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Degradation of Kapok Fiber-Reinforced Polyester Composites with Calcium Carbonate Filler in Seawater

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Abstracts: This research investigates the development of polyester composites reinforced with kapok fiber and CaCO3 filler to reduce environmental impact. Natural fibers, valued for their lightweight, fire resistance, cost-effectiveness, and biodegradability, are commonly used in composites. However, synthetic fibers, despite their superior mechanical properties, raise environmental concerns. The study focuses on the composites' performance, stability, and endurance in harsh conditions. Kapok fibers were manually extracted, and CaCO3 powder was sieved to 80 mesh. Composite samples were created with varying volume fractions of CaCO3 powder and kapok fibers, using polyester as the matrix. Tensile and bending tests, according to ASTM D3039 and ASTM D790 standards, were conducted on these samples. Additionally, water absorption was assessed gravimetrically, and mechanical strength degradation was studied via immersion in artificial seawater at different temperatures. Results indicated that water absorption increased with higher fiber volume fractions in the composite, primarily due to the water-absorbing capabilities of natural fibers and CaCO3 particles, surpassing that of polyester resin. Increased immersion temperatures accelerated water diffusion, impacting interactions between water molecules, polymer chains, and natural fibers. Furthermore, an increase in the CaCO3 filler volume fraction negatively affected the mechanical strength of kapok polyester-fiber composites. This was attributed to the agglomeration of CaCO3 powder, disrupting the polyester resin's viscosity and damaging the bond between kapok fiber and the matrix. The composites exhibited their highest tensile strength with 10% CaCO3 filler before immersion, but this strength decreased following exposure to high temperatures.

Keywords: Polyester, Composite, CaCO3, Kapok Fiber.

1. INTRODUCTION

The increasing use of composites in manufacturing and construction industries is driven by their versatility in combining materials with diverse properties. Composites can be classified into particle-reinforced, fiber-reinforced, and structural composites (K. Kaw, 2006). They find applications in car bumpers, vehicle bodies, airplane bodies, and boat bodies. Fiber-reinforced composites, which require a balance of strength and lightness (Dinesh et al., 2020), can use either natural or synthetic fibers.

Synthetic fibers offer superior mechanical properties but contribute to environmental concerns due to high CO2 emissions (Aruchamy et al., 2020; Sethuraman et al., 2020). In contrast, natural fibers, such as kapok, possess advantages like lightweight, non-abrasive properties, fire resistance, cost-effectiveness, and biodegradability (Ochi, 2008; Ramanaiah et al., 2012). These fibers are considered promising for green composites, where one or both components (matrix and reinforcing fiber) are bio-based. However, natural fibers have weaknesses like water absorption, low melting points, and interfacial adhesion issues (Kalia, 2009).

Researchers are extensively exploring the use of natural fibers as reinforcement to create lightweight, strong materials as alternatives to metals (Sanjay et al., 2018). Natural fibers offer advantages like abundance, easy processing, strength, and environmental decomposability (Herlina Sari et al., 2018). They also have a lower specific weight compared to glass fiber, low CO2 emissions, and biodegradability, making them ideal for sustainable applications (Indran & Raj, 2015; Sreekumar and Thomas et al., 2008).

Natural fiber-reinforced polymers are gaining prominence due to their benefits, including low weight and cost, minimal equipment damage, good mechanical properties, sustainability, abundance, flexibility, biodegradability, and reduced health risks (Arpitha et al., 2017; Bajpai et al., 2014). However, natural fiber composites are sensitive to moisture, causing significant degradation in mechanical properties compared to synthetic fiber composites due to their higher water absorption (Won et al., 2008).

To enhance the mechanical properties of natural fiber-reinforced polymer composites, researchers are incorporating nanoscale fillers. Inorganic fillers like talcum powder (TP), CaCO3 (CC), and bio-fillers like eggshell powder (ESP) increase tensile strength by reducing the crystallization index of natural fibers and minimizing the formation of new hydrogen bonds in the composite (Leite et al., 2017). These fillers also improve wear resistance, water absorption resistance, and overall mechanical properties. CaCO3 fillers, in particular, enhance the tensile modulus, microhardness, and bending strength of polyester matrix composites (Jafari & Farzaneh, 2013).

