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Loading-dependent mechanical performance of alkali-treated areca nut husk fiber reinforced polyester composites modified with *Uncaria gambir* extract

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Abstract: Natural fiber-reinforced polymer composites often experience mechanical performance limitations due to weak interfacial bonds between hydrophilic fibers and hydrophobic matrices. This study experimentally examined the effect of alkali treatment and modification using *Uncaria gambir* extract (UGE) on the mechanical properties and interface morphology of polyester composites reinforced with areca nut husk fiber (ANHF). Four composite configurations were prepared with a constant fiber weight fraction of 40 wt.% after alkali treatment using 6% NaOH for 24 hours, while the remaining 2 wt.% UGE was selectively applied as a fiber surface treatment, matrix additive, or a combination of both. Tensile and flexural properties were evaluated in accordance with ASTM standards, while interface morphology was examined using scanning electron microscopy (SEM). The results showed that alkali-treated composites without UGE addition had the highest tensile strength, which was attributed to increased fiber surface roughness and mechanical interlocking mechanisms. Conversely, fiber surface modification using UGE significantly increased flexural strength, indicating better stress distribution under flexural loading due to increased interface continuity. However, the addition of UGE to the matrix caused a decrease in tensile strength, which was thought to be related to a reduction in matrix stiffness. SEM observations confirm the presence of distinct interface morphology differences according to the treatment applied. These findings indicate that UGE serves primarily as a bio-based interfacial modifier, enhancing flexural performance, while its effectiveness is strongly governed by the mechanical loading mode.

Keywords: natural composites; renewable material; natural fibers; engineering materials

1. Introduction

Natural fiber-reinforced polymer composites are gaining attention as a sustainable alternative to synthetic fiber-based composites because they have low density, are renewable, and are more environmentally friendly [1]. Natural fibers used as reinforcing materials in composites can be obtained from various sources, such as jute [2], flax [3], hemp [4], ramie [5], kenaf [6], sisal and henequen [7], pineapple leaf [8], banana and abaca [9], palm leaf [10], coconut [11], cotton [12], oil palm biomass (OPB) [13], rice straw and husk [14], wheat straw [15], maize, oat, barley and rye [16], bamboo [17], sugarcane bagasse [18] and cymbopogon nardus [19]. In general, these various natural fibers have shown mechanical properties that are close to those of certain synthetic fibers, thus having great potential to be developed as composite reinforcements in various engineering applications.

However, the use of natural fibers as composite reinforcements still faces several limitations, particularly related to the quality of the fiber-matrix interface bond. Hydrophilic natural fibers tend to have low compatibility with hydrophobic polymer matrices, which can reduce stress transfer efficiency and composite mechanical performance. Natural fibers also have several disadvantages, such as being prone to mold and bacteria, which can cause them to become brittle and easily decay [20]. This will certainly affect the quality of composites that use natural fibers as reinforcements. One step taken to overcome this disadvantage is to add natural additives. *Uncaria gambir* extract (UGE) is one additive that has been extensively studied and has antibacterial properties [21]. Research results reveal that UGE can fight bacteria and significantly inhibit the growth of fungi [22]. The presence of UGE in PVA also shows the same antibacterial properties in composites due to the presence of catechins [23]. In addition to its antibacterial properties, UGE in PVA can improve the bond in composites, as indicated by high tensile strength values [24], [25]. However, the role of UGE in thermoset matrix natural fiber composites, particularly polyester, is still not fully understood. Furthermore, most previous studies reported an increase in mechanical properties in general without distinguishing the material's response to different types of loading, even though tensile and flexural properties are controlled by different stress transfer mechanisms. The potential trade-off between increased interface continuity and decreased matrix stiffness due to the addition of bio-based additives has also rarely been discussed systematically.

