

EFFECT OF NaOH AND Ca(OH)₂ ALKALI TREATMENT TIME ON *PURUN TIKUS* FIBER PROPERTIES

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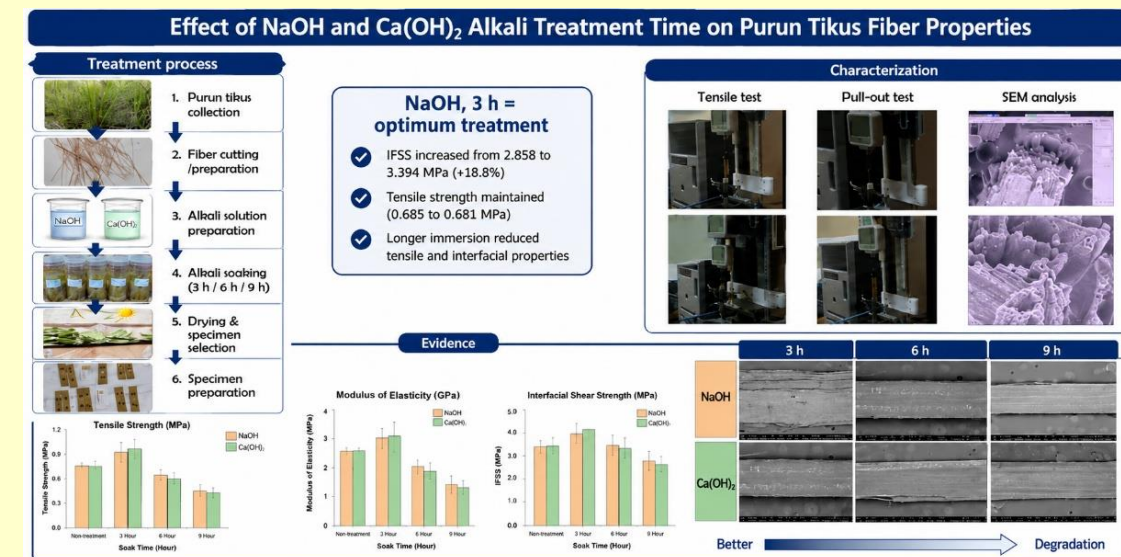
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Abstract

Purun tikus fiber (*Eleocharis dulcis*) is a promising natural fiber for environmentally friendly composite reinforcement due to its abundant availability and relatively high cellulose content. However, the presence of lignin, hemicellulose, and surface impurities can reduce interfacial bonding and mechanical performance. This study investigated the effect of alkali treatment type and immersion time on the mechanical and morphological properties of single *purun tikus* fibers. The fibers were treated in 5 wt.% NaOH and Ca(OH)₂ solutions for 3, 6, and 9 h, with three specimens prepared for each treatment variation. Mechanical characterization included single-fiber tensile testing based on ASTM C1557 to determine tensile strength, strain, and elastic modulus, pull-out testing to evaluate interfacial shear strength, and field emission scanning electron microscopy (FESEM) analysis to observe surface morphology changes. The results showed that the most favorable treatment condition was obtained using NaOH for 3 h. Under this condition, the interfacial shear strength increased from 2.858 MPa to 3.394 MPa, corresponding to an improvement of approximately 18.8%, while the tensile strength was maintained at 0.681 MPa, close to that of the untreated fiber (0.685 MPa). In contrast, longer immersion times of 6 and 9 h reduced the tensile strength and interfacial performance, indicating fiber degradation due to excessive alkalization. FESEM observations confirmed that short-duration alkali treatment improved surface cleanliness and roughness, whereas prolonged treatment caused cracks and structural damage. These findings indicate that controlled NaOH treatment is more effective than Ca(OH)₂ in improving the suitability of *purun tikus* fiber as a composite reinforcement.



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Keywords: fiber, alkalization, NaOH, Ca(OH)₂

Introduction

Natural fibers have attracted increasing attention as reinforcing materials for polymer composites because they are abundant, lightweight, relatively inexpensive, biodegradable, and derived from renewable resources. In composite systems, fibers function as the main load-bearing phase, while the matrix transfers and distributes the applied load between fibers. Therefore, the suitability of natural fibers as reinforcement is strongly influenced by their tensile properties, surface characteristics, fiber–matrix adhesion, and resistance to damage during processing [1], [2]. However, most lignocellulosic fibers contain cellulose, hemicellulose, lignin, pectin, waxes, and other surface impurities. The presence of non-cellulosic components may reduce compatibility with polymer matrices, especially hydrophobic matrices, and can limit stress transfer at the fiber–matrix interface [3–5].

