Degradation of Polyester Composite Reinforced E-Glass Fiber with Calcium Carbonate Filler in Seawater

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Abstracts: This research is concerned with composite materials that use $CaCO_3$ as a reinforcing material in construction, especially subsea panels and buildings. Composite materials are solids composed of two or more materials that have their own properties and improve their properties when combined. In this experiment, glass fiber and $CaCO_3$ were mixed in a certain composition and tested to evaluate the water absorption and mechanical strength of the composite. The results show that increasing temperature can increase water absorption, while the addition of $CaCO_3$ has a significant effect on water absorption due to its hydrophilic properties. Water absorption in polyester composites is influenced by aging temperature and Calcium Carbonate powder filler, affecting tensile and bending strength. As aging temperature increases, mechanical strength decreases, composite having the highest tensile strength. Bending tests show debonding and splitting fault patterns due to glass fiber arrangement. With a deeper understanding of how CaCO3 affects composite materials under various conditions, this research can provide valuable insights for developing more durable composite materials suitable for construction applications, especially in corrosive environments such as subsea buildings.In this research, it proposes theoretically model to controlling on half-wave voltage of modulator, in which it uses studying the factor of effective of refractive index difference Δn_{eff}, this using analysis distribution of refractive index for LN material before and after voltage is applied (i.e., with and without applied voltage), and uses analysis a factor of propagation constant difference $\Delta\beta$, before and after voltage is applied where it depends on the wavelength, these leads to it controls on the half-wave voltage $V\pi$ using a reduction half-wave voltage up to 1V. Also, it achieves an excellent solution for solve the fundamental optical mode with effective factor is n_{eff}. Finally, the modulator with an effect effective refractive index difference Δn_{eff}have lower applied voltage up to 10V, and with an effective propagation constant difference the operating voltage is from value of 20V.

Keywords: Polyester Composite, Fiberglass, $CaCO_3$, Mechanical Properties

1. INTRODUCTION

Composites can be described as a competitive alternative material to replace metallic materials in their use as infrastructure. Besides, composites offer a wide range of characteristics that are great for building applications. Composite materials are non-uniform solid materials produced by joining two or more mechanically bonded materials together. Each material in the composite retains its properties, and when combined, its combined properties enhance its properties as an individual solid material [1]. In general, composites consist of two phases, namely matrix and reinforcement. The matrix serves to bind the reinforcement, which in turn increases the strength of the composite [2] (Ayu et al. 2020).

Today, composites composed of fibers and polymers are the most popular and widely applied type of composites in various types of industries [3]. They have the potential to replace conventional metals in structural applications such as aerospace, automobiles, and the manufacture of wind turbine blades [4], [5]. The most well-known fiber-reinforced polyester composites in which a continuous fiber material embedded in a polyester matrix is a combination of natural fiber-reinforced polyester (NFRP), glass fiber-reinforced polyester (GFRP) and carbon fiber-reinforced polyester (CFRP) [6], [7]. Glass fiber has shown its performance as a polymer reinforcement (GFRP) at a fairly low price compared to carbon fiber (CFRP).

West Nusa Tenggara Province has a very high calcium carbonate (CaCO3) composition below 50%. The utilization of CaCO3 is mostly used as a concrete mixture. A broader development of CaCO3 is needed. For this reason, in this research, CaCO3 is used as a filler in the epoxy matrix of a composite hybridized with glass fiber.

The most interesting issues for composite materials are long-term stability, performance, and maintenance under aggressive environments. The urgency of this research is to introduce polymer composite technology by utilizing useless waste for the manufacture of panels and construction of underwater buildings and provide a technological/scientific breakthrough on composite durability due to CaCO3 filler in its application in corrosive environments. This research will support local government programs in the provision of cheap and environmentally friendly goods, facilities, and infrastructure with high performance.

2. METHOD

In this research glass fiber and CaCO3 were mixed in a certain composition and tested to evaluate the water absorption and mechanical strength of the composite. Besides, there are several independent variables, dependent variables, and controlled variables that were used.

