

The Influence of Seawater Immersion on the Mechanical Properties and Structure of Carbon Fiber/Polyester-PEG Composite Materials

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ABSTRACT

Carbon fiber, polyester, PEG, immersion test, mechanical properties

Structural batteries are a crucial solution in dealing with issues related to vehicle electrification to achieve energy sustainability in the future. However, the development of structural battery technology for electric-powered ships is still very limited. In particular, there are limitations in evaluating the mechanical performance of carbon fiber/polyester-PEG based composite materials for structural battery applications in electrically powered vessels. The main focus of this research is to fabricate carbon fiber/polyester-PEG to further observe the performance of its mechanical properties and structure when interacting with seawater. The value of the addition of polyethylene glycol (PEG) to the fabrication carried out was varied by 0%, 10%, and 25%. The results show that addition of 10% PEG produces a composite with the highest tensile strength, increasing the toughness of the polyester material. However, all samples experienced a decrease in tensile strength after immersion due to decreased bond performance between the fibers and seawater. The addition of 25% PEG resulted in a significant decrease in mechanical properties because high levels of PEG content made the sample easily brittle and destroyed when interacting with seawater. This research can provide initial studies on the development of carbon fiber/polyester-PEG composite materials that can be used as structural battery building materials for future electric boat applications.

1. INTRODUCTION

Despite having experienced the climate change since the beginning stage, the human activity has significantly severe the effect by the unstoppable increase of CO₂ emissions during the last century.[1] Several countries (e.g., China, the United States, the EU27, India, Russia, Japan, Germany, Canada, Iran, and Indonesia) were nominated as the world's largest CO₂ emitters in 2020.[2-6] In such the report, transportation sector contributes as the largest donator especially for international aviation and maritime sector.[4] In maritime sector, the International Maritime Organization (IMO) has stated to decrease the greenhouse gas emissions as high as 50% by 2050. Recently, the government of Indonesia has also increased target for the greenhouse emissions reduction through the Enhanced Nationally Determined Contribution (ENDC) pursuing the vision for net-zero emission by 2060. Transition of energy become as unavoidable demand to reduce the severe effect of the climate changes. For this purpose, electrification of transport sector can be effective ways to restrict emissions of CO₂. Electrification of the vehicle such as aircraft, car, or even sea vessels are highly needed to provide the better sustainability of the future world.

The structural batteries, multifunctional device that can store the energy while providing mechanical properties, are attracting many interests to solve the electrification problem in transportation sector.[7] In particular, the concept that was first developed by US Army Research Laboratory (ARL) in 2007, enables to construct the architectures of transportation while in the same time acts as energy storage and loadbearing application.[8] Previous work claimed that application of structural battery can reduce the

total weight up to 30%, optimizing the performance and efficiency of the vehicle.[9] During the last decade, several intensive efforts had been conducted to adjust such the technology especially for the aircraft and road vehicle application.[10-12] On the other hand, development of structural battery as the potential technology for switching the propulsion machine to battery-powered vessels still be limited. Whereas, it is known that utilization of the fibre reinforced polymer (FRP) as the most developed materials for structural battery in sea vessel application will face serious problems caused by marine environment, which can degrade its physical and chemical properties.[13]

The utilization of composite materials for marine structures application had been extensively studied since the 1980's. It is well known that composite materials (especially FRP) become as the main consideration in the nautical industry, especially to construct Hulls, Ships, and Submarines.[14] This fact can be attributed to the advantages of composite technology such as its lightweight, high strength and stiffness, the possibility to manufacture it in automatization, and relatively fast fabrication process. Aramid, carbon, and glass are the most commonly used fibre type for marine application not only due to its promising mechanical properties but also its corrosion resistance in marine environment. In addition, several resins had been also considered for the application in marine environment such as vinyl ester, polyester, epoxy, and phenolic.