Kapok (Ceiba pentandra) fibers are widely used as natural reinforcement, but they have limitations due to high viscosity, low crosslinking density, and susceptibility to cracks in harsh environments (Sanjay et al., 2018). Detecting cracks in Natural Fiber Composites (NFC) early is crucial for structural safety and reliability (Ramesh et al., 2013). This research investigates kapok fiber-reinforced polyester composites with CaCO3 filler in a chloride-containing solution, focusing on their performance, stability, and long-term behavior in aggressive environments. Understanding how these composites perform in such conditions is vital for their practical applications.

2. METHOD

There are three types of variables in this study: independent variables, dependent variables, and controlled variables. The variation in soaking time for the polyester composite, namely 1 day, 7 days, and 15 days, is the independent variable. Tensile properties, bending strength, impact toughness, and fracture surface morphology of the composite are the dependent variables. The volume fraction of glass fiber is the variable under control. Tensile, flexural, opacity, and FESEM strength were all determined for each sample. The design of the experimental work is shown in Table 1.

Tabel 1. Experimental design of the laboratory work

Sampel	Kapok fibre (ply)	$CaCO_3$	Temperature	Exposure time (days)
A	5	5	25	1
B				7
C				15
D		10	35	1
E				7
F				15
G		15	50	1
H				7
I				15

3. Result and Discussion

3.1. Water absorption

In this research, the primary focus is on investigating the impact of varying CaCO3 filler content, soaking temperature, and duration of water absorption on the rate of water absorption and the mechanical strength degradation, including tensile and bending properties, of polyester composites reinforced with kapok woven fibers. The results of the water absorption tests conducted on the polyester-kapok composites reveal changes in the specimen's weight before and after immersion in seawater for 500 hours at different temperatures, as shown in Figures 1, 2, and 3.

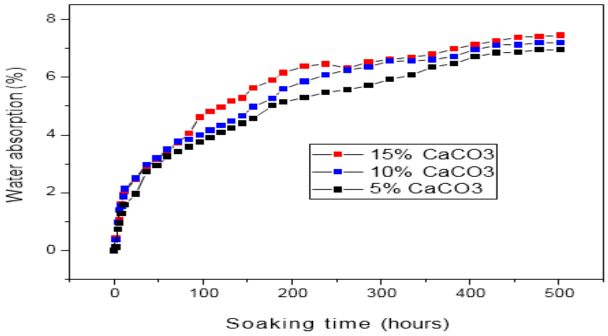

Fig 1. Water absorption of polyester-kapok composite at room temperature.

Figure 1 demonstrates that the water absorption of polyester-kapok composites with varying CaCO3 filler content at room temperature is influenced. The composite containing 5% CaCO3 filler exhibits the lowest water absorption, measuring 6.95%, in contrast to the 10% and 15% CaCO3 filler polyester-kapok composites, which absorb water to saturation levels of 7.2% and 7.46%, respectively. Initially, all CaCO3 filler fractions (5%, 10%, and 15%) in the polyester-kapok composites experience a significant increase in water absorption (around 50 hours of immersion). Subsequently, water absorption begins to decrease until it reaches saturation. The saturation point marks a condition where the composite can no longer absorb water, maintaining a constant water content (Ashori, 2010).

The water absorption in these composites is influenced by the natural fibers and CaCO3 particles, which possess higher water-absorption capabilities compared to the polyester resin itself. Natural fibers, known to absorb water by 11% - 12% (Lokantara et al., 2009), contribute to the increased water absorption in natural fiber-reinforced composites compared to pure polyester. A higher fiber volume fraction in the composite leads to greater water absorption. Additionally, the matrix-fiber bonding creates gaps that enable water to enter capillaries (Dhakal et al., 2006). Moreover, natural fibers contain lignocellulose and other impurities that readily absorb water, further elevating water absorption levels.

