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Alkali Treatment and Concentration Effects on Epoxy/Agel Fiber Composite Mechanical Properties for Marine Engineering Use

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***Abstract*—** The development of polymeric composite materials in shipbuilding has increased tremendously. To improve the sustainability of ship hulls, natural fibers have gained increasing attention as an alternative reinforcement material in polymer composites due to their renewability, low cost, and favorable specific mechanical properties. One of the natural resources that can be used as fiber reinforcement is agel leaf fibers (ALF). ALF is harvested from *Corypha gebanga* plant that is available throughout Indonesia. However, the inherent hydrophilicity and presence of non-cellulosic constituents on natural fiber surfaces often result in poor interfacial bonding with hydrophobic polymer matrices, limiting composite performance. This study investigated the effect of NaOH concentration and alkali treatment duration on the tensile properties of agel fiber-reinforced epoxy composites. Agel fibers were treated with NaOH solutions at concentrations of 1%, 2%, 3%, 4%, and 5%, with immersion durations of 1, 2, 3, and 4 hours, then rinsed with distilled water until reaching a neutral pH (7.5–8.5). The treated fibers were used as reinforcement in epoxy matrix composites fabricated using a self-made resin transfer equipment. Tensile testing was conducted at a loading speed of 5 mm/min to evaluate tensile strength and elongation at break for each treatment combination. Results showed that both NaOH concentration and treatment duration influenced mechanical performance in different manner. The mechanical properties of composites improved with longer duration. However, the effect of alkali concentration is more complex. The strength and stiffness behaves inversely with ductility. So the optimum parameter of ALF's alkalization should be decided carefully depending on the priority of the mechanical characteristics required in the application. Based on the dataset obtained from the experiment, it is suggested that alkali treatment in 1% NaOH for 4 hours, yields a good balance of tensile strength, modulus of elasticity, and maximum elongation among all combinations.

Keywords—agel leaf fiber (ALF); alkali treatment; epoxy composites; mechanical properties; NaOH concentration

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I. INTRODUCTION

Ships are the primary means of transportation connecting not only islands but also areas bordered by rivers. In the past, water transportation was made of wood due to its abundant availability and easy fabrication. Even today, wooden ships are still widely used, especially by fishermen and for short-distance crossings (rivers, lakes, and short straits) [1]. Wooden ships that were previously rowed now use motorized engines. With the increasing population, the demand for wooden ships has also increased. This massive growth has a negative impact on the environment because wood takes a long time to be harvested before being used as raw material for ship hulls. Excessive tree felling can disrupt the balance of the ecosystem. Therefore, various efforts are being made to find alternative raw materials to replace wood. One of these is the development of polymeric composites. Ship hulls made of fiberglass-reinforced plastic (FRP) composites have been fabricated to improve the fishing abilities [2]. FRP ship hulls show a lack of leakage issues and biofouling attack, thus increasing usage life.

FRP is classified as a synthetic fiber made of melted glass. Along with carbon and aramid fiber, synthetic fibers are used because they have good mechanical properties. However, synthetic fiber bring up new environmental issues due to their non-biodegradable nature. Consequently, researchers return to natural fiber, such as pineapple leaf, banana branch, ramie, and bamboo, due to their biocompatibility and biodegradability. Even though natural fibers are susceptible to microorganisms and are not suitable for a hydrophobic matrix, they exhibit high strength-to-weight ratio [3]. Furthermore, the source of natural fibers are abundant and relatively cheap, compared to synthetic fiber.

Natural fiber can be classified into cellulose-rich extracted from plant, protein-based originally made from animal (silk, wool), and mineral-based such as basalt fiber and wollastonite) [4]. This research will mainly talk

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about cellulosic plant-based natural fiber. The plant-based fiber is also known as lignocellulosic fibers because of the content of cellulose and lignin. Lignin, together with hemicellulose are the amorphous matrix embedding the crystalline cellulose. So, the mechanical properties of natural fibers (plant-based) is based on the crystallinity of cellulose content. To eliminate the lignin and hemicellulose, alkaline treatment with sodium hydroxide is often done, resulting higher crystallinity content, higher hydrophobic degree (higher surface contact angle), and better surface roughness. Immersion in 5 wt% NaOH has improved the interfacial compatibility with the hydrophobic epoxy, resulting better tensile, flexural, and impact strength as reported for epoxy-Typha fiber composites [5]. Further improvement of surface properties can be obtained by giving silane treatment to the alkalized fiber. The specific tensile strength of alkalized jute-reinforced composite was improved by 5% and 10% silane treatment. However, similar silane treatment decreased the specific tensile strength of alkalized-ALF-reinforced composites [6].