The development of structural battery as the 'mass-less' energy storage drastically increases in the recent years. In general, structural battery was commonly developed using carbon fiber (CF)/polymer-based composite (CFRP) attributed to its advantages including high specific strength and stiffness, high fatigue resistance, and lightweight.[15-17] Moreover, the microstructure of CF is similar with graphite material hence potential for battery anode application.[18] The polymer matrix based on Polyester is considered to be suitable for the application of structural battery in marine environment as the ability to reduce the possibility for chemical corrosion while providing high mechanical and stability.[19] However, the capability of ionic conductivity cannot be achieved such by the mentioned polymer. In this case, blending the polymer matrix with ionic-conducting component to produce a solid polymer electrolyte (SPE) that can guarantee both the ion conduction and load transfer functions was widely developed.[20-23] Modification of the polymer matrix through the addition of conductive component (e.g., ionic liquids (ILs), polyethylene glycol (PEG), polyethylene oxide (PEO), etc.) could be an effective way to develop advance SPE. However, it was known that the improvement of ionic conductivity could result in the decrease of mechanical properties, and vice versa.

Previously, vinyl ester matrix had been successfully modified to increase the concentration of ions such by incorporating polyethylene glycol (PEG) as the conductive component.[21, 24-27] The cross-linked vinyl ester networks could provide the structural performance, while the amorphous PEG delivered the long-range ion transport. However, it was known that vinyl ester is costly and need a careful preparation during the production or treatment.[14] Polyester is another type of resin that also widely used as matrix in boat structure. Polyester is more affordable than vinyl ester and epoxy resin while still providing comparable performance to both resins. To the best of our knowledge, there is a limit work to evaluate the mechanical performance of multifunctional composite material based on carbon fibre/polyester-PEG, especially for marine environment application.

The objective of this research is to investigate the potential of carbon fibre/polyester-PEG as the composite electrode in structural battery for future electric sea vessels application. The fabricated FRP for structural battery technology in this research contributes to the ongoing pursuit for the potential replacement of Li-ion batteries as the most widely used energy sources in electric vehicle. This development will be a part for supporting energy transition in order to reduce the severe effect of climate change caused by the CO₂ emissions.

2. MATERIALS AND METHODS

2.1 Materials

This research utilized key materials including pure carbon fiber 3K 200 gsm Plain Weave (thickness of 0.27 mm), Polyethylene Glycol 400 USP, YUKALAC® 157 BQTN-EX Series resin, and methyl ethyl ketone peroxide (MEKP) catalyst.

2.2 Fabrication Technique

The synthesis process began with preparing the necessary equipment and materials, such as roller brushes, regular brushes, spatulas, beakers, stirring rods, measuring cups, mold boards, YUKALAC® 157 BQTN-EX Series resin, and methyl ethyl ketone peroxide (MEKP) catalyst. First, carbon fiber sheets with thickness of 0.27 mm were cut into 25 cm x 12.5 cm pieces, totaling 9 sheets. A resin-catalyst mixture was prepared by combining 300 ml of YUKALAC® 157 BQTN resin with 1% catalyst. The target thickness for the fabricated composite material was 2.5 mm to meet ASTM D3039 standards for tensile testing of symmetric fiber composites.

The fabrication process began by evenly coating the mold board with wax or a release agent. The resin-catalyst mixture was then slowly applied to the mold surface. The pre-cut carbon fiber sheets were placed onto the resin-coated mold and subsequently covered with additional resin to ensure even coating. A roller brush and spatula were used to evenly distribute and impregnate the resin into the fiber sheets. This layering process was repeated nine times to achieve a composite thickness of 2.5 mm. After layering, the sample underwent a curing process at room temperature for approximately 24 hours. Once fully cured and hardened, the sample was cut into four pieces of 25 cm x 2.5 cm, referred to as PEG 0% samples.

Subsequent fabrication involved creating samples with Polyethylene Glycol 400 (PEG) incorporated into the polyester resin. The process followed the same steps as for PEG 0% samples, with the difference being the inclusion of PEG in the polyester resin at concentrations of 10% and 25%. PEG was mixed into the polyester resin using a stirrer for 30 minutes. The resulting products were designated as PEG 10% and PEG 25% samples.

