Mechanical Characterization of NaOH-Treated Agel Fiber-Cotton Composites

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ABSTRACT

Composites comprising two or more distinct materials are fabricated to enhance the mechanical properties of the constituent materials. A common approach for generating composites is vacuum infusion. This technique enables the infusion of two materials utilizing a vacuum. In the field of composite science, textile composites have emerged as an important new development. Agel rope, derived from twisting agel fibers, exhibits inferior bending strength and elongation compared to ropes fabricated from synthetic fibers. Moreover, agel rope is susceptible to bacterial decay. This study aims to characterize the mechanical properties of textile composites comprising woven agel rope subjected to NaOH treatment. Specimens in the longitudinal (warp) show maximal load-bearing capacity, as determined by experimental results. Samples treated with 5% NaOH tolerated peak loads of 51.12 N prior to failure, with an associated deflection of 3.18%. Specimens in the transverse (weft) of the woven cotton demonstrated maximum load of 40.75 N at 0.9% deflection. The maximum stress was 25.67 MPa. Similar to agel rope, NaOH treatment removes adhering contaminants from cotton fibers, thereby enhancing their strength. However, NaOH concentrations exceeding 7.5% extract cellulose, damaging the fiber ultrastructure.

Keywords: *Agel fiber, bending test, biocomposite, warp and weft direction, woven*

I. Introduction

Composite materials are engineered combinations of two or more constituents with significantly different physical or chemical properties [1], [2]. The constituents retain their individual identities in the composite yet combine synergistically to produce a material with enhanced structural or functional capabilities. Textile composites are a subclass of composites where the reinforcing phase comprises fibrous materials that have been processed into fabric constructions. The fibers may be naturally occurring substances like cotton or flax or synthetic polymers like polyester or aramid [3]. The fiber mats can be tailored in their areal dimensions and three-dimensional architecture to meet specific design requirements. The matrix phase, frequently a thermoset or thermoplastic resin, serves to bind the fibers together, facilitate load transfer between them, and protect them from environmental damage [4], [5]. Through judicial selection of the fiber and resin phases, textile composites with superior mechanical performance compared to their constituents can be synthesized [6]. Textile composites comprise a class of composite materials where the reinforcing phase is constituted by fibers processed into planar textile constructions. The fibers may derive from naturally occurring polymers such as cellulose or protein or synthetic polymeric materials, including polyester, aramid, and glass. These fibers are manufactured into planar textile mats or fabrics via weaving, knitting, or nonwoven processes [7]–[9].

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The mats can be fabricated over a wide range of in-plane dimensions and shapes as per design specifications. An interfacing matrix phase, commonly an epoxy, polyester, or vinyl ester thermoset resin, serves to bond the fibers and transfer loads between them. The resin also protects the fibers from environmental damage [10]. Through optimization of the fiber architecture and resin chemistry, textile composites with superior mechanical performance compared to the individual constituents can be actualized. Initial textile composite developments focused on two-dimensional (2D) mats with in-plane fiber alignment. While exhibiting appreciable in-plane mechanical properties, these composites displayed inferior out-of-plane bending strength perpendicular to the mat plane. Recent innovations in three-dimensional (3D) weaving technology have enabled textile preforms with fibers oriented along the thickness direction [11], [12]. The resulting 3D textile composites demonstrate enhanced out-of-plane mechanical properties relative to their 2D counterparts. These improvements expand the application space of textile composites to more complex load-bearing structures, such as those found in aerospace, automotive, and sporting contexts. However, 3D weaving is more intricate and costly than 2D weaving. The fiber crimping inherent to woven 3D preforms can also compromise composite strength.

Agel rope refers to cordage manufactured from fibers derived from processing leaves of the Arenga pinnata palm species, known as agel [13]. Compared to ropes synthesized from synthetic polymer fibers like nylon or polyethylene, agel ropes exhibit inferior mechanical performance in terms of lower bending strength at break and reduced extensibility or elongation at break. Agel ropes are also susceptible to biodegradation mediated by cellulolytic bacteria and fungi, limiting service lifetimes [14]. Chitosan is a natural cationic biopolymer produced by deacetylation of chitin; a structural polysaccharide found in crustacean shells. Owing to its antibacterial properties, chitosan can form a protective coating on the surface of agel ropes. This surface modification with chitosan serves a dual purpose. First, the coating acts as a barrier to moisture and microbial attack, enhancing durability. Second, the chitosan coating strengthens and toughens the agel fibers by improving load transfer between fibrils [15]. Consequently, the breaking strength and elongation at break of the agel ropes are increased.

Agel fiber rope-reinforced epoxy composites exhibit deficiencies in mechanical performance and flammability resistance stemming from the intrinsic properties of the agel fibers [13], [16]. Specifically, the relatively low bending strength and stiffness of the agel fibers compared to synthetic polymer fibers limits the attainable mechanical properties of the resulting composites [17]. Additionally, the high cellulose content of natural agel fibers renders the composites more flammable. To ameliorate these shortcomings, a sodium hydroxide (NaOH) alkaline solution treatment was investigated. Immersion in a 5% NaOH solution has been shown to improve the mechanical properties of biocomposites reinforced with sisal fibers, another natural cellulosic fiber. The NaOH treatment dissolves the amorphous constituents of the fiber cell wall, thereby exposing the crystalline cellulose microfibrils that bear load. This increases the bending strength and modulus of the individual fibers. Additionally, the NaOH modifies the fiber surface topography, enabling improved mechanical interlocking and adhesion with the epoxy matrix. The net result is an enhancement in the strength, stiffness, and toughness of the agel fiber/epoxy composite. The NaOH treatment also increases the thermal stability and reduces the flammability of the natural fibers.

As summarized in the preceding literature review, sodium hydroxide (NaOH) alkaline treatment has proven effective in enhancing the mechanical properties of natural fiber-reinforced polymer composites. Building on these prior investigations, the author aims to

explore the influence of NaOH processing on the flexural properties of plain woven agel-cotton biocomposite textiles fabricated with epoxy resin matrices. The research will focus specifically on composites reinforced with balanced plain weave fabrics comprising agel and cotton fiber ropes. The objective is to characterize the flexural strength and associated behavior of these textile biocomposites after subjecting the constituent agel and cotton fiber ropes to controlled immersion in NaOH solutions. Systematic variation of the NaOH treatment parameters, including concentration, temperature, and duration, will elucidate the optimal conditions for improved flexural performance. Standard three-point bend testing will quantify enhancements in ultimate flexural strength. Inspection of the fracture surfaces via scanning electron microscopy will provide additional insight into the microstructural mechanisms underlying the mechanical improvements. The knowledge gained from exploring NaOH treatments can guide further optimization of agel-cotton biocomposite textiles to expand their utility in structurally demanding applications.

II. Material and Methods

1. Materials

The raw materials utilized in this research consist of 1 mm diameter rope produced from Arenga pinnata (agel) fibers, Ripoxy R804 J500 epoxy resin, sodium hydroxide (NaOH) solutions for alkali treatment, and deionized water as a solvent. The woven agel rope can be seen in Figure 1. The instrumentation for experimentation incorporates an ESP32 microcontroller unit for test automation and data logging, a BMP180 pressure transducer for vacuum monitoring, relay modules and solid-state relays for electro-mechanical system control, a 12 V 3 A DC power supply for energizing vacuum components, a DC-DC buck converter to furnish suitable logic voltages, printed circuit boards for constructing circuits, female pin headers to enable removable connections, solenoid-actuated pneumatic valves to regulate vacuum, a Bourdon tube vacuum gauge for secondary pressure measurement, pneumatic fittings and valves machined from brass stock, a vacuum chamber equipped with a quick-seal lid to house the composite samples, a rotary vane mechanical vacuum pump for evacuating the chamber, and a diaphragm-style vacuum pump for degassing the resin prior to infusion. The integration of these devices and hardware implements the vacuum-assisted resin infusion process and acquisition of relevant experimental data.

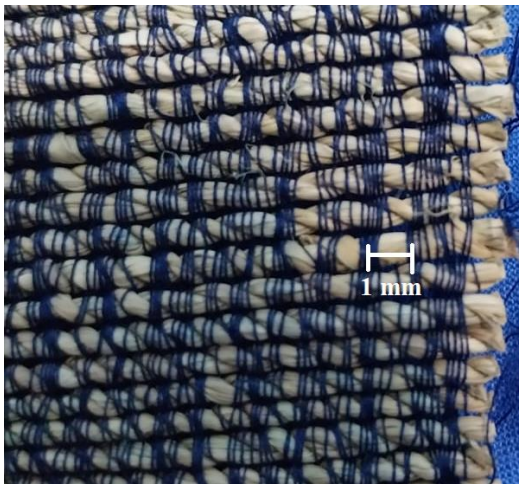

Fig. 1. Woven from agel fiber with a weave distance of 1 mm.

2. Method

Alkaline treatment with sodium hydroxide (NaOH) solutions involves immersing the material in an aqueous mixture containing dissolved NaOH. This processing is hypothesized to enhance the strength of natural fiber-reinforced composites by removing weak constituents such as lignin, fats, waxes, and other impurities from the fiber surface. These components act as defects that degrade the mechanical performance of the fibers and the fiber/matrix interfacial adhesion. This investigation will explore the effects of NaOH concentration on the flexural properties of composites reinforced with agel (Arenga pinnata) fiber ropes. The ropes will be subjected to soaking in solutions containing 0% (untreated control), 2.5%, 5%, and 7.5% NaOH prior to composite fabrication. The treated fiber ropes will be incorporated into an epoxy matrix via 3D printing. The resulting biocomposites will undergo three-point bending tests to evaluate the flexural strength and modulus.

The textile preform was fabricated by plain weaving agel fiber ropes with cotton threads into a balanced fabric with dimensions of 25 x 25 cm. Alkaline treatment solutions were prepared by dissolving sodium hydroxide (NaOH) in deionized water at concentrations of 0% (control), 2.5%, 5%, and 7.5% w/v. The finished woven mats were immersed in the NaOH solutions at ambient temperature for 1 hour. Subsequently, the treated mats were removed from the solutions and thoroughly rinsed with water until the pH was neutralized to 7. After drying, the mats were infused with epoxy resin using an automated Internet of Things-enabled vacuum-assisted resin transfer molding apparatus programmed to a vacuum pressure of -0.6 bar. The molded biocomposites measured 25 x 25 x 0.3 cm. Visual inspection of the samples was conducted to ascertain absence of voids. The weight fractions of the constituents in the cured composites are shown in Equation 1

$$W_i = \frac{m_i}{m_{tot}} = W_i = \frac{50}{180} = 0.28 \quad \text{..... (1)}$$

where Wi is mass fraction of the printed composite material, m_i is mass of a specific component or material within the composite, and m_{tot} is total mass of the composite material.

The composite laminate was machined into rectangular flexural test specimens with dimensions of 2.5 cm x 25 cm x 0.3 cm along the warp (agel fiber) and weft (cotton fiber) directions per ASTM D790-03 standard, as shown in Figure 2. The specimens were subjected to three-point bending tests following the procedures outlined in ASTM D790-03. The equations for bending stress, bending strain, and bending modulus of elasticity can be seen in equations 2-3

$$\sigma = \frac{3FL}{2bd^2} \quad \text{..... (2)}$$

$$\varepsilon = \frac{6\delta.d}{L^2} \quad \text{..... (3)}$$

$$E_b = \frac{L^3 m}{4bd^3} \quad \text{..... (4)}$$

where σ is stress (MPa), F is force (N), b is cross-sectional width (mm), d is specimen thickness (mm), L is cross-sectional length/span (mm), ε is strain, δ is deflection, E_b is the bending elastic modulus (MPa), and m is the tangent slope of the deflection load curve (N/mm).

Load-displacement data was collected during the tests, and the flexural stress-strain response was calculated. The test results were analyzed to characterize the flexural

properties, including the flexural strength, flexural modulus, and flexural strain to failure of the hybrid composite in the longitudinal and transverse directions. The data provides quantitative insight into the bending behavior and mechanical performance of the agel-cotton fiber-reinforced composite laminate.

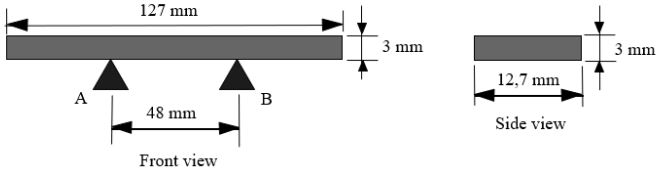

Fig. 2. ASTM D790-03 standard bending test specimen

III. Results and Discussions

The flexural test is an experimental method used to determine the mechanical properties of a material. It enables the quantification of the structural integrity and elastic modulus of a specimen under an applied moment. By incrementally loading a prismatic sample in pure bending until fracture arises, the flexural rigidity, bending strength, compressive strength, and ductility can be obtained. This flexural analysis produces stress-strain data by measuring the load-displacement response. The flexural stresses present in the longitudinal (warp) and transverse (weft or filling) directions of the woven cotton fabric specimen are shown in Figure 3 and 4. The warp direction indicates the threads running lengthwise in the fabric, while the weft or filling threads run perpendicular, across the width. Figure 3 and 4 displays the magnitudes of the bending stresses generated along each of these orthogonal axes when the test load is applied. This allows comparison of the stress-strain response and flexural properties in the two primary orientations of the fabric weave.

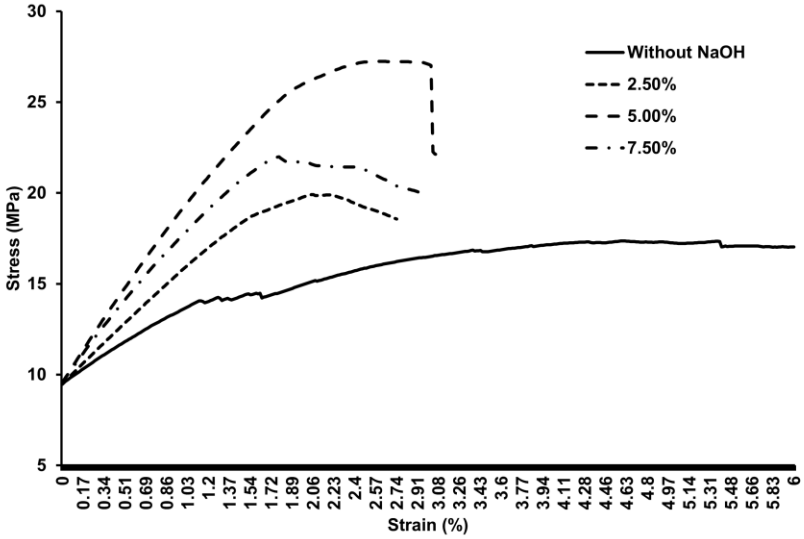

Fig. 3. Warp (Agel) direction bending test stress

From Figure 3, it is observed that the specimen in the longitudinal (warp) shows the greatest strength against the load. The maximum load that can be borne is 51.12 N with a deflection of 3.18% in specimens treated with 5% NaOH. Pretreatment with aqueous sodium hydroxide (NaOH) solutions induces notable enhancements in bending strength properties of natural fiber composites, as substantiated experimentally in this study. The alkali

treatment process removes lignins, waxes and other hydrophobic constituents coating the exterior surfaces of the fiber cell walls [18]. This augments accessibility for interfibrillar bonding with the epoxy resin matrix. Incremental improvements in interfacial adhesion permit more efficient load transfer and mitigate crack propagation at the interface. Our findings demonstrate that a 5% NaOH molar concentration elicits optimal mechanical performance enhancements. This is attributed to the balance between cleansing fiber surfaces of impurities versus causing excessive fiber damage or corrosion at higher alkali contents.

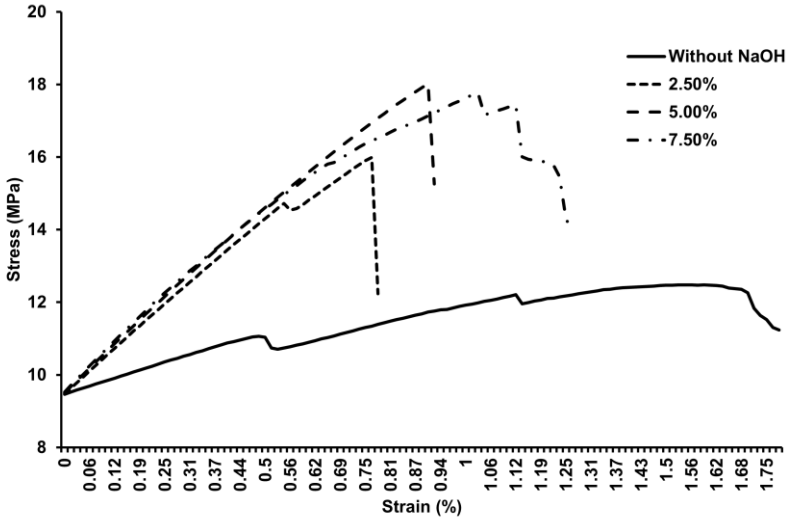

Fig. 4. Weft (Katun) direction bending test stress

Experiments conducted on cotton fiber composites with weft directions show a maximum sustainable load of 40.75 N under 5% NaOH pretreatment, corresponding to a deflection at failure of 0.9%. This equates to a maximum bending stress of 18 MPa along the principal axis. As with other natural fibers, sodium hydroxide (NaOH) solutions induce surface modifications that enhance mechanical performance. The alkaline treatment removes non-cellulosic components like waxes, oils, and impurities that may otherwise compromise interfacial bonding with the polymer matrix. However, excessive exposure to highly concentrated NaOH solutions can damage the cellulose molecules comprising the load-bearing fraction of the cotton fibers [19]. Our observations indicate that NaOH concentrations exceeding 7.5% initiate degradation of the cellulosic components, outweighing the benefits of surface cleaning. This results in inferior mechanical properties compared to the optimal 5% NaOH concentration. A comparison of bending stress and strain is shown in Figure 5 and 6.

Alkaline solutions induce the removal of lignin, hemicellulose, waxes, oils, and other surface impurities on the natural fibers. This cleansing of non-cellulosic components exposes the crystalline cellulose microfibrils and increases surface roughness. The enhanced mechanical interlocking and physical interactions between the purged, roughened fiber surfaces and the matrix lead to improved stress transfer efficiency and inhibition of de-bonding at the interface [20]. Optimization of the NaOH concentration is necessary to remove sufficient surface impurities while avoiding excessive fiber damage.

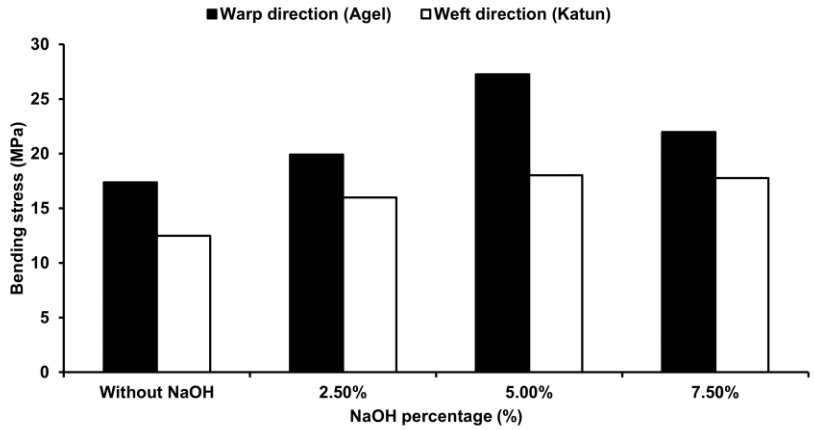

Fig. 5. Comparison of bending stress between warp and weft directions

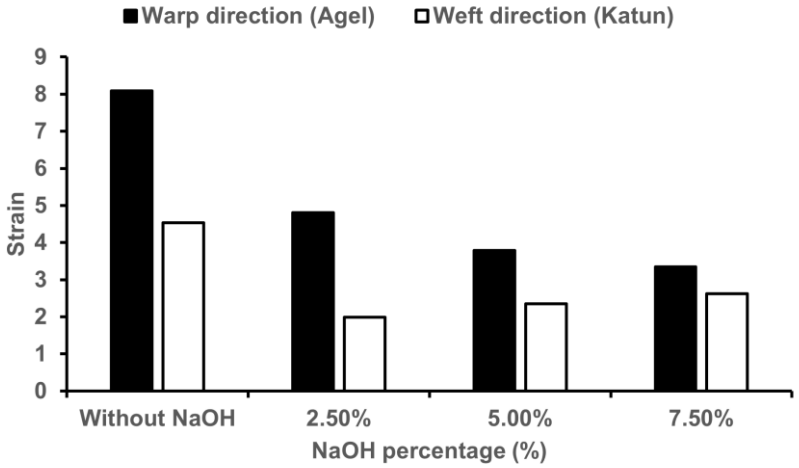

Fig. 6. Comparison of bending strain between warp and weft directions

The degradation of lignin and hemicellulose components that occurs at higher NaOH molarities is believed to cause this decline in bending attributes. As the alkalinity intensifies, the breaking down of unstructured, matrix-binding fractions exceeds the threshold required for efficient fiber surface cleansing. This causes excessive disruption and solubilization of the reinforcing constituents within the fiber cell walls. Consequently, the ensuing loss of structural integrity outweighs any incremental benefits of matrix-fiber adhesion improvements. Detailed chemical analysis could elucidate the NaOH concentrations at which cellulose degradation mechanisms are activated.

The modulus of elasticity is shown in Figure 7. Figure 7 shows that the maximum elastic modulus value of 21.5 MPa was exhibited by woven fabric specimens oriented in the weft direction after being subjected to a 7.5% NaOH alkaline treatment. Specimens with a woven structure tested in the warp direction and processed with 2.5% and 5.0% NaOH resulted in an identical elastic modulus of 20 MPa. Untreated control samples measured in both the warp and weft orientations were found to have significantly lower modulus of elasticity values compared to the NaOH-treated counterparts. Overall, these results indicate that bending stiffness of the woven natural fiber composite material studied here can be enhanced through alkaline exposure, with an optimal NaOH concentration between 5-7.5% for maximizing the reinforcement effect.

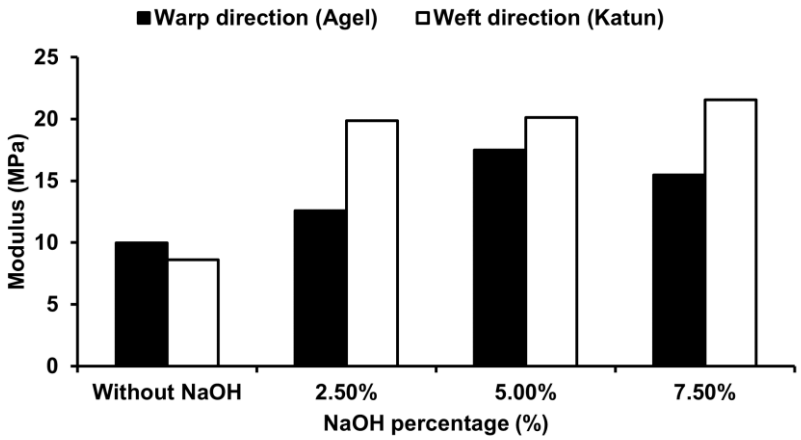

Fig. 7. Comparison of Modulus of elasticity between warp and weft directions

IV. Conclusions

In summation, the specimen exhibiting optimal bending properties consisted of a woven fabric architecture oriented along the warp direction that was processed with a 5% NaOH alkaline treatment. This sample demonstrated a peak load at failure of 51.12 N and a maximum strain of 3.18%, correlating to an ultimate bending strength of 32.20 MPa. The declining fiber strength observed with 7.5% or higher NaOH concentrations is hypothesized to arise from excessive degradation of the lignin and hemicellulose components within the natural fibers caused by the highly caustic nature of solutions with elevated sodium hydroxide content. This chemical damage to the internal structural matrix leads to compromisation of mechanical integrity. Overall, these conclusions showcase the potential to enhance bending performance of natural fiber composites through mild alkali exposure while highlighting the importance of determining an ideal processing window to maximize benefits and avoid excessive fiber disruption.

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