days	6	43.3	43.0	0.69
	8	46.7	45.4	2.78
	10	49.7	48.6	2.21
40 days	6	43.3	39.4	9.01
	8	46.7	42.6	8.78
	10	49.7	47.8	3.82
60 days	6	43.3	32.2	25.64
	8	46.7	41.6	10.92
	10	49.7	47.4	4.63

Figure 2 represents the mass gain rate (%) of 6, 8 and 10 mm thick GFRP composite at different immersion periods. A trendline is obtained for each represented graph considering a third-order polynomial equation.

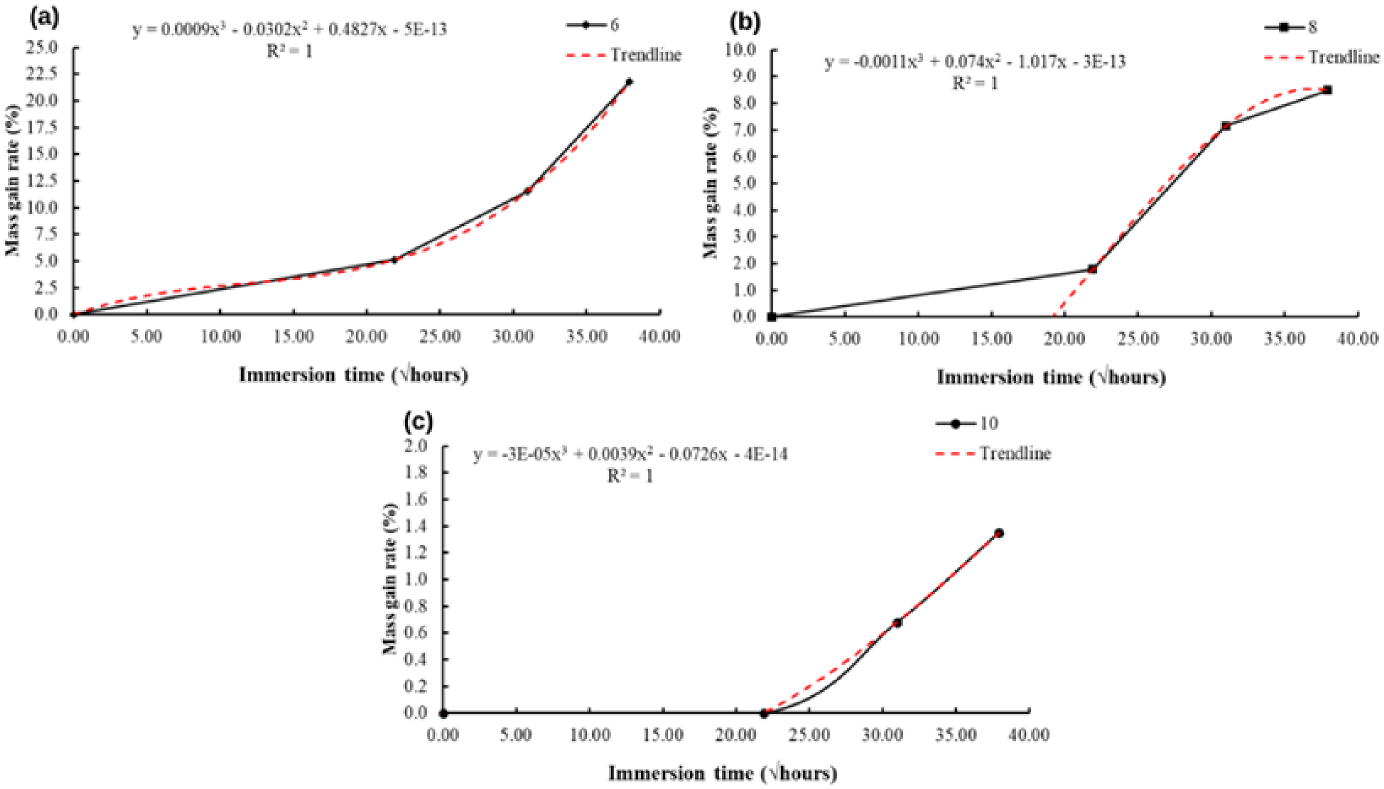

Figure 2: Results of mass gain rate for **(a)** 6mm; **(b)** 8 mm; **(c)** 10 mm GFRP composites.

