Effect of Immersion into Seawater and Freshwater to Mechanical Properties of Vinyl ester Carbon Composite

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Abstract

The carbon-vinyl ester composites have been developed as a base material for Amphibious floats. This composite has properties of strength, lighter weight and resistant to corrosion. This research aimed to know the effect of immersion into seawater and freshwater to the mechanical properties of vinyl ester carbon composite manufactured from vacuum assisted resin infusion (VARI) method. Experiments have been carried out on the tensile, compressive, and shear tests. A 10-day duration for immersion is required in seawater and freshwater. The results exhibit that the Ultimate Tensile Strength of the composite with seawater immersion declined by 1.27% compared to the composite without immersion treatment. Meanwhile, the composite in freshwater obtains above 3.56%. Compressive and shear strength decrement was observed more significantly than their tensile strength. The compressive strength for freshwater and seawater treatment declined to 17.89% and 16.7%, respectively. Additionally, the shear strength for freshwater and seawater treatment decreased by 27.87 % and 25.77%, respectively.

Keywords: *Immersion, Seawater, Freshwater, Mechanical Properties, Vinyl ester Carbon Composite*

1. Introduction

Amphibious aircraft can take off both on land and on the water without changing the structure of any part so that they are suitable for several special missions on land and sea (Dawei et al., 2011). Amphibious aircraft commonly operate in lakes, seashores, and seawater. Amphibious aircraft floats used to be made of aluminium which was resistant to corrosive environments such as seawater. However, it can be developed from a polymer matrix composite (Abdurohman et al., 2019). It is imperative that this aircraft shall be parked within the garage in order to prevent material damage due to environmental circumstances.

Polymer Matrix Composite (PMC) is a type of composite with a polymer base material such as epoxy, vinyl ester and unsaturated polyester, which is commonly used as a protective layer in various types of marine vehicles such as hulls, patrol boats, minesweepers and domes on ships. The source of vinyl ester resin is available in Indonesia easily. The reasons for using composite materials are lightweight, corrosion resistance in manufacturing, and no extra expensive tools (Chakraverty et al., 2013; Davies, 2016). Vinyl ester is chosen because of its superior relative to phthalic polyester in seawater due to steric inhibition and the faster drying process of vinyl ester (Firdosh et al., 2018)

Apart from several advantages of using composites, a concern is the loss of stiffness, strength and other mechanical properties caused by water absorption in composites (Gargano et al., 2018). Amphibious floats are in direct contact with seawater and freshwater when not flying and during the flushing process to reduce the effects of high salinity seawater. Water can consequently seep into the polymer matrix and the fiber interface region. Water can be present in composites as free molecules which exit in free volume between their polymer chains and bond chemically depending on the matrix properties of the polymer and the fiber interface region (Gargano et al., 2018). The amount and rate of water absorption by polymer composite materials depend on many factors, including fiber volume fraction, location, type and size of defects (e.g. cracks, voids) (Bian et al., 2012; Pavlidou et al., 2005) and environmental conditions such as humidity, temperature, and applied stress (Gautier et al., 1999; Mourad et al., 2010; Pavlidou et al. 2005; Ray 2006).

Based on the study of (Alia et al., 2013) the tensile strength of vinyl ester resin composites after immersion in seawater for nine months is reduced by 12%. Then, the study of (Mittal et al., 2015) using glass/vinyl ester composites showed that the tensile strength of the composites after immersion in seawater was reduced by 7%. In previous research (Koshima et al., 2019) using Plain-woven CFRP (T700S 12K TORAY) and vinyl ester resin (RIPOXY R-802 Showa Denko K.K.), the tensile strength and compression strength decreased significantly after immersion for 100 days.

Later on, for compressive properties, (Arhant et al., 2016), used material C/PA6 pre-impregnated tape from Celanese (CFR-TP PA6 CF60-01), and the fibre HTS45 12 K carbon. Compressive strength Carbon-polyamide decreased significantly just below 50 % from 0 to 1% water content. Hereafter, for shear stress, (Tual et al., 2015) Study on CFRP composites (UD carbon, epoxy) exposed to seawater show that ILSS is reduced by 20-30%.

In another study, a test was conducted to determine the effect of immersion in deionized water for 28 days on the ILSS value and flexural strength of carbon vinyl ester resin composites showed that the value were decreased (Dell'Anno & Lees, 2012). Based on the study (Alvarez et al., 2007), the higher decrease of composite's mechanical properties observed in the case of vinyl ester matrix after the samples were immersed in distilled water at different temperatures.

The vacuum-assisted resin infusion (VARI) method has been widely used in the marine, energy, infrastructure, aerospace and defense industries. VARI is a closed molding method that can be used in making complex and flexible products based on fiber-reinforced polymer composite. The molding on the VARI is similar to those in the Hand Lay-up method so they are easy to modify and clean. (Hsiao et al., 2012).