In this study, areca nut husk fibers (ANHF) were used as reinforcement. Areca nut husks contain approximately 15–30% fiber [26], with a cellulose content of about 53.2% [27]. Although ANHF has been extensively studied as a composite reinforcement material, there is still a research gap regarding the effectiveness of chemical treatment and the use of natural additives that have the potential to modify the fiber-matrix interface more optimally [28], [29]. Therefore, this study aims to examine the effect of alkali treatment and modification using UGE on the mechanical behavior of ANHF-reinforced polyester composites, focusing on the dependence of mechanical performance on the type of loading. Tensile and flexural properties were evaluated to reveal the role of mechanical interlocking and interface continuity under different loading conditions, while interface morphology was analyzed using scanning electron microscopy (SEM). The novelty of this research lies in clarifying the role of UGE as a bio-based interface modification agent and confirming that its effectiveness on the mechanical properties of composites is highly dependent on the loading mode applied.

2. Material and methods

2.1 Material

ANHF was obtained from areca nut shell waste and used as a reinforcement in composites. The resin used was orthophthalic unsaturated polyester, and the catalyst used was methyl ethyl ketone peroxide. Sodium hydroxide (NaOH) was used for alkali treatment of fibers, while UGE was used as a bio-based additive for interface modification (from PT. Andalas Sitawa Fitolab).

2.2 Preparation sample

This study was conducted using an experimental method with the creation of four types of composite samples differentiated based on fiber treatment and matrix composition. In all samples, ANHF fibers were first cleaned and dried, then soaked in a 6 wt.% NaOH solution for 24 hours at room temperature to increase surface roughness and remove non-cellulose components. After soaking, the fibers were rinsed with distilled water until they reached a neutral pH and then dried at room temperature until they reached a dry condition.

The sample preparation process is shown in Figure 1. Sample 1 is a composite made from ANHF treated only with NaOH for 24 hours without further UGE treatment. The fibers serve as reinforcements with a weight fraction of 40 wt.%, while the matrix consists of 60 wt.% polyester resin. Sample 2 has the same fiber treatment as Sample 1, specifically 6 wt.% NaOH immersion for

24 hours. The difference lies in the matrix, where the resin is added with 2 wt.% UGE additive to the total weight of the matrix. Sample 3 was made using ANHF which, after treatment with 6 wt.% NaOH and drying, was then immersed in a 2 wt.% UGE solution for 24 hours at room temperature and dried again. The composite was then made without adding UGE to the matrix. The composition of this composite consisted of 40 wt.% fiber and 60 wt.% resin. Sample 4 is a combination of fiber and matrix treatment. ANHF was treated with 6 wt.% NaOH for 24 hours, dried, then immersed in a 2 wt.% UGE solution for 24 hours and dried again. Next, the fibers were used to make composites with a matrix to which an additive of 2 wt.% UGE was added to the total weight of the matrix.