The developed equation for 6, 8 and 10 mm GFRP composites are given by Eq. [5], Eq. [6] and Eq. [7], respectively. The term y_i in the equations represents the mass gain rate (%) value for ith thickness, and x represents the immersion period (square root of hours). All the developed equations possess high prediction accuracy ($R^2 = 1$) within the experimented limits. The average standard deviations are determined to be 0.634, 0.436 and 0.031 for 6, 8 and 10 mm GFRP composites. When immersed in seawater, the composites absorb water molecules and release the soluble solid particles. Therefore, the mass gain rate represents the net effect of mass gained due to water absorption and the mass lost due to the oozing out of soluble solid particles from the composite. The average mass was recorded as 78, 112 and 148 g for 6, 8 and 10 mm thick fabricated untreated GFRP samples.

$$y_6 = 0.0009x^3 - 0.03025x^2 + 0.4827x - 5e^{-13} \tag{5}$$

$$y_8 = -0.001x^3 + 0.074x^2 - 1.107x - 3e^{-13} \tag{6}$$

$$y_{10} = -0.0003x^3 + 0.0039x^2 - 0.072x - 4e^{-14} \tag{7}$$

Table 3 provides the results for mass change in composites due to water absorption. The thinner composites were seen to absorb more amount of water. Therefore, it could be said that the thicker composites with a greater number of lamina layers prevent the degradation as the salt particles do not tend to penetrate much in such compositions. The unit used for the immersion period in Figure 1 and Figure 2 is the square root of hours, as it provides a neat and regular curve. Moreover, the prediction within the experimental limits of response value for a small immersion period gets easier. The reduction in the hardness value and increase in the water absorption values with the increase in the immersion period is mostly the result of microstructure evolution manifesting as an increase in porosity. The microstructural analysis was conducted using the SEM and EDX analysis to affirm the assumption.

Table 3: Mass gain rate results for 6, 8 and 10 mm seawater treated GFRP composites.

Immersion time (days)	**Composite thickness (mm)**	**Mass of untreated specimen (g)**	**Mass of seawater treated specimen (g)**	**Mass gain rate(%)**
20	6	78	82	5.13
	8	112	114	1.79
	10	148	148	0.00
40	6	78	87	11.54
	8	112	120	7.14
	10	148	149	0.68
60	6	78	95	21.79
	8	112	122	8.93
	10	148	150	1.35

The results of the SEM analysis for 6, 8 and 10 mm thick GFRP composites concerning different seawater immersion periods are depicted in Figure 3, Figure 4 and Figure 5 respectively. Irrespective of the thickness, the severity of the composite degradation is observed to increase with the increasing period of immersion. The fiber cracks are visible in the presented SEM images. The SEM images were also analyzed using the ImageJ software to determine the degradation of fibers characterized by the change in the diametric values. The average diameter of the fibers in untreated samples was 16 µm. The average diameter value was reduced to 15.5, 14 and 11 µm considering the GFRP samples subjected to 20, 40 and 60 days of seawater immersion. Changes in diameter values were reported to be greatest in samples with a thickness of 6 mm and smallest in samples with a thickness of 10 mm. On average, the reduction in diametric values was observed to be 3.13, 12.5 and 31.25%. The fiber cracks and the reduction of diametric value confirm the degradation of the fibers used in the composites. Moreover, the SEM images also evidently show the severe matrix erosion at the surface and matrix-fiber debonding. The matrix degradation in the GFRP samples was because of the leaching action, wherein the distributed salt particles (present in the seawater) leach away to form a porous structure [31]. Irrespective of the thickness, the matrix degradation is observed to have initiated in the 20 days aged samples. Nevertheless, the matrix erosion was low in the case of 10 mm samples compared to the counterparts for any given period of immersion time. The phenomenon observed in all the samples was the leaching of the matrix due to salt present in the seawater medium, causing porosity and leading to further penetration of the seawater into the composites. The second stage is wherein the matrix-fiber debonding occurs, and the component functional stability starts weaking. In this phase, the matrix degrades and exposes the fibers to the seawater medium, which causes cracks in them. With a further increase in immersion time, the matrix erosion increases, causing greater damage to the composites. The salt particles could not damage the 10 mm thick composites much, probably due to the higher amount of fibers and matrix layers than their counterparts. However, an increased immersion time than the selected period in the current study can cause damage to it and thus can be considered as a future scope of the present work. The matrix degradation is also caused due to the hydrolysis (breaking of the crosslinking bondage) of polyesters caused due to the presence of sodium hydroxide and potassium salt [28, 32]. The reinforcing phase provides the mechanical strength in a fiber composite compared to the matrix material [33]. In this study, it is the glass fibers. Therefore, to further examine the reason for the reduction of hardness caused due to severe degradation of fiber material, particularly after 60 days of immersion period, EDX analysis was conducted. Glass fibers are made up of silica [SiO_2] and other material oxides like calcium [CaO], magnesium [MgO]and aluminum [Al_2O_3][33]. The EDX analysis aimed to observe the reduction in the atomic weights of these elements and to check for the formation of calcium carbonate [$CaCO_3$].

