Effect of Seawater Immersion on Mechanical Properties of Glass/Vinylester Composites for Marine Application

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Abstract. Composites are widely used in marine applications due to their lightweight, resistance to corrosion and fatigue, and high specific stiffness and strength. This study used vinylester resin and woven cloth e-glass fiber fabricated by VARI methods. The composites were immersed in natural sea water for 6 months. The weight gain and mechanical properties of composites before and after seawater immersion were evaluated. The failure characterization and spectroscopy analysis of the composites were observed using SEM and FTIR. The results exhibit the degradation of mechanical properties after sea water immersion. The highest degradation occurred in the compressive properties of the composites with a 60% decrease in compressive strength.

INTRODUCTION

Fiber reinforced polymer composites are widely used in marine, aerospace, and construction applications due to their high specific stiffness and strength, lightweight, and resistance to corrosion and fatigue.[1-5] Vinyl ester and polyester are the common matrix used for marine application.[5] Vinylester is used because it is better at avoiding the diffusion of seawater into the composite and still has high cross-linking when the material is immersed in seawater.[5] In addition, vinyl ester resin is very resistant to hydrolysis that can result from immersion in seawater.[6] Study on vinyl ester resin exhibit that this tensile strength reduced by 12% after 9 months immersed in sea water.[7]

One of the fibers that are widely used as composite reinforcement is glass fiber. Study of Garima Mittal et al. exhibits that the tensile strength and modulus of glass/vinyl ester composites reduced by 7% and 4% respectively after sea water immersion.[8] Other study exhibits that the tensile stiffness of glass/polyester composites was reduced 10% after 2 years immersion in sea water.[9] The tensile strength of glass/polyester composites modified cadmium sulfide was decreased by 13.8% after sea water immersion. Study of Garcia et al. exhibits the reduction of tensile strength by 23.81% for glass/epoxy composites after 90 days immersion in sea water.[10] The tensile strength of quasi isotropic e-glass/epoxy composites was reduced by 27% after artificial sea water exposed and the composites failure after sea water immersion caused by failure of fiber and crack of fiber/matrix interface.[11] Other study exhibits 47% reduction in tensile strength of e-glass/epoxy composites after sea water (65 ^{0}C) immersion for 60 to 90 months.[12]

Most of the studies used woven roving or unidirectional type glass fibers and have not found any references using glass woven cloth fibers. Hence, this study uses e-glass woven cloth fibers as composites reinforcement. This paper studies about the effect of sea water immersion on the mechanical properties of e-glass/vinylester composites. The objectives of this study are to evaluate the durability of e-glass/vinylester composites after immersed in sea water. The material used in this study are vinyl ester resin as matrix and woven cloth e-glass fabric as reiforcements. This study uses ASTM D3039 standards for tensile properties, ASTM D6641 for compressive properties, and ASTM D3518 for shear properties.

The 8th International Seminar on Aerospace Science and Technology – ISAST 2020
AIP Conf. Proc. 2366, 040005-1–040005-11; https://doi.org/10.1063/5.0060163
Published by AIP Publishing. 978-0-7354-4123-1/$30.00