However, this process also has several drawbacks, such as requiring consumables that cannot be reused, the potential of air leakage during the process depending on the worker's skill level. The pressure not as intense as other resin transfer molding processes or the autoclave bagging process, so that air void is still unavoidable (Hsiao et al., 2012). This method uses vacuum pressure to move the resin into the laminate. Previously the material was placed in the mold and other supporting materials and then vacuumed before the resin was added (Abdurohman et al., 2018). This writing better knows the effect of seawater using the VARI method, which is usually used for ships but is used in aircraft parts. Then, It used quality seawater from North Java, Indonesia with a salinity level of 2.5% and groundwater from the Center for Aviation Technology, Rumpin, Bogor, West Java, Indonesia.

The purpose of this study is to determine the effect of immersion in seawater and freshwater on the mechanical properties of the carbon-vinyl ester composite such as the value of the strength of the tensile test, compressive test and shear test. Similar research has been carried out by (Figliolini et al., 2014), but this research utilized materials from different manufacturers which are easily available in Indonesia. The condition and the quality of seawater also different from the reference which took place in the USA.

Materials that used in this research are reinforced carbon and matrix vinyl ester resin. Considering there was no carbon/vinyl ester material in the previous research, however, the research used data comparisons for a single similar material, such as composite, vinyl ester, carbon composite, and with same treatment in seawater and water.

2. Methodology

Flow process of the research, shown in Figure 2-1

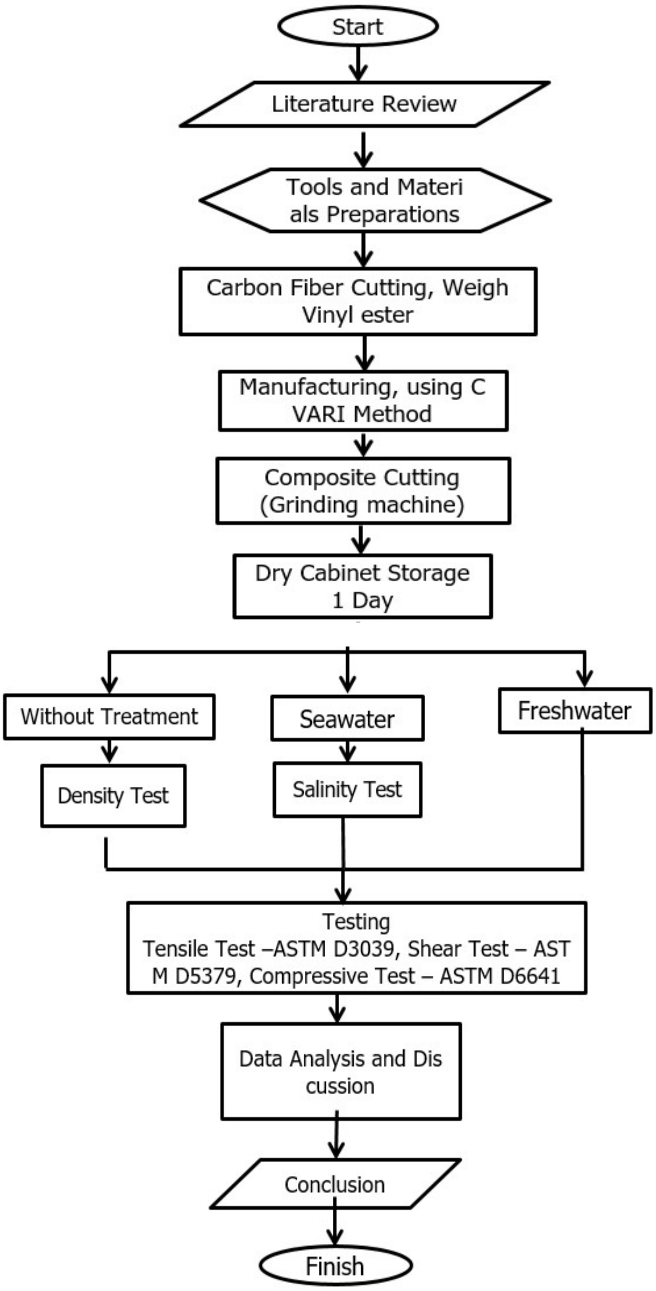

Figure 2-1: Flow Process Diagram of the research

2.1 Material and processes

The material under study is vinyl ester resin (Ripoxy R-804 J500). This material has the main property of excellent chemical resistance, especially against acids, alkali and solvents, also resistance to corrosion (K. Abdurohman et al., 2020; Bunsell & Renard, 2005). Before usage, it must be mixed with a catalyst (MEKPO or cumene hydroperoxide) and a promoter (cobalt naphthenate or promoter D). The chemical bonds can be seen in Figure 2-2 and Figure 2-3. The amount of catalyst was 2.5% by volume of the resin. The reaction temperatures are at, 60-90^{o}C and heat will be generated. The heat generated is sufficient to react to the resin so that the maximum strength is obtained according to the desired mold shape (Gautier et al., 1999). Properties resin can be seen in Table 2-1 and Table 2-2. Ripoxy R-804 J500 normally used for Marine (yachts and boats) (SHOWA DENKO K.K, n.d.). All materials were obtained from PT. Justus Kimiaraya with Carbon Twill 3 K Tow. Volume fraction fibre and resin is 50%:50%.