Purun tikus fiber (*Eleocharis dulcis*) is a locally available lignocellulosic fiber that grows abundantly in swamp areas of South Kalimantan. This fiber has potential as an alternative reinforcement material because of its availability, natural origin, and cellulose-based structure [3], [6]. Previous research on hybrid composites reinforced with Alabio duck feathers and *purun tikus* fiber showed that alkalization using 5% NaOH affected the wear, hardness, and void characteristics of polyester-based composites [7]. This finding supports the relevance of *purun tikus* fiber as a local natural reinforcement candidate. However, composite-level properties alone are not sufficient to explain the fundamental contribution of the fiber. Therefore, single-fiber characterization is required to understand the tensile response and interfacial behavior of *purun tikus* fiber after chemical treatment.

Alkali treatment is one of the most commonly used chemical modification methods for natural fibers because it can modify the chemical composition and surface morphology of lignocellulosic fibers. This treatment removes part of the hemicellulose, lignin, waxes, pectin, and residual surface impurities, resulting in a cleaner and rougher fiber surface. Such surface modification is generally beneficial for natural fibers because it can improve mechanical interlocking between the fiber and matrix and enhance the stress-transfer capability in composite systems [5], [8]. Previous studies have reported the positive effect of alkali treatment on several natural fibers, such as jute, date palm, bamboo, and other lignocellulosic fibers. Roy et al. showed that mild alkali treatment improved the mechanical reliability of jute fibers by modifying the fiber surface and reducing non-cellulosic components [9]. Oushabi et al. reported that alkali treatment of date palm fibers improved surface roughness and contributed to better interaction between the fiber and polyurethane matrix [10]. Similar findings were also reported by Elmoudnia et al. and Verma and Goh, who explained that controlled alkaline treatment can improve fiber morphology, stiffness, and compatibility with polymer matrices, while excessive treatment may cause fiber degradation and reduce mechanical performance [11], [12]. Therefore, previous studies indicate that alkali treatment can be effective for improving natural fiber performance, but its effect strongly depends on the fiber type, treatment condition, and immersion duration.

Previous studies have also provided quantitative evidence that alkali treatment can significantly affect the mechanical and interfacial performance of natural fibers. Roy et al. reported that mild alkali treatment increased the tensile strength and elongation at break of jute fibers by approximately 82% and 45%, respectively, while reducing the fiber diameter by about 37% [9]. Oushabi et al. investigated date palm fibers treated with NaOH concentrations of 0, 2, 5, and 10 wt.% and found that alkali treatment improved the fiber–polyurethane interfacial behavior, with 5 wt.% NaOH reported as the optimum concentration for bond characteristics [10]. In a pull-out-based study on bamboo fibers, Liu et al. showed that appropriate NaOH treatment improved bonding performance while maintaining the initial mechanical properties of the fibers [13]. For $\text{Ca}(\text{OH})_2$ treatment, Anggono et al. reported that sugarcane bagasse fibers treated with 14% $\text{Ca}(\text{OH})_2$ at 60–70°C for 4 h produced the highest tensile strength of 11.30 MPa in bagasse fiber–polypropylene composites when 5 cm fibers were used at a fiber/PP weight ratio of 25/75 [14]. These quantitative findings indicate that alkaline treatment can improve tensile and interfacial performance, but the optimum condition depends strongly on alkali type, concentration, treatment duration, and fiber characteristics.

Sodium hydroxide (NaOH) is widely used for natural fiber treatment because of its strong ability to remove non-cellulosic components and modify fiber surface morphology. In terms of tensile performance, NaOH treatment can improve fiber stiffness and stress-transfer capability when the treatment condition is properly controlled. The removal of hemicellulose, lignin, waxes, and surface impurities can increase the relative cellulose content and produce a cleaner fiber surface, which contributes to better tensile response [9], [10], [12]. However, the effectiveness of NaOH strongly depends on treatment concentration and immersion time. Insufficient treatment may leave residual impurities on the fiber surface, whereas excessive treatment can damage the cellulose-rich load-bearing structure, resulting in reduced tensile strength and premature failure [10], [12].

In relation to pull-out behavior and interfacial adhesion, alkali treatment can improve the interaction between natural fibers and polymer matrices by increasing surface roughness and mechanical interlocking. A rougher and cleaner fiber surface provides better contact with the matrix, allowing more effective stress transfer across the fiber–matrix interface. Previous pull-out-based studies have shown that appropriate alkali treatment can increase interfacial bonding strength, while over-treatment may weaken the fiber or interface due to structural deterioration [13], [15]. Therefore, the effect of alkali treatment should not only be evaluated from tensile properties, but also from interfacial shear strength obtained through pull-out testing.