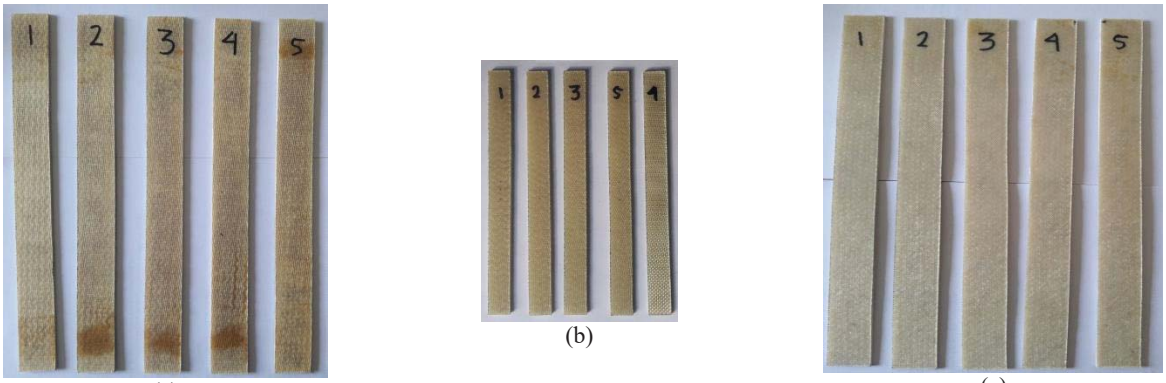
MATERIALS AND METHODS

Materials

This study used woven cloth e-glass fiber 135 gsm commercially named EW-135 cloth as reinforcement. The vinylester resin of Bisphenol A (Ripoxy-R802 EX-1), hardener of methil ethyl ketone peroxide (MEPOXE) 3% and promoter of cobalt naphthenate (P-EX) 0.3% were used as matrix material. Ripoxy R-802-EX-1 was manufactured by Showa High Polymer Co., Ltd, Japan which has 4-6 dPa.s/25 ^{0}C viscosity and 22-32 min geltime. These materials are used to manufacture hybrid composites using vacuum assisted resin infusion (VARI) process. The fiber, matrix and consumable materials for VARI process used in this study were procured from Justus Kimia Raya, Indonesia.

Composites Manufacturing

The e-glass/vinylester composites were manufactured using VARI process. This process used a set of vacuum pump from vacmobile – New Zealand to distribute resin from reservoir to the laminate area. The laminate consist of 23 layers of woven cloth e-glass fabric in 0/90 woven fiber orientation. The composite laminates were fabricated on flat glass mould. The composites were cured for 24 hours in room temperature. The test samples were cut from composites panels according to ASTM standards for water absorption, tensile, shear, and compressive tests. The samples were post-cured at 120 ^{0}C for 3 hours in an oven. The density of e-glass/vinylester is 1.68 g/cm^3. The volume fraction of fiber and matrix are 43% and 57%, respectively. The mass fraction of fiber and matrix are 62% and 38% respectively.



(a) (b) (c)

FIGURE 1. Specimens for (a) tensile test, (b) compressive test, and (c) in-plane shear test

Water Absorption

Water absorption testing was carried out according to the ASTM D570 standard[13] by immersing the composite in a container filled with sea water with a salinity of 32 $^0/_{00}$ (part per thousand) for up to 6 months. Sea water was changed every month. The sample dimensions length x width are 60x60 mm. Before testing, the samples were dried in an oven with a temperature of 120 ^{0}C for 3 hours to remove the water content in the composites. After that the sample was weighed using analytical balance precisa ES320A with sensitivity 0.1 mg then the sample was immersed in a container filled with sea water. The sample was first weighed after 24 hours of immersion. Samples were taken back from the container at the end of the first week, wiped with tissue paper and weighed. The next sample was weighed every two weeks. Figure 1 shows the water absorption or water diffusion process of Fickian law. The water absorption percentage is calculated as follows.

$$WaterAbsorption = \frac{P_W}{P_D} x100 \quad (1)$$

where P_W and P_D are wet and dry mass of sample respectively.

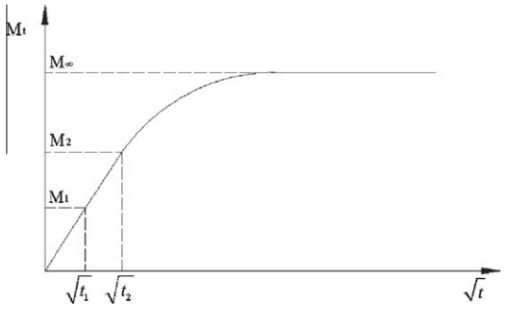


FIGURE 2. Diffusion process of Fickian[14]

Mechanical Test

The tensile tests were undertaken according to ASTM D3039 standard. The sample size was 250 mm x 25 mm x 2.5 mm. The test used constant crosshead speed of 2 mm/min. The tensile properties were recorded using Tensilon RTF 2410 with capacity of 100 kN. The tensile test was performed for dry samples and wet samples immersed in sea water for 2, 4, and 6 months.

The In-plane shear tests were performed using the same UTM for tensile test Tensilon RTF 2410, Japan. The shear strength has been investigated as per ASTM D3518. The sample size was 250 mm x 25 mm x 2.5 mm. The fiber orientation is arranged in $\pm 45^0$ when the composites panels were manufactured. The shear test was conducted for dry and wet samples immersed in sea water for 6 months. This is done because of the limited availability of material and the immersion time of six months is sufficient to represent the effect of sea water diffusion.