Independent variable: variation in the length of immersion of polyester composites namely; 1 day, 7 days, and 15 days. shown in Table 1

Dependent variable: Tensile characteristics, bending strength, impact toughness, and morphology of the composite fracture surface.

The number of test samples for each test is:

- Tensile test, 27 samples.

- Bending test, 27 samples.

- SEM photographs of 9 samples of tensile test failures.

Controlled variable: volume fraction of glass fiber (glass)three different temperature.

3. RESULT AND DISCUSSION

3.1. Water Absorption

In this research, water absorption testing was carried out on fiberglass-reinforced polyester composites with CaCO3 powder filler at three different temperature conditions to obtain the maximum ability of the composite to absorb water. The measurement results of the composite water absorption rate at 50 °c are shown in Figure 1 below.

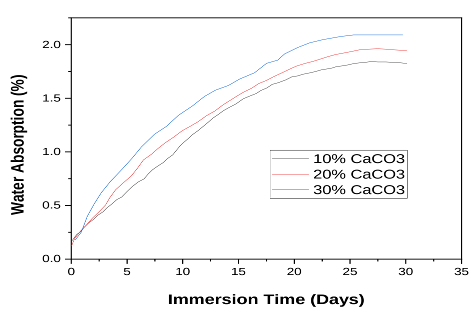

Fig. 1: The relationship between soaking time and weight gain of a polyester composite at 50°c.

Furthermore, the water absorption values at the saturated state of the composite are presented in table 1.

Table 1. Maximum weight (saturated weight) of polyester composites at various temperatures.

Sample	Soaking temperature		
	27	40	50
FC10	1.295	1.357	1.418
FC20	1.715	1.823	1.926
FC30	1.827	1.943	2.091

This research examines the level of water absorption in polyester composites reinforced by glass fiber with variations of Calcium carbonate (CaCO3) powder filler of 10%, 20%, and 30% at 50°C. The results showed that the water absorption of these composites increased with the increase of immersion time and CaCO3 volume fraction. At 27°C, samples with CaCO3 volume fractions of 10%, 20%, and 30% experienced an increase in weight at saturated conditions of 1.295%, 1.715%, and 1.827%, respectively. Meanwhile, the water absorption rate graph shows that the composite follows the Fickian diffusion pattern in various types of composite specimens at aging temperatures. However, the behavior of the desorption curve may not follow the Fickian diffusion pattern. The addition of CaCO3 powder causes a significant increase in water absorption, so it is recommended to use it in composites with an optimal volume fraction. Refers to previous findings by [8] who reported irregular behavior in water absorption in composites using nanosilica fiber and kenaf fiber. This shows the importance of consistency in material composition to obtain consistent results in water absorption properties.

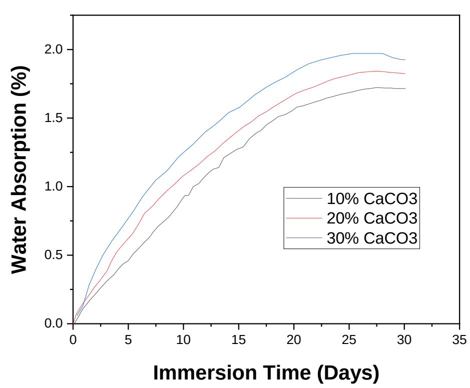

Fig 2: The relationship between soaking time and weight gain of a polyester composite at 40°c.

Figure 2 shows the increase in water absorption of polyester composites with 10%, 20%, and 30% CaCO3 at 40°C. The water absorption is affected by the immersion temperature and time, reaching a saturation point after a certain time. Composites with more CaCO3 absorb more water due to the hydrophilic nature of CaCO3. Increasing the soaking temperature (aging) of polyester composites causes an acceleration of the diffusion process in the composite, as well as increasing the time to reach equilibrium [9]. This anomaly can occur, especially for prolonged aging due to more complex interactions between water molecules and the polymer chain structure of the polyester. Water molecules interact with the polymer chain to form many hydrogen bonds [10]. In addition, the addition of excessive CaCO3 fillers (sometimes tending to agglomerate) can result in the formation of voids, reducing mechanical strength and allowing for water cluster areas, where water is absorbed and remains as free water.