In addition to NaOH, calcium hydroxide ($\text{Ca}(\text{OH})_2$) can also be used as an alkaline treatment agent. Compared with NaOH, $\text{Ca}(\text{OH})_2$ generally provides a milder alkaline environment, which may produce different effects on tensile properties and interfacial adhesion [16], [17]. Anggono et al. reported that $\text{Ca}(\text{OH})_2$ treatment of sugarcane bagasse fibers influenced the tensile performance of bagasse fiber–polypropylene composites, indicating that calcium hydroxide can modify lignocellulosic fibers and affect their reinforcing

ability [14]. However, because $\text{Ca}(\text{OH})_2$ has lower alkalinity and different reactivity compared with NaOH , its ability to improve fiber surface roughness, tensile response, and fiber–matrix adhesion may vary depending on fiber type and treatment duration. Therefore, comparing NaOH and $\text{Ca}(\text{OH})_2$ treatments is important to determine which alkaline treatment is more effective in maintaining tensile integrity while improving interfacial bonding. Although many studies have discussed alkali treatment of natural fibers, research focusing specifically on *purun tikus* fiber is still limited. Most available studies have emphasized composite-level properties, while the fundamental behavior of single *purun tikus* fibers after alkaline treatment has not been sufficiently discussed. In particular, the combined effects of alkali type and immersion time on tensile strength, strain, elastic modulus, interfacial shear strength, and surface morphology remain unclear. This gap is important because the performance of natural fiber composites is strongly affected not only by the strength of the fiber itself but also by the quality of adhesion at the fiber–matrix interface.

Therefore, this study aims to evaluate the effect of alkali treatment using NaOH and $\text{Ca}(\text{OH})_2$ with immersion times of 3, 6, and 9 h on the mechanical and morphological properties of single *purun tikus* fibers. The novelty of this work lies in the comparative evaluation of two alkaline solutions under controlled soaking durations to determine their influence on tensile strength, strain, elastic modulus, interfacial shear strength, and surface morphology. The results are expected to provide a clearer understanding of the optimum alkali treatment condition for improving the suitability of *purun tikus* fiber as a potential reinforcement material in polymer composites.

Research Methods

This study used an experimental approach to evaluate the effect of alkali treatment type and immersion time on the mechanical and morphological properties of single *purun tikus* fibers (*Eleocharis dulcis*). The fibers were cleaned, mechanically separated into single fibers, and treated using 5 wt.% sodium hydroxide (NaOH) and 5 wt.% calcium hydroxide ($\text{Ca}(\text{OH})_2$) solutions for 3, 6, and 9 h. Untreated fibers were used as control specimens. After treatment, the fibers were rinsed with distilled water to remove residual alkali and dried under ambient conditions. Three specimens were prepared for each treatment variation.