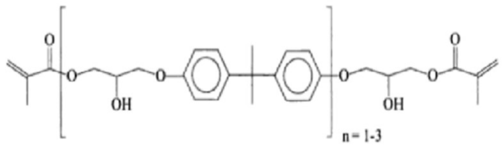

Figure 2-2: Vinyl ester (Bunsell & Renard, 2005)

Table 2-1: Properties of liquid resin (SHOWA DENKO K.K, n.d.)

Properties	**Unit**	**Value**
Property	-	Clear yellow
Appearance	dPa s/25 C	2-3,5
Gel time	Minutes	16-25

*Gel time: 25 ℃ 55%MEKPO 1.5phr Co-Oct(8%) 0.5phr 2.

Table 2-2: Properties of cured resin (SHOWA DENKO K.K, n.d.)

Property	**Unit**	**Laminate**	**Test method**
Flexural strength	MPa	184	ASTM D790
Flexural modulus	GPa	7,6	ASTM D790
Tensile strength	MPa	111	ASTM D638
Tensile elongation	%	2	ASTM D638

Laminated constitution: 3 mats (450g/m2),glass contents 30% Laminate

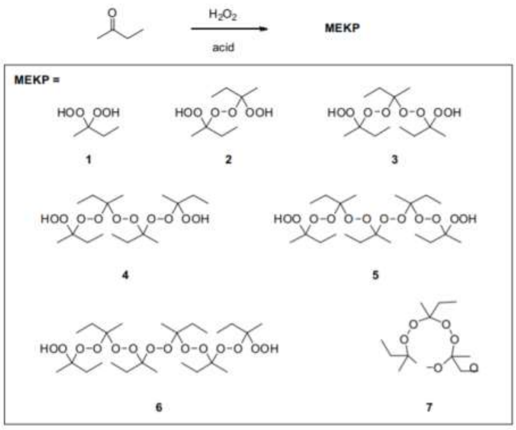

Figure 2-3: Methyl ethyl ketone peroxide (MEKP) (Graham et al., 2011)

Figure 2-3 shows the Methyl ethyl ketone peroxide (MEKP). It is an organic peroxide formed by reaction of methyl ethyl ketone (MEK) with hydrogen peroxide (Graham et al., 2011).

The fibre used in the manufacture of composites is carbon fibre (carbon fibre) twill HDC-522-3K obtained from Toko FRP. Carbon fibre is suitable for applications where strength, stiffness, lightness and fatigue resistance are required. In addition, carbon fibre can also be used in applications that require high-temperature resistance, Figure 2-4 shows the stitch.

Fiber Carbon HDC-522-3K

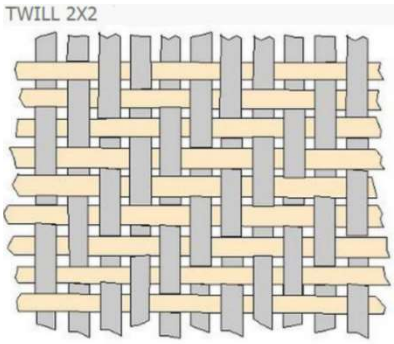

Figure 2-4: Fiber Carbon HDC-522-3K (TokoFRP, n.d.)

Spesification of the Fiber Carbon as follows, Table 2-3

Table 2-3 : Fiber Carbon HDC-522-3K specification (TokoFRP n.d.)

Weave	2/2 Twill	
Carbon thickness (mm)	0,30 +- 0.05	
Tensile Modulus	230 GPa	
Construction (thread count/inch)	Warp: 13 +- 0.5 Carbon 3K	Weft: 14 +- 0.5 Carbon 3K
Carbon Fabric Weight (g/M²)	220 +- 6.5	

2.2 Manufacturing Process

Composite production using the VARI method begins with cutting 30 cm x 30 cm carbon fiber and other materials. the next step is applying wax to the table and then piling up the fibers and installing other supporting materials. The working principle of Vacuum Infusion is to take the advantage of the constant pressure from the vacuum device, which helps distribute the resin to all parts of the fiber. Vacuum Infusion has an inlet and an outlet. Next step, the channel is then used as the resin inlet, while the late channel is used as the place for the vacuum device to be placed and the resin outflow. Vacuum Assisted Resin Infusion (VARI) can be seen in Figure 2-5.