Indonesia is the house of various natural fibers, among of them is agel fiber. The fiber is extracted from the leaves of palm tree (Corypha gebanga) that is easily found throughout Indonesia, especially in Bali, Java, and Lombok. Agel leaves are traditionally used as roof materials, rope, fashion item (hats, bags), furniture, and other handicrafts. Agel leaf fiber (ALF) is extracted mechanically from the harvested leaves to separate the fiber bundle from soft tissue. Then, the fiber is washed and dried before subjecting into chemical treatment which is normally done to modify the surface properties. ALF reinforced polymer composites has been developed for structural application such as body material for unmanned aerial vehicle [7] and for the hull of fishing boat [1].

The commonly used of synthetic composite matrix, such as epoxy and unsaturated polyester have hydrophobic surface that is more suitable for hydrophobic synthetic fiber. In contrast, natural fiber has hydrophilic nature due to large amount of hydroxyl group, causing them to have poor surface interaction with polymeric matrix. Heat treatment using hot steam is commonly used to minimize the hydrophilicity by increasing the surface contact angle on bamboo [8] and wood [9]. Alkaline treatment using potassium hydroxide (KOH) has been reported to successfully remove the hydroxyl group and the amorphous lignin and hemicellulose of ramie fiber [10]. Sodium hydroxide is another alkaline used often in chemical fiber treatment. Tensile testing was done on untreated and treated ALF by NaOH at various concentrations and duration. The highest tensile strength for fiber bundle is 1464 MPa and was obtained for ALF treated by 4 wt% NaOH for 1 hour. Longer duration of immersion in alkali solution can cause damage to the fibers, resulting lower mechanical strength [11]. Immersion of knaf fiber in 6 wt% of NaOH for 24 hours produced lower tensile strength for the nude fiber and the respective composite even though those method successfully produce impurities-free fiber [12].

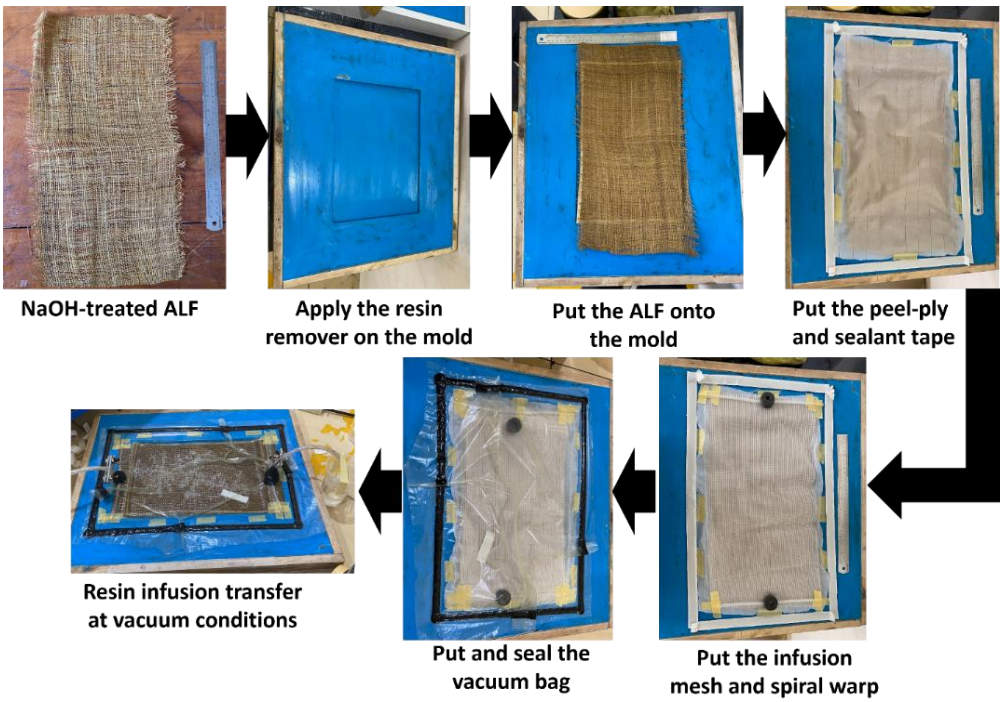


Figure 1. Fabrication of epoxy/ALF using resin infusion transfer method

Despite the reports of mechanical properties's improvement on ALF by alkali treatment, most existing studies have focused on single-fiber mechanical characterization or have tested on limited range of immersion; short-time or very long time of immersion (12-24 hour). A systematic investigation on more practical range of NaOH concentration and immersion durations (1-4 hours) to the surface compatibility with hydrophobic polymeric matrix has not been well discussed. Moreover, the combined effect of both parameters to the ALF reinforced epoxy composite, rather than single fiber level, has not been

comprehensively reported.

