



<https://doi.org/10.31217/p.40.2.7>

Effects of Long-term Seawater Ageing on the Mechanical Properties of FFRP

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ARTICLE INFO

Original scientific paper

Received 3 November 2025

Accepted 29 December 2025

Key words:

Flax fibers

FFRP

Mechanical properties

Seawater weathering

ABSTRACT

This study examines the effects of natural seawater ageing on mechanical properties, including tensile strength and flexural strength of flax fiber reinforced polymers (FFRP). Specimens were subjected to a natural seawater environment in periods of exposure of one, three, and five months, as well as a reference group that was kept at room temperature. Water absorption and desorption were measured by weighing the specimens before and after exposure. All submerged specimens exhibited varying levels of microorganisms and adhering algae attachment to the material surface, as well as subsequent growth rates. Both tensile and flexural properties were measured and compared to reference specimens, which were kept in a dry room environment. Various levels of ultimate tensile strength (UTS) degradation were observed for different exposure times for tensile testing specimens, as well as a reduction in maximum flexural strain for three-point bending test specimens.

1 Introduction

Reinforcements for marine composite structures are primarily made from glass fibers due to their strength-to-cost and workability characteristics [1]. With the rise of environmental awareness in recent years, a new generation of composite materials has emerged, minimizing harmful impacts, becoming candidates to replace or reduce the use of synthetic fibers in composites across various fields, including the marine sector [2]. The possibility of using flax, bamboo, coconut, and other natural fibers in marine composites is being researched [3,2].

Various researchers have addressed issues of the influence of seawater on the mechanical properties and behaviour of different materials, such as CFRP, GFRP, shipbuilding steel and bio composites [4-7]. Natural seawater was found to affect CFRP tubes negatively, in respect to their load bearing capability and crashworthiness [4], as well as [5] in which a comparison of glass fiber and carbon fiber specimens exposed to natural seawater found glass fiber composites to be more brittle

and to have a bigger change of elastic modulus in comparison to carbon fiber composites after an ageing period of 60 days.

Vukelić et al. [6] investigated the mechanical behavior of welded specimens made of shipbuilding steel for periods of up to 36 months and found that exposure time between three and six months showed a significant influence of specimen mass loss and that sea splash environment generally showed the most negative impact on corrosion rates due a combination of both chemical and mechanical degradation of material. Senthilrajan et al. [7] compared bending strength and the natural frequency was given for polymer composites reinforced with natural constituents such as jute and aloe vera after a 12-day period of exposure to water and seawater.

Fiber-reinforced composites are used in marine applications as lighter alternatives to metals. Compared to traditional materials, they are not highly susceptible to chemical corrosion. They are also a good alternative to wood, which degrades when exposed to biological agents found in seawater [2].

Natural fibers have many benefits when used as a constituent in composite materials, including their organic nature, low cost, low density and biodegradability. Other advantages include negligible health hazards during manufacturing processes as well as safer working conditions for humans compared to conventional composites [8].

Several prominent applications of natural fiber-reinforced composites exist in the marine sector. Some mentioned by Hawary et al. [2] are boat hulls decks and masts, including some yachts, sailing boats as well as racing boats. Haramina et al. suggested that bio-composites such as flax- and hemp-fiber composites could be applied in various marine structures, including ship and boat components, offshore and underwater structures [9].

Despite the mentioned advantages, natural fiber composites come up short in terms of mechanical strength. They are also hydrophilic, which results in a decrease of mechanical properties once exposed to a marine environment [10].

The matrix material type plays a crucial role in determining the composite's performance. Four main types of thermoset matrices, which are epoxy, polyester, vinyl ester, and phenolic, are used in composites manufacturing processes [11, 12].

The long-term interaction between natural fiber-reinforced polymers and the marine environment remains underexplored. However, various studies showed the significance of hydrothermal aging on the mechanical properties of flax fiber reinforced polymers [13,14,15]. The investigation provided valuable insights into how the hydrothermal aging of specimens exposed to distilled water at temperatures of 40 °C and 60 °C affects their mechanical properties. Furthermore, Apolinario's experimental research in which glass fiber - polyester specimens as well as flax fiber polyester specimens were submerged in a bath at a regulated temperature of 30 °C for a period of six months, showed a tensile modulus decrease by 37%, and an increase of ultimate stress 34% relating to flax fiber reinforced specimens [16]. Rothenhäusler's study investigated different flax fiber modifications, and the effect different chemical treatments, such as sodium hydroxide, silane, and siloxane, have on moisture sensitivity, water absorption, and therefore the mechanical properties of prepreg flax fibers and epoxy specimens [17]. Similarly, Prabhu and colleagues investigated how treating flax and bamboo fibers with NaOH affected composite performance. After immersing the treated samples in distilled water for 135 days, they found that alkaline treatment with 5% NaOH enhanced mechanical properties: in bamboo fiber composites, hardness increased by 3.57%, tensile strength by 47%, and flexural strength by 7.36%, while in flax fiber composites, the corresponding improvements were 2.43%, 20.72%, and 13.85%. These results align with the trends observed by Rothenhäusler. Furthermore, the study demonstrated that specimens

treated with sodium hydroxide maintained greater tensile strength after prolonged water exposure than untreated dry specimens [17,18].