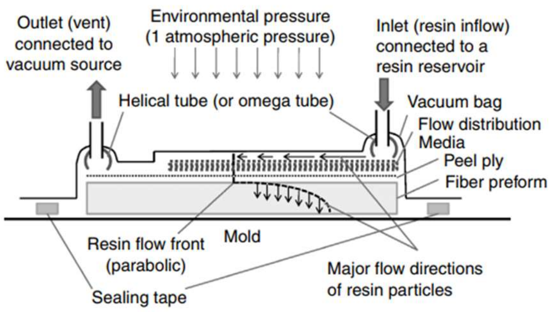

Figure 2-5: Vacuum assisted resin infusion (VARI) (Hsiao & Heider, 2012)

For Tensile testing, sample was prepared with ASTM D3039 testing standards. Sample size was 25 mm x 250mm x 2.5mm, for (width x length x thickness). Then, Shear Testing, Compressive Testing sample was prepared with ASTM D5379, ASTM D6641. Sample size was 20mm x 76 mm x 2.5 mm, 13 mm x 140mm x 2.5 mm, for (width x length x thickness). All testing used five specimens. Cutting the specimen using grinder. After cutting, the specimens were immersed in seawater and freshwater for ten days. It uses quality seawater from North Java Indonesia with a salinity level of 2.5% and groundwater from the Center for Aviation Technology, Rumpin, Bogor, West Java, Indonesia.

Tests were carried out using Universal Testing Machine (UTM) Tensilon RTF-2410 Japan. In this test the properties were taken: tensile stress, ultimate tensile strength, and modulus of elasticity. Constant crosshead speed 2 mm / minute. Testing is carried out on room temperature 24^0 C and 54% RH.

3. Result and Analysis

3.1 Density Results

Density testing is used to determine the average density value of the Twill / Vinyl ester Fiber Composite Density composite. The average of composite density obtained from this experiment was 1.348 g/cm³. The data showed that the composite's density was lesser than aluminum's density, 2.7 g/cm³. With the same float design, the composite material weighs less than aluminum, so there is no need to reduce passengers' or cargo weight capacity. The density test function is a basic control tool after production (Jędral., 2019). The aluminum's density 2.7 g/cm³

3.2 Mechanical Testing

3.2.1 Tensile Test Results

In the tensile testing process, see Figure 3-1

Figure 3-1: Tensile Test according ASTM D6641

Figure 3-2 shows the tensile test strength of the composite at the different treatments. It can be seen that the maximum point stress average for specimens before immersion is 527.62 MPa, immersed specimens in seawater are 520.904 MPa, decreased 1.27% than before treatment, and the immersed specimens in freshwater are 546.44 MPa, increased 3.56% than before treatment. Seawater was reducing 1,27%, meanwhile water was increasing 3,56%.

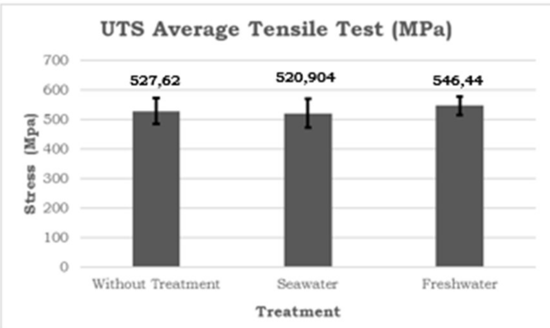

Figure 3-2: Graph of stress - treatment of Composites without treatment, Seawater immersion treatment, and Freshwater immersion treatment.

In this tensile test, there was an increase in immersed specimens in freshwater, which should have decreased before immersion. It also happens because, in the preparation process, the test sample cut manually using a grinder machine without conditioning so that residual stress occurred. The specimens immediately tested so that the specimen's maximum point stress value before immersion was smaller than that of the specimen after freshwater immersion. It can happen because of the presence of different residual stress on each specimen due to sample preparation. The water immersion process affected large number of voids, decreasing bonding between fibre and matrix, mesoscale free volume, capillarity of fibre, and microcrack as a consequences of debonding of fibre and resin. On the other hand, the effect of water caused plasticization of the matrix has been done by (K. Abdurohman & Adhitya, 2019; Fitriah et al., 2017; Sari et al., 2020). Graph Stress Strain Tensile Strength can see in Figure 3-3. Furthermore, with seawater immersion, the composites were gradually increasing absorption, then it will be affected in voids, interfacial bonding strength, and properties of the reinforced material. Later on, The Glass/Vinyl ester reduced the tensile strength from (Mittal et al., 2015) around 280 MPa to 260 MPa. Other studies conducted(Alia et al., 2013), with pure vinyl ester resin being immersed in seawater during 9 month it was reduces the ultimate tensile strength 12 %, from 120.95 N to 107 N. Moreover, this research was approach similar single materials, as a result of development research.