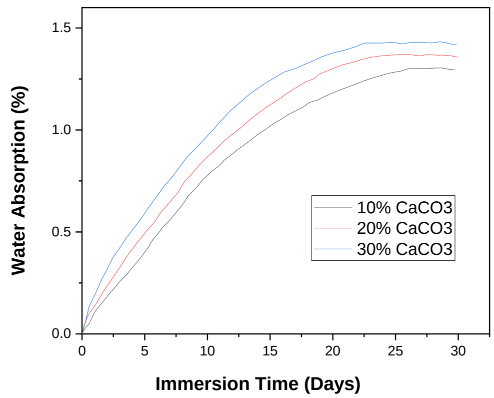

Fig 3: Water absorption of polyester composites with different aging temperatures.

The water absorption of fiberglass-reinforced polyester composites with varying volume fraction of CaCO3 filler (10%, 20%, and 30%) at 27 °C (room temperature) is shown in Figure 4.3. From Figure 4.3, it can be seen that all samples show similar absorption curves to the composite water absorption at 40°C and 50°C. Basically, there is no clear difference between the absorption curves for all samples, it is just that there is a difference in the respective weight of water absorbed. Figure 4.3 shows that the polyester-fiberglass composite with 10% CaCO3 filler weighs 1.295%, the composite with 20% CaCO3 filler weighs 1.715% and the 30% CaCO3 composite weighs 1.827%. Thus, CaCO3 particles have a significant effect on water uptake under these conditions. This may be due to the very large volume fraction incorporated into the composite. This result is different from the results presented by [11] In which, CaCO3 particles had no significant effect on the water uptake of PVC/CaCO3 composites, due to their poor solubility in water and low water diffusion in the material. However, small differences started to appear after 40 days of immersion. At this stage, diffusivity appears as an increasing function with respect to the filler content.

A different phenomenon was reported by [12]. They added a small amount of nanoparticles which tended to decrease the water uptake and diffusion rate. However, for cases where the particles are reactive to water, water uptake tends to increase as the filler content increases. Another factor is the agglomeration of fillers that form water clusters on the polymer also increases water uptake. In this study, since NCC was treated using fatty acids that tend to be hydrophobic and not reactive to water, particle agglomeration is likely to be the cause of increased water uptake, especially at high NCC content (i.e. 5% by weight).

The effect of temperature on water absorption in polyester composites with fiberglass fibers and CaCO3 filler is obvious. Compared to 30°C, the water uptake increased from 1.295% to 2.091% after 30 days of immersion at 50°C. The explanation for this sharp increase can be attributed to the higher activity of water molecules at higher temperatures. In addition, at low temperatures, polymeric materials become hard like glass, while at high temperatures, polymeric materials become soft like rubber. This makes the composite more susceptible to the penetration and attack of water molecules at 50°C, so no saturation or equilibrium level is reached sooner and water absorption continues to increase. This phenomenon cannot be explained by Fickian diffusion and indicates irreversible damage that gets worse with time and water absorption. The hydrophilic nature of CaCO3 and the larger interface may play a role in this phenomenon [11].

3.2. The Effect of Aging Temperature on Tensile Strength.

The characteristics of polyester composites are highly dependent on various parameters such as the type of reinforcement/filler, size of reinforcement/filler, environment and aging temperature. The tensile characteristic response of 25% fiberglass reinforced polyester composites with 5%, 10% and 15% Calcium Carbonate powder filler volume fraction variation is presented in table 2 below.