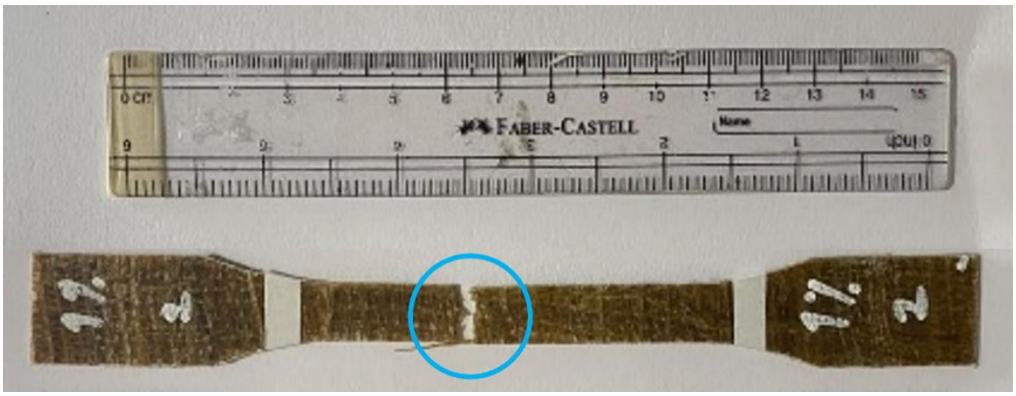


Figure 2. Tensile testing specimen

So, the objective of this study is to investigate the effect of NaOH concentration (1%, 2%, 3%, 4%, and 5%) and treatment duration (1, 2, 3, and 4 hours) on the mechanical properties of ALF-reinforced epoxy composite. The results will provide a practical procedure to optimize the parameters to fabricate the ALF-reinforced composite into a structural application product.

II. Method

A. Fiber Preparation

Alkali treatment was done to remove the impurities and the amorphous structure of lignin and hemicellulose from the cellulosic AFL. NaOH solutions were prepared by dissolving into distilled water at 1%, 2%, 3%, 4%, and 5% of weight. The woven ALF were cut into 35×25cm in size before being immersed into the solution for 1, 2, 3, and 4 hours. The woven ALF were then rinsed with distilled water until the pH is netral (7.5 – 8.5). After rinsing, alkalized ALF were dried in the oven at 60°C for 4 hours. Before use, ALF were kept in airtight storage with silica gel to avoid excessive humidity.

B. Composites Fabrication

The polymer matrix composite was prepared using the vacuum resin infusion method (Error! Reference source not found.). The ALF was stacked on top of the mold in a single direction, where the warp thread is in the 0° direction. Prior to stacking, the mold was greased to ease the collection of finished products. Then, peel-ply fabric, infusion mesh, and vacuum bag were put on top of the ALF fabric, respectively. The stack was connected to one inlet and one outlet for the resin flow. The mixture of epoxy resin and its hardener was transferred onto a mold made of silicon rubber by suction pressure of -0.5 bar. The resin infusion was done for 5 minute to ensure the wetting process for all parts of woven fiber while the vacuum condition was maintained during 24 hours curing process. The mass fraction of reinforced fiber can be calculated as the ratio of fiber's mass to the total mass of composites [13].

C. Mechanical Characterization

Tensile testing was executed using Universal Testing Machine with capacity of 10 kN. The ALF-epoxy composites were cut following the ASTM D638 about tensile testing for reinforced-polymeric composite materials in the warp thread direction (0° direction). A specific testing standard is used to minimize any external effects. Each sample has a gauge length and width of 80 mm and 10 mm. Tensile testing was repeated 3 times to ensure data reliability. The tensile testing was done with a 5 mm/s loading velocity. Observation using light microscope was done to investigate the presence of fabrication defects such as void and the cause of composite failures.