2.3 Material Characterizations

Fourier-transformed Infra-red Spectroscopy (Bruker Alpha II) were used to investigate the functional groups present on surface of composite samples. Tensile strength test was performed by using 250 N Instron Universal Testing Machine (5985 Series) to determine mechanical properties of the samples. Tensile test sample was prepared according to ASTM D3039 standard. For conducting the testing, specimen was prepared with a dimension of 250 mm in length, 25 mm in width, and 2.5 mm in thickness (tolerance of the dimension was $\pm 1-4\%$). The composite samples were glued onto mounting tab (paper) by using epoxy resin. During the test setup, the specimen is securely mounted in a universal testing machine (UTM), and strain measurement devices, such as extensometers or strain gauges, are attached to measure elongation accurately. The test is conducted under controlled environmental conditions to ensure consistency. The tensile test was then conducted at a strain rate of 5 mm min⁻¹. Each sample, prepared with PEG concentrations of 0%, 10%, and 25%, was duplicated, resulting in a total of six samples for measurement. The fabricated samples were labeled as PEG 0%_1, PEG 0%_2, PEG 10%_1, PEG 10%_2, PEG 25%_1, and PEG 25%_2.

3. RESULT AND DISCUSSION

Figure 1a) shows the fabrication results of the carbon fiber/polyester-PEG composite material using the wet hand layup method. In this study, carbon fiber was used as the reinforcing material and polyester/PEG as the adhesive matrix. From visual observations, it can be seen that the 0% PEG sample (Figure 1a, left) produced the driest condition after the curing process in ambient conditions for approximately 24 hours. The observations also showed that the addition of 10% and 25% PEG (Figure 1a, middle and right) made the surface of the resulting composite material wetter for the same curing duration of 24 hours. The wetness level of the composite material surface increased with the addition of more PEG into the resin. In the chemical synthesis mechanism, PEG is strongly absorbed and trapped within the interconnected spatial network structure of the curing polyester resin.[28] However, the excess amount of PEG added will cause some of it to not participate in the curing process within the sample. As a result, the PEG that does not undergo the curing process will remain in a liquid phase. The fabricated samples were then prepared for tensile testing with dimensions of 25 cm x 2.5 cm x 2.5 mm using tolerance for the dimension of $\pm 1-4\%$ according to ASTM D3039 (Figure 1b).

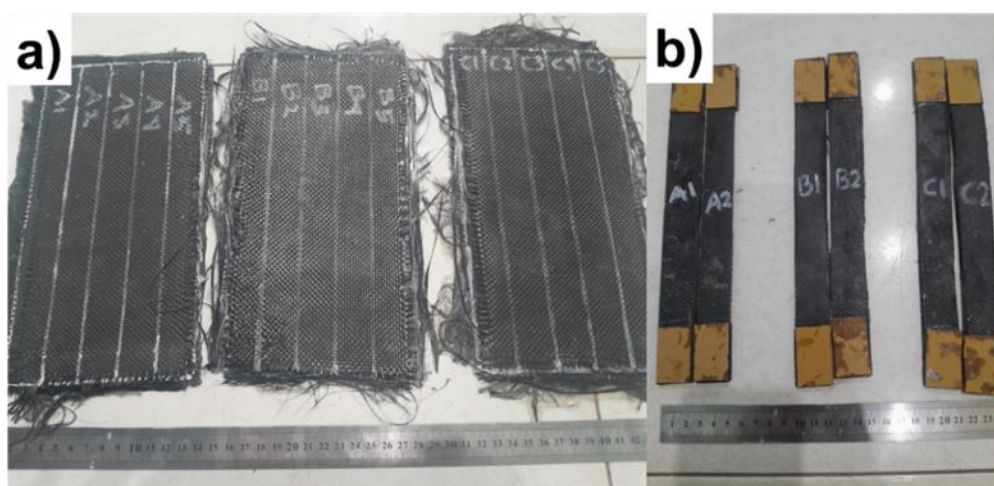


Figure 1. a) Fabricated composite material with the addition of 0% (left), 10% (middle) and 25% (right) PEG. b) Tensile testing samples in accordance with ASTM D3039.