Alkali treatment involves fibrillation of bundles into small groups while removing any impurities such as hemicellulose, lignin, and waxes [19]. By removing the impurities, alkali treatment generates a rough surface on the fibers. Additionally, due to the reduction of fiber diameter, an improvement in aspect ratio can result in better adhesion between fiber and polymer matrix [9].

Xu et al. [20] investigated flexural and tensile properties of flax fiber reinforced polymers by employing an accelerated weathering environment that replicated outdoor conditions, including UV radiation, condensation, and water spray, in which specimens of different thicknesses (2 and 4 mm) were exposed up to 1500 hours. Results of their investigation showed a decline in tensile strength of 17% and 38% respectively as well as a decline in flexural strength of 24% and 52%, respectively.

Wang and Petru [21] investigated the effects of different surface treatments (including alkalization, silanization, acetylation, and alkali-silanization) on the dynamic mechanical behavior of FFRP under hygrothermal ageing. After 16 days of hygrothermal ageing in distilled water at 60 °C, the results showed that acetylation-treated FFRP exhibited excellent damping performance, as well as the best water intake performance.

Existing research has largely focused on natural fiber reinforced polymers such as jute, hemp or sisal [22,23]. Additionally, the studies explored short-term exposure and material exposure to sweet water as well as distilled water [2,24,25]. To the best of the author's knowledge, no prior work has focused on the effects of natural seawater ageing on the mechanical properties of flax fiber reinforced polymers. This study aims to fill this gap by investigating the effects of long-term fiber-reinforced polymer composites. The results provide insight into the durability of these bio-composites under real-life marine environment conditions, which has not been comprehensively addressed in the existing literature. Presented findings relevant for the development of sustainable composite materials in marine and coastal applications, where environmental exposure and long-term performance are critical considerations.

Previous research has begun to address composite materials in maritime contexts. Recently, Načinović et al. [4] studied the crashworthiness of carbon/epoxy composite tubes before and after exposing them to natural seawater for 30 days. In 2025, Kopic & Mihaljec reviewed nearly 1,000 studies on the environmental ageing of structural materials used in the maritime sector and emphasized a research gap, noting that the long-term natural marine environment ageing of modern composite materials remained underexplored in shipbuilding and marine engineering [26].

2 Materials and Methods

In this study, commercially available 300 g flax fiber twill, 50 g flax fiber unidirectional, and 150 kg/m³ end-grain balsa core, as well as IB2 Bio Epoxy resin, were used. Two types of specimens were the first of which was a 2 mm thick specimen made for tensile testing, which was made with two layers of flax fiber twill and two layers of unidirectional flax fiber in between them. The second type of test specimen was a 7 mm thick specimen made with two layers of flax fiber twill and a balsa core sandwich in between. The chosen method of production was vacuum infusion, before which the materials were dehumidified for two hours at 80 °C in an OV301 Composites curing oven.

The epoxy resin chosen was IB2 Epoxy Infusion Bio Resin with approximately 38% resin plant-derived content [27]. Overall, a total of 40 specimens were manufactured using the vacuum infusion process. Twenty for tensile testing and twenty for three-point bending testing. In both cases, they were divided into four groups of five specimens each: a dry reference group, which was kept at room temperature, and groups submerged in seawater for one, three, and five months, respectively. Specimens were cut out with an AccuStream waterjet cutter at the operational speed of 200 mm/min and an operating pressure of 3800 bar, after which the specimens were placed in a metal cage kept at a depth of three meters for a period of one, three, and five months, respectively. All of the specimens that were exposed to natural seawater (30 specimens) were submerged at once and kept at the sea bottom near the port of Torpedo in Rijeka, Croatia. Specimens were brought to the surface only to be taken out and for periodical visual checkups (every three weeks).

Figure 2 shows the sea temperature variations during the time of natural seawater exposure. The data was



Figure 1 Material dehumidification in the curing oven

collected via DHMZ (State Hydrometeorological Institute). Each data point represents the natural seawater temperature, which was taken every Monday at 8 AM at a depth of one meter in the period from April 7th to September 8th near the port of Torpedo.

Table 1 shows the mechanical properties of flax fiber itself, out of which the used flax fiber twill and unidirectional are made, the difference lying in fiber orientation and weave pattern.

Table 2 shows the mechanical properties of balsa core, epoxy resin, and flax fiber itself and two flax fiber reinforcements, which were used in two different configurations: a 2 x 2 twill weave and a unidirectional (UD) arrangement.

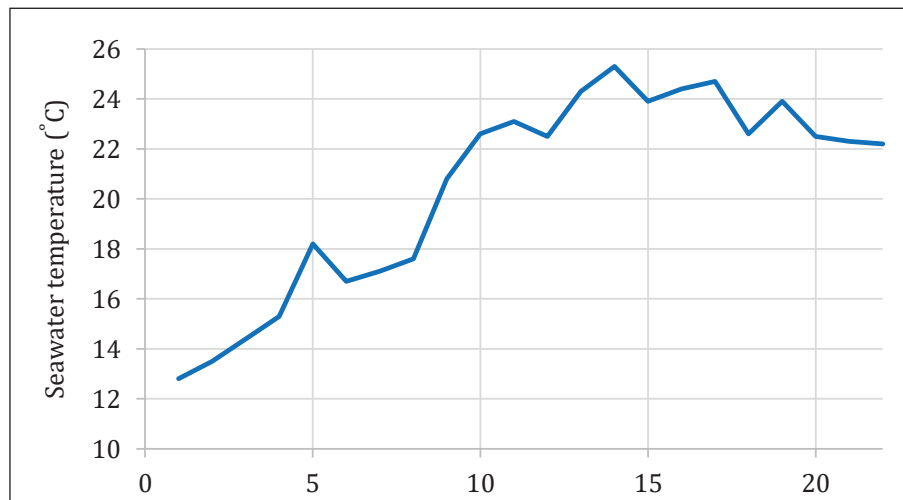


Figure 2 Seawater temperature changes [28]