D. FTIR Characterization

The agel leaf fiber (ALF) was characterized by Fourier Transform Infra-Red (FTIR) Thermo Scientific before and after soaking in alkali solution. The fiber samples were cut into small pieces so it can fit the sample holder. The infrared spectra were recorded at room temperature in the region of 400 – 500 cm-1. Chemical characterization using FTIR was done to evaluate the effectiveness of alkali treatment on the chemical structure of agel fiber.

III. Results and Discussion

A plate of epoxy/ALF agel composite was successfully made using vacuum resin infusion method. Each composite plate consists of 4 plies of woven fiber. The average mass ratio of ALF is 35 wt%. Prior to composite fabrication, the ALF undergoes alkali treatment with NaOH 5 wt% with various immersion duration (1; 2; 3; 4; and 5 hours) as several precedent study recommend [13]–[15]. **Figure 2** showed the epoxy/ALF composite sample condition after the tensile test. The fracture occurred in the middle of the gauge length. The fracture line is perpendicular to the loading direction or along the direction of weft fiber. It demonstrated that the cause of failure is mainly due to tensile stress rather than interlaminar shear [16]. The fracture area indicated a fibrous surface where the fiber was pulled out of the matrix. The matrix failed first then the load was transferred to the fiber.

Figure 4 showed the fracture area from the side view and top view. The side view of the fracture indicates a small portion of delamination or the separation between matrix and fiber reinforcement. Delamination is commonly caused by weak interfacial adhesion between

the epoxy matrix and ALF [17]. The interfacial shear stress transfer from the continuous matrix to the woven fiber is unsufficient, causing the stress to be unevenly distributed to the fibers. Meanwhile, the top view shows visible protruding fiber bundles out of the matrix. The fiber is pulled out due to debonding at the fiber-matrix interface. Fibers were sliding out of the matrix and protruding above the fracture planes [18]. Consequently, the failure occurred before reaching the maximum stress of the fiber. It implies that the alkali treatment only partially eliminated the impurities such as hemicellulose and lignin, causing difficulties in resin impregnation.

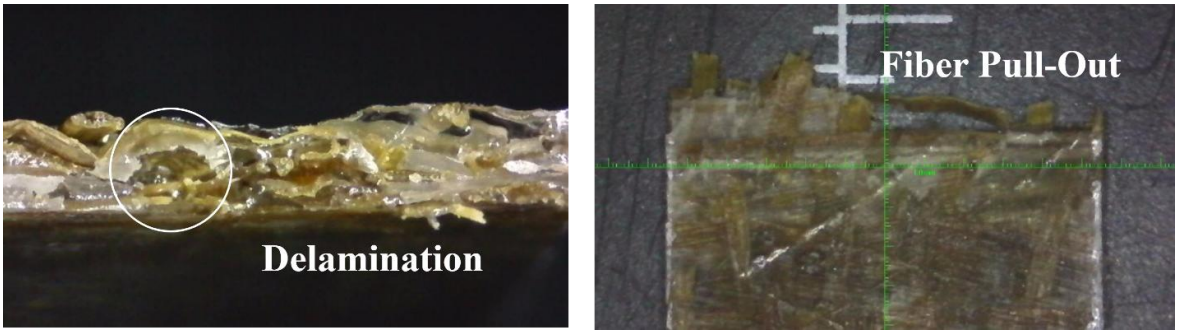


Figure 3. Section area (left) and fracture plane (right) of epoxy/ALF composites

During tensile testing, the gradually increasing load and the specimen grip displacement were recorded. The load was then converted into stress (MPa) and the displacement was used to compute the materials' strain. The stress – strain curve of epoxy/ALF can be seen in **Figure 4**. All epoxy composites reinforced by ALF treated at various durations and concentrations give a similar curve pattern. The stress – strain graph of epoxy/ALF composites gives bilinear or two elastic regions separated by a plateau, called the knee-point, resulting in a yield point that is well observed on the graph. The slope of each region is different. The slope for the first elastic region is less steep than the latter. This is a typical shape for natural fiber composites made by the resin infusion technique [19]. At lower load, composites' behaviour follows the Rule of Mixtures, where the load is distributed to both fiber and matrix in a proportion equivalent to the composition percentage. Since the polymeric matrices have lower strength, they are unable to withstand higher loads and fail before the fiber. Then the load shifts to fibers, resulting in a plateau shape normally recognized as a yield point. The yield point for all samples was found at a low value of stress-strain. All samples failed abruptly without a noticeable plastic region, demonstrating brittle failure.