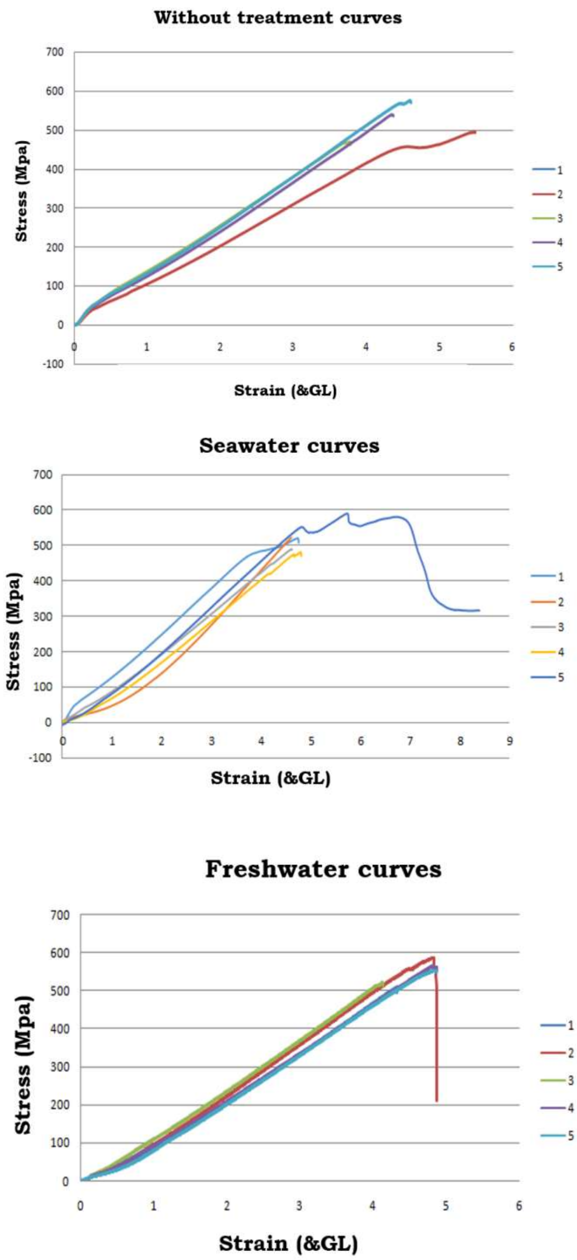

Figure 3-3: Graph of Stress-Strain Tensile Testing of Composites without treatment, Seawater, Freshwater.

In previous research, (Koshima et al., 2019) using Plain-woven CFRP (T700S 12K TORAY) and vinyl ester resin (RIPOXY R-802 Showa Denko K.K.), the tensile strength decreased significantly after immersion for 100 days around 22 %, from approximately 600 MPa to 460 MPa. The difference in the type of material will affect the material properties, confirmed by the results of research (Sivakumar) who has researched the mechanical properties of carbon vinyl ester composites with 2 different types of carbon fiber. The first type of fiber is Cytec Fiberite supplied W-5-322 which produces a tension strength of

436.6 MPa, a tensile modulus of 47.5 GPa,. The second type of fiber is Devold LT650 which produces a tension strength of 1125.7 MPa, a tensile modulus of 56.7 GPa,

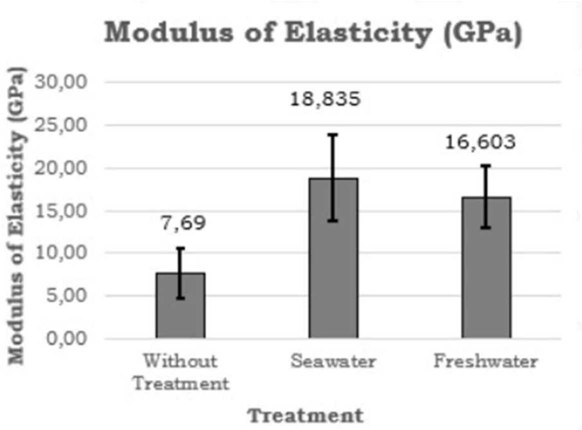

Figure 3-4: Graph of Modulus Elastisity Tensile Testing of Composites without treatment, Seawater immersion treatment, and Freshwater immersion treatment

From Figure 3-4 the data shows the value of modulus elasticity without treatment, seawater, and freshwater are 7.69, 18.835, and 16.603, respectively. Then, the diffusion water increases the composites stiffness, which indicates an increase in the modulus of elasticity after immersion by seawater and freshwater. The increase in stiffness due to seawater is higher than freshwater because of dissolved salt molecules in seawater. (Fitriah et al., 2017; Zhao et al., 2016). The results of this test was also corroborated by research from (Figliolini et al., 2014) that have increasing data of modulus elasticity by 3.2% from 120 GPa to 124 GPa. This could happen since the size of carbon fiber is different and this is affecting the adhesion between fiber and resin. Besides, the materials come from different factories which have special treatment and production technology for their products (Figliolini et al ., 2014).

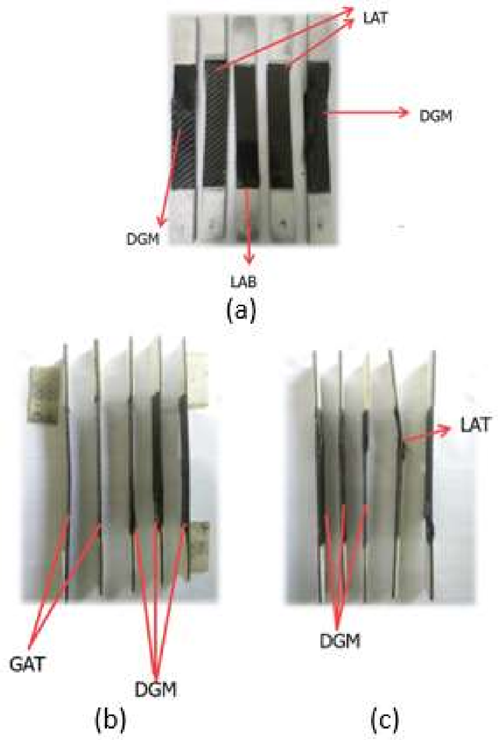

Figure 3-5: Failure Pattern of Tensile Testing of a. Untreated Composites, b. Seawater Absorption, c. Fresh Water Absorption.