Figure 2 illustrates the water absorption of polyester composites immersed at 35°C, with varying 5%, 10%, and 15% CaCO3 filler content. The water absorption rates are 7.87%, 7.34%, 7.59%, and 7.87%, respectively. Comparatively, polyester composites with 5% filler exhibit the lowest average water absorption when compared to room-temperature immersion composites. This suggests that an increased percentage of CaCO3 filler in the polyester-coconut cake composite results in greater seawater absorption by the composite. This indicates that CaCO3 particles react with water, leading to increased water uptake with higher filler content (CaCO3). Additionally, factors such as filler agglomeration, which forms water clusters in the polymer, contribute to increased water absorption. Moreover, elevating the immersion temperature from room temperature to 35°C also leads to increased water absorption in the composite. Consequently, the water absorption of the composite is influenced by multiple factors, including immersion temperature, filler volume fraction (CaCO3), and the treatment of natural fibers.

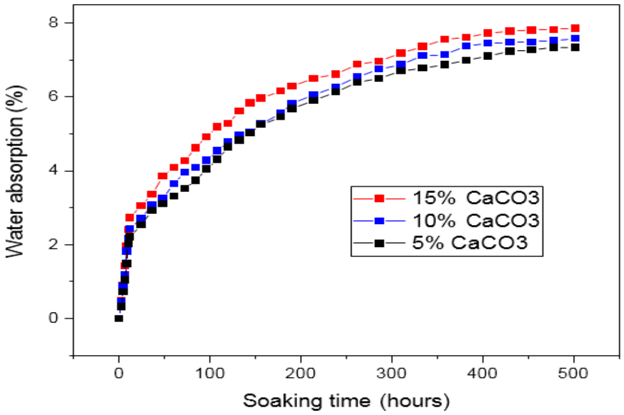

Fig 2. Water absorption of polyester composite-polyester with temperature of 35 °C

The research focused on the water absorption rate of kapok fiber-reinforced polyester composites with CaCO3 powder filler at a temperature of 50°C, as illustrated in Figure 3. The water absorption behavior of these composites adheres to Fickian diffusion across all specimen types at the aging temperature. Water absorption in composite resins follows a diffusion process, where water molecules infiltrate the spaces between resin matrix molecules, causing displacement and degradation of the resin matrix.

Figure 3 depicts the relationship between water absorption and soaking time for polyester-kapok composites. The water absorption patterns at 50°C align with those observed at room temperature and 35°C. After 500 hours of immersion, the water absorption remains constant at 7.788% for 5% CaCO3 polyester-kapok composites, while for 10% CaCO3 and 15% CaCO3 polyester-kapok composites, the constant weights are 8.157% and 8.628%, respectively. The curve between weight gain and the square root of time is linear, with water absorption at <50 hours showing a proportional relationship to the square root of time. Beyond 50 hours, water absorption starts to decelerate, indicated by a gradual decrease in the curve's gradient until equilibrium is reached.

The low water absorption in polyester-kapok composites with CaCO3 filler is attributed to the low water absorption properties of polyester resin as a composite material. Water absorption primarily occurs through kapok fibers, which have a high lignocellulose content that enhances water absorption. Elevating the immersion temperature of polyester composites accelerates the diffusion process within the composite and shortens the time to reach equilibrium. This phenomenon becomes more pronounced during prolonged aging, where complex interactions between water molecules and the polyester polymer chain structure take place.

Water molecules form numerous hydrogen bonds with the polymer chains, intensifying their interactions. Consequently, an increase in the volume fraction of CaCO3 powder filler augments both the interaction of the polymer chain structure with water molecules and the interaction between water molecules and natural fibers. Regarding the maximum permissible water absorption value according to JIS A5905-2003, which is <25% for composite boards, it is evident that all types of polyester-kapok composites with CaCO3 filler examined in this study meet the minimum water absorption standard.