In general, FTIR testing is used to determine the success of the mixing process of PEG and polyester resin in the fabricated composite material. The FTIR transmittance spectra of the 0% PEG, 10% PEG, and 25% PEG samples are shown in Figure 2. As can be seen in the spectrum of the 0% PEG sample, the transmittance valleys at wavenumbers 2931 cm^{-1} and 1070 cm^{-1} are due to the presence of aliphatic hydrocarbon groups (CH stretching) and the unsaturated bonds of the C=C group. Additionally, the aromatic ring at wavenumber 1596 cm^{-1} and the carbonyl group (C=O) at 1735 cm^{-1} are also clearly visible in the resulting FTIR spectra.[29] In the spectra of the 10% and 25% PEG samples, several new absorption valleys appear, such as at wavenumbers 3435 cm^{-1} and 2881 cm^{-1} , which are caused by the stretching vibrations of the O-H and C-H groups.[28] The sharpness of some spectra in the region from 1443 to 1340 cm^{-1} originates from the combination bands of C-O-H and O-C-H deformations. Additionally, the spectra in the region from 1100 to 500 cm^{-1} can be attributed to the vibrational modes of the C-O and C-C groups.[29] The formation of valleys at specific wavenumbers characteristic of both PEG and polyester resin is evidence of the successful mixing process of these two materials.

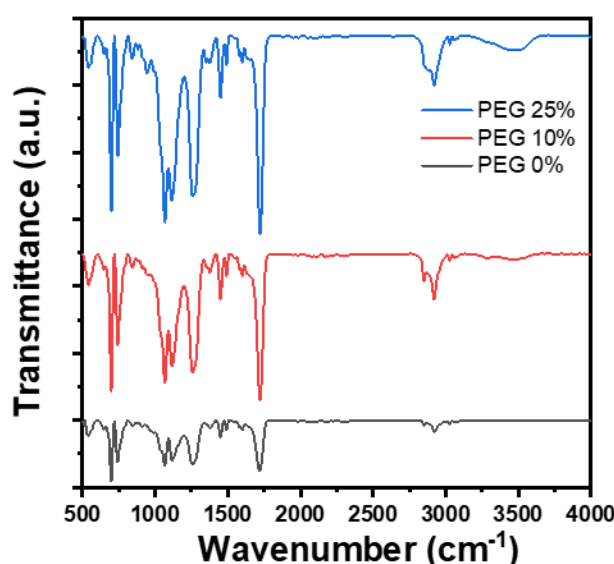


Figure 2. Spectrum of FTIR test results for 0% PEG, 10% PEG and 25% PEG samples.

The tensile strength test of the material was subsequently conducted on all the produced samples using an Ultimate Tensile Machine (UTM). The test results of the samples using the UTM on the fabricated composite material can be seen in Figure 3a. In this measurement, each sample with 0% PEG, 10% PEG, and 25% PEG was replicated (labeled as 1 and 2) to enhance the accuracy of performance evaluation by

averaging the results. The tensile test results for each sample pair (PEG 0%, PEG 10%, and PEG 25%) show variations, likely due to microstructural differences or testing uncertainties. PEG 0% samples exhibit moderate differences, indicating minor inconsistencies in material properties or processing. PEG 10% samples show greater deviation, possibly due to localized structural differences, uneven carbon fiber distribution, or slight specimen preparation discrepancies. PEG 25% samples have the least variation among the modifications. Despite these differences, all variations remain within an acceptable range for the material and testing conditions. Figure 3b clearly shows the comparison of the average value of tensile strength for all the samples fabricated in this study. The tensile strength values for the 0% PEG, 10% PEG, and 25% PEG samples are 265.3 MPa, 274.5 MPa, and 258.2 MPa, respectively.

From this data, it can be seen that the addition of 10% PEG increases the tensile strength of the composite by 9.2 MPa compared to the 0% PEG sample. The addition of PEG as a soft material in this case can enhance the toughness of the unsaturated polyester material, which is inherently very rigid and brittle.[29] Additionally, the 10% PEG addition maintains good adhesion between the fiber and the polymer matrix used. However, the addition of 25% PEG results in a decrease in the material's tensile strength by 16.3 MPa. In this case, the excessive use of PEG can lead to a softer matrix system after the curing process, thereby reducing mechanical performance, particularly in terms of tensile strength.[20]