Table 3-1: ASTM D3039 failure pattern

Failure Pattern	Characteristic Failure Pattern

LAT/LAB	Lateral At Top/Bottom
DGM	Edge Delamination, gage, Multiple Areas
GAT	Gage at tab

From Figure 3-5 determining the pattern of damage that occurs visually, it can be seen in ASTM D3039 which can be seen in Table 3-1. Edge delamination could occur when the adhesion between fiber and matrix not strong enough. It is caused by adding a catalyst to the resin while the mixing process accelerates the time and produced induced tension. The lateral at top and bottom occurred when the fiber apart from the matrix. It is caused by the void that occurred during the resin mixing process. To prevent the void appeared, ensure the vacuum infusion method took place in the cleanroom and used the proper vacuum machine that provides stable pressure. (Tododjahi et al., 2018).

3.2.1 Tensile Test Simulation

Tensile test simulation using progressive failure was performed to be a comparison for the tensile test result. The maximum load versus axial displacement in the simulation will be compared to the tensile test result. In progressive failure simulation, load can be captured within specified time. Tensile test specimen was modeled in finite element software. Here is geometry of tensile test specimen.

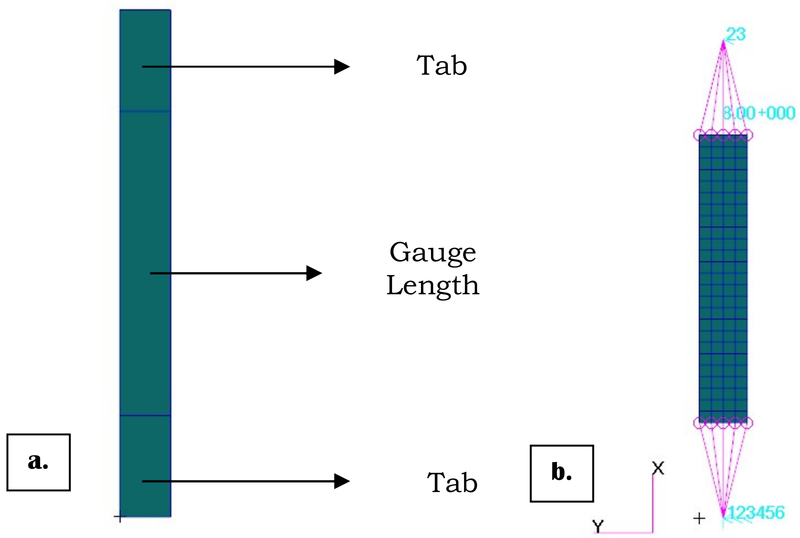

Figure 3-6: Tensile Test Specimen Model, a. Geometry Model, b. Finite Element Model

Based on Figure 3-6 a. there are three areas in the model, two-tab areas and one gauge length area. Since the simulation is only focuses on gauge length area, those two-tab areas are not modeled. Figure 3-6 b. shows the finite element model of the tensile test specimen. Gauge length area was modeled using 2D element called CQUAD. There are 100 elements of CQUAD in the gauge length area. Meanwhile in the tab areas, two rigid body elements (RBE2) were connected to specified nodes. The lower RBE2 was used as boundary condition, and the upper RBE2 was used to pull the model up to certain height. Boundary condition used 123456 in the lowest nodes. This value means that no translation and rotation are allowed for those nodes.

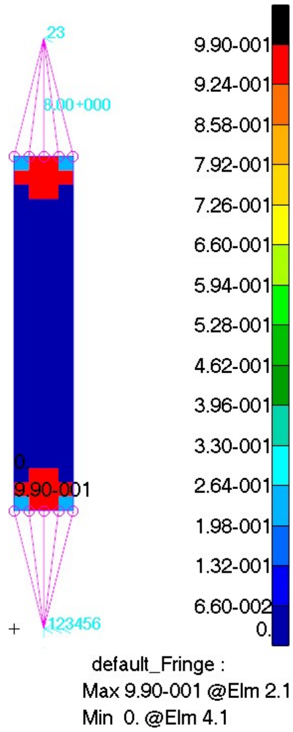

Figure 3-7: Total Damage for Progressive Failure Distribution

Figure 3-7 shows that in the specified time the total damage value is almost one which means the specimen is failed. Furthermore, the red area covers side to side specimen. It indicates that the specimen is not only failed but also breaks apart.