Table 2. Tensile strength, elongation and modulus elasticity values of composites

s	Temperature (°c)	Tensile strength (MPa)	Elongation (%)	Modulus Elasticity (GPa)
FC10	Dry	104.42 ± 5.1	4.6 ± 1.1	7.43 ± 0.67
	30	96.73 ± 7.5	6.21 ± 1.7	6.62 ± 0.6
	40	82.04 ± 6.6	5.06 ± 1.3	6.85 ± 0.47
	50	77.28 ± 7.8	5.86 ± 2.06	6.23 ± 0.56
FC20	Dry	116 ± 5.1	4.1 ±1.12	8.5 ± 0.51
	30	98.01 ± 4.5	5.04 ± 1.01	8.56 ± 0.48
	40	90.13 ± 8.3	4.13 ± 0.93	8.41 ± 0.48
	50	87.15 ± 6.6	4.08 ± 1.08	8.32 ± 0.31
FC30	Dry	102 ± 5.43	4.21 ± 1.3	10.34 ± 0.45
	30	79.87 ± 5.9	4.76 ± 1.7	9.51 ± 0.5
	40	71.63 ± 7.3	4.22 ± 2.1	9.53 ± 0.39
	50	71.02 ± 6.6	3.06 ± 1.01	9.39 ± 0.67

The tensile strength of polyester composites reinforced with 25% glass fiber, with the addition of 10%, 20%, and 30% Calcium Carbonate (CaCO3) powder filler after immersion at 30°C, 40°C, and 50°C is shown in Figure 4.4. The results show that the composite with 10% CaCO3 exhibits a tensile strength of 104.42 MPa, which is higher than the 30% glass fiber composite without filler which only reaches 50.82 MPa [13]. However, after the addition of 10% CaCO3, the tensile strength of the sample increased to 116 MPa, while with the addition of 30% CaCO3, the tensile strength dropped to 102 MPa from the previous condition.

This increase in tensile strength is related to the high integrity level of the glass fiber reinforcement in polyester with 10% CaCO3. The use of a higher volume fraction of CaCO3 (20%) reduced the tensile strength to 102 MPa, but was still higher than previously reported results [13]. In the condition with 30% CaCO3, the small decrease in tensile strength was due to a decrease in the viscosity of the polyester resin, which resulted in the resin being difficult to wet the glass fibers and many fibers were lifted [14].

These results are also consistent with research by[15] which showed that the addition of CaCO3 filler to GFRP (glass fiber reinforced polymer) composites reduced the tensile strength after immersion in water for 30 days.

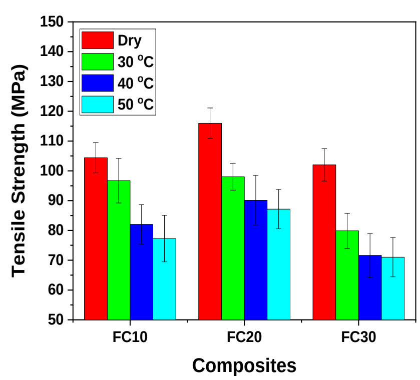

Fig 4: Tensile strength of polyester composites with variation of CaCO3 filler at different aging temperatures

The effect of soaking temperature on polyester composites with fiberglass fibers and Calcium Carbonate (CaCO3) powder filler is very visible in the significant decrease in tensile strength of the composite. This decrease is caused by damage to the interfacial bond between the components in the composite, namely reinforcement (fiberglass fibers), matrix, and CaCO3 filler, due to water penetration into the composite. Composites with 10% CaCO3 experienced a decrease in average tensile strength of 7.36%, 21.43%, and 35.12% after immersion in seawater at temperatures of 30°C, 40°C, and 50°C respectively compared to dry conditions. Composites with 20%

CaCO3 decreased in tensile strength by 15.51%, 22.3%, and 24.87%, while composites with 30% CaCO3 decreased by 21.7%, 29.77%, and 30.37% at the same temperature.