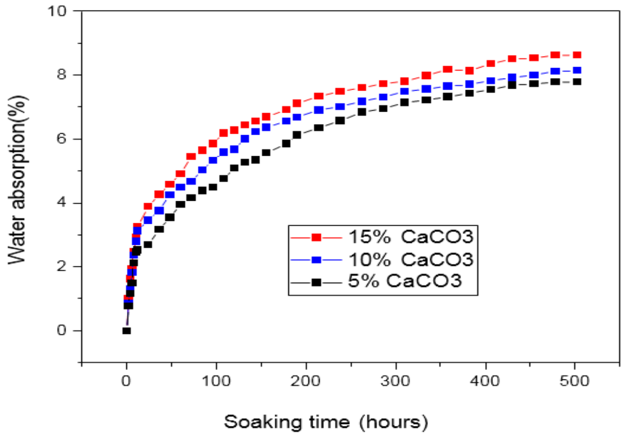

Fig. 3. Water absorption of polyester-polyester composites with a temperature of 50 °C.

3.2. The Effect of Caco3 Powder Filler and Water Absorption Temperature On the Water Absorption Rate of Composites.

The Research reveals that increasing the CaCO3 powder content in kapok fiber polyester composites leads to higher water absorption over longer soaking times. Kapok fibers have a natural affinity for water absorption, and this behavior is influenced by factors like the type of matrix and filler used. CaCO3 particles tend to agglomerate, increasing their water absorption reactivity. Smaller CaCO3 particle sizes exacerbate this tendency. Elevated soaking temperatures also intensify water absorption due to increased micro-crack formation and potential hydrolysis of unsaturated polyester chains, which enhances water molecule penetration into the composite (Aziz & Mohamed, 2016).

3.3. The Effect of Soaking Time and Temperature On Tensile Strength

Table 2 The value of tensile strength, elongation and elastic mosulus of composites.

Sample	Tensile strength (MPa)	Elasticity modulus (MPa)	Elongation (%)
KH5F	50.285 ± 5.03	2587 ± 237	2.4986 ± 0.53
KH10F	56.285 ± 7.22	2877.6 ± 261	2.7738 ± 0.27
KH15F	52.394 ± 2.97	2828 ± 304	2.9988 ± 0.34
K1H5F	48.125 ± 4.81	2573.2 ± 273	2.8361 ± 0.27
K1H10F	45.392 ± 2.18	2870.6 ± 264	2.3481 ± 0.38
K1H15F	41.083 ± 4.37	2801 ± 188	2.083 ± 0.52
K7H5F	49.54 ± 4.8	2251,3 ± 293	2.8834 ± 0.28
K7H10F	48.451 ± 6.6	2482 ± 285	2.6432 ± 0.51
K7H15F	43.624 ± 5.1	2580.6 ± 290	1.9872 ± 0.24

K15H5F	43.098 ± 6.53	2082.4 ± 302	2,1481 ± 0.31
K15H10F	37.6 ± 3.49	2260.8 ± 264	2,1986 ± 0.53
K15H15F	35.184 ± 4	2252 ± 203	2,1983 ± 0.24

The results shows that polyester composites reinforced with kapok fibers and CaCO3 powder filler exhibited varying tensile strengths. The highest strength was in composites containing 10% CaCO3 powder, indicating excellent fiber-matrix-filler interaction, as shown by minimal fiber deformation or voids. However, increasing filler content beyond 10% led to reduced tensile strength due to lowered resin viscosity and interface bond disruption. Prolonged soaking also weakened tensile strength, as water damaged the interface. These findings align with prior research on filler effects in polyester-fiber composites.

Moudood et al. investigated the impact of fiber moisture on jute fiber-reinforced epoxy composites. They found that excessive moisture weakened the fiber-matrix interface, increased microstructural porosity, but optimal tensile strength occurred under low humidity conditions. Water molecules enhanced fiber tensile strength but adversely affected the interface and overall microstructure. Water also increased strain at break and decreased Young's modulus due to its plasticizing effect on natural fibers. Similar results were reported by Sari et al. with different materials, linking immersion time of corn husk fiber-reinforced polyester composites to reduced interfacial bonding due to water penetration.