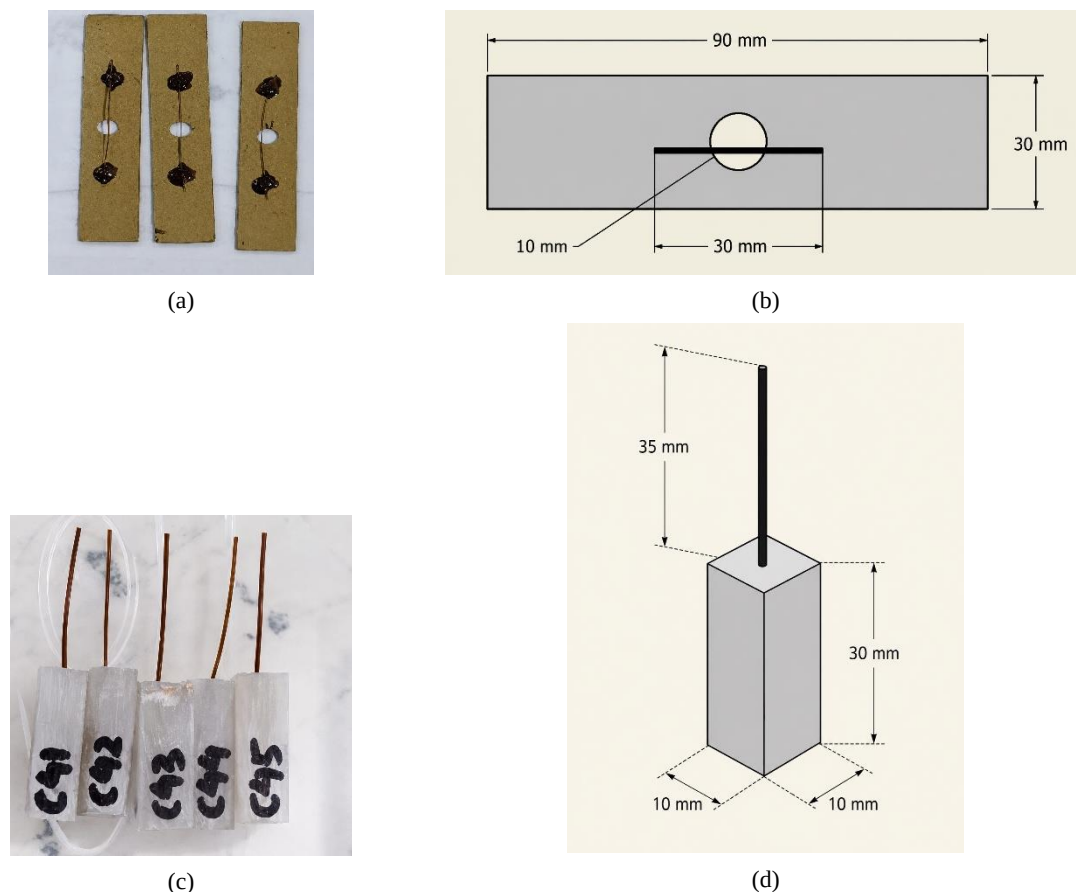


Figure 1. Schematic illustration of *purun tikus* single-fiber testing: (a) tensile test specimen, (b) tensile test configuration, (c) pull-out test specimen, and (d) pull-out test configuration.

Single-fiber tensile specimens were prepared according to ASTM C1557 by mounting each fiber on a paper frame to maintain axial alignment during testing. The effective gauge length was 30 mm. Tensile testing was performed using an IMADA CO. LTD ZTS testing machine with a 50 N load capacity and 0.2% accuracy. Before loading, the middle part of the paper frame was cut so that the tensile force was applied only to the single fiber. The tensile strength, strain, and elastic modulus were obtained from the recorded load–displacement data. The cross-sectional area of the fiber was measured from FESEM images using ImageJ software. Each image was calibrated using the scale bar in the micrograph, and the fiber boundary was manually traced to obtain the cross-sectional area. Because *purun tikus* fibers have irregular cross-sectional shapes, the measured area was used directly for tensile strength calculation. For pull-out analysis, the equivalent fiber diameter was calculated using Eq. (1):

$$d_{eq} = \sqrt{\frac{4A}{\pi}} \quad (1)$$

where d_{eq} is the equivalent fiber diameter and A is the cross-sectional area measured using ImageJ.

The single-fiber pull-out test was conducted to evaluate the interfacial shear strength (IFSS) between the fiber and epoxy matrix. One end of the fiber was embedded in the matrix at a controlled embedded length, while the free end was pulled axially until interfacial failure occurred. The maximum pull-out force was used to calculate IFSS using Eq. (2):

$$IFSS = \frac{F_{max}}{\pi \cdot d_{eq} \cdot L} \quad (2)$$

Where F_{max} is the maximum pull-out force, d_{eq} is the equivalent fiber diameter, and L is the embedded fiber length.

Surface morphology was observed using field emission scanning electron microscopy (FESEM; FEI–Quanta FEG 650). The samples were mounted on double-sided carbon tape and observed under low-vacuum conditions. FESEM observation was carried out at 20 kV accelerating voltage, spot size of 5, dwell time of 1 μ s, working distance of 10 mm, and vCD detector for BSE imaging. The morphology analysis focused on surface cleanliness, roughness, fibrillation, cracks, peeling, and structural damage caused by different alkali treatments and immersion times. The morphology analysis focused on surface cleanliness, roughness, fibrillation, cracks, peeling, and structural damage caused by different alkali treatments and immersion times. The results were analyzed comparatively by comparing untreated fibers with NaOH- and Ca(OH)₂-treated fibers at different immersion times. The mechanical properties and IFSS values were presented as average values with standard deviation, while FESEM observations were used to support the interpretation of the mechanical behavior.

Results and Discussion

The results of this study generally showed that alkali treatment affected the mechanical and morphological properties of *purun tikus* fiber depending on the type of alkali solution and immersion time. In general, short-duration treatment improved fiber performance, whereas prolonged treatment tended to reduce the mechanical properties due to structural degradation. This trend is consistent with previous studies on alkali-treated natural fibers, which reported that moderate treatment improves surface condition and fiber–matrix interaction, while excessive treatment may damage the fiber structure and lower its load-bearing capability [10], [18]. Therefore, the discussion in the following sections focuses on the effect of alkali type and immersion time on tensile strength, strain, elastic modulus, interfacial shear strength, and surface morphology of *purun tikus* fibers under controlled conditions.

1. Tensile Strength

Fig. 2 shows the tensile strength