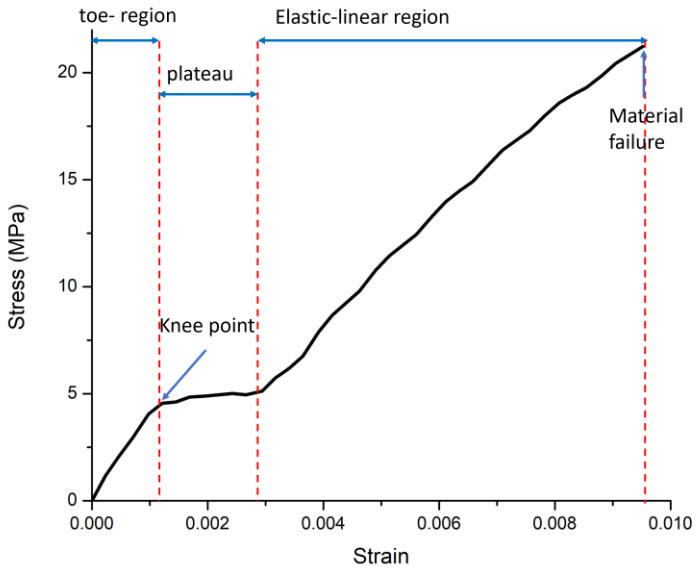


Figure 4. Example of stress – strain curve of epoxy/ALF composites

Another possibility of the "knee-point" is the phenomenon of fiber straightening that is specifically found on long-fiber-reinforced composites fabricated by the resin infusion method, as found in composites reinforced by woven flax fiber [20]. The long fiber is often found with kink bands or slip plane defects that can influence the stress transfer after matrix failure. Even though flax is classified as a bast fiber and ALF is a leaf fiber [4], a similar phenomenon can be seen as both fibers used are chemically treated and have a main composition of cellulose. The elastic region after the knee-point corresponds to the fiber's loading or strain hardening. The material failure happens when both matrix and fiber reinforcement cannot withstand a higher load.

Error! Reference source not found. shows the ultimate tensile stress, maximum elongation, and modulus of elasticity of epoxy composites whose fibers are treated in NaOH 5 wt% for various durations (1 – 4 hours). At a constant concentration of NaOH, the tensile strength and modulus of elasticity of composites show an increasing trend with longer soaking duration. But the maximum elongation remains unchanged. It can be interpreted as alkali exposure does not influence the ductility of composites as much as strength and rigidity. Longer duration of immersion causes more impurities to be eliminated, leaving the crystalline cellulose [20]. The fiber surface becomes rougher after alkalization, improving the surface contact with the matrix [21], resulting in better tensile strength. The maximum duration of alkali treatment experimented in this research is not excessive to avoid cellulose degradation that can occur at more than 12 hours of immersion [11]. Among our set of samples, the composites using ALF with 4 hours of immersion obtained the highest tensile strength. It is similar to the optimum alkalization parameter found for *Borassus flabellifer* leaf fiber [22] and sugar palm fiber as reinforcement for epoxy matrix [23].

Mechanical properties improvement is achieved due to physical surface change. Longer alkali exposure continues to increase fiber surface roughness and cellulose crystallinity. A slight dip in tensile strength for ALF treated for 3 hours is a common phenomenon found in alkali-treated natural fiber composites. It is likely due to a transitional balance between the positive effect of impurity removal and early-stage microfibril disruption, as long duration (above 12 hours) damages the cellulose chain [11]. The sign of cellulose microfibril degradation can be seen through relatively constant strength at a medium duration of soaking (5 – 8 hours)[23].