The data obtained from the supplier for the glass fibers were taken as the benchmark for its various constituents. The glass fiber used in the present study comprised 55.2 % silica, 18.7 % calcium oxide [CaO], 8% aluminium oxide [Al_2O_3], 7.3 % of lithium oxide [Li_2O], and 4.6 % magnesium oxide [MgO] as its major constituents. As assumed, the atomic weight of silica and magnesium and aluminum oxides has decreased in the seawater-treated specimen irrespective of the composite thickness.

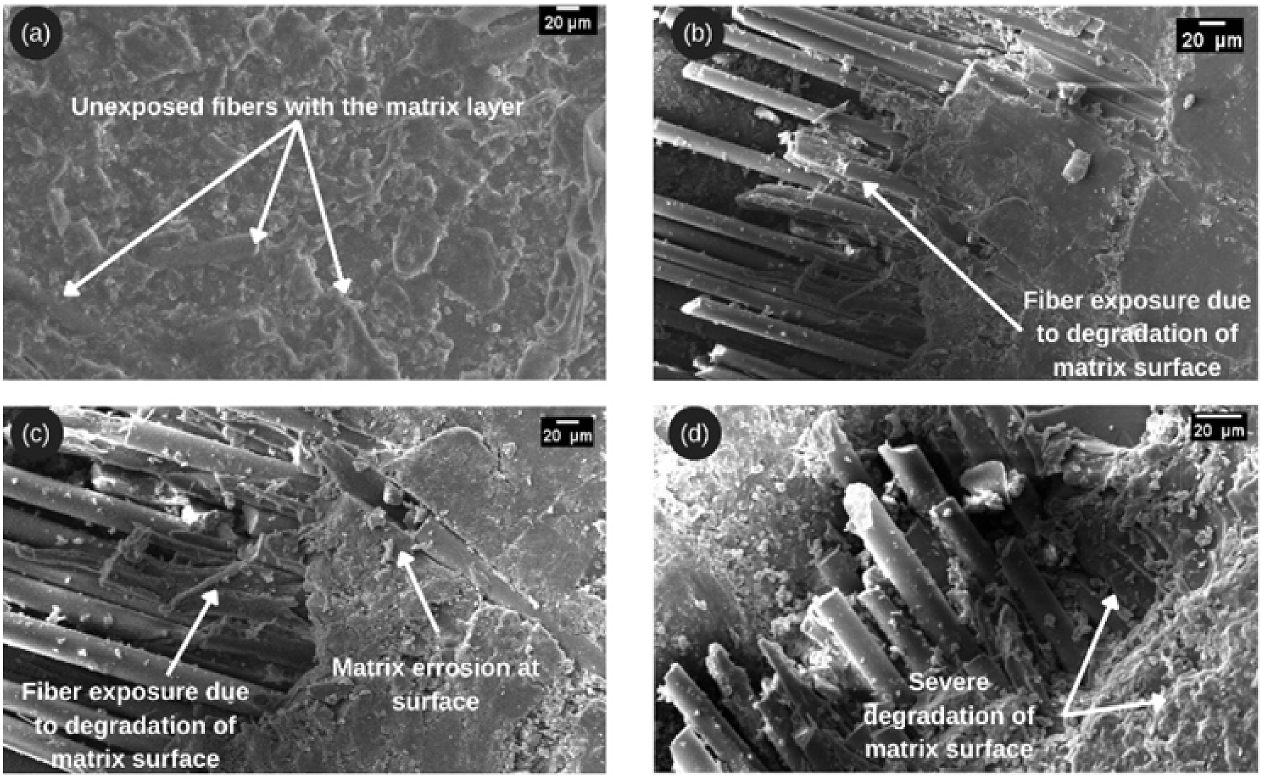

Figure 3: SEM results for 6 mm GFRP: **(a)** untreated; **(b)** 20 days seawater treated; **(c)** 40 days seawater treated; **(d)** 60 days seawater treated.