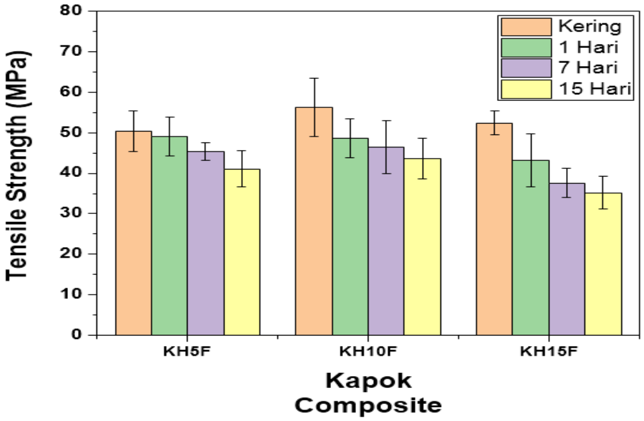

Fig. 4. Tensile strength of polyester composites with different immersion temperatures.

Shahbakhsh et al. discovered that a 5% CaCO3 volume fraction provided optimal mechanical properties in composites. Adding 5% CaCO3 by weight significantly improved tensile, flexural, and compressive moduli compared to carbon fiber/epoxy composites. This enhancement stemmed from restricted polymer chain mobility due to CaCO3 treatment, as well as reactions between amino groups on CaCO3 nanoparticles and epoxy groups in the matrix. Additionally, the higher modulus of CaCO3 nanoparticles contributed to the composite's increased elastic modulus.

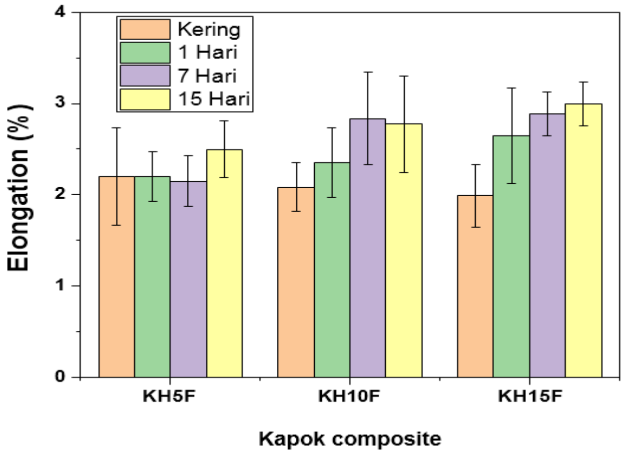

Fig. 5. elongation of polyester composites with different immersion temperatures.

The addition of CaCO3 powder filler in polyester composites reduces deformation. The highest strain, 2.99%, was in K15H15F composite at room temperature. Unsoaked polyester composites had the highest average elongation due to optimal stretching conditions. Water facilitated stretching in these composites. CaCO3 powder improved composite ductility but also increased fiber mobility during fracture, as observed in KH15F composite. See Table 1 and Figure 5 for elongation values of each composite sample.

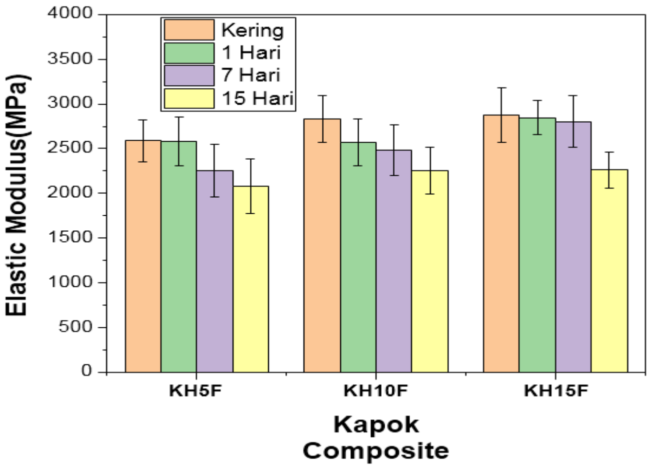

Fig 6. elastic modulus of polyester composites with different soaking temperatures

In kapok composites with CaCO3 filler, the load is focused on the fiber ends dispersed in the matrix, resulting in weakening of the fiber-matrix interface. The elastic modulus of polyester composites increased significantly with an increase in CaCO3 volume fraction from 5% to 15%. This indicates that kapok fiber and CaCO3 are able to reduce polyester resin deformation and tensile strain. K0H15F (15% CaCO3) has the highest elastic modulus, 2877.6 MPa, while KH5F (5% CaCO3) with 15 days immersion has the lowest modulus, 2082.4 MPa. The stiffness of the composite is influenced by the water absorption rate of kapok fiber which is still higher than that of polyester-CaCO3.

