

Nnamdi Azikiwe University Journal of Civil Engineering (NAUJCVE)

Volume-3, Issue-3, pp-14-28

www.naujcve.com

Research Paper

Open Access

Effects of Stacking Sequence and Mercerization treatment on Mechanical and Physical Properties of Hemp and Ramie-Reinforced Hybrid laminated Composites

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Abstract: Natural fibers are available in low cost, easily renewable and they are rich in cellulose. As the natural fibers possess impurities over the surface, poor wettability and interfacial bonding are the main drawbacks encountered during fabrication process. In the current research work, hemp (H) and Ramie (R) fibers were treated with different sodium hydroxide (NaOH) concentrations (2%, 4%, 6%, and 8%) for 5 hr at room temperature. The effects of stacking sequence (HHRRHH, HRHRHR and RRHHRR) and surface modification on physical and mechanical properties of hemp and ramie fiber-reinforced epoxy composites were investigated with a 30 wt.% of fiber loading. The attributes such as water absorption, density, tensile properties, flexural properties, impact energy, hardness were examined. From the experimental results, the 6% NaOH-treated composites compared with untreated fiber composites disclosed an increase of 13.69% for tensile strength, 17.27% for flexural strength, 14.29% for impact energy and 16.67% for hardness with stacking sequence of RRHHRR. The surface morphology of the untreated, treated reinforcements, and fracture surface of the tensile-tested hybrid composite samples were examined by scanning electronic microscope.

KEYWORDS: Mercerization treatment; hybrid composite; mechanical properties; stacking sequence; natural fibers

Date of Submission: 19-06-2025

Date of acceptance:20-05-2025

1. INTRODUCTION

Due to the demand for eco-friendly and growing environmental awareness, the usages of natural fibers in composites fabrication were increasing day by day. Polymer composites reinforced with natural fibers have played an important role for a long time due to their merits such as low density, non-abrasive, biodegradability, high specific strength, and modulus and low cost ((Okoronkwo et al. 2021), (Okoronkwo et al. 2022)). The key problem in the

application of natural fibers is weak adhesive bonding between the hydrophobic matrix and hydrophilic fiber. The high polar surface of natural fibers induces an incompatibility problem with many synthetic matrices, which imply poor mechanical properties in a composite material Pickering according to ((Okoronkwo et al. 2019) (Ezeokpube et al. 2021)). It is necessary to enhance the bonding between the reinforcement and matrix and fiber dispersion either by chemical or

physical methods. Preliminary tests conducted by researchers have confirmed that alkali treatment has been found to be the most feasible one among various chemical treatments (Okoronkwo et al. 2025).

It is known that natural fibers possess some limitations compared with those common fibers such as glass and carbon, where it is having more inferior mechanical properties and a higher water absorption (Dashtizadeh et al. 2017). Therefore, an introduction of hybrid bio-composites consists of two or more fibers in one matrix is seen as a solution to enhance the natural fiber-reinforced polymer composites' properties. (Nurazzi et al. 2021) stated that hybridizing one natural fiber with another natural fiber/synthetic fiber in one matrix will improve its thermal and mechanical than the individual fiber composites (Okoronkwo et al. 2024). This has shown that hybrid composites are more reliable for various applications, besides being more environmentally friendly.

The hybridization of natural fiber-based reinforced polymer composites can be done through a combination of natural–natural fibers, natural–synthetic fibers, natural fiber with carbonaceous materials, and natural fiber with metal (Okoronkwo et al. 2022). Due to their varied properties and considerations of interfacial adhesion, hybrid natural fiber composite materials are facing difficulties in fabrication. Composites are manufactured in a variety of ways, such as through basic mixing and open or closed moulding. Many factors can affect the interactions between the fiber and matrix, for example, and could be mild owing to the existing van der Waals forces, hydrogen bonding, and weak electrostatic interactions. In addition, a good interaction could also exist due to the chemical interactions between those materials. Therefore, studies on hybrid natural fiber composites keep increasing in order to discover the ability of hybrids to be a possible alternative, replacing various petroleum-based products.

Hemp is another renowned bast fiber crop, an annual plant in the Cannabis family that cultivates in temperate climates. As a European Union subsidy for non-food agriculture, many current initiatives are progressing for its development in Europe. PP composites with hemp fibers were functionalized by the reactions of melt grafting using glycidyl methacrylate (GMA) and were manufactured via batch mixing. The fibers and PP matrix modifications and various compatibilizer additions were conducted to enhance the interactions of the fiber–matrix. In comparison with the unaltered composite, chemical bonding between the fiber and the polymer (PP/Hemp) resulted in improved fiber distribution in the PP matrix

as well as higher interfacial adhesion in the modified composite. Matrix and fiber modifications highly influenced the phase behavior and thermal stability of the composites. The alterations in the crystallization behavior and spherulitic morphology of PP in the composites were analyzed due to the hemp fibers' nucleating effect. Additionally, with increasing modified hemp content, a significant rise in the PP isothermal crystallization rate (120–13 8° C) was observed. All composites demonstrated a higher tensile modulus (about 2.9 GPa) and lower elongation at break when compared to plain PP. Still, compatibilization with modified PP (10 phr) boosted the stiffness of the composites due to better fiber–matrix interfacial adhesion.

(Ramesh et al. 2021) fabricated hybrid composites using carbon, alkaline-treated and untreated hemp fibers and investigated their properties. The hybrid composites possessed maximum tensile, flexural, impact, and shear strengths of 61.4 MPa, 122.4 MPa, 4.2 J/mm², and 25.5 MPa, respectively. In addition, from the composites' mechanical properties, the alkaline-treated composites exhibited better performance (Ramesh et al. 2021). (Thiagamani et al. 2019) fabricated hybrid bio-composites using the green epoxy matrix, reinforced with sisal (S) and hemp (H) fiber mats via the cost-effective hand lay-up method and hot press employing different stacking sequences. As the stacking sequence was changed, the tensile strength varied slightly, where the intercalated arrangement (HSHS) hybrid composite demonstrated a maximum tensile modulus compared with the other hybrid counterparts. Hybrid composites (SHHS and HSSH) possessed a compressive strength that was 40% more than the other layering configurations, and the HHHH sample had the maximum ILSS of 4.08 MPa (Thiagamani et al. 2019).