This decrease can be explained by the fact that at high temperatures, water molecules enter the polymer chain more easily, resulting in more conformation (relaxation) of the polymer molecules. These results are consistent with previous research which states that the addition of fillers in polyester composites causes a decrease in tensile strength due to stress concentration in the matrix, when the fillers gather and the matrix cannot wet the fibers because the resin cannot enter between two adjacent fibers [16].

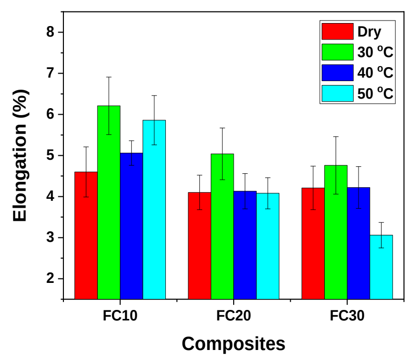

Fig 5: Elongation of polyester composites with variation of CaCO3 filler at different aging temperatures.

Figure 5 shows that the addition of Calcium Carbonate powder filler to polyester composites with fiberglass fibers provides significant strengthening and durability, but reduces the elongation or deformation of the composites. The FC10 (30 °C) composite has the highest strain of 6.21%, especially at 30 °C, which is the optimal condition for polyester to undergo molecular stretching and water diffusion easily. However, at 40°C and 50°C aging temperatures, the strain of the FC10 composite decreased to 5.06% and 5.86%. This decrease is due to the increase in matrix toughness which may be caused by the agglomeration of CaCO3 powder in the polyester matrix.

On the other hand, FC30 composites with CaCO3 content of about 30% showed an increase in brittleness, resulting in very low strain at fracture, such as in the 50 °C aging FC30 composite with elongation at fracture of 3.06%. This shows that the elongation of polyester composites with fiberglass fibers and CaCO3 filler at 30% by weight is still higher than that of composites with 10% and 20% by weight filler. However, a reduction in mobility and an increase in brittleness occurred when the CaCO3 content was increased, resulting in fewer fibers available for elongation before break.

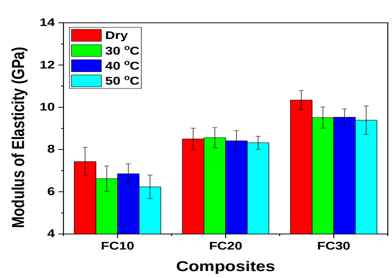

Fig 6: Elastic modulus of polyester composites with a variation of CaCO3 filler at different aging temperatures.

Figure 4.6 shows that the elastic modulus of polyester composites with fiberglass fibers and Calcium Carbonate (CaCO3) powder filler increases significantly with an increase in CaCO3 volume fraction. This increase in Young's modulus indicates that fiberglass fibers and CaCO3 can inhibit the deformation of polyester resin in the composite, maintaining tensile strength and strain (Fu et al. 1988). Under these conditions, the composite with 20% CaCO3 (FC20) has the highest elastic modulus of 9.53 GPa at 40°C, while the lowest belongs to the composite with 10% CaCO3 (FGs) at 6.23 GPa. The addition of CaCO3 filler seems to increase the embrittlement of the polyester matrix, possibly due to the formation of double hydrogen bonds between water molecules (-OH) and the molecular chain structure.

3.3. Bending Strength

From the data obtained, it can be seen that the bending strength of fiberglass composites is influenced by fiber volume fraction and temperature. The highest bending strength was found in the composite with 20% volume fraction at dry temperature, reaching 124.36 MPa, while the lowest occurred in the composite with 10% volume fraction at 50°C, which was 95.79 MPa. In addition, it is seen that composites soaked at a certain temperature have a lower bending strength compared to dry composites, this is due to debonding of the specimen surface which affects stress transfer.

The bending modulus also shows a similar pattern, where the value increases with an increase in testing temperature and then decreases when the temperature is increased again. This occurs due to the hygroscopicity effect on the matrix which has a positive effect on the stiffness of the composite, but after a point, the hygroscopicity no longer has a positive effect on the stiffness of the composite.