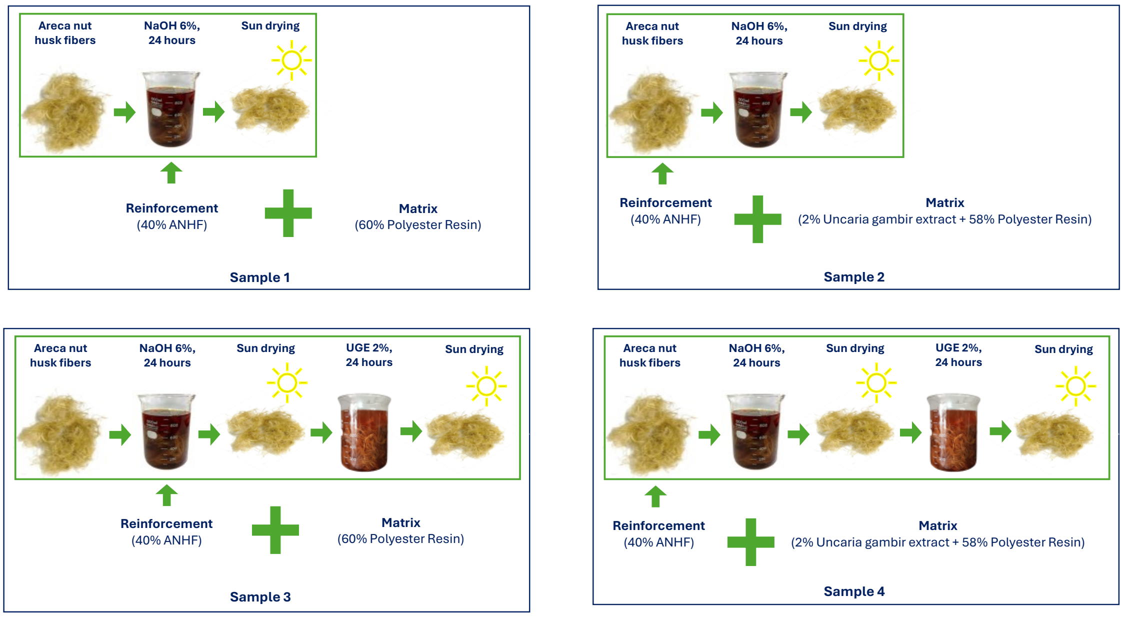


Figure 1. Schematic diagram of sample preparation for all samples

The sample board manufacturing process was carried out using the hand-lay-up method. The resin and catalyst mixture was stirred homogeneously before impregnating the fibers. The composite panels were made with dimensions (width x length x thickness) of 300 mm x 200 mm x 2.5 mm for tensile test samples and 100 mm x 185 mm x 4 mm for flexural tests. After molding, the composite is left to cure at room temperature until the curing process is completed, then removed from the mold and cut to the dimensions of the test specimen.

2.3 Mechanical testing

Specimens and tensile testing procedures refer to ASTM D 3039, while specimens and flexural testing procedures denotes ASTM D 7264. Five specimens were made for each test for each sample with different fractions. Tensile and flexural properties tests are performed using a Universal Testing Machine HT-2402 Series (10-50 kN) with a maximum capacity of 50 KN. The tensile and flexural test processes are presented in Figure 2.

2.4 Microstructural and surface morphology analysis

Microstructural and surface morphology analysis of ANHF was performed using Scanning Electron Microscopy (SEM) ZEISS Auriga Crossbeam system (from Carl Zeiss Microscopy, Munich, Germany). Samples were taken from the fracture surface resulting from mechanical testing to identify the failure mechanism and interface bonding quality due to variations in fiber and matrix treatment.

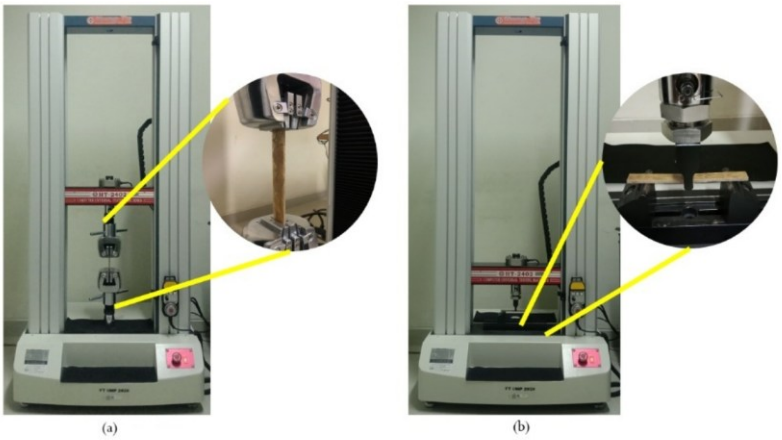


Figure 2. Tensile testing machine used in this study. (a) Tensile strength test and (b) Flexural strength test

3. Results and discussion

Figure 3 shows the tensile strength and elastic modulus of ANHF-polyester composites with various fiber and matrix treatments. Composites treated with alkali without the addition of UGE showed the highest tensile strength values compared to other variations. This increase is associated with the effect of NaOH treatment, which is able to remove lignin, hemicellulose, and surface impurities, thereby increasing fiber roughness and improving mechanical interlocking between the fibers and the polyester matrix. Conversely, the addition of UGE to the matrix causes a decrease in the tensile strength of the composite. This phenomenon indicates a decrease in matrix stiffness due to the presence of bio-based additives, which potentially disrupts the continuity of the matrix phase and the efficiency of stress transfer under tensile loading conditions. Under tensile loading, the matrix plays a dominant role in transferring stress along the fiber direction, so that a decrease in matrix stiffness has a direct impact on the decrease in composite tensile strength. UGE applied as a fiber surface treatment did not show a significant increase in tensile strength compared to the alkali control sample. This performs the increase in interface continuity due to UGE does not directly contribute to improved tensile performance, which is more controlled by matrix stiffness and axial load transfer efficiency.