3.3. Bending Strength

Testing the bending strength of polyester composites using the three-point bending method with three repetitions on each specimen variation. The aim is to obtain the average test results and standard deviation, which are then displayed in Table 3.

During the three-point bending test, the bending stress increases from the center of the composite to the outside, with the center zone of the composite being at the neutral axis. Figure 4.7 shows that the polyester composites reinforced with Kapok fiber and CaCO3 powder filler variations without immersion in seawater have the highest bending strength, especially the KH10F composite with a bending strength of 78.755 ± 7.6 MPa. This is due to the strong interfacial bonding between kapok fiber - CaCO3 powder and polyester matrix. However, in dry condition, the composite experienced a decrease in bending strength when the volume fraction of CaCO3 powder reached 15% in the polyester composite, to 72.917 MPa from the previous (10%

CaCO3) which reached 78.755 MPa. This is thought to occur because the ability of polyester to fill fiber voids/gaps decreases, causing poor interfacial bonding (Sapuan et al. 2020; Sari et al. 2021) so that the flexural and tensile strength of the composite decreases as well, this has been confirmed by the observation of macro photos and SEM.

Table 3 Bending strength of polyester-kapok composite with CaCO3 filler.

Sample	Bending strength (MPa)	Bending Modulus (MPa)
KH5F	68.266 ± 8.32	3982.5 ± 279
KH10F	78.755 ± 5.68	4226.8 ± 428
KH15F	72.917 ± 7.6	4782.4 ± 296
K1H5F	67.594 ± 6.53	3894.3 ± 462
K1H10F	69.352 ± 6.42	3571.6 ± 324
K1H15F	61.273 ± 4.89	4255.9 ± 445
K7H5F	66.316 ± 4.87	3624.7 ± 345
K7H10F	67.265 ± 7.1	3474.12 ± 388
K7H15F	55.092 ± 5	3762.4 ± 383
K15H5F	61.548 ± 5.48	3473.6 ± 438
K15H10F	60.164 ± 6.14	3250.8 ± 463
K15H15F	49.734 ± 8.33	3751.6 ± 453

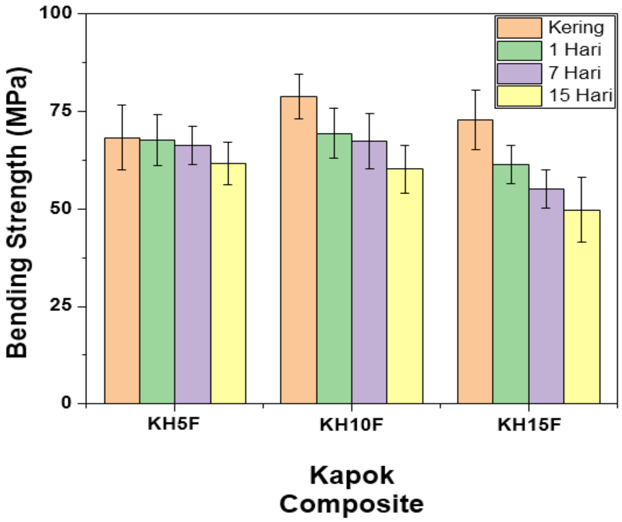

Fig. 7. Bending strength of kapok reinforced polyester composites with different aging temperatures.