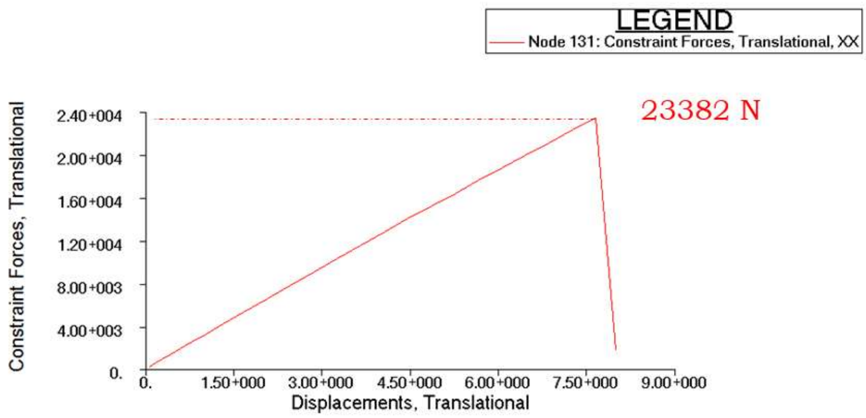

Figure 3-8: Load vs displacement in x direction graph

This graph starts with linear line which indicates that material is in elastic zone. In 23382 N of load, it starts to fail and there is no yielding area. Based on Figure 3-8 above stress is obtained by dividing maximum load with the section area. From the calculation, the stress is 500.15 MPa.

3.2.2. Compressive Test

After that, proceed with the compressive test shows in (Figure 3-9).

Figure 3-9: Compressive test ASTM D6641

Figure 3-10 shows the compressive strength of the composite at different treatments. The data obtained from the Maximum point stress test for pre-immersion were 133.02 MPa, specimens immersed in seawater were 110.796 MPa and specimens immersed in freshwater were 109.214 MPa. Furthermore, Seawater and water were reducing 16,7 %, 17,89%. This test is like the one done by (K. Abdurohman & Adhitya, 2019), where there will be a decrease in compressive strength after immersion. Furthermore, for compressive properties, (Arhant et al., 2016), polyamide decreasing significantly just below 50% from 0 to 1% water content. Hereafter, for shear stress, (Tual et al., 2015) Study on CFRP composites (UD carbon, epoxy) exposed to seawater show that ILSS is reduced by 20-30%. Then, the study from (Shivakumar et al., 2006) a compression strength was 262.1 Mpa.

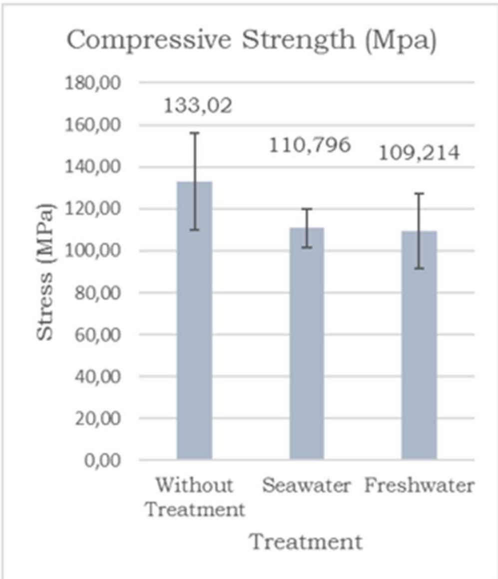

Figure 3-10: Graph of Stress-Strain Testing Compressive on untreated Composites, Immersion in Seawater, Freshwater, Percentage Comparison of Average Compressive Test

In previous research, (Koshima et al., 2019) using Plain-woven CFRP (T700S 12K TORAY) and vinyl ester resin (RIPOXY R-802 Showa Denko K.K.), the compression strength decreased significantly after immersion for 100 days around 14 %, from around 370 MPa to 300 MPa. The value was different due to the previous research using different plain-woven carbon 12K, in this research use carbon 3K.

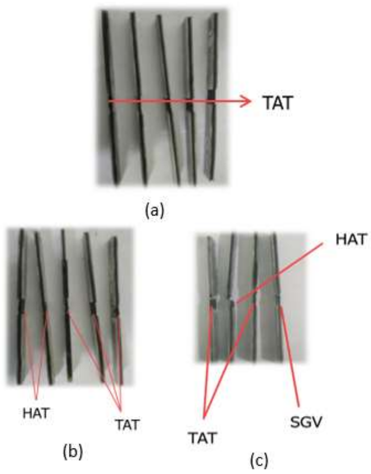

Figure 3-11: Failure Pattern of Compressive Testing of Untreated Composites, Seawater Absorption, Fresh Water.