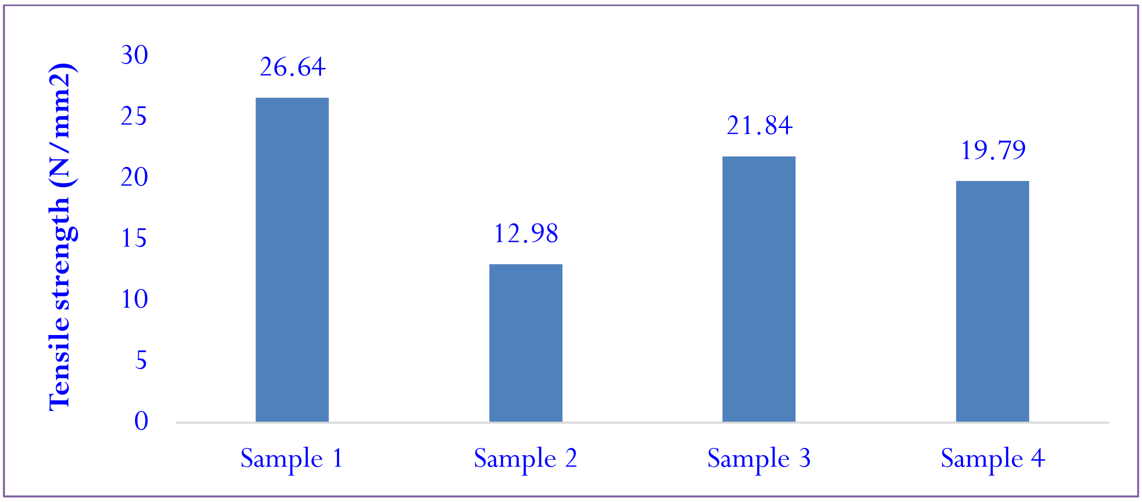


Figure 3. Tensile strength of all samples

As opposed to tensile behavior, flexural test results show that composites with UGE treatment on the fiber surface produce the highest flexural strength. This increase confirms that interface modification using UGE can improve stress distribution under flexural loading conditions, where tensile and compressive stress gradients occur simultaneously on the specimen cross-section. Under flexural loading, composite failure is controlled not only by matrix stiffness, but also by the quality of the interface bond that is capable of resisting delamination and fiber pull-out. The presence of UGE on the fiber surface is thought to improve interface continuity, thereby increasing the composite's ability to resist flexural deformation before failure occurs. In contrast, adding UGE to the matrix does not provide a significant increase in flexural strength and, in some cases, shows a decrease in performance. This reinforces the indication that UGE is more effective as an interface modification agent than as a matrix additive in polyester-ANHF composite systems.