The compressive tests were conducted according to ASTM D6641 standard. The sample size was 140 mm x 13 mm x 3 mm. The test was undertaken using compression jig as per ASTM D6641 by Tesilon RTF 2410. The compressive test was conducted for dry and wet samples immersed in sea water for 2, 4, and 6 months.

FIGURE 3. Tensilon RTF 2410 universal tesing machine

Scanning Electron Microscopy (SEM) Obervations

The observations of composites microstructure were caried out using SEM. Scanning Electron Microscopy (SEM) was performed in the CMPFA lab, Faculty of Engineering, University of Indonesia. The observation was focused on the surface fracture of the tensile test samples before and after immersion in sea water.

Spectroscopy Observation

Spectroscopic analysis was performed using FTIR (Fourier Transform Infra Red). The infrared spectrum is a plot between the transmittance and the frequency or wave number. This spectrum also shows the number of absorption peaks (bands) at a characteristic frequency or wave number.[15] Measurements in the infrared spectrum are carried out in the middle infrared light region (mid-infrared), namely at a wavelength of 2.5-50 μm or a wave number of 4000-200 cm^{-1}.[16] If a certain frequency of infrared radiation is passed to a sample of an organic compound, there will be frequency absorption by that compound.[16] A detector placed on the other side of the compound will detect the frequency passed through the sample not absorbed by the compound.[16] The number of frequencies that pass through the compound (which is not absorbed) will be measured as the percent transmittance.[16] The 100 percent transmittance means no IR frequencies are absorbed by the compound.[16] The 100 percent transmittance means no IR frequencies are absorbed by the compound.[16]

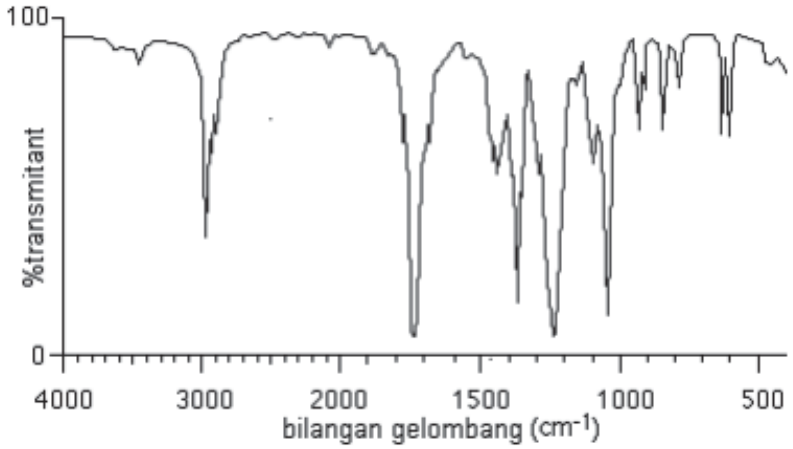


FIGURE 4. Example of FTIR chart

RESULTS AND DISCUSSION

Water Absorption

The test results show that seawater diffusion in the e-glass/vinyl ester composite sample follows Fick's law. Sea water absorption increases significantly at the beginning of immersion process and reaches a saturation point which tends to stabilize after 192 hours. This is consistent with several references even though the stabilization points are different.[5,17] The time needed to reach saturation is faster than some references because the edges of the composite is not coated with resin. Seawater enters from this side apart from diffusion through the matrix, so the time needed to reach saturation is faster. The maximum weight gain of e-glass/vinyl ester composites is 0.69%.

There are several causes of seawater absorption into the composite. According to Ying Hu et al., the absorption of water into the composite is caused by diffusion of water through the matrix and by capillary diffusion through debonding of the fiber and matrix interfaces.[18] According to Anxin Ding et al. absorption of water into composites without voids is caused by free volume between polymer molecular chains, infiltration of polymer molecular structures which increases swelling, and capillarity in the fiber / matrix interface.[19] According to Alessi et al.[20] the water absorption mechanism into the composites is related to the diffusion of water into the free volume of the meso scale, the capillarity of the fibers, and the micro cracking due to fiber-matrix debonding. Other researchers stated that water absorption in composites was caused by voids, micro cracks, matrix, and fiber / matrix interfaces.[21]