(Norizan et al. 2021) investigated the effects of chelators, white rot fungi, and enzyme treatments towards hemp fiber separation from bundles and enhanced the hemp fibers' interfacial interaction with the PP matrix. The findings indicated that treated fiber composites had a greater interfacial shear strength than untreated fiber composites, a conclusion that was corroborated by a large body of literature ((Olaniran et al. 2019), (Norizan et al. 2021), (Wang et al. 2019), (Amin et al. 2021), (Lobo et al. 2021) and (de Araujo Alves Lima et al. 2020)). This demonstrates that the white rot fungal treatment increased the interfacial attachment of hemp fiber to PP. Composites made of chelator concentrate treated hemp fibers exhibited the maximum tensile strength, measuring 42 MPa, a 19% improvement above composites made of untreated hemp fiber. Additionally, hemp fiber reinforced PP

composites showed fascinating recyclability. The findings demonstrated that despite the high number of reprocessing cycles, the mechanical properties of hemp fiber/PP composites were well maintained. Newtonian viscosity reduced as the number of cycles increased, indicating a decline in chain scissions and molecular weight caused by reprocessing. Another possible explanation for the decrease in viscosity was the shortening of the fibers during reprocessing.

Ramie is a plant from the Urticaceae (*Boehmeria* spp.) family that comprises approximately 100 species. The exploitation of ramie is for use as textile fiber with two limiting factors: production regions as well as a need for more considerable pre-treatment than other commercial bast fibers ((Okoronkwo et al. 2022) and (Okoronkwo et al. 2025)). Ramie fiber/sugar palm fiber reinforced epoxy hybrid composites were manufactured using a combination of melt mixing and injection moulding techniques (Razali et al. 2018). Numerous ramie fiber/PP composites were manufactured by changing the fiber length, content, and pre-treatment technique. Increments in fiber length and content were associated with significant increases in tensile, flexural, and compression strengths. Nonetheless, they negatively affected the elongation behavior and impact strength of composites.

The major problem with employing natural fibers is that they are incompatible with a polymer matrix, which reduces the mechanical performance ((Ilyas et al. 2020), (Nurazzi et al. 2021), (Ilyas et al. 2021) and (Aisyah et al. 2021)).

In the present investigation, hemp and ramie fiber-reinforced epoxy composites were fabricated with 30 wt. % fiber loading and the fibers are subjected to mercerization treatment at different NaOH concentrations (2%, 4%, 6%, and 8%) to study the physical and mechanical properties of hemp and ramie fiber-reinforced hybrid composites.

II. MATERIALS AND METHODS

2.1. Materials

Mechanical performance of hybrid composites was extremely relied on direction of the fibers. Compared with the randomly aligned short orientations of fibers, the unidirectional fibers were giving better strength (Devireddy and Biswas 2017). In the current research work, the unidirectional hemp and ramie fibers were selected as reinforcement material. The continuous ramie fiber was extracted from its parent tree and the unidirectional hemp fiber was purchased from the local commercial sources. The unidirectional hemp and ramie fibers were cut into 30 cm length and prepared in layers form as shown in Fig. 1. The polymer matrix used is a combination of LY 556 Liquid Epoxy and HY 951 Hardener, which were provided by Nycil industrial CCRD limited, Nigeria. The sodium hydroxide (NaOH) procured from Ikenna chemicals Nigeria limited, Nigeria, was used for surface treatment of the hemp and ramie fibers.

2.2. Fiber treatment

The main problem with untreated natural fibers is poor interfacial bonding between the matrix and reinforcement. To overcome this problem, the continuous hemp and palmyra fibers were drenched separately in four different alkali concentrations (2%, 4%, 6%, and 8%). These fibers were retained to immerse in the NaOH concentrations for 5 h at room temperature as recommended for better penetration of the NaOH solutions into the reinforcement material (Hamidon et al. 2019). Fig. 2 shows the chemical treatment of hemp and ramie fibers. The treated hemp and ramie fibers were removed from the NaOH solution and thoroughly cleaned with running tap water to remove any excess of NaOH over the fiber surface. Finally, the cleaned hemp and ramie fibers were dried in an oven at 70°C for 4 h to remove the remaining moisture content.