Composites with high CaCO3 fillers and immersed in water generally experience a decrease in tensile properties, except for sample KH5F after 1 and 7 days of immersion which has a bending strength stress almost the same as the dry one. Water can make the fiber swell, fill the gap between fiber and matrix, and improve mechanical properties (Boubakri et al. 2009). However, overall, water absorption weakens the bonding of the fiber to the matrix, affects cohesion, and results in cracks. Water can also form hydrogen bonds, affecting composite dimensions and interfacial bonds, causing a decrease in mechanical properties. Water absorption is divided into free water and bound water, and these interactions affect the mechanical properties of the composite.

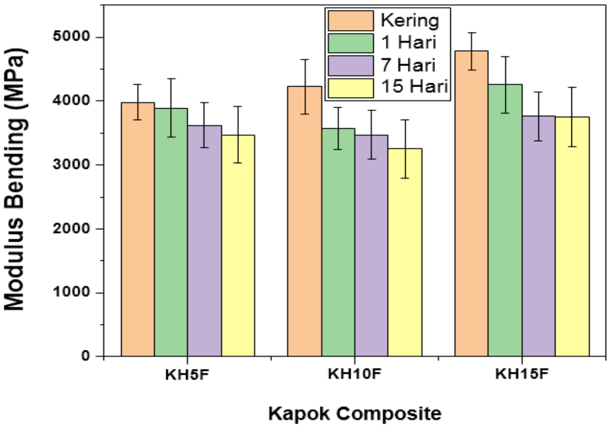

Fig 8. Bending modulus of kapok reinforced polyester composites with different aging temperatures.

Figure 8 shows the bending test results of polyester composites before and after immersion in seawater at various temperatures. Pure polypropylene has limited water absorption and does not undergo significant mechanical changes. However, polypropylene composites with wood powder have higher water absorption, resulting in mechanical degradation due to fiber swelling and interface and matrix damage. Water absorption also affects the stress transfer between the wood powder and the polymer matrix.

3.4. Fracture Pattern of Kapok Fiber-Polyester Composite with Caco3 Filler

Figures 9, 10, and 11 show the fracture of polyester-kapok composites without immersion, after 7 days, and 15 days of immersion, respectively. Composite fracture occurred more frequently in kapok fibers with high volume fraction content of CaCO3 powder, especially in 90° fibers. Failure analysis indicated kapok fiber failure and fiber pull-out, especially in the kapok fiber arrangement in the unidirectional (0°) position. The bond between the fiber and the matrix is critical to the mechanical properties of the composite, and the strength of the interface is affected by the water absorption and wettability of the fiber. A weak interface can lead to composite damage (Clyne & Jones, 2001; Wang et al., 2002).

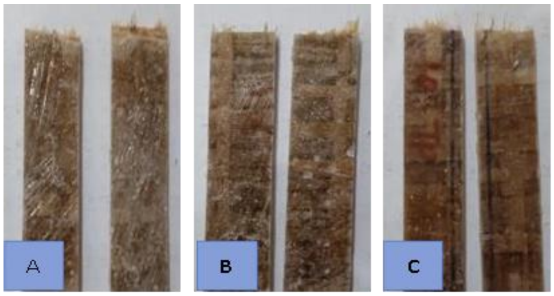

Fig. 9. Polyester-kapok composite specimens with no immersion; (a) 5% CaCO3, (b) 10% CaCO3, (c) 15% CaCO3.

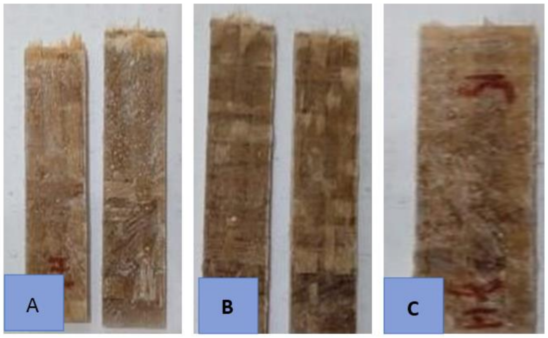

Fig 10. Specimens of polyester-kapok composite with 7 days of immersion; (a) 5% CaCO3, (b) 10% CaCO3, (c) 15% CaCO3.