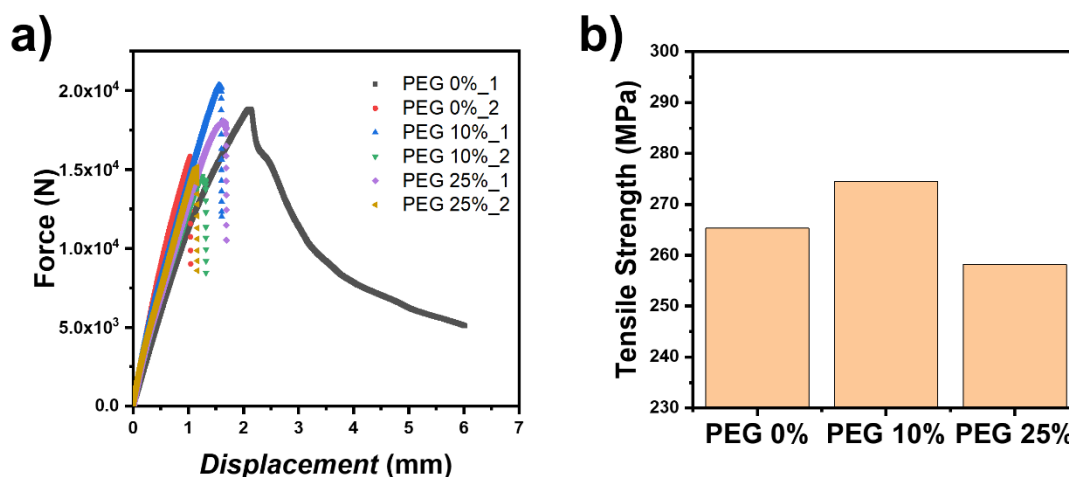


Figure 3. a) Tensile test results curve and b) comparison of material strength values for 0% PEG, 10% PEG and 25% PEG samples without seawater immersion.

The tensile strength test of the material was subsequently conducted on samples that had undergone treatment by immersion in seawater for 14 days. The test results of the samples using the UTM on the composite material that had been treated by immersion in seawater are presented in Figure 4a. Similar to the tensile strength measurements before the immersion test, which showed slight variations likely due to microstructural differences or testing uncertainties, the observed variations remain within an acceptable range for the material and testing conditions. Figure 4b clearly shows the comparison of the average tensile strength values for all the fabricated samples after being immersed in seawater. It can be seen that the tensile strength values for the 0% PEG, 10% PEG, and 25% PEG samples after immersion in seawater are 264.3 MPa, 269.4 MPa, and 237.6 MPa, respectively.