Table 1 Properties of flax fiber [29]

| Flax fiber | | | | | | |
|----------------|------------------------|-----------------------|-------------------------|-------------------------|---------------------------|-------------------------|
| Density (g/cc) | Tensile strength (MPa) | Elastic modulus (GPa) | Specific strength (s/g) | Specific modulus (" /g) | Elongation at failure (%) | Moisture absorption (%) |
| 1.4 | 800-1500 | 60-80 | 571-1071 | 43-57 | 2.7-3.2 | 8-12 |

Table 2 Properties of used materials [26,30]

| | Flax Fiber Twill | Flax Fiber UD | Balsa core | IB2 Epoxy Resin |
|------------------|----------------------|---------------------|-----------------------|------------------------|
| Aerial weight | 300 g/m ² | 50 g/m ² | N/A | N/A |
| Density | N/A | N/A | 150 kg/m ³ | 1160 kg/m ³ |
| Tensile strength | N/A | 214 ±13 MPa | 1.1 MPa | 68.0 MPa |

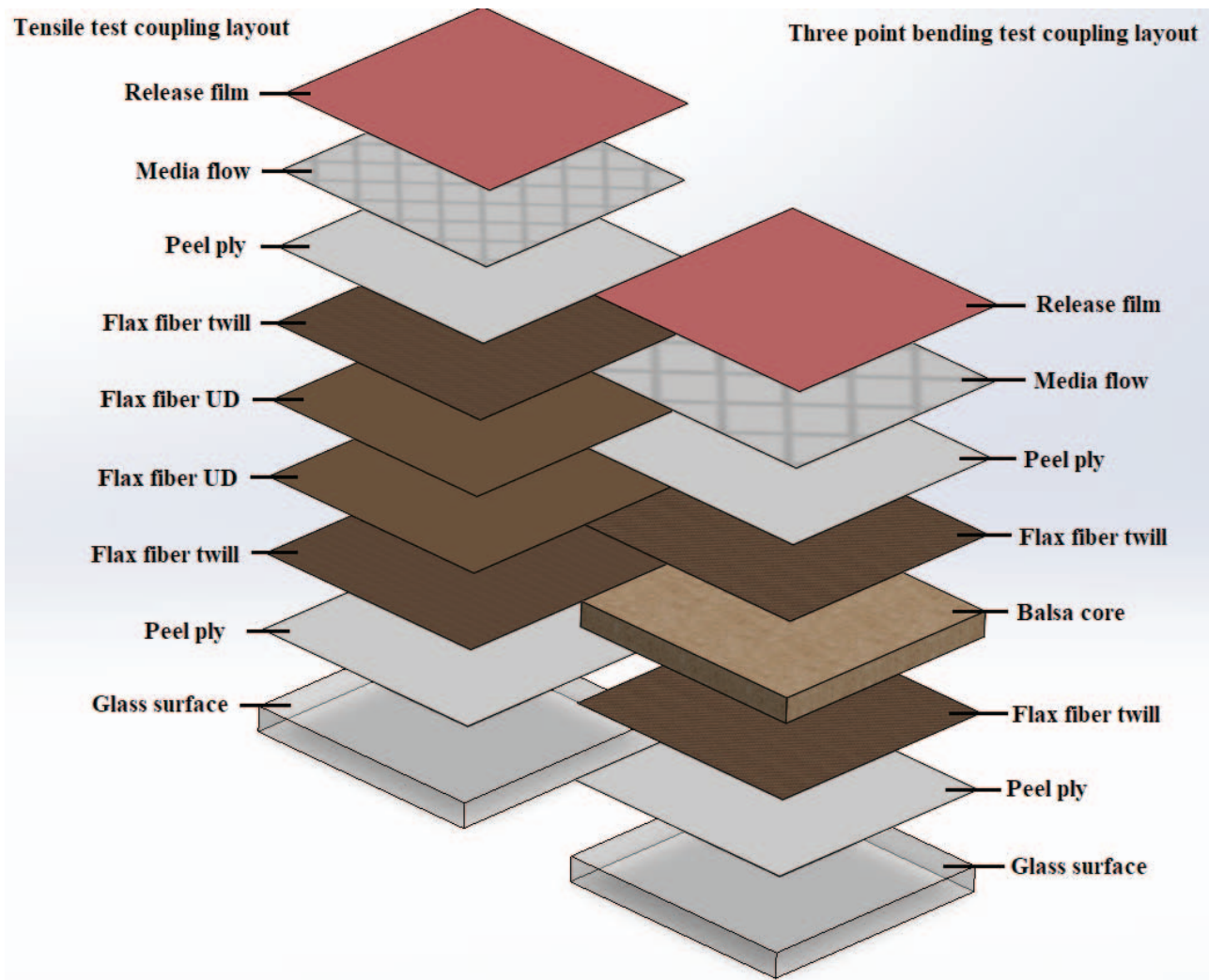


Figure 3 Laminates layout

Figure 3 shows the laminate layout plan for each type of specimen as well as the testing setup in which specimens were held.