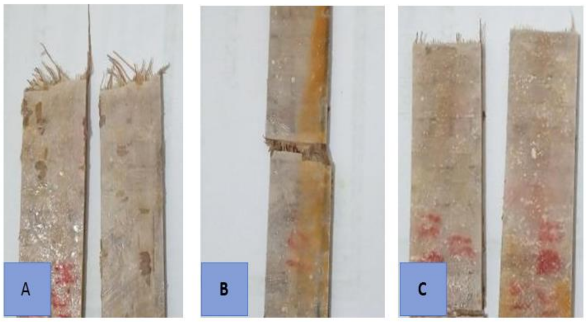

Fig 11. Specimens of polyester-kapok composite with 7 days of immersion; (a) 5% CaCO3, (b) 10% CaCO3, (c) 15% CaCO3.

3.5. Fracture Morphology Analysis (SEM).

SEM observations of the tensile tested samples without immersion, after 7 days, and 15 days of immersion are depicted in Figures 12, 13, 14. Figure 12 shows that increasing the CaCO3 powder content results in agglomeration that weakens the polyester-CaCO3- cotton fiber interfacial bond. This results in a decrease in mechanical strength, with optimal conditions at 10% CaCO3. Sample KH10F has the highest tensile strength.

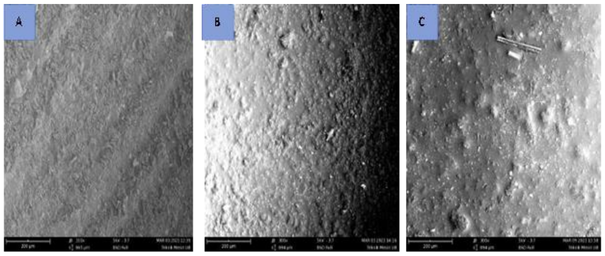

Fig. 12. SEM photographs of the fracture morphology of polyester composites without soaking with fillers (a) 5% CaCO3, (b) 10% CaCO3 and (c) 15% CaCO3.

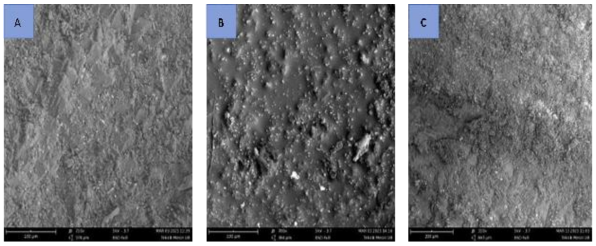

Fig. 13. SEM photographs of the fracture morphology of polyester composites immersed for 7 days with fillers (a) 5% CaCO3, (b) 10% CaCO3 and (c) 15% CaCO3.

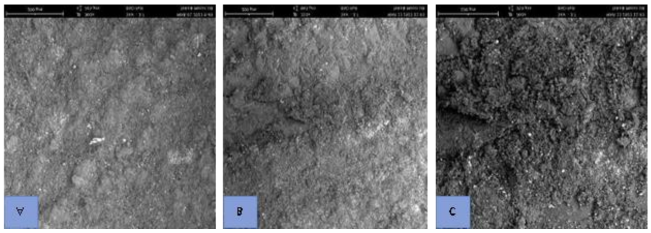

Fig. 14. SEM photographs of the fracture morphology of polyester composites immersed for 15 days with fillers (a) 5% CaCO3, (b) 10% CaCO3 and (c) 15% CaCO3.

CONCLUSION

This research investigates the effect of aging temperature on kapok fiber-polyester composites with CaCO3 filler. The water absorption, tensile and bending strength of the composites were strongly influenced by the aging temperature and CaCO3 content. In general, the mechanical strength of the composites decreased as the aging temperature increased. The KH10F composite without soaking has the highest tensile strength (56.285 MPa), while the KH15F composite with soaking at 50°C has the lowest strength (35.184 MPa). The fracture pattern of the composites is straight with 90° fiber spacing. SEM morphology showed CaCO3 agglomeration and a less dense surface with increasing CaCO3. Overall, aging temperature with CaCO3 affects the water absorption and mechanical properties of the composites.

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