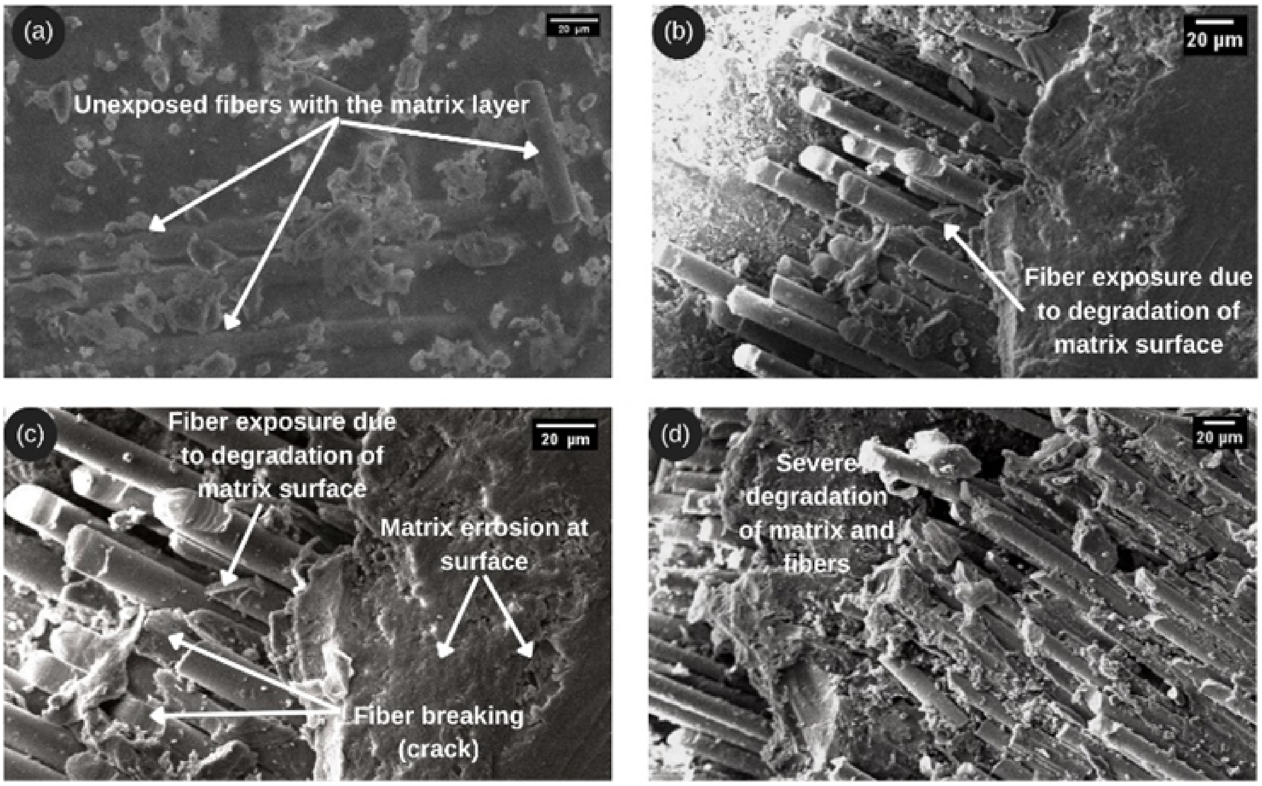

Figure 4: SEM results for 8 mm GFRP: **(a)** untreated; **(b)** 20 days seawater treated; **(c)** 40 days seawater treated; **(d)** 60 days seawater treated.