The standard vacuum infusion manufacturing process was applied, in which the release agent was first applied to the glass surface, after which the material

was laid out. Materials used include peel-ply (Nylon 66) fabric, which was used to create a rough finish and soak up excess resin. Over the laid-out material, a release film bag was placed to seal the mold from ambient air, allowing a vacuum to be achieved within the bag.



Figure 4 Vacuum infusion process

Exposure times of one, three, and five months were chosen to compare the degradation of the composite's mechanical properties. Specimens that were submerged for three and five months were weighed before and after exposure.



Figure 5 Submersion set-up

After being submerged, they were weighed twice, once one hour after being removed from natural seawater and once 24 hours after that.

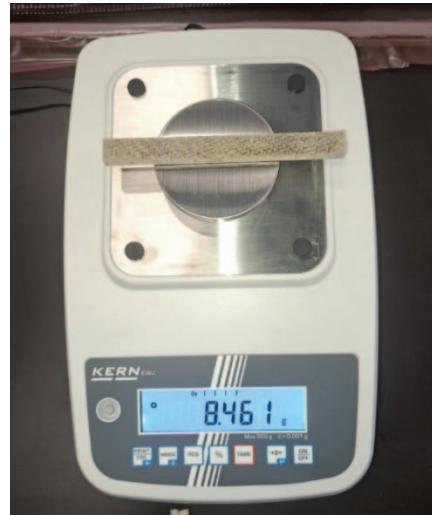


Figure 6 Reference group bending test specimen weight in.

Weighing was performed using the KERN EWJ 600-3 precision scale, which has a linearity of 0.001 grams. Both tensile tests and bending tests were carried out to observe the failure patterns and mechanical properties of FFRP after prolonged exposure to natural seawater in a maritime environment.

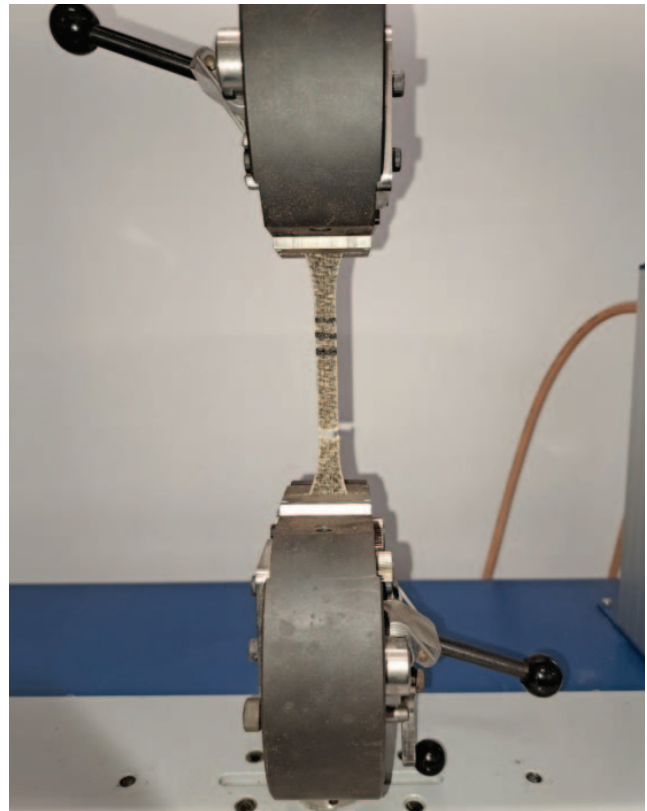


Figure 7 Fractured specimen after tensile testing.

Tensile tests were carried out on the Hegewald & Peschke Inspekt 20-1 testing machine with a loading capacity of 20kN in the scientific laboratory of the Maritime Faculty in Rijeka with the crosshead speed of 3 mm/min. Overall, 20 specimens were tested for tensile strength, five of which were kept at room temperature and considered the reference group, and five were submerged for one, three, and five months, respectively.