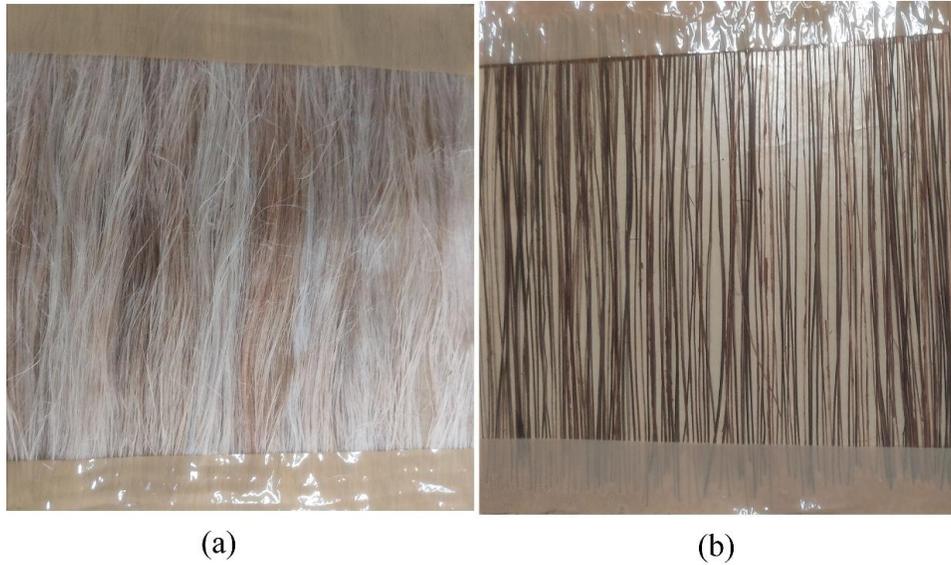


Fig. 1. Unidirectional hemp and ramie fibers



Fig. 2. Chemical treatment of hemp and ramie fibers

2.3. Composites preparation

The fabrication of hemp and ramie fiber-reinforced hybrid composite laminates was done by a simple hand lay-up process. All the composite samples were prepared with 30% fiber loading with four different alkali concentrations (2%, 4%, 6%, and 8%). The designation and NaOH concentration of fabricated composites samples are listed in Table 1. Firstly, the

continuous hemp and ramie fibers were chopped into 30 cm length to prepare into a layer's form. The layers were kept in three modes of stacking sequences HRHRHR (Hemp-Ramie- Hemp-Ramie-Hemp-Ramie), HHRRHH (Hemp-Hemp-Ramie-Ramie-Hemp-Hemp), and RRHHRR (Ramie-Ramie-Hemp-Hemp-Ramie-Ramie) by maintaining the relative weight ratios of the hemp and ramie as 1:1, 2:1, and 1:2. Epoxy and hardener were blended with weight ratio of 10:1,

respectively. Each layer was pre-impregnated with matrix and placed one layer over the other layer in the mould until it achieved the thickness of 4 mm. The bubbles trapped between the layers were removed by moving the steel roller over the composite. Finally, the composite laminate was compressed by placing the mould release sheet and allowed to cure at room temperature for 48 h. The required samples were prepared as per dimensions given in the ASTM standards to investigate the physical and mechanical behaviour. Fig. 3 shows the fabricated composite laminate and samples as per ASTM standards.

2.4. Mechanical behaviour of hybrid composites

Tensile test was conducted under the uni-axial loading conditions of the fabricated samples. Tensile properties were evaluated with a cross head speed of 5 mm/min using ASTM D3039–76 standards. The test was performed using tensometer with sample gage length of 50 mm.

The flexural properties of the prepared composite samples were evaluated using three-point

bending test on the same tensometer. Flexural strength was determined as per ASTM D790 standard with a speed of 5 mm/min. Impact energy of the fabricated specimens was evaluated by low velocity Izod impact tester supplied by VEEKAY test lab, India, as per ASTM D 256 standard. The specimen dimension for the impact test was 64 mm × 12.7 mm × 4 mm. Leitz micro-hardness tester was used to evaluate the Vickers hardness of the specimens as per ASTM D785 test standard.

Table 1. Detailed designation and NaOH concentration of fabricated composites.

Composite	Stacking sequence	Fiber loading	NaOH concentration
CI	HRHRHR	15wt.% Hemp + 15 wt.% Ramie	Untreated
C2			2
C3			4
C4			6
C5			8
C6	HHRRHH	20wt.% Hemp + 10 wt.% Ramie	Untreated
C7			2
C8			4
C9			6
C10			8
C11	RRHRRR	10 wt.% Hemp + 20 wt.% Ramie	Untreated
C12			2
C13			4
C14			6
C15			8



Fig. 3. Fabricated composite laminate and specimens.

2.5. Physical behavior of hybrid composites

The experimental densities of composite specimens were measured by Archimedes method as per ASTM D 792–91 standard. Knowing the mass and volume of the samples, the density is calculated using the Eq. (1).

$$\text{Density } (\rho) = \frac{\text{Mass}(m)}{\text{Volume}(V)}$$

The percentage of water absorption of the fabricated specimens was determined as per ASTM D570–10 standard. The dimension of the specimens was 60 mm × 10 mm × 4 mm. The value of water absorption was evaluated using the following Eq. (2).

$$\text{Water absorption}(\%) = \frac{(W_1 - W_0)}{W_0}$$

where W_0 is the initial weight of sample and W_1 is the weight of water-absorbed sample.

2.6. Scanning electron microscopy (SEM)

The cross-section and fiber surface of untreated and treated fibers were taken using a scanning electron microscope model JEOL JSM-6480LV. Before conducting the SEM evaluation, the samples were placed on aluminum stubs coated with gold by means of a plasma sputtering apparatus.

III. RESULTS AND DISCUSSION

3.1. Density and water absorption

The experimental density of the untreated and NaOH-treated hemp and ramie fiber-reinforced composites is shown in Fig. 4. For NaOH-treated composites, there was some slight increase in the density values of the hybrid composites as the NaOH concentration was increased. The alkali treatment eliminates the impurities, which contribute to decrease the fiber volume; hence, the densities of treated fiber composites increases as compare to untreated fiber composites. On the other hand, the density values of the composites did not appear to be affected by the stacking sequence of the fibers. Fig. 5 represents the effect of stacking sequence and NaOH concentration on the water absorption properties of fabricated composites. In all the stacking sequences, the water absorption of the NaOH-treated hemp and ramie fiber-reinforced hybrid composites was lower than that of the untreated fiber-reinforced composites. This may be due to strong intermolecular reinforcement and matrix bonding that decreased the rate of water absorption in hybrid composites. The composites with stacking sequence RRHHRR with a slight positive tendency was observed compared with the other two stacking sequences.