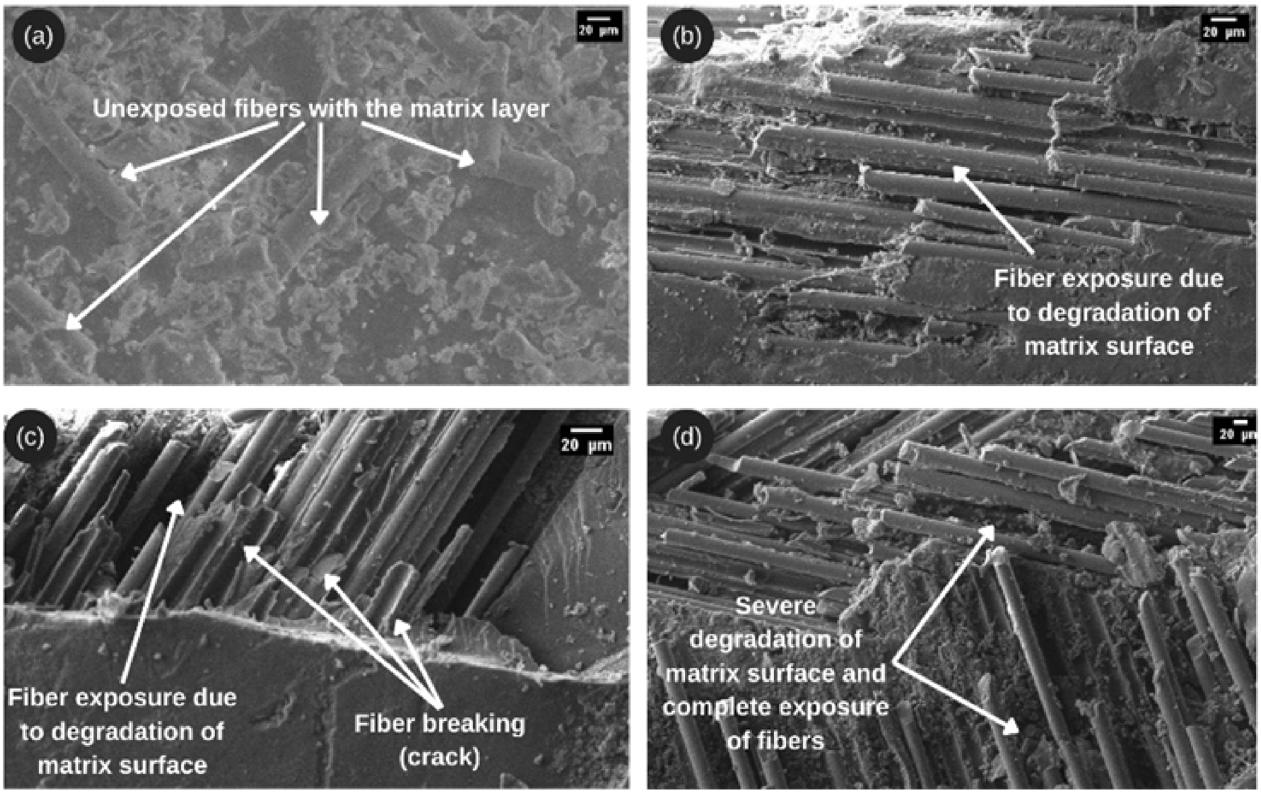

Figure 5: SEM results for 10 mm GFRP: **(a)** untreated; **(b)** 20 days seawater treated; **(c)** 40 days seawater treated; **(d)** 60 days seawater treated.

The loss was seen to be higher for the 6 mm thick composite, followed by the 8 and then the 10 mm thick samples. The EDX results support all the results obtained for the hardness test and SEM analysis. The results justify the decrease in the diameter values of the fibers, increasing the porosity. The atomic weight of silica is seen to reduce on average by 45.50, 42.75 and 36.68 % in 6, 8 and 10 mm composites. The average reduction in the atomic weight of aluminum oxide is by 3.08, 2.16 and 1.73 % for 6, 8 and 10 mm thick composites. Considering the average reduction in the atomic weights of magnesium oxide for 6, 8 and 10 mm composites, it was recorded as 2.47, 0.825 and 0.65 %. The traces of potassium chloride [KCl] and potassium bromide [KBr] salts in good percentages were found in 6 and 8 mm thick composites, which might have caused the leaching effect. As mentioned earlier, the salts could not penetrate much into the 10 mm thick material. Figure 6, Figure 7 and Figure 8 illustrate the results of the discussed EDX analysis. In addition, the calcium carbonate is in higher proportion in the 6 mm composites and least in the 10 mm composite. The average atomic weight of the CaCO3 is determined to be 34.49, 33.49 and 23.25 % in the 6, 8 and 10 mm composites, respectively. The EDX results and all other tests justify the degradation in the GFRP composites subjected to seawater conditions. The EDX results also justify the importance of the thickness of the composites when used for marine conditions.