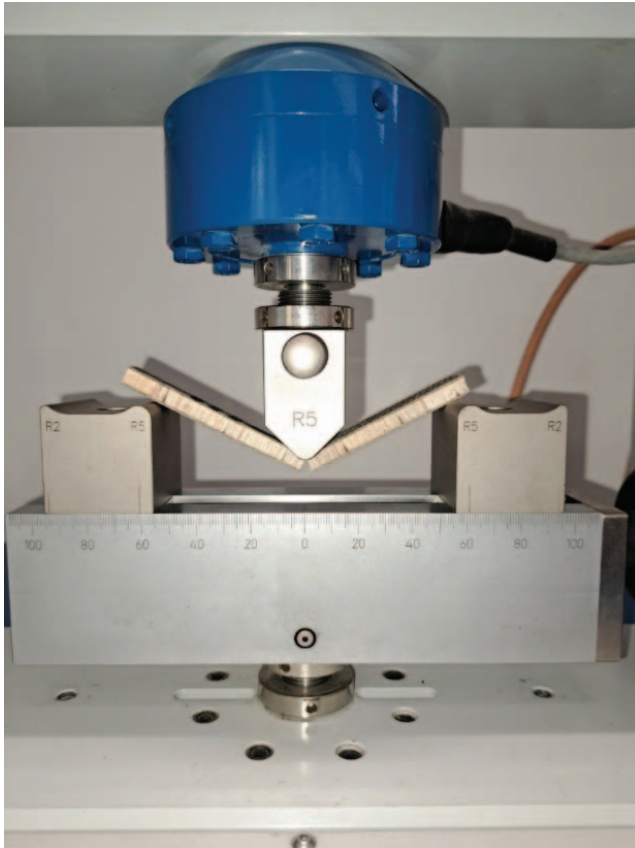


Figure 8 Fractured specimen after three point bending testing.

Three-point bending tests were conducted on a Hegewald & Peschke Inspekt 20-1 testing machine, with a loading capacity of 20 kN, in the laboratory of the Faculty of Maritime Studies, Rijeka, using a crosshead speed of 10 mm/min. Overall, 20 specimens were tested for their bending properties, with five specimens kept at room temperature as reference points and five specimens exposed to natural seawater for one, three, and five months, respectively.

3 Results

3.1 Mass measurements

All specimens that were exposed to natural seawater saw some form of bio growth in the form of algae and marine organisms. A noticeable color change was noted between the reference group and the group, which was submerged for five months, the latter having a darker tone. A slighter tone change is noted in between every group, in which each one being exposed for longer ended up having a slightly darker tone.



Figure 9 Close-up of bio growth accumulation.

Presented in Figure 10 are the recorded mass and mass gain and loss due to water absorption and desorption for both the tensile test group and the bending test group.

In Figure 10, a) and b), the Average mass gain is shown for 3-month and 5-month exposure groups for both tensile test specimens and three-point bending test specimens.

Blue area named Weight in #1 represents the weight taken before any exposure. The orange area named Weight in #2 represents the weight taken one hour after taking specimens out of seawater. The gray area named Weight in #3 represents the weight taken one day after taking specimens out of seawater. Before the second weight in, any leftover sand and loose bio growth was cleaned with a damp towel.

On average, the three-point bending test group, which was exposed for three months, saw a mass increase of 91% and a subsequent mass decrease of 12.85% the day after. The five-month exposure group specimens, on average, showed a mass increase of 99.5% and a subsequent mass decrease of 12.8% the day after. The tensile test group didn't show such high mass variation; on average the three-month exposure group showed a mass increase of 13.5% and a subsequent mass loss due to water loss of 7.4%. The five-month exposure group specimens showed a mass