Table 3-2: ASTM D6641 failure pattern

Failure Pattern	Characteristic Failure Pattern
TAT	Lateral At Top/Bottom
HAT	Edge Delamination, gage, Multiple Areas
SGV	Gage at tab

Figure 3-11 shows failure pattern Compressive Testing at different treatment. In determining the pattern of damage that occurs visually, it can be seen in ASTM D6641 which can be seen in Table 3-2. Edge delamination could occur when the adhesion between fiber and matrix not strong enough. It is caused by adding a catalyst to the resin while the mixing process accelerates the time and produced induced tension. The lateral at top and bottom occurred when the fiber apart from the matrix. It is caused by the void that occurred during the resin mixing process. To prevent the void appeared, ensure the vacuum infusion method took place in the cleanroom and used the proper vacuum machine that provides stable pressure. (Tododjahi et al., 2018) SGV (Long, Splitting, Gage, Various) had occurs when the matrix between the bundle of fiber was damaged, followed by the damage of fiber so that it is actually broken (Kosim Abdurohman & Marta, 2018).

3.2.3. Shear Testing

Furthermore, the data obtained from the shear test of Maximum point stress before immersion were 29.97 MPa, specimens immersed in seawater were 20.50 MPa, and specimens immersed in freshwater were 19.24 MPa can see in Figure 3-12. Furthermore, Seawater and water were reducing 25,77 %, 27,87%. From this shear test data, the value of the Maximum point stress has decreased. This shear test data is almost the same which shows that the shear strength will decrease after immersion (Pavlidou et al., 2005). The reduction in the composite's shear strength after exposure to seawater suggest that the interface between fiber and matrix are degraded by absorbed moisture (Figliolini et al., 2014). The difference in the type of material will affect the material properties, confirmed by (Dell'Anno et al., 2012). The test was carried out using 2 different types of carbon fiber, one general-purpose sizing (GPF) and the second vinyl ester sizing (VEF). From the two types of materials, it was shown that water immersion reduced the ILSS value from 26 MPa to 25 MPa in GPF and 33 MPa to 27 MPa at VEF.

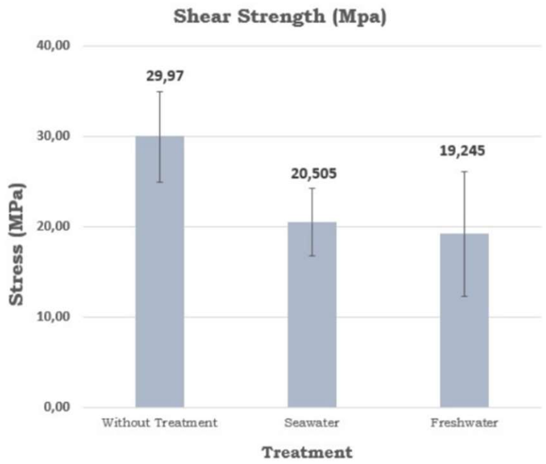

Figure 3-12: Graph of Stress-Strain Testing Shear on untreated Composites, Immersion in Seawater, Freshwater, Percentage Comparison of Average Compressive Test

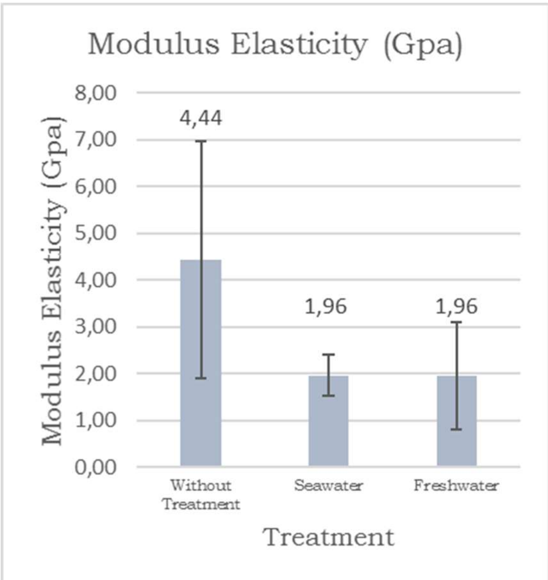

Figure 3-13: Graph of Modulus Elasticity Testing Shear on untreated Composites, Immersion in Seawater, Freshwater, Percentage Comparison of Average Compressive Test

In Figure 3-13 the data shows the value of modulus elasticity testing shear without treatment, seawater, and freshwater are 4.44, 1.96, and 1.96, respectively. There was a downward trend similar and the value is greater than the literature from 5.45 GPa to 5.25 GPa. This difference in value occurs since the type of fiber used has different sizes (Figliolini et al., 2014).

4. Conclusions

The experiments conducted to investigate the effects of seawater and freshwater absorption in ten days on mechanical properties of composites carbon/vinyl ester composites. The results have shown that Ultimate Tensile Strength (UTS), shear strength, and compressive strength in seawater and freshwater was decreased after being exposed. The value of stress-strain simulation in tensile strength nearly same with the simulation. Furthermore, the absorption of seawater and freshwater in the composites were gradually increased with the immersion. Then it will affect the voids, interfacial bonding strength, and properties of the reinforced material. This data will be used for the N219 Amphibious aircraft material database. It will be a consideration for the future development for float construction. In contrast, the specimen's UTS value in freshwater increased due to inadequate of specimen preparation. These phenomena will be investigated in the future research.

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Contributorship Statements

All authors contribute to manufacturing, testing, and analysis.

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