Table 3. Composite bending strength

Designation Sample	Temperature (°c)	Bending Strenght (MPa)	Modulus Bending (GPa)
FC10	Dry	123 ± 4.72	14.63 ± 0.5
	30	112.3 ± 4.56	14.26 ± 0.7
	40	98.51 ± 3.77	15.07 ± 0.5
	50	95.79 ± 4.5	14.01 ± 0.5
FC20	Dry	124.36 ± 2.1	17.28 ± 0.6
	30	118.82 ± 2.2	17.44 ± 0.4
	40	110.84 ± 2.6	17.41 ± 0.7
	50	103.39 ± 3.2	16.82 ± 0.3
FC30	Dry	110.32 ± 3.47	18.53 ± 0.4
	30	105.31 ± 4.64	19.17 ± 0.5
	40	98.21 ± 3.5	18.26 ± 0.6
	50	100.18 ± 4.25	18.2 ± 0.6

During the three-point bending test, it is observed that the increase in bending stress occurs from the center zone of the composite towards the outer side. The center of the composite acts as the neutral axis, while the top and bottom layers experience the maximum bending stress.

Figure 7 shows that the bending strength trend of polyester composites reinforced with glass fiber and Calcium Carbonate powder filler is affected by aging temperature. The FC20 composite without soaking has the highest bending strength, indicating a strong interfacial bond between the fiberglass fibers, Calcium Carbonate powder, and polyester matrix. On the other hand, the FC30 composites without soaking had the lowest bending strength, thought to be due to a decrease in the ability of polyester to fill the gaps in the fibers, resulting in less effective interfacial bonding.

Another study by Ruizhe Si et al showed that the addition of silicate filler can improve the bending strength of basalt fibers. The filler helps to improve the bond between the basalt fiber and the matrix, which in turn improves the reinforcing effect of the basalt fiber and the overall mechanical properties of the composite [17].

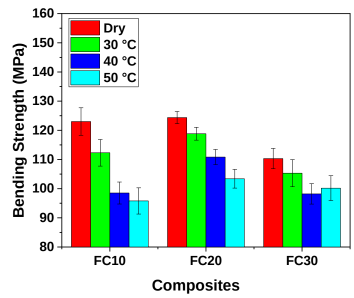

Fig 7: Bending strength of polyester composites with a variation of CaCO3 filler at different aging temperatures.

In composites subjected to immersion, there is a decrease in strength as the immersion temperature increases. The soaking process results in the absorption of water by the composite, mainly through small cavities and fibers, which is ultimately responsible for the increase in weight of the polyester composite. This results in the plasticization of the composite and modification of the internal structure. This plasticization causes damage to the bonding of the fibers with the matrix and secondary bonds between the polymer chains, which are important for the cohesion of polymeric materials[9].

Water absorption affects the mechanical cohesion of composites in two stages: first, water absorption without cracks results in a decrease in strength, and then water absorption above the saturation level causes significant crack development, which contributes to a large increase in weight and a decrease in mechanical strength of polyester composites.

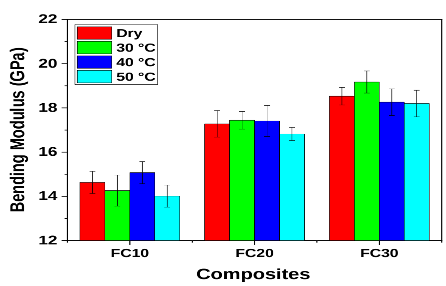

Fig 8: Bending modulus of polyester composites with CaCO3 filler variation at different aging temperatures.

Figure 8 shows that the elastic modulus of the composites increases as the fiber volume fraction increases, both in the unsoaked and soaked conditions at 30, 40, and 50 °C. However, in the FC10 Composite, there was a decrease in elastic modulus at 30 °C, then increased at 40 °C, and decreased again at 50 °C. In the FC20 and FC30 Composites, there was an increase in elastic modulus at 30°C, but then decreased at 40°C and 50°C. This shows that soaking temperature affects the elastic modulus behavior of composites with different fiber volume fractions.