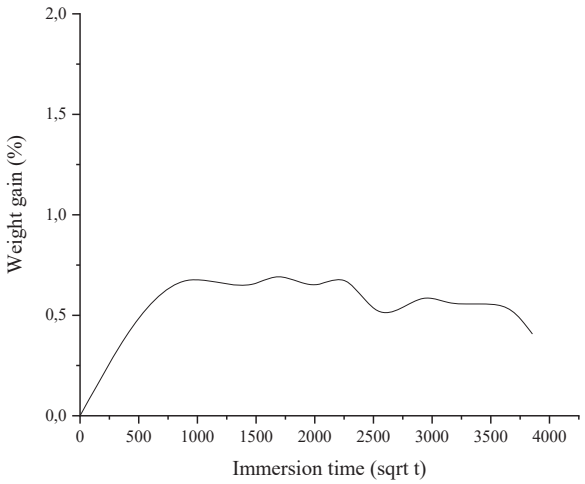


FIGURE 5. Sea water diffusion

Tensile Properties

The test results exhibited the tensile properties difference between the composites without immersion and after immersed into seawater. The composite without immersion has a tensile strength value of 175±11 MPa and a modulus of elasticity of 7.9±0.5 GPa. The composite after immersion in sea water for 2 months had a tensile strength value of 152.6±7 MPa and a modulus of elasticity of 8±0.4 GPa. There was a decrease in the value of tensile strength and an increase in the modulus of elasticity by 13% and 1%, respectively. After 4 months of immersion in sea water, the composite had a tensile strength value of 138.2±6 MPa and a modulus of elasticity of 6.5±0.3 GPa. There was a decrease in the value of tensile strength and modulus of elasticity by 21% and 18%, respectively. The composite after immersion in sea water for 6 months has a tensile strength value of 130.3±6 MPa and a modulus of elasticity of 7.86±0.4 GPa. There was a decrease in the value of tensile strength and modulus of elasticity by 26% and 1%, respectively. Decrease in the tensile strength of glass / vinylester composites also occurred in several references although the percentage of reduction was different, some were smaller[8] some were bigger.[22]

The decrease in tensile strength due to the immersion of sea water is getting bigger with the longer the immersion time by 2, 4, and 6 months. The elastic modulus was almost unchanged after 2 months of immersion and decreased significantly after 4 months of immersion. Meanwhile, after 6 months of immersion, the modulus of elasticity decreased almost unchanged as it was without immersion. This is in accordance with several references that conducted studies on the effect of sea water immersion on composite properties where there was a significant decrease in tensile strength and a very small change in the modulus of elasticity.[23] The decrease also occurs at the break point strain. The composite without immersion breaks in the average strain of 5%, whereas after sea water immersion the breaks in the average strain of 3% -3.9%. This shows that the composite strains after immersion are shorter than without immersion. The decrease in the value of the break point strain also occurs in reference to glass composite research with immersion of sea water accompanied by heat.[12]

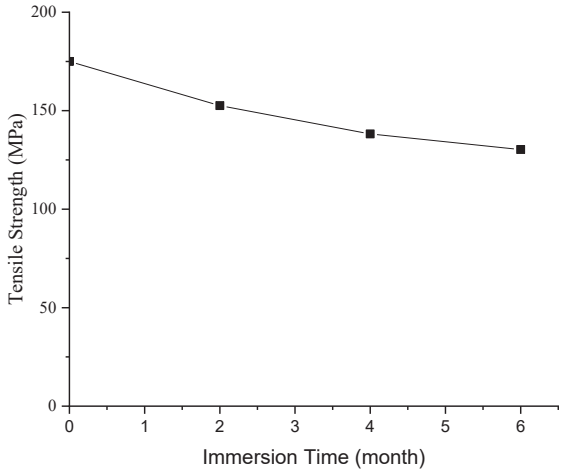


FIGURE 6. Tensile strength of e-glass composite before and after sea water immersion

Shear Properties

The test results showed the difference between the shear properties of the composites without immersion and after immersion. The test results without immersion showed the composite shear strength of 91.5±3.7 MPa and a shear modulus of 3.5±0.14 GPa. The composite after immersion in sea water for 6 months has a shear strength value of 76.6±9.6 MPa and a shear modulus of 3.4±0.4 GPa. There was a decrease in the value of shear strength and shear modulus by 16% and 1%, respectively. These results indicate a significant decrease in shear strength while a very small decrease in shear modulus.

Based on ASTM D3518, the maximum shear strength is determined at a shear strain of less than 5% or at a shear strain of 5%, so the shear strength value above must be redefined. The maximum shear strength values at 5% shear strain for composites without immersion and after 6 months of immersion were 73.75±5 MPa and 68.3±9 MPa. There was a decrease in the maximum shear strength by 7% after 6 months of immersion in seawater.