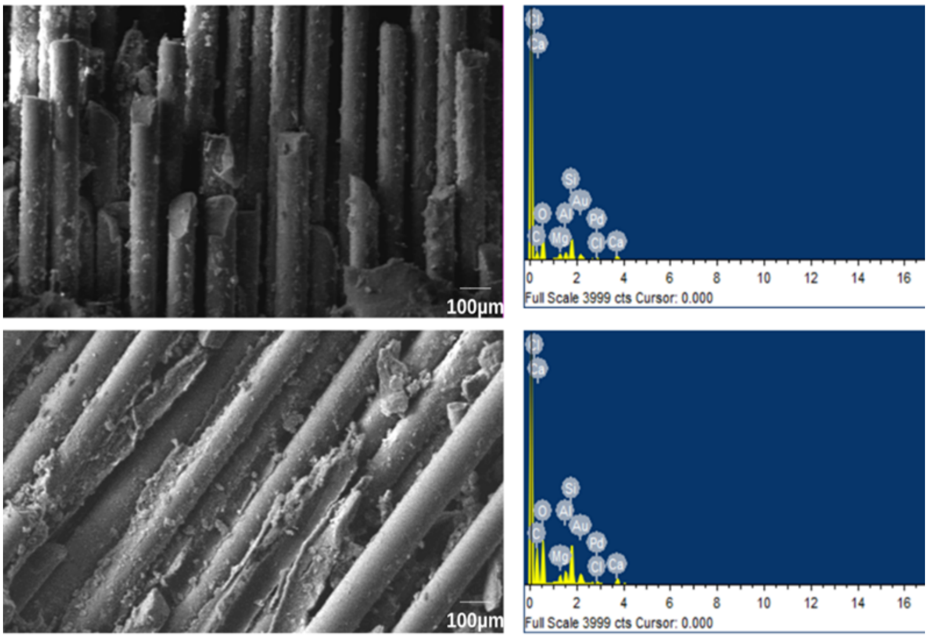

Figure 6: EDX results for 60 days seawater treated 6 mm GFRP at two different positions.

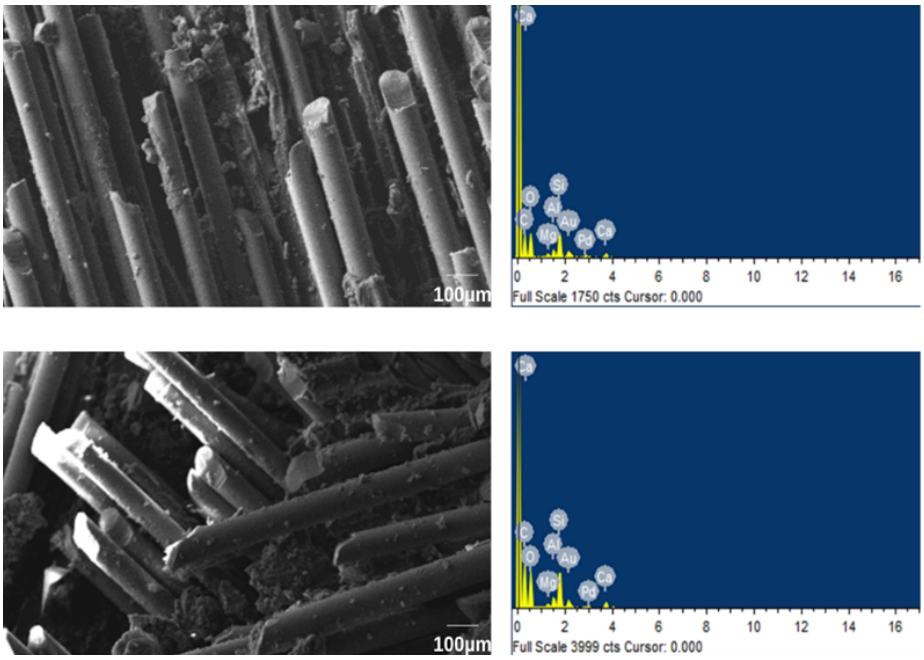

Figure 7: EDX results for 60 days seawater treated 8 mm GFRP at two different positions.