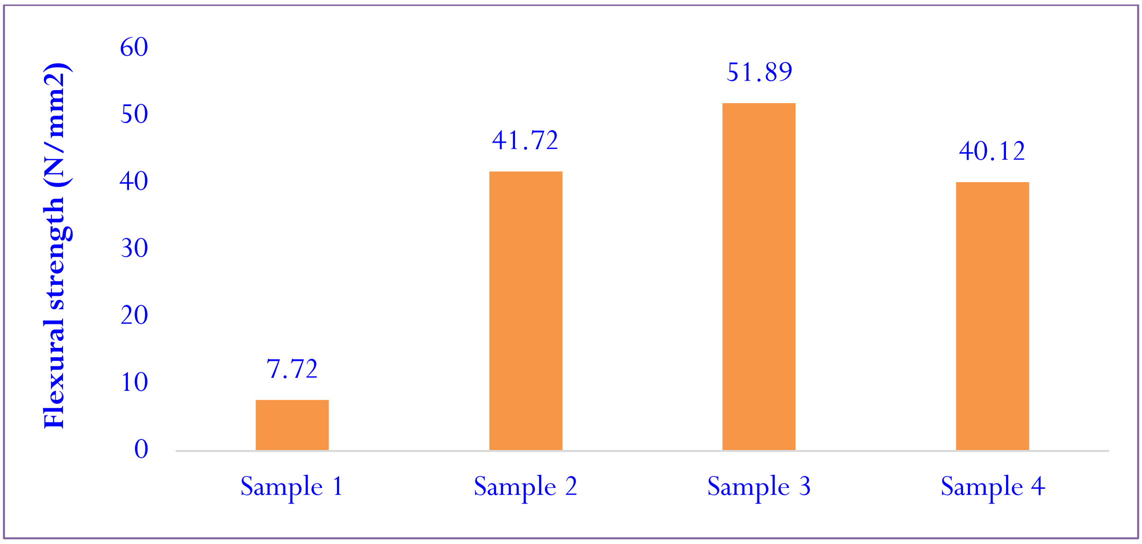
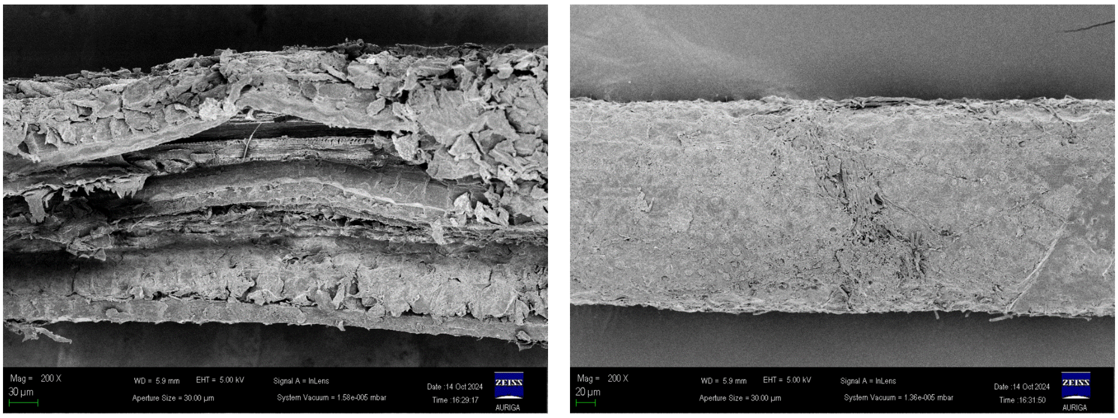


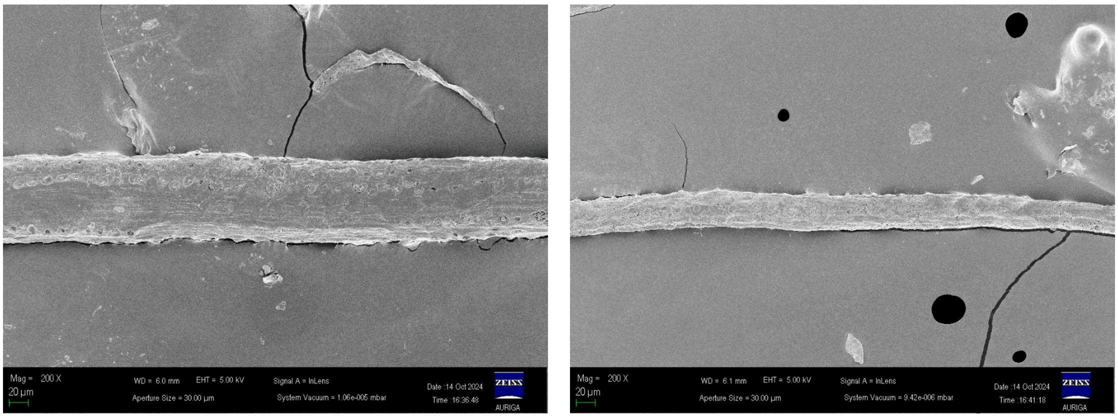
Figure 4. Flexural strength of all samples