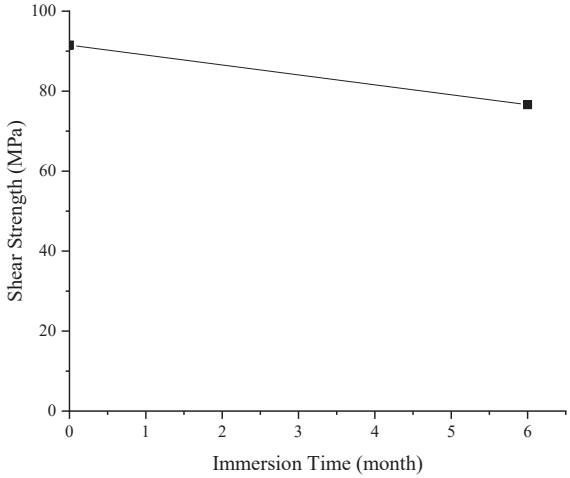


FIGURE 7. Shear strength of e-glass composite before and after sea water immersion

Compressive Properties

The test results showed the difference between the compressive properties of the composites without immersion and after immersion. Composites without immersion have a compressive strength and modulus value of 214.2±10.6 and 29.4±1.5 GPa respectively. After 2 months of immersion in sea water, the composite has a compressive and

modulus strength value of 87±2.2 MPa and of 17.1±0.4 GPa respectively. There was a decrease in the compressive strength and modulus values by 59% and 42% respectively. Composites after immersion in sea water for 4 months have a compressive strength and modulus value of 89.3±6.5 MPa and 24.4±1.8 GPa respectively. There was a decrease in the compressive strength and modulus values by 58% and 17% respectively. The composites immersed in sea water for 6 months had a compressive strength and modulus value of 86.5±6.3 MPa and 19.6±1.4 GPa. There was a decrease in the compressive strength and modulus values by 60% and 33% respectively.

The decrease in strength and compressive modulus due to sea water immersion is quite significant. When compared with the tensile and shear properties, the compressive properties experience a greater decrease after immersion. This shows that the compressive properties of the e-glass/vinylester composite are very sensitive to the seawater environment. The high value of the decrease in the compressive strength of the e-glass composites due to sea water immersion also occurs in several research references.[19,24] The decrease also occurred at the break point strain. The composite without immersion, break in the average strain of 1.57% while after immersing the break in the average strain is 0.4% - 0.5%. This shows that the composite strain due to the compressive load after immersion is shorter than without immersion, which means that the immersion causes the composite to become more brittle. The composite failure mode due to compressive load is almost the same, the composite fracture with the fault direction of 45^0 when viewed from the side.