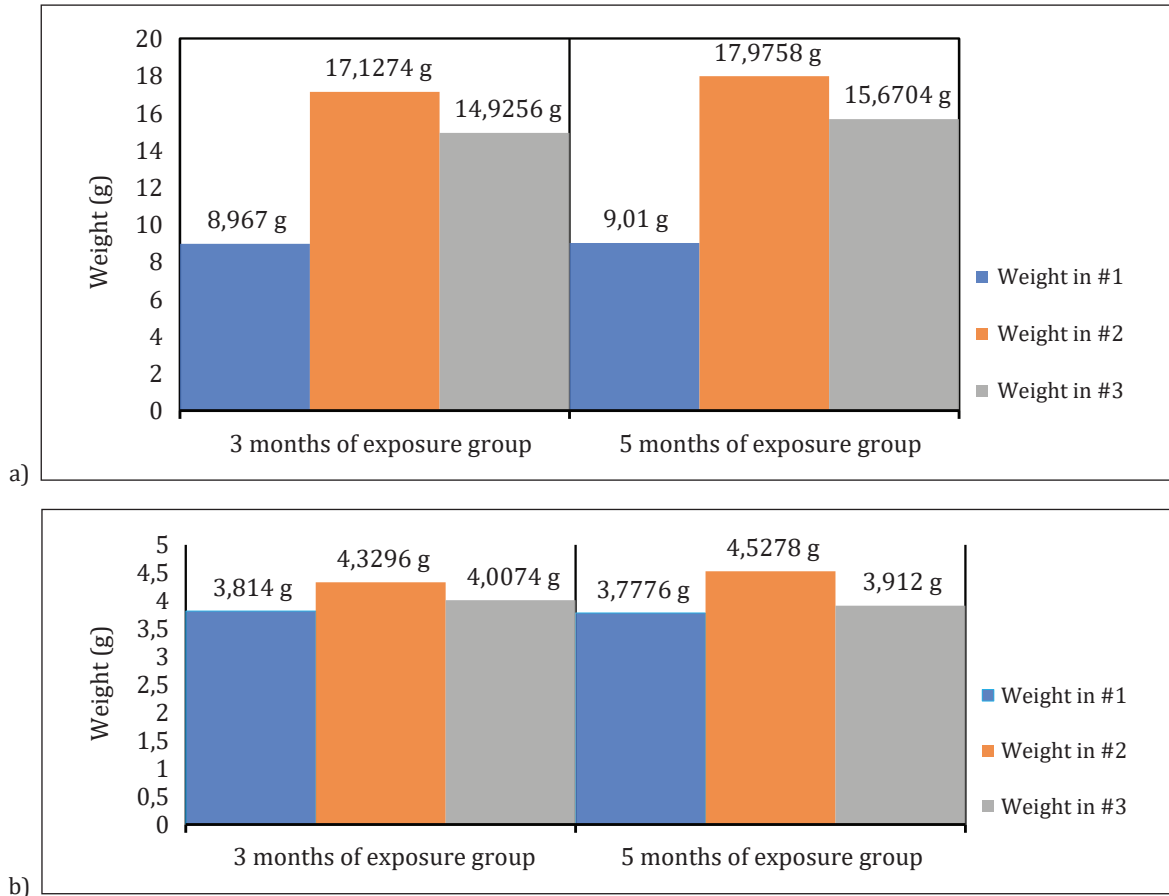


Figure 10 Specimen mass gain chart a) three-point bending test group, b) tensile test group

increase of 19.8% on average and a subsequent mass decrease of 13.6%.

Figure 11 presents the normalized weight measurements of the specimens at three measurement time points. For each specimen, the initial (pre-immersion)

weight was defined as 100%, and subsequent measurements are expressed as percentages relative to this value.

At the time of testing, bending test groups that were exposed for 3 and 5 months exhibited average mass increases of 58.4%, 66.4% and 73.9%, respectively,

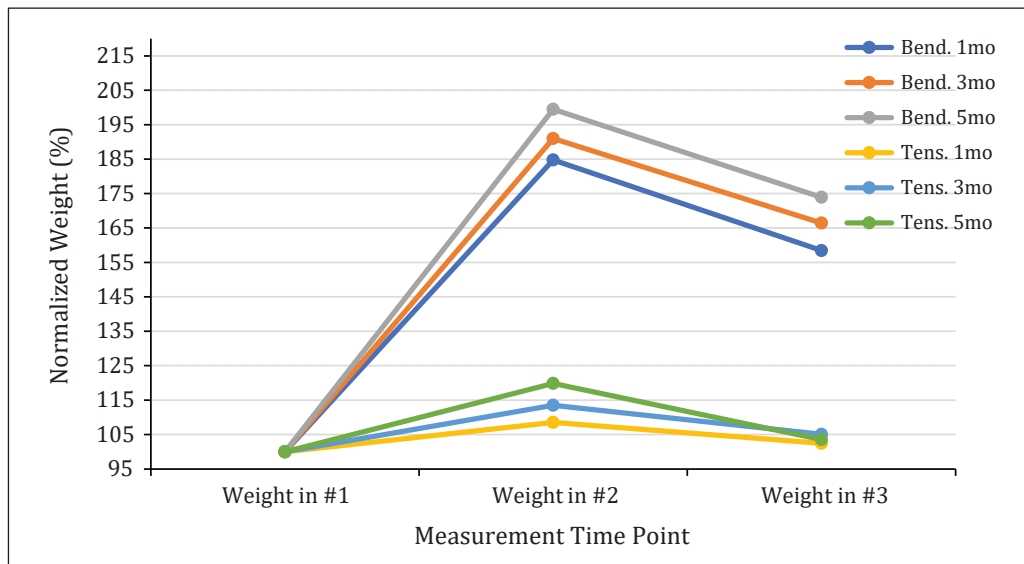


Figure 11 Normalized weight of different specimen groups

whereas the tensile test groups showed much smaller increases of 2.4%, 5% and 3.6% for groups exposed for periods of one, three, and five months.

Tensile test results are presented in two forms of diagrams. The first is a column graph that compares average UTS and strain values. The second one is a stress-strain diagram in which stress represents a value calculated by a testing machine. Similarly, bending tests were represented by two diagrams, the first one being a column graph in which average maximal loading strength and average displacement values are compared. The second one is a load-displacement curve in which load-displacement curves are compared between each specimen group. Load represents the force value provided by the machine while displacement represents the physical position change recorded by the machine.

3.2 Tensile properties and stress-strain curves

Diagram shown in Figure 12 shows average UTS and strain values of FFRP specimens, which were submerged for different durations.

Reference groups had an average UTS of 133 MPa, and a fall of 9.58% for UTS is noted for the one-month group compared to the reference group. Further exposure showed a fall of 31.63% for UTS in contrast to the reference group. The group of specimens that were submerged in natural seawater for the longest had the lowest UTS of 73.364 MPa, as expected, and showed the biggest fall in UTS compared to the dry reference group. When it comes to strain the reference group had an average strain of 3.74% while the group which was submerged for one month saw an increase in average strain of 28.34%, the group which was submerged for three