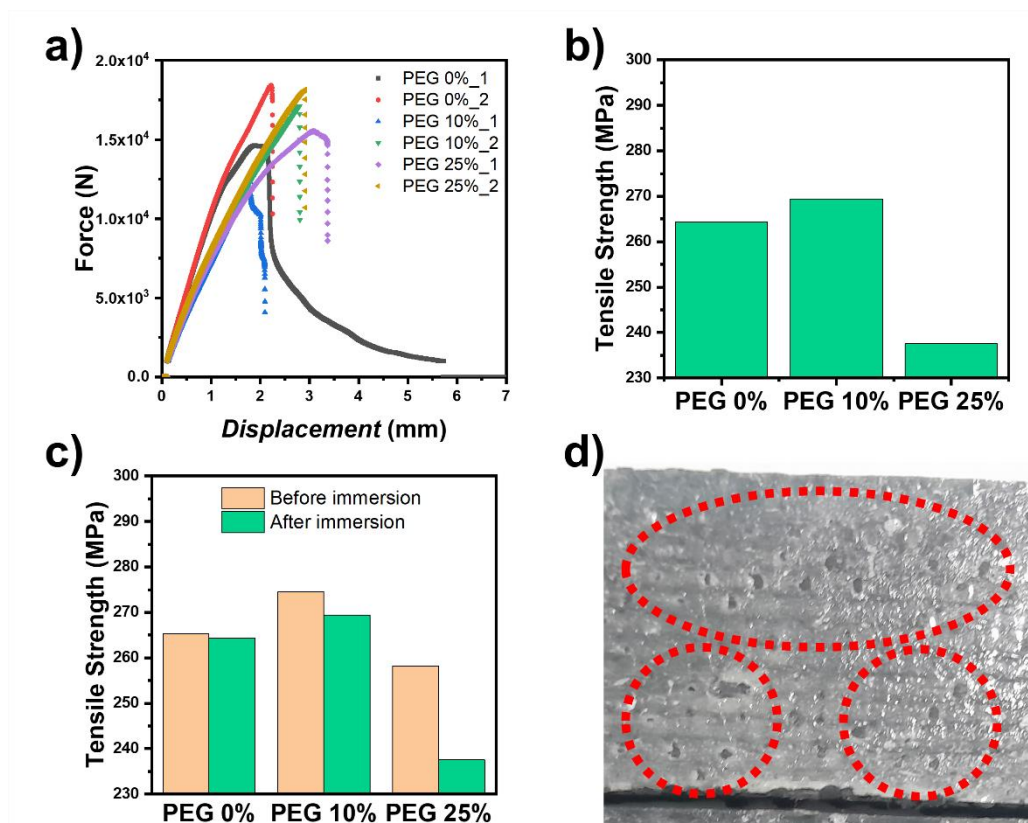


Figure 4. a) Tensile test results curve and b) comparison of material strength values for 0% PEG, 10% PEG and 25% PEG samples after the seawater immersion process. c) Comparison of tensile strength values for 0% PEG, 10% PEG, and 25% PEG samples with and without the seawater immersion process. d) Results of surface observations of the 25% PEG sample after the seawater immersion process.

Figure 4c shows that all samples experienced a decrease in tensile strength after being immersed in seawater. Sequentially, the decreases in tensile strength values for the 0% PEG, 10% PEG, and 25% PEG samples were 0.93 MPa, 5.12 MPa, and 20.63 MPa, respectively. The decrease in tensile strength of the composite material after the immersion process can be attributed to seawater infiltrating and permeating the composite material, subsequently weakening the bond between the fiber and the matrix.[30] It is well known that PEG is highly water-soluble, meaning that when exposed to seawater, it gradually dissolves and leaches out from the polyester matrix.[31] Over time, this leaching process creates microvoids or porosity, which can be visibly identified as white spots on the sample surface. As a result, the diffusion of PEG weakens the adhesion between the carbon fiber reinforcement and the surrounding polyester resin, potentially leading to delamination and mechanical degradation. Consequently, the mechanical properties, specifically tensile strength, decrease. This reduction in tensile strength is more significant in samples with high PEG content, namely the 25% PEG sample. PEG can degrade more severely compared to polyester when interacting with seawater. In this context, samples containing PEG become brittle and easily disintegrate, thereby reducing the mechanical properties of the material.[32] Due to its solubility, white spots resulting from PEG dissolution by seawater will clearly appear on the surface of the sample, creating numerous degradation pits/holes around these areas (Figure 4d). This phenomenon becomes more pronounced with higher PEG content. The degraded PEG then becomes an entry point for seawater into the sample. The more and larger the entry points for seawater into the sample, the more significant the weakening of the fiber-matrix bond, leading to a greater decrease in tensile strength. This is why the 25% PEG sample experienced a drastic reduction in tensile strength compared to the 0% PEG and 10% PEG samples, with an 8% decrease.

Figure 5a shows the FTIR spectrum of the 25% PEG sample under seawater immersion treatment (red graph) and without seawater immersion treatment (black graph). It is clear from the displayed figure that the two samples show significant differences in their spectra, particularly in the characteristic valleys of

PEG. The wavenumbers 3435 cm^{-1} and 2881 cm^{-1} , which arise due to the stretching vibrations of the O-H and C-H groups, exhibit a significant increase in transmittance values after 14 days of seawater immersion. These valleys, which are characteristic of PEG, show an increase in transmittance after the immersion process, and the valley at wavenumber 3435 cm^{-1} is almost unnoticeable.