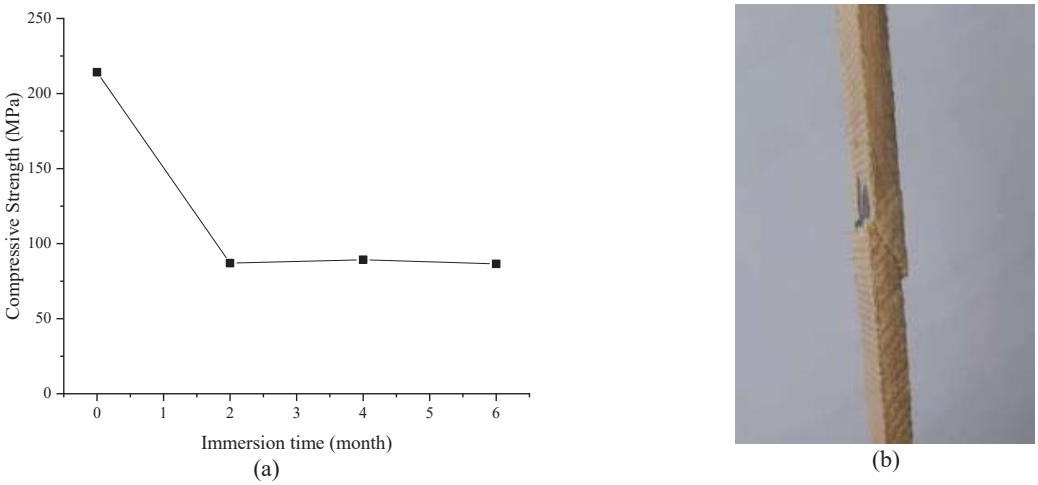
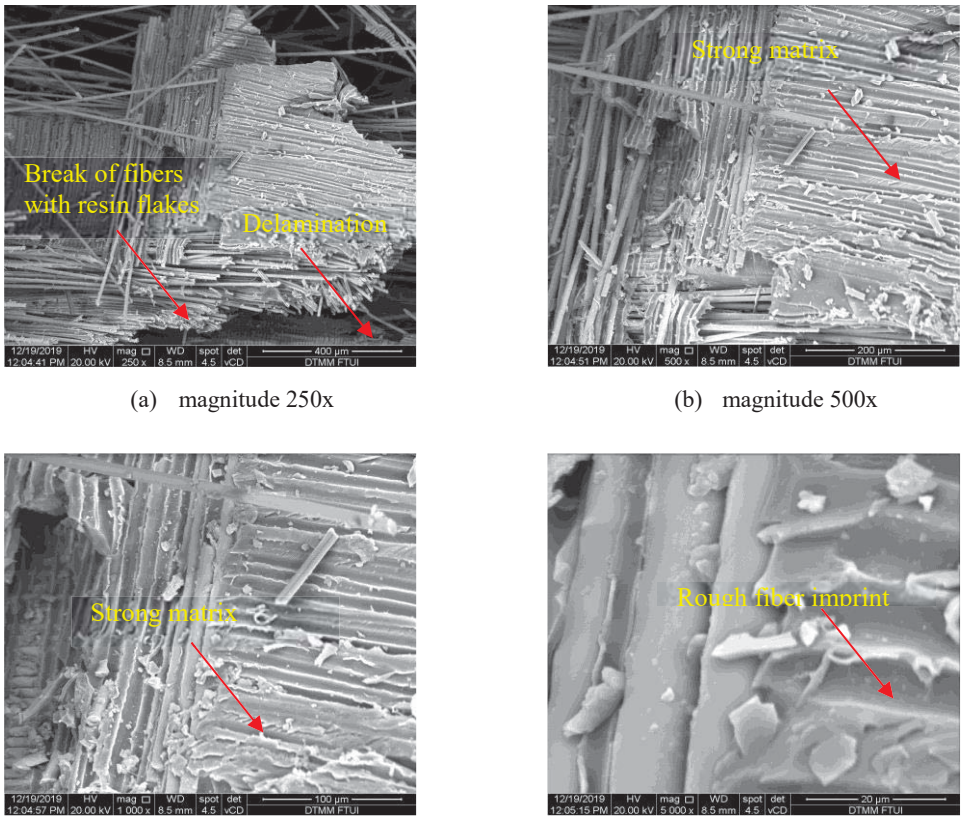


FIGURE 8. (a) compressive strength of e-glass/vinyl ester and (b) compressive failure mode.

Scanning Electron Microscopy (SEM)

SEM tests were carried out on e-glass/vinylester composites before and after sea water immersion. SEM was used to conduct microstructural observations of composite damage.[2] The figure below shows the SEM results of the fracture of the e-glass/vinylester composite without and after seawater immersion for 2 months after the tensile test. SEM micrograph was only performed on tensile test samples because only in this test the sample failed to break into two fractures. This fracture surface was carried out by SEM photos to determine the effect of seawater diffusion on the fiber/matrix bonds visible in the fault section. Failure in the compressive and shear test did not cause the specimen to break in two parts so that it was not possible for SEM photos. However, the fracture section of the tensile test specimen represents an analysis of the effect of seawater diffusion on fiber / matrix bonds because all specimens undergo the same treatment, even though the degradation values for each of the mechanical properties are different.

Figure 9 shows the SEM results of the e-glass/vinylester composites without immersion. Figure 9 (a) shows the number of resin flakes in the fractured fiber. This shows a good adhesion bond between fiber and resin in the composite sample without immersion. Figures 9 (b) and (c) show the strong matrix fracture surface for the non-immersion composite characterized by a rough surface such as streaks. Figure 9 (d) shows the fiber imprint failure on the composite in the form of fiber traces on a matrix that looks a bit rough. Other studies on woven roving fiber composites have also demonstrated this type of failure.[25]