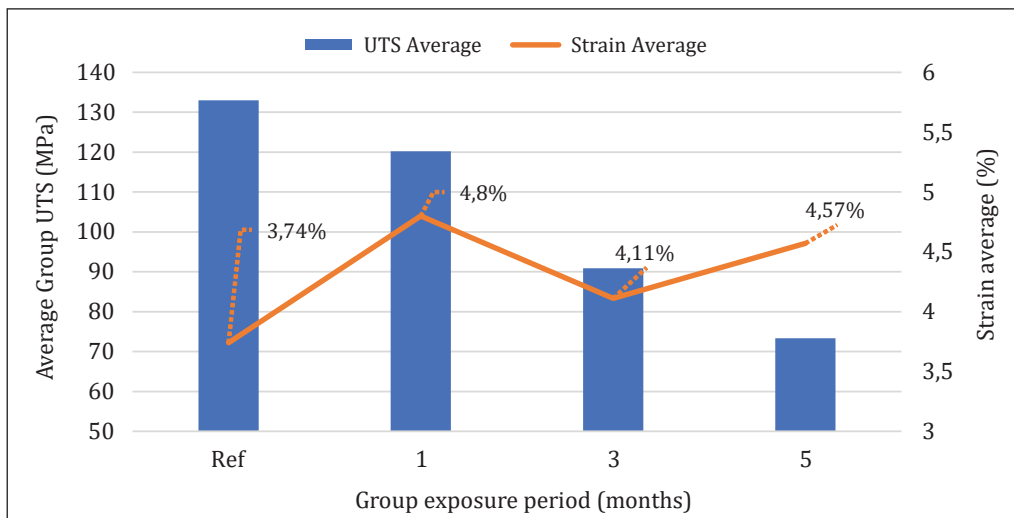


Figure 12 Comparison of average group UTS and Strain values.

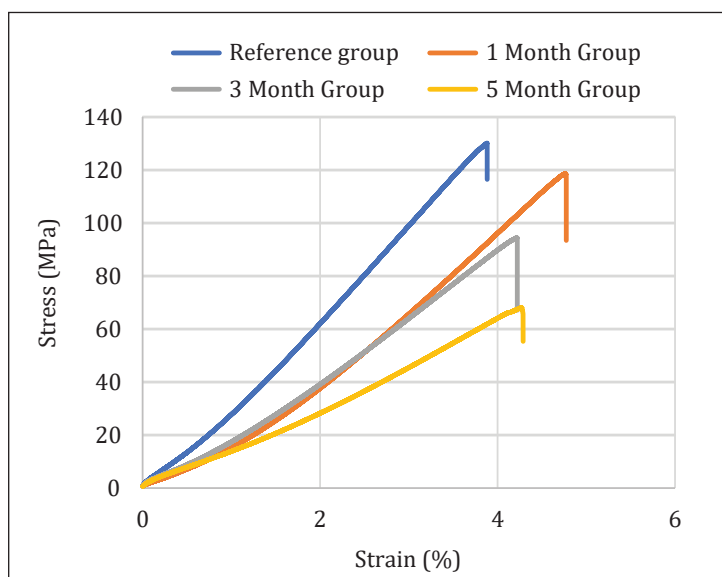


Figure 13 Comparison of average group stress-strain curves

months saw a decrease in average strain value compared to the one month group by 14.375%. The longest exposed group had an average strain of 4.57%.

Comparison of average Stress-Strain curves shows significant degradation of UTS for each of the ageing groups of specimens. Likewise, it shows overall improvement in levels of strain for all three groups, which were submerged. The one-month group showed the highest levels of strain improvement. The last two groups showed a decrease in strain values but still an overall improvement compared to the reference group. Tested specimens showed linear elastic behavior up until failure with little to no plastic deformation, which indicates that the material failed abruptly. The shown

stress-strain curve comparison for each group represents hand-selected samples that were visually identified as the most representative of the group's behavior.

3.3 Bending properties and load-displacement curves

Diagram shown in Figure 14 shows a comparison of different group maximal load values with a comparison of average group displacement values.

The reference group had a load average of 186.34 N while the 1-month exposed group showed a decline in loading stress of 13.65%. The three-month exposure group showed further decline, resulting in a lower aver-

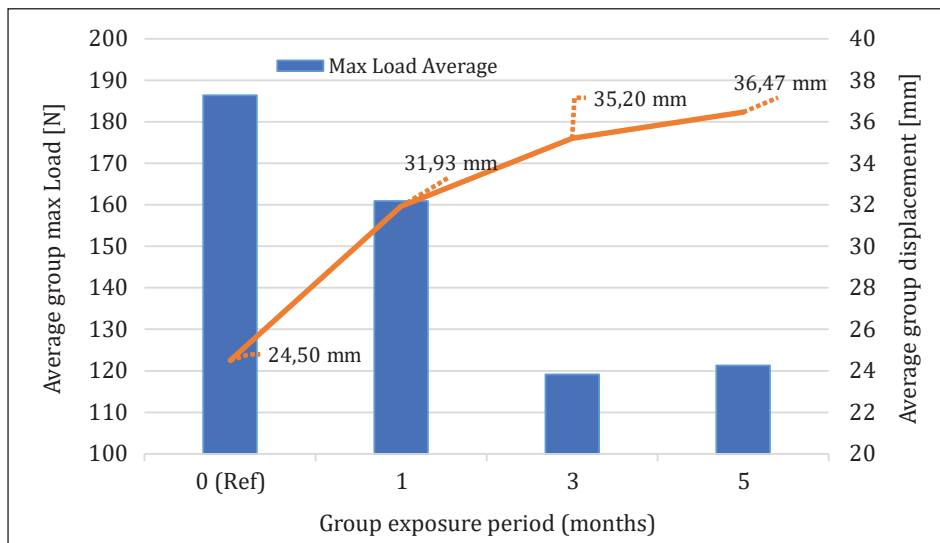


Figure 14 Comparison of average maximal group Loading stress and Displacement values