3.2. Tensile properties

Figs. 6 and 7 depict the influence of alkali treatment and stacking sequence on tensile strength and tensile modulus of fabricated composite samples. Significant difference was found in the results between the alkali-treated and untreated composites. Improvement in the tensile properties of alkaline-treated hybrid composites

is observed due to the removal of impurities such as fats, pectin, and lignin along the fiber surface, which improves the fiber bonding with matrix. Up to 6% NaOH concentration, the tensile characteristic of the composite samples increases and then decreases at 8% NaOH concentration. At 6% NaOH concentration, the maximum tensile strength is found 85 MPa, 75 MPa, and 68 MPa with stacking sequence of RRHHRR, HRHRHR, and HHRRHH, respectively. Compared with the untreated composites, the alkali-treated composites at 6% NaOH concentration improve the tensile strength up to 18.06%, 17.24%, and 13.64% with stacking sequence of RRHHRR, HRHRHR, and HHRRHH, respectively. At 6% NaOH concentration,

the maximum tensile modulus is found to be 4.98 GPa, 4.74 GPa, and 4.50 GPa with stacking sequence of RRHHRR, HRHRHR, and HHRRHH, respectively. Compared with the untreated composites, the alkali-treated composites at 6% NaOH concentration improve the tensile modulus up to 13.69%, 12.78%, and 12.86% with stacking sequence of RRHHRR, HRHRHR, and HHRRHH, respectively.

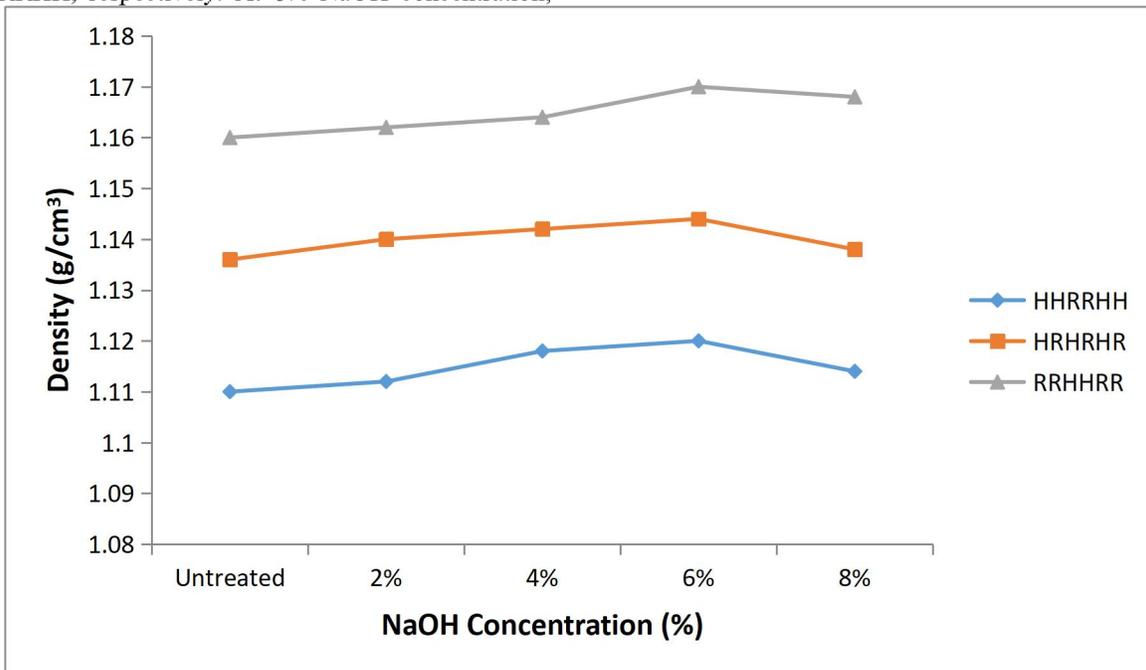


Fig. 4: Experimental density of the untreated and NaOH-treated hybrid composites.