(a) magnitude 250x

(b) magnitude 500x

(c) magnitude 1000x

(d) magnitude 5000x

FIGURE 9. SEM micrograph of dry e-glass/vinyl ester

Figure 10 shows the SEM results of e-glass/vinylester composites after 2 months of immersion in seawater. Figure 10 generally shows the brittle failure of the composite after immersion in sea water which is characterized by a smooth fracture surface in the fiber or matrix. The smooth fracture surface is visible on the fiber imprint surface in Fig. 10(b) and the fiber fault surface in Fig. 10(d). Figure 10(a) shows the smooth surface of the fractured fiber without resin flakes. This indicates poor adhesion bonding between fiber and resin after sea water immersion. Sea water immersion causes degradation of the matrix, fibers, and fiber/matrix interfaces. The degradation of the matrix is characterized by the absence of a strong matrix fracture surface as shown in Fig. 7. Degradation of the fibers is observed by the number of breaks in the fibers as shown in Fig. 10 (d).

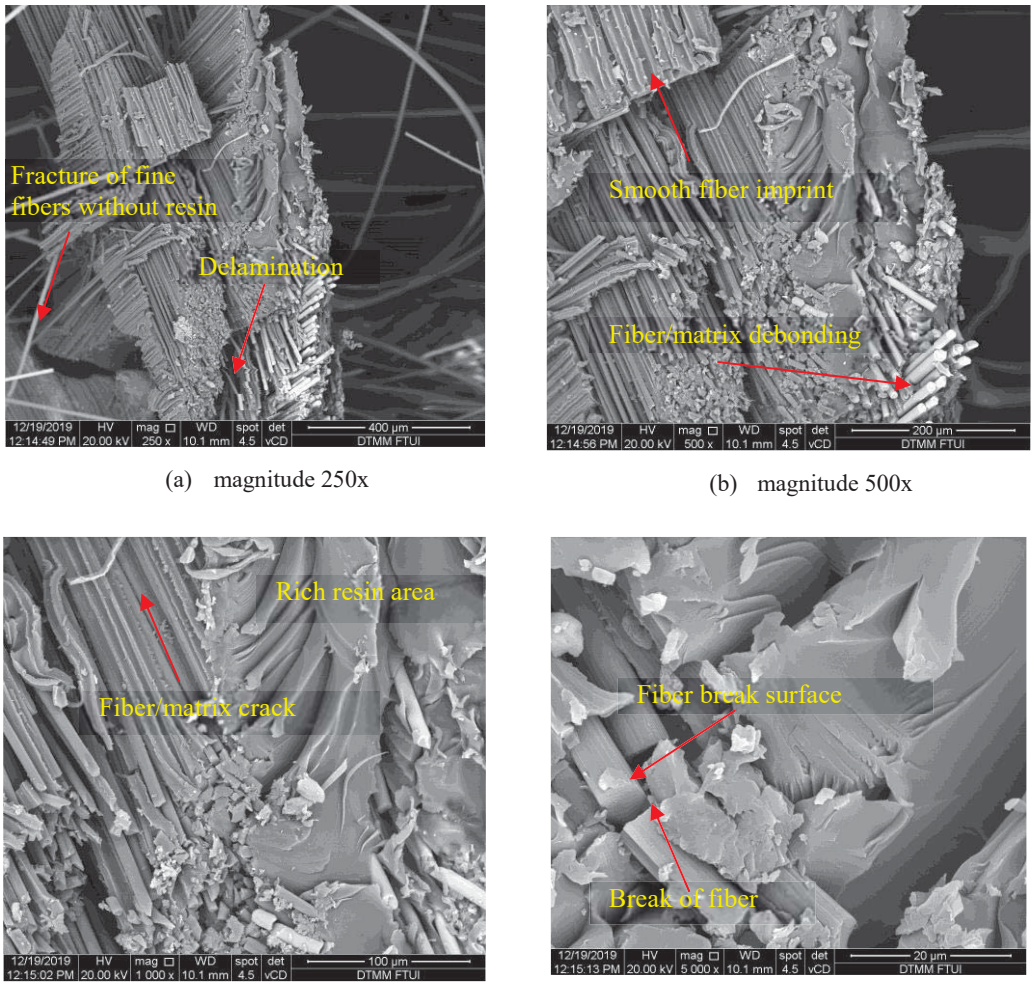


FIGURE 10. SEM micrograph of e-glass/vinylester after seawater immersion

Fourier Transform Infra Red (FTIR)