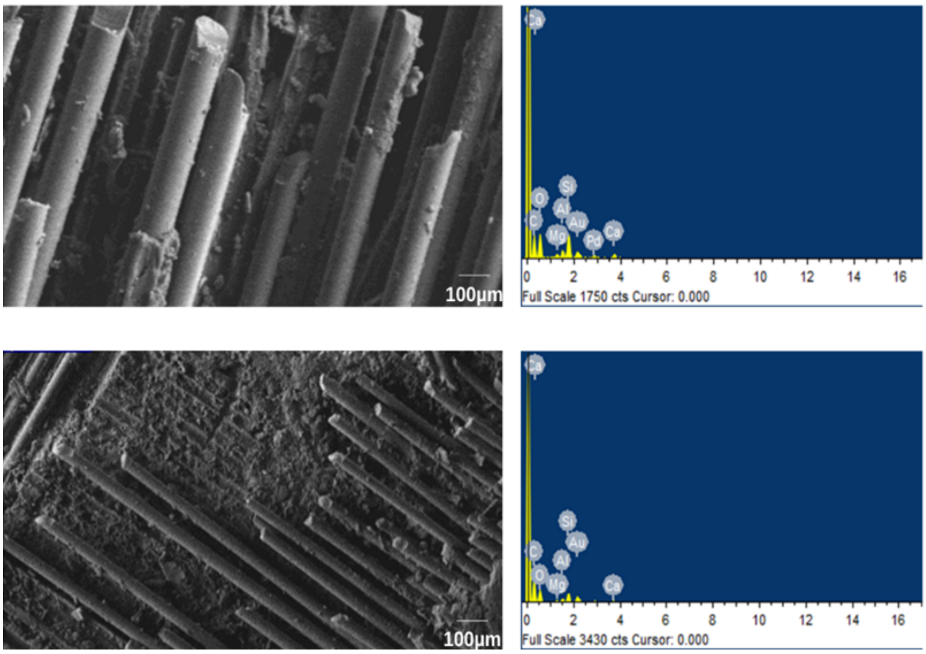

Figure 8: EDX results for 60 days seawater treated 10 mm GFRP at two different positions.

4 Conclusion

This research aimed to determine the influence of seawater on the hardness of 6, 8, and 10 mm thick composites made of glass fiber and isophthalic polyester, a new generation of marine composites. This is one of the first studies to explore the impact of seawater immersion on marine composites' hardness. The investigation revealed the vulnerability of polyester resin to hydrolysis, which resulted in the leaching out of ester species with hydroxyl end groups and debonding at the matrix-fiber interfacial region. The change in the mass of glass fiber reinforced unsaturated polyester composites after immersion in seawater was due to two effects: water absorption and soluble particle extraction. The matrix swelled and eroded due to the absorbed moisture. The matrix erosion due to leaching exposed the glass fibers, which were then damaged by the salts in the artificially prepared seawater. Due to the dissolving of silica and its other primary elements, the exposure of the fibers to seawater caused a reduction in their diameter. The material's loss of functional stability resulted from physical damage and/or irreversible chemical deterioration, as demonstrated by the material's progressively decreasing hardness values after prolonged exposure to seawater. The SEM and EDX analyses of the specimen after immersion in seawater revealed that the fiber/matrix interface had been severely degraded, with the degree of damage increasing with immersion time.

Declaration of Competing Interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Funding Declaration

This research did not receive any grants from governmental, private, or nonprofit funding bodies.

Author Contribution

Ritesh Bhat: Conceptualization, Methodology, Data curation, Writing- Original draft preparation, Visualization, Validation. **Nanjangud Mohan**: Supervision, Validation, Writing- Reviewing and Editing. **Sathyashankara Sharma**: Supervision, Validation, Writing- Reviewing and Editing. **Suma Rao**: Supervision, Validation, Writing- Reviewing and Editing.

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