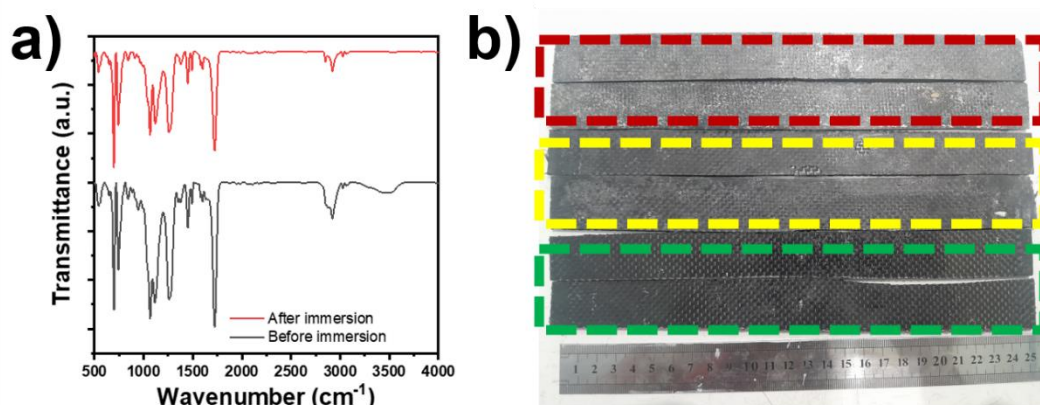


Figure 5. a) Spectrum of FTIR test results for 25% PEG samples with seawater immersion process (red graph) and without seawater immersion process (black graph). b) Surface morphology of the 0% PEG Sample (green dotted line), 10% PEG Sample (yellow dotted line), and 25% PEG Sample (red dotted line) after the seawater immersion process.

The transmittance values from the FTIR results are inversely related to the concentration of functional groups in the sample. High transmittance values indicate that the sample allows most of the infrared radiation to pass through. In this context, the concentration of functional groups at specific wavelengths tends to be low because less energy is absorbed by these functional groups. Based on the obtained spectral data, we can conclude that the PEG in the sample underwent significant degradation after the seawater immersion process. Visually, we can clearly see that the surface of the 25% PEG sample experienced degradation after seawater immersion, marked by the appearance of white holes evenly distributed across the sample surface (Figure 5b). The concentration of this degradation decreases in the 10% PEG sample and is almost non-existent in the 0% PEG sample, which retains the same color and surface condition as before the immersion process. This is additional evidence that the PEG structure in the composite material is significantly affected by interaction with seawater. This is an important note considering that PEG is an additive for the composite material. The degradation of PEG due to seawater immersion requires further investigation so that its potential to improve the conductivity of composite materials can be maintained.

This study serves as a preliminary exploration of the potential use of carbon fiber/polyester-PEG composites as electrode materials for structural batteries in future electric sea vessels. While the impact of PEG addition on the mechanical properties of the composite has been identified in this study, further development and in-depth investigation are necessary to fully understand its feasibility and optimize its performance before transitioning to real-world applications. For future development, it would be valuable to further investigate the long-term durability of the composite materials in marine environments, particularly their ability to maintain optimal performance under various stress conditions. Additionally, potential manufacturing processes, such as Vacuum-Assisted Resin Infusion (VARI), are considered promising fabrication methods for future development, offering advantages in enhancing composite quality, mechanical performance, and scalability for practical applications.

4. CONCLUSION

The fabrication of carbon fiber/polyester-PEG composite materials has been successfully achieved using the wet hand lay-up method, with FTIR testing clearly indicating the characteristics of polyester and PEG in the transmittance spectrum of the fabricated samples. The addition of PEG resulted in samples with 10% and 25% PEG content having surfaces that remained wet after the curing process due to some PEG not hardening and remaining in a liquid phase. Adding 10% PEG yielded the highest tensile strength, as the appropriate amount of PEG can enhance the toughness of the otherwise rigid and brittle unsaturated polyester. However, excessive PEG content reduced tensile strength by making the matrix too soft, thereby decreasing mechanical performance. Immersion in seawater reduced the tensile strength of the composite

materials, as seawater infiltration weakened the fiber-matrix bond, especially in samples with high PEG content that became more fragile and susceptible to degradation. The PEG in the samples experienced significant degradation after seawater immersion, creating voids that resulted in higher transmittance spectra in FTIR testing.

DECLARATION OF COMPETING INTEREST

There are no conflicts to declare.

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