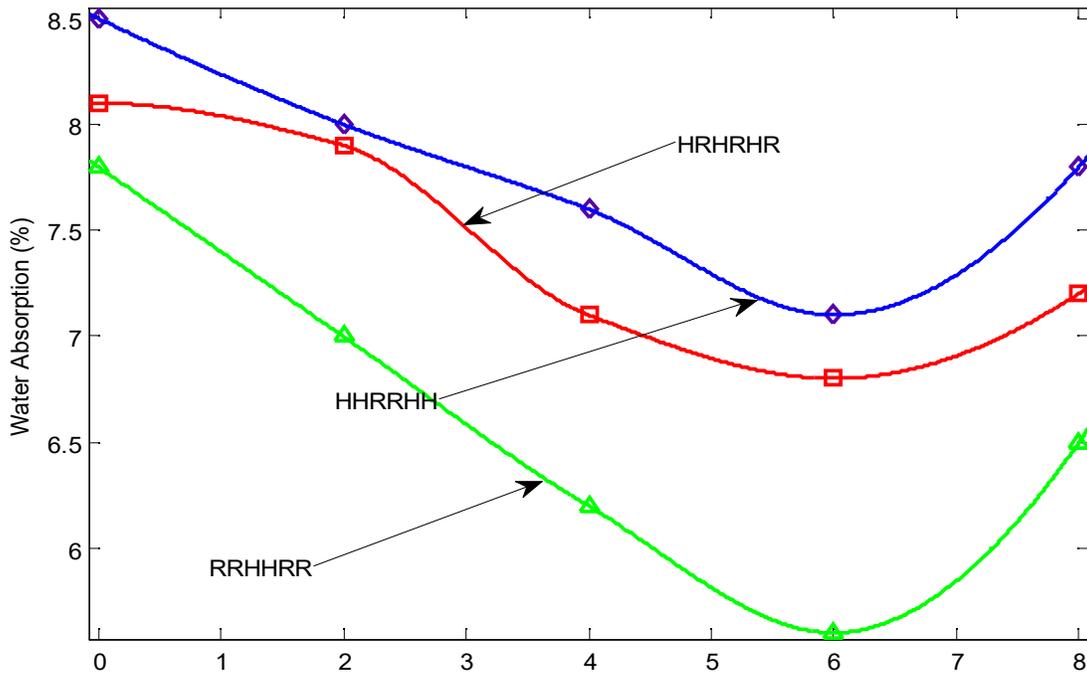


Fig. 5: Effect of NaOH concentration on water absorption of hybrid composites

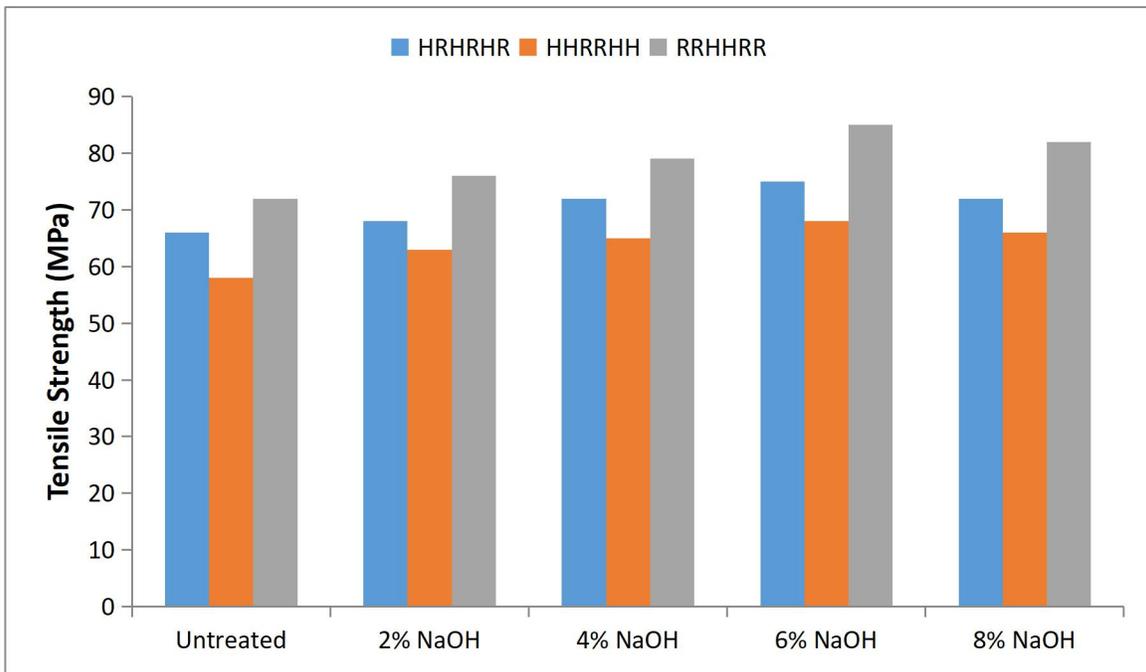


Fig. 6: Effect of NaOH concentration on tensile strength of hybrid composites.

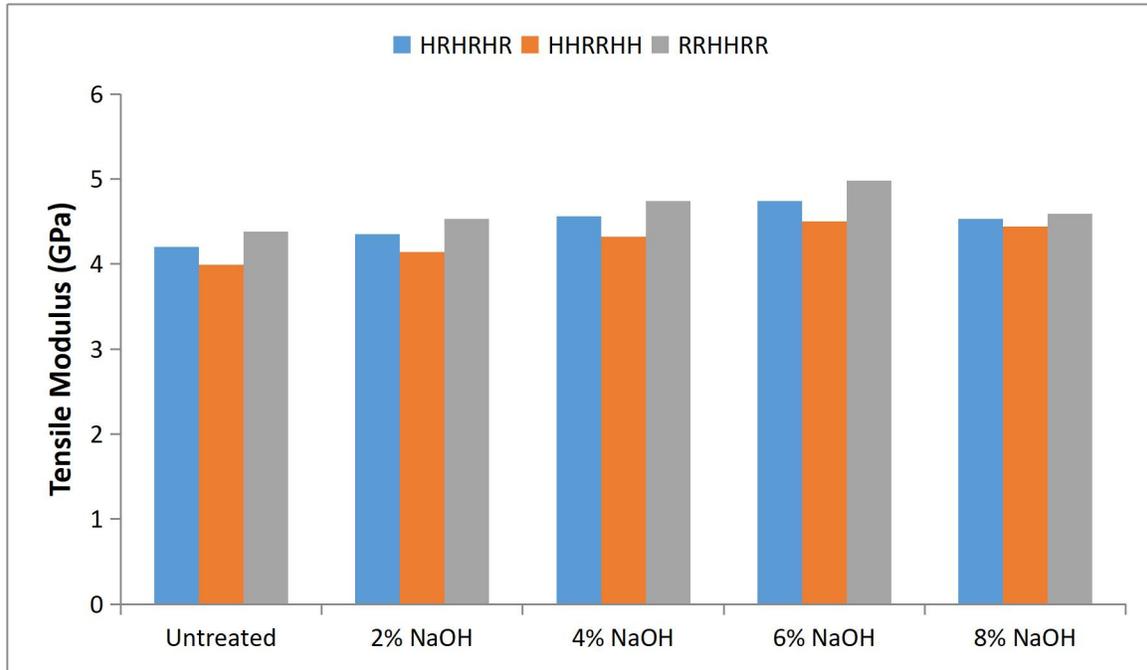


Fig. 7: Effect of NaOH concentration on tensile modulus of hybrid composites.