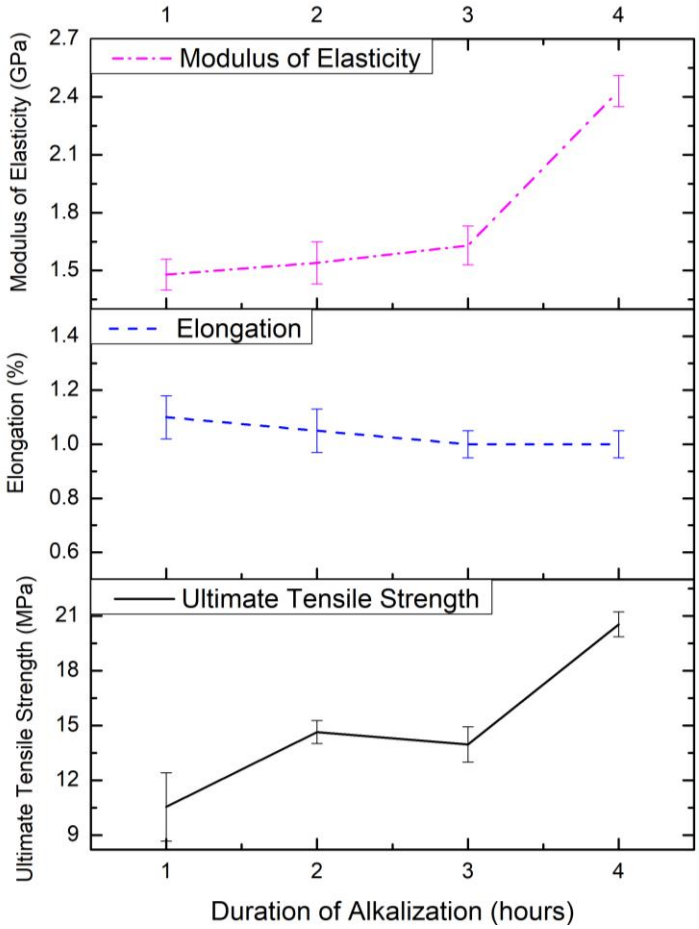


Figure 5. Modulus of elasticity, maximum elongation, and ultimate tensile stress of epoxy composite reinforced by ALF treated in NaOH 5 wt% for various durations

Based on the previous explanation, 4 hours of alkali treatment gives the optimum soaking duration to achieve the best mechanical properties. Figure 6 shows the mechanical properties of epoxy composite reinforced by ALF treated in various concentrations of NaOH for 4 hours of immersion. The effect of alkali concentration is more complex than immersion duration. The tensile strength progressively decreases at lower concentrations of 1 – 3 wt%, then rises sharply at 5 wt%. At low concentration, impurities such as hemicellulose and lignin are removed partially, causing surface irregularities. This is consistent with a previous report that found the tensile strength is inconsistent with the increase of alkali concentration [24]. A sudden jump in tensile strength at 5 wt% can be justified by a previous report that recommends a similar concentration [13]. A higher concentration of NaOH eliminates more amorphous lignin and hemicellulose, resulting in a high tensile strength and lower density, indicating a better alkalization effect [25]. Due to the elimination of soluble substances, the mass ratio of cellulose is improved, as

seen in composites reinforced by treated ALF in 5 wt% NaOH.

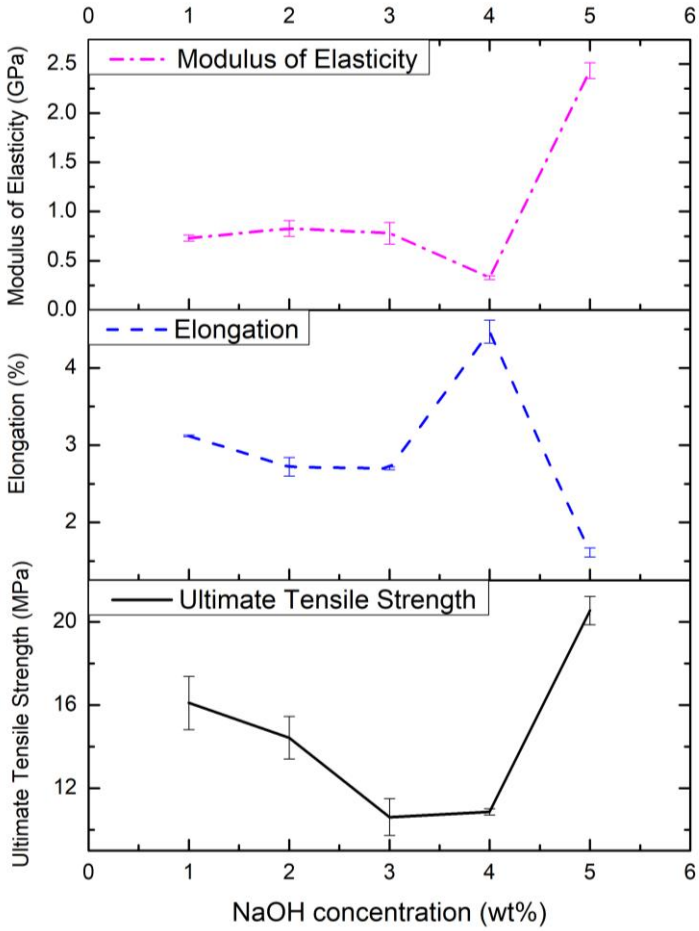


Figure 6. Modulus of elasticity, maximum elongation, and ultimate tensile stress of epoxy composite reinforced by ALF treated in various concentration of NaOH for 4 hours immersion