FTIR testing was carried out on e-glass/vinylester composites without immersion and after immersion in sea water. Non-immersion glass composites showed a wave absorption for the C-H bond strain at 2921.56 cm^{-1} and the C=C bond strain at 1508 cm^{-1}. Meanwhile, the wave absorption for the Ar-H bending bond varies at 699, 799, and 926.9 cm^{-1}. The glass composites after immersion showed a wave absorption for the O-H bond strain at 3403 cm^{-1} where this bond was not clearly visible in the composite without immersion. This indicates that the O–H groups are connected by hydrogen bridges due to seawater absorption that occurs during the immersion process. This is evidence of the hydrolysis reaction of the composite matrix which causes degradation of the fiber/matrix interface. In addition, in the composites after sea water immersion, the wave absorption for the C = C bond strain in areas 1508 and 1606 cm^{-1}, and for the C-H bending bond at 1456 cm^{-1}. Meanwhile, the absorption wave for the C-O bond occurs at 1011 cm^{-1} and the Ar-H bond bending on 698, 759, and 827 cm^{-1}. Figure 9 shows the change in the IR frequency absorbed by the composite after immersion in sea water, especially in the Ar-H, C-H, and O-H bond areas. This shows that seawater immersion affects the molecular bonds in the composite matrix.

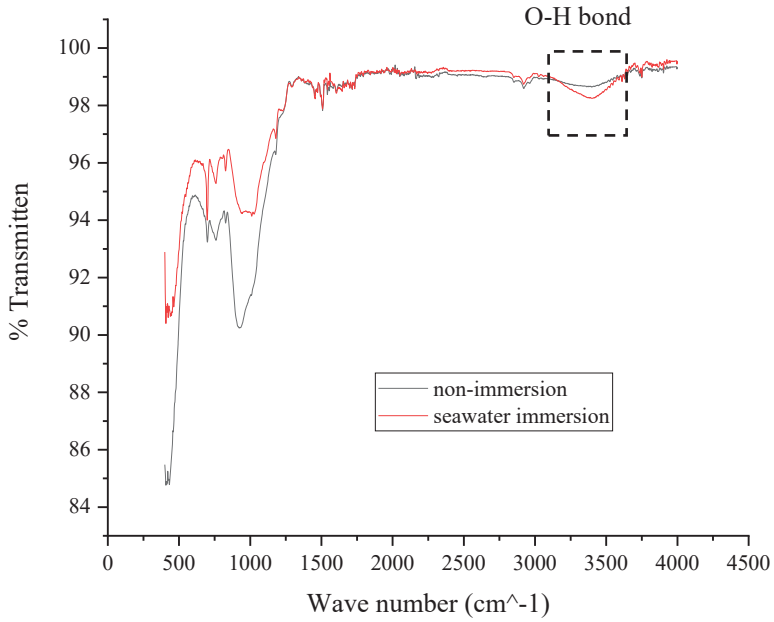


FIGURE 11. FTIR results for e-glass composites before and after sea water immersio n

CONCLUSIONS

Study on the effect of sea water on the strength of the composite as a seaplane float material has been carried out. The matrix used in this study is vinylester resin because it is better at avoiding seawater diffusion, still has a high cross-linking when the material is immersed in seawater, and is highly resistant to hydrolysis that can arise from sea water immersion. The fiber used is e-glass fiber. The composites are made with vacuum assisted resin infusion technology. Sea water immersion treatment was carried out on composite specimens with immersion time of up to 6 months. Tests carried out are gravimetric test, tensile test, shear test, and compressive test. Observations were also carried out on composites before and after se water immersion with SEM and FTIR.

The results showed that the maximum weight gain of e-glass/vinylester composites was 0.69%. The values of tensile, compressive, and shear strength of the e-glass/vinylester composite without sea water immersion were 175±11 MPa; 214.2±10.6 MPa; and 91.5±3.7 MPa respectively. Degradation of mechanical properties due to immersion in sea water occurs in composites. In the glass composite, there was a decrease in the maximum tensile, compressive, and shear strength by 26%, 60%, and 16% respectively. The highest decrease occurred in compressive strength. Degradation of mechanical properties occurs because seawater immersion causes plasticization, matrix and fiber degradation, fiber/matrix debonding, hydrolysis, and cracks. This indication is evident from the microstructural analysis of the fracture surface using SEM and FTIR test results.

ACKNOWLEDGMENTS

Many thanks go to composites workshop and material testing laboratory of Pustekbang – LAPAN for the technical support of composites manufacturing and testing facilities used in this study.

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