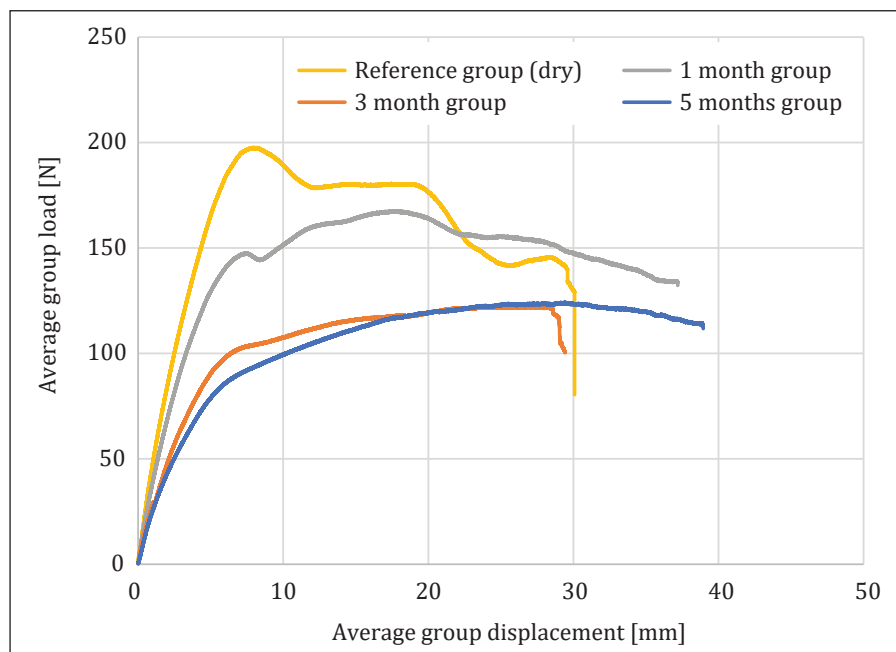


Figure 15 Comparison of average Load-Bending curves

age loading stress of 36.6% compared to the reference group. The five-month exposure group showed a slight increase in average loading stress of 121.3 N. Displacement levels were recorded as well; the reference group had an average displacement value of 24.5 mm. The group that was submerged for one month showed a displacement distance increase of 30.32%. The group that was submerged for 3 months showed further increase in displacement distance and had an average displacement that was 43.67% higher compared to the reference group. Lastly, the group that was submerged for 5 months exhibited further increase in displacement levels at 36.47 mm.

A comparison of the flexural properties of FFRP specimens that were submerged for different time periods is presented in Figure 15. A degradation of loading capacity is noted for specimen groups that were submerged for longer periods. The Load-Bending curves shown for each group (reference group, one month, three months, and five months natural seawater immersion) represent hand-picked specimens that were visually identified as most representative of the group's overall behavior.

4 Discussion and conclusions

The research presented here aims to improve knowledge and provide better data on the effects of seawater ageing on tensile and bending properties of flax fiber reinforced polymers. Tensile test specimens, which were submerged for five months, showed the lowest UTS value at 73.36 MPa. In comparison to the reference group, which had a UTS of 132.96 MPa, the five-month group showed a 44.8% drop in UTS value. Bending test specimens, which were submerged for three months, showed the lowest Loading force record at 119.1 N compared to the reference group, which had an average loading force of 186.3 N, resulting in a drop of 34.9%. It is noted that the five-month group had a slight rise in the loading force of 1.8%

The reduction in mechanical properties observed in this study is consistent with previous investigations on the hydrothermal ageing of flax fiber composites. Kollia et al. [13] reported a 15–30% decrease in tensile strength and up to 35% reduction in modulus for flax/bio-based epoxy composites after 180 days in distilled water at elevated temperatures, attributing the losses to fiber/matrix interface degradation. Similarly, Apolinario Testoni et al. [16] found a 37% drop in tensile modulus for flax/polyester specimens after six months of water immersion at 30 °C, accompanied by a partial recovery after drying, suggesting reversible plasticization. Xu et al. [21] also reported notable losses under UV-water cyclic ageing up to 24% and 52% in the flexural strength and modulus and up to 17% and 38% in the tensile strength and modulus, which supports the notion that

environmental factors beyond moisture alone, such as UV radiation and oxidative effects, further compromise flax-reinforced composites.

Future research should focus on developing a comprehensive numerical model for structural analysis, which would enable more accurate predictions of material degradation and structural performance over time. In this study, a five-month exposure period was used as an initial step toward understanding the effects of the real marine environment on flax fiber-reinforced polymers. However, since vessels and offshore structures typically remain in service for much longer periods, extended exposure tests are essential. Longer-term experiments would provide more realistic insights into the progressive degradation mechanisms and enable better calibration of predictive models for long-term structural integrity assessment.

Acknowledgments: This work has been supported by the University of Rijeka within the project UNIRI-ZIP-2103-2-22 “Marine Composite Material Recycling and Re-use (M-COMARE)”.

Author Contributions: Research, A.B; Writing, A.B; Data collection, A.B; Data curation, A.B; Supervision, G.V; Validation, G.V; Final approval, G.V.

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