Similar to tensile strength, the modulus of elasticity also gives a jump at 5 wt% NaOH. It can be seen as a transition into higher fiber stiffness once the amorphous hemicellulose is removed. It is in agreement with a sudden drop in elongation indication a shift into brittle behaviour of the fiber. The elongation behaves inversely compared to strength and modulus of elasticity. It is commonly known that rigid materials tend to be more brittle. Even though treatment at 5 wt% NaOH improves the strength and rigidity, increasing the NaOH concentration beyond the threshold may induce embrittlement. Then it suggest that the maximum concentration for alkali treatment is 4 wt%. The complete data of tensile properties of epoxy/treated-ALF are presented in **Table 1**.

TABLE 1
COMPLETE DATA OF TENSILE PROPERTIES OF EPOXY/ALF COMPOSITES

Materials	Concentration of NaOH (wt%)	Immersion Duration (hours)	Ultimate Tensile Strength (MPa)	Maximum Elongation (%)	Modulus of Elasticity (GPa)
1H-5%	5	1	10.54 ± 1.87	1.10 ± 0.08	1.48 ± 0.08
2H-5%	5	2	14.64 ± 0.63	1.05 ± 0.08	1.54 ± 0.11
3H-5%	5	3	13.96 ± 0.96	1.00 ± 0.05	1.63 ± 0.10
4H-5%	5	4	20.54 ± 0.68	1.00 ± 0.05	2.43 ± 0.08
4H-4%	4	4	10.86 ± 0.15	3.67 ± 0.25	0.33 ± 0.02
4H-3%	3	4	10.61 ± 0.89	2.25 ± 0.03	0.78 ± 0.11
4H-2%	2	4	14.43 ± 1.02	2.26 ± 0.15	0.83 ± 0.08
4H-1%	1	4	16.1 ± 1.28	2.36 ± 0.07	0.73 ± 0.03

Even though the maximum limit of concentration in alkali treatment of agel is found to be 4 wt%, it is recommended to use 1 wt% of NaOH. The strength-ductility trade-off should be considered carefully to avoid material brittleness and to obtain a balance between both mechanical properties. The 1 wt% of

NaOH is able to balance all mechanical properties, obtaining both good strength, stiffness, and ductility, though not at the best value.

The phenomenon found at 5 wt% where the tensile strength and modulus of elasticity increased sharply while the elongation dropped indicates that different mechanical properties can respond differently to the alkali concentration. The optimum concentration can be decided based on which property is prioritized for the application (strength vs ductility). Given the context of marine engineering applications such as ship hulls, both high strength and high ductility are required. Combining the two experiments of duration and concentration variation, it suggests that alkali treatment for agel leaf fiber is done at 1 wt% NaOH for 4 hours to balance the tensile strength, maximum elongation, and modulus of elasticity.

IV. CONCLUSION

This study confirms that alkalization duration and NaOH concentration influence the mechanical performance of Agel Leaf Fiber (ALF)-reinforced epoxy composites through distinct mechanisms: longer immersion time steadily improved strength and stiffness without sacrificing ductility, whereas concentration exerted a more complex, threshold-dependent effect in which gains in strength and rigidity beyond a certain level came at the cost of increased brittleness, confirming that concentration is the more decisive and sensitive parameter governing the fiber-matrix interface. Given that marine structures such as ship hulls require both load-bearing strength and resistance to cyclic wave-induced stresses, alkalization at 1 wt% NaOH for 4 hours is recommended as the most application-appropriate protocol, since it provides a balanced mechanical profile rather than the isolated highest strength value; however, as fractographic evidence revealed persistent interfacial debonding and fiber pull-out even under this favorable condition, future work should explore complementary surface modification strategies such as silane coupling alongside long-term durability and moisture-exposure testing, to further strengthen the fiber-matrix bond and validate the composite's reliability under real marine service conditions.

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