3.4. Fracture Pattern of Polyester Composite

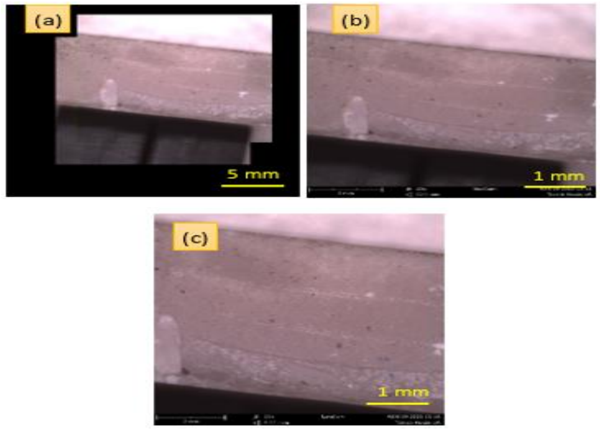

Fig 9: macro photos of polyester composite fractures with different aging temperatures; (a) 5% Calcium Carbonat powder filler, (b) 10% Calcium Carbonat powder filler, (c) 15 Calcium Carbonat powder filler.

Figure 9 shows that the fractures in the polyester composite have different patterns depending on the volume fraction of Calcium Carbonate powder. The higher the volume fraction of CaCO3 powder, the more splitting multiple fracture areas. Failure analysis of the composite samples showed fiber debonding and fiber pull out, especially in composites with 25% CaCO3 powder. In Figure 4.10c, it can be seen that these composites cannot wet the fiberglass fibers well, which is in line with the low tensile and bending test results. Although composites with multiple area faults generally have high strength, their use as building structures can make it difficult to predict the direction of crack propagation.

Fig 10. macro fracture photos of polyester composites with different CaCO3 fillers

3.5. Morphology Analysis

SEM observations in Figure 11 show that the higher the volume fraction of glass fiber, the more fibers clump together (agglomeration). This is due to the very small size of the glass fiber so the polyester matrix has difficulty entering the gaps in the fiber due to the increased viscosity due to the addition of Calcium Carbonate powder filler. As a result, the bonding of the fiber interface with the matrix is weak, and this results in a decrease in mechanical strength.

In addition, SEM observations also showed that the glass fibers failed and fiber pull-out occurred in all polyester composite samples. Water absorption by the composites accelerated the mechanical failure of the polyester composites. This failure can be attributed to the weak bond between the glass fibers, polyester resin, and Calcium Carbonate powder, as well as the voids that appeared during the non-optimal composite fabrication process.

Fig 11. SEM photographs of fracture morphology of polyester composites with different aging temperatures; (a) and (b) 15% Calcium Carbonate powder filler, (c) and (d) 5% Calcium Carbonate powder filler.

CONCLUSION

The effect of aging factors such as aging temperature on the bending tensile strength and water absorption of polyester composites has been investigated and reported. The water absorption of polyester composites is highly dependent on aging temperature and Calcium Carbonate powder filler. This greatly affects the tensile and bending strength of the polyester-calcium Carbonate powder composites. Overall, the mechanical strength of polyester composites decreased as the aging temperature of polyester composites increased. The highest tensile strength was possessed by the FGd composite without soaking at 116 ± 5.1 MPa then decreased to 71.02 ± 6.6 MPa for the FGt composite after soaking in seawater at 60 °c. The same trend occurred in the bending test results. Failure of polyester composite samples experienced debonding, fiber pulls out of glass fiber with splitting multiple area fault pattern due to the unidirectional arrangement of glass fiber. SEM morphology photos show the presence of fibers that are unable to be wetted with polyester Matrix - Calcium Carbonate powder. Finally, the aging temperature with Calcium Carbonate powder filler is the factor that determines the volume of absorbed water and mechanical properties.

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