The SEM results in Figure 5 show clear differences in the morphology of the fiber-matrix interface due to variations in fiber treatment and the addition of UGE. Sample 1, which was only treated with NaOH, showed a rough fiber surface with open fibrils and indications of delamination and fiber pull-out, reflecting the dominance of interlocking mechanical bonds due to the removal of lignin and hemicellulose. In Sample 2, the addition of UGE to the matrix resulted in a more homogeneous and compact interface, with reduced voids around the fibers, indicating increased physical adhesion between the fibers and the matrix. Sample 3, which involved UGE immersion of the fibers after alkali treatment, showed a smoother and coated fiber surface, indicating the formation of a polyphenol layer on the fibers that improved chemical compatibility with the resin, although microcracks were still observed around the interface. The combination of UGE treatment on the fibers and matrix in Sample 4 resulted in the most continuous and compact interface morphology with minimal defects, indicating synergy between the modification of the fiber and matrix surfaces. Overall, the SEM images confirm that alkali treatment plays a major role in increasing the surface roughness of the fibers, while the presence of UGE, both on the fibers and in the matrix, contributes to improving the quality and continuity of the fiber-matrix interface.

Mechanical testing results show that the addition of UGE as an additive tends to reduce tensile strength, while UGE treatment of fibers significantly increases flexural strength. In tensile testing, the failure mechanism is dominated by load transfer parallel to the fiber direction, where matrix stiffness and mechanical interlocking play a major role. The addition of UGE, especially in the matrix, is thought to reduce the stiffness and intrinsic strength of the matrix due to increased plasticity, thereby reducing tensile load transfer efficiency despite improved interface quality. A similar phenomenon has also been reported in PVA/UGE biocomposite films, where increased flexibility and toughness of the material are not always accompanied by an increase in maximum tensile strength [30]. On the other hand, in flexural tests, the material experiences a combination of tensile, compressive, and shear stresses that are highly sensitive to interface quality and crack propagation resistance. UGE treatment of the fibers results in a more continuous interface and improves fiber-matrix chemical compatibility through catechin phenolic group interactions, thereby making the flexural stress distribution more uniform and suppressing delamination. Thus, UGE functions primarily as an effective bio-based interface modification agent in improving flexural performance, but is not always advantageous for applications dominated by tensile loading.



Sample 1 Sample 2



Sample 3 Sample 4

Figure 5. SEM micrographs of all samples at 200x magnifications

4. Conclusion

This study examines the effect of alkali treatment and UGE addition on the mechanical properties and morphology of the interface of ANHF composites with a polyester matrix. Tensile testing results show that NaOH treatment without UGE addition produces the highest tensile strength, which is associated with increased fiber surface roughness and a dominant mechanical interlocking mechanism. Conversely, the addition of UGE as an additive, particularly in the matrix, tended to reduce tensile strength due to decreased matrix stiffness despite improved interface quality. In flexural testing, UGE treatment of the fibers significantly increased flexural strength, indicating that improved chemical compatibility and interface continuity were more effective in withstanding combined tensile-compressive stresses and inhibiting delamination. SEM observations confirm that the combination of alkali treatment and UGE produces a more continuous and defect-free fiber-matrix interface, although the best interface conditions do not always correlate with maximum tensile strength. Overall, this study confirms that the effectiveness of UGE as a bio-based additive in ANHF-polyester composites is highly dependent on the type of loading applied. These findings provide new insights into the role of natural additives in modifying the interface of natural fiber composites, and serve as an important basis for designing environmentally friendly composites with mechanical performance tailored to specific structural applications.

Author's declaration

Author contribution

Rahmat Azis Nabawi: conceptualization, methodology, investigation, formal analysis and writing-original draft. **Syahril**: methodology, validation, and writing-review & editing. **Hairul Abral**: methodology and validation.

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Data Availability

The raw data used to support the findings of this study are available from the corresponding author upon reasonable request.

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Competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

Ethical clearance

Ethical approval is not applicable to this manuscript.

AI statement

The authors confirm that this article is an original work and that no artificial intelligence tools were used in the preparation of the manuscript, including writing, editing, or generating tables and figures.

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