3.3. Flexural properties

The influence of alkali treatment and stacking sequence on flexural strength and flexural modulus of the fabricated composites are shown in Figs 8 and 9. The Fig. displays the average flexural strength related with maximum and minimum values of each sample. The 6% NaOH-treated composites showing maximum flexural strength values of about 116 MPa, 101 MPa, and 129 MPa with stacking sequence of HRHRHR, HHRRHH, and RRHHRR, respectively. The percentage improvement from untreated to treated composites with 6% NaOH concentration in HRHRHR, HHRRHH, and RRHHRR layering hybrid composites are found 14.85%, 9.78%, and 17.27%, respectively. The 6% NaOH-treated composites showing maximum flexural modulus values of about 7.42 GPa, 6.86 GPa,

and 7.84 GPa with stacking sequence of HRHRHR, HHRRHH, and RRHHRR, respectively. This could be due to the alkali treatment improves the bonding between reinforcement and epoxy matrix and permitting the matrix to enter into reinforcement in the fabrication, which leads to the high load carrying by the reinforcement during testing conditions. The decrease in flexural strength at 8% NaOH concentration may be due to the higher NaOH concentration that may damage the fiber cellulose which will reduce the strength. On other hand, lower flexural strength was observed when the hemp fiber was used as outer layer and ramie fiber as the inner layer. This may be due to the lower intrinsic properties of hemp fiber as related to ramie fiber (Narayana, Rao, and Rao Devireddy 2020).

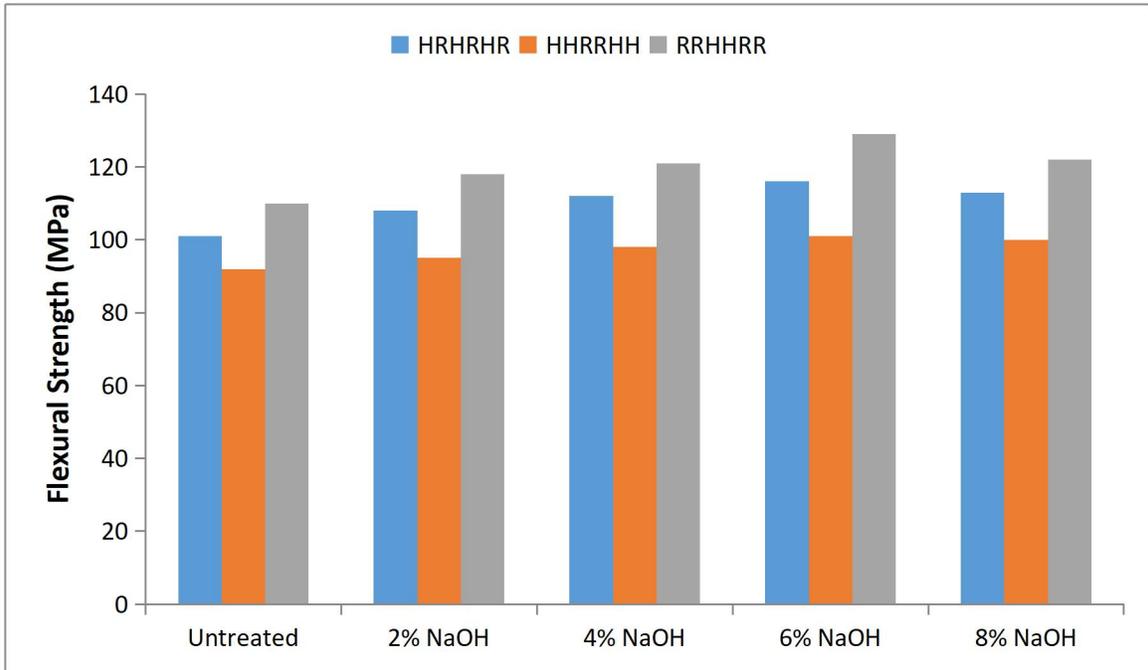


Fig. 8: Effect of NaOH concentration on flexural strength of hybrid composites.

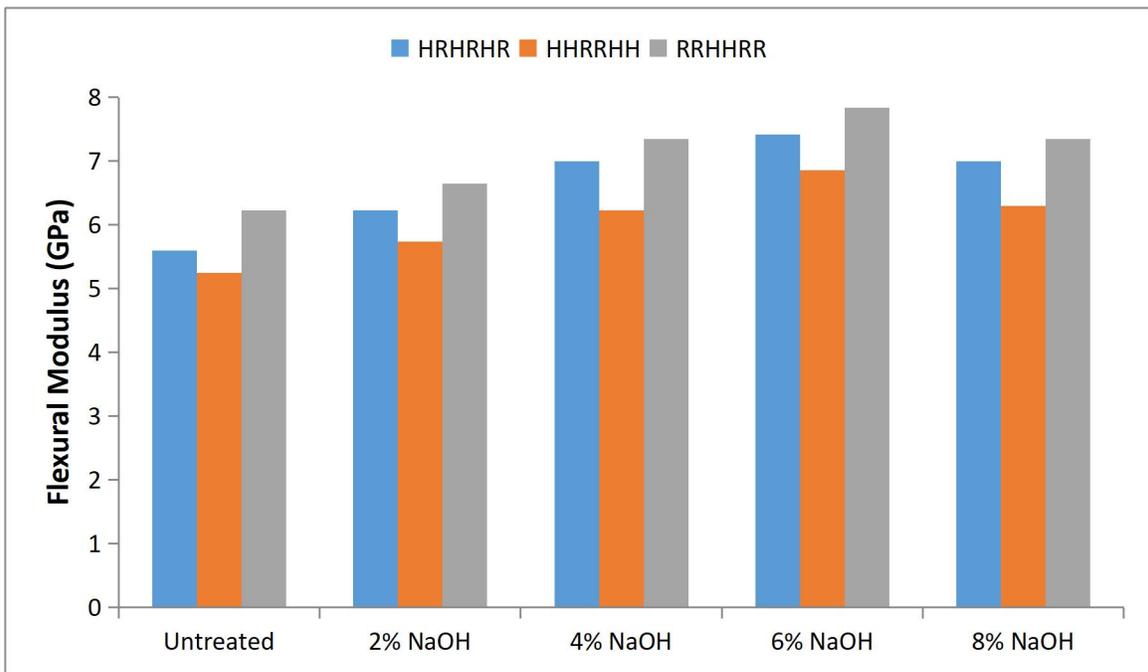


Fig. 9: Effect of NaOH concentration on flexural modulus of hybrid composites.

3.4. Impact energy

It is known that the impact strength of composite material is depending on the interfacial bonding, fiber pullout, fiber, and matrix properties. Fig. 10 illustrates the impact energy values of tested composites in

graphic form. The alkali treated hybrid composite samples was shown better values as compared to untreated composites. Up to 6% NaOH concentration, the impact of energy values of fabricated hybrid composites are progressively increased to the maximum value and then decrease at 8% NaOH

concentration. And the similar trend also occurred for all the stacking sequences. From the Fig., it is detected that the impact energy of HRHRHR, HRRRHH, and RRHHRR composites is high about 14.29%, 10.64%,

and 14.06%, respectively, as compared to untreated composites. A similar trend of observations were found with other researchers.

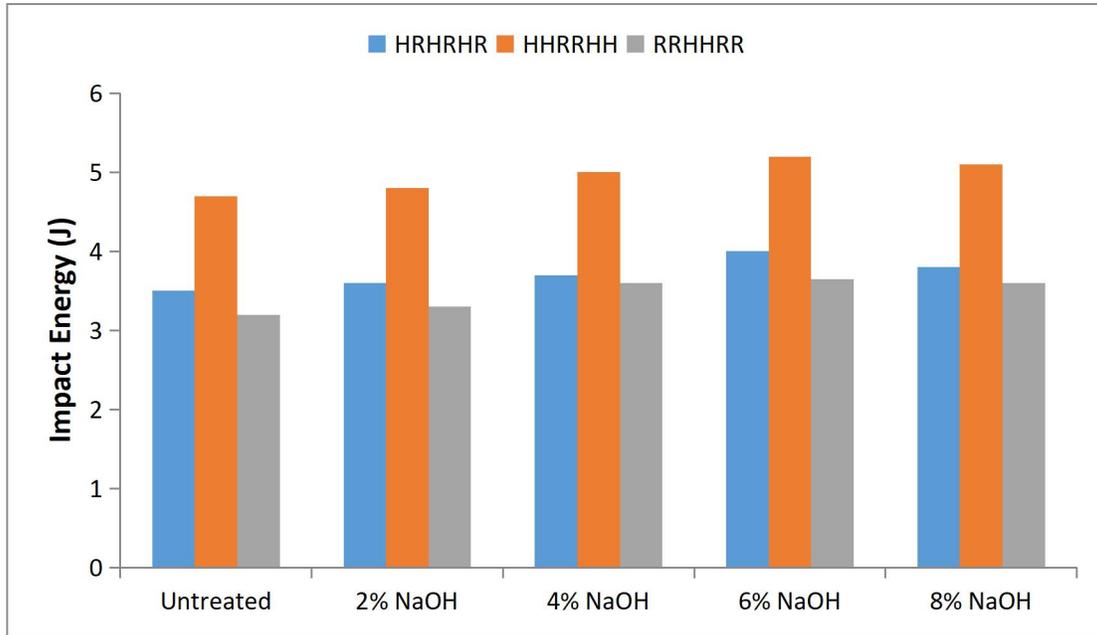


Fig. 10: Effect of NaOH concentration on impact energy of hybrid composites.

3.5. Hardness

The Vickers hardness of untreated and NaOH-treated composites with different stacking sequence is present in Fig. 11. Similar to the tensile and flexural properties, the NaOH treatment increases the hardness of composite as compared to untreated composites. The

hybrid composites fabricated with 6% NaOH treated to hemp and ramie fiber contribute to the better performance of compared to the untreated and other NaOH treatments. At 6% NaOH concentration, the maximum hardness values for the composites with stacking sequence HRHRHR, HRRRHH, and RRHHRR are 45 Hv, 42.5 Hv, and 49 Hv, respectively.

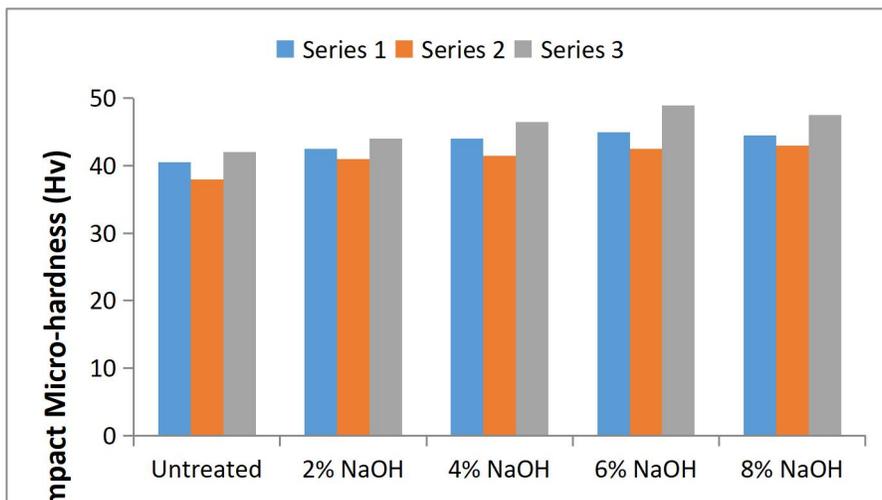


Fig. 11: Effect of NaOH concentration on hardness of hybrid composites

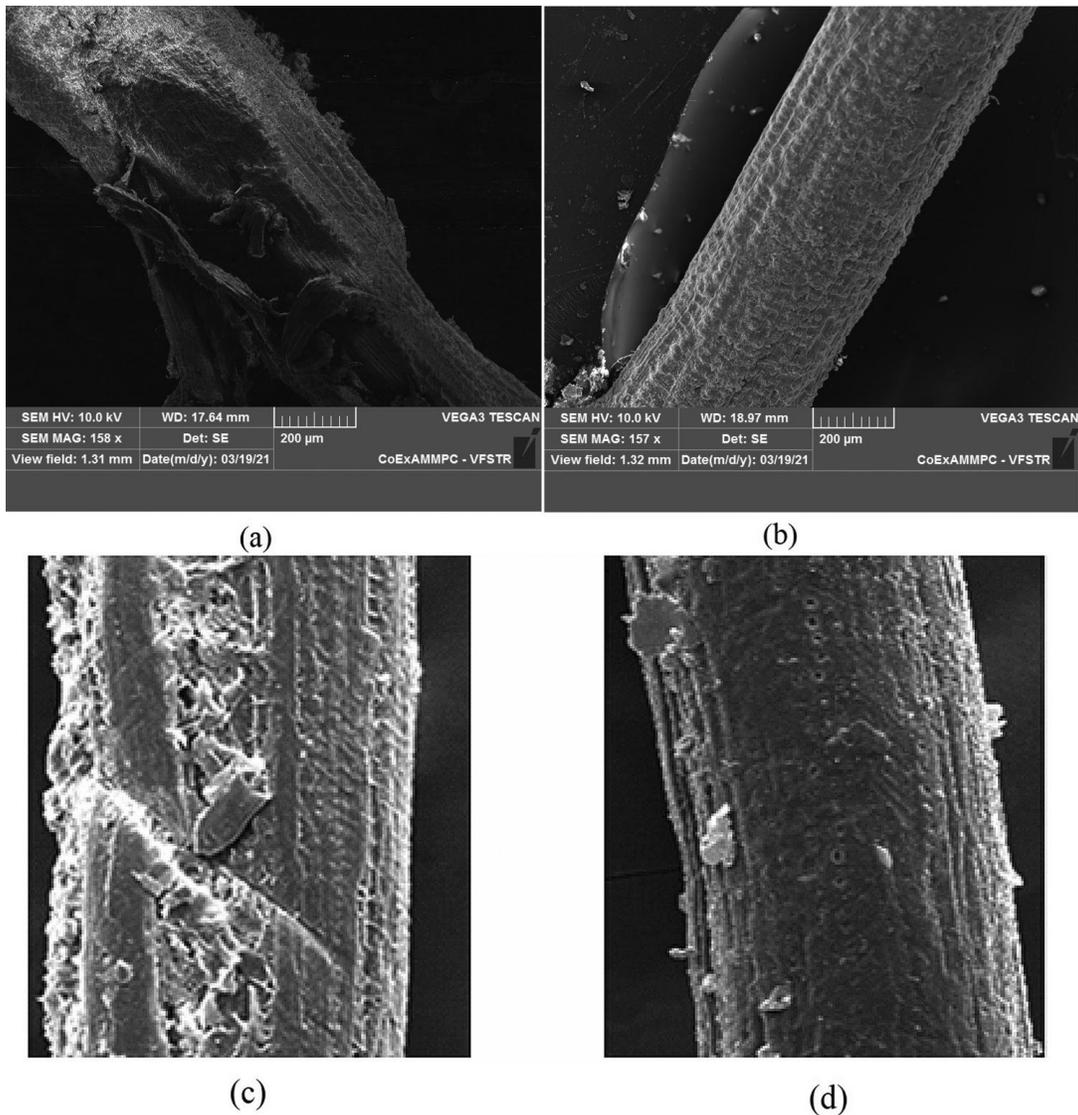


Fig. 12. Surface morphology of (a) untreated hemp fiber and (b) treated hemp fiber with 6% NaOH solution (c) untreated ramie fiber and (d) treated ramie fiber with 6% NaOH solution.

3.6. Scanning electron microscopy (SEM)

To study the effect of NaOH concentration on hemp and ramie fiber, surface morphology was carried out on the untreated and treated fibers and their hybrid composites using scanning electron microscopy. The surface morphology photographs of both untreated and alkali-treated hemp fiber surfaces are exhibited in Fig. 12. As expected, the surface morphology of treated fibers was different to those untreated fibers in terms of their level of smoothness and roughness. Clearly from Fig. 12(a), the impurities were found on the surface of the untreated hemp fiber. On the other hand, Fig. 12(b) displays hemp fiber after 6% NaOH treatment, which was soaked for 5 h. The impurities have been removed

from fiber surface and also show some scrapes along the surface.

IV. CONCLUSION

The effect of mercerization and stacking sequence on physical and mechanical behavior of hybrid composites has been studied and the following conclusions were drawn:

Compared with the untreated composites, the 6% NaOH-treated hybrid composites increased the flexural strength (+17.27%), tensile strength (+13.69%), impact energy (+14.29%), and hardness (+16.67%) of hybrid composites.

- SEM investigation revealed that load carrying capacity by the fiber and interfacial bonding improved after NaOH treatment which attributed to the increment in mechanical behavior of hybrid composite.
- The investigation exhibited that 8% NaOH concentration would damage the fiber strength, which plays a main role as far as the mechanical properties were decreased.

Highlights

- Preparation of hybrid composite materials reinforced with hemp and ramie fibers.
- A comprehensive discussion on effect of alkali treatment on physical and mechanical properties of hemp and ramie fiber-reinforced composites.
- Improvement of mechanical properties of the elaborated materials.

Disclosure statement

The authors declare that there is no conflicting interest in the publication of this paper.

Acknowledgements

This paper was not supported by any financial grant from granting bodies, the authors really appreciate by acknowledging Engr. Dr. George. O. O., and others for their active inputs and contributions needed for the outcome of this publication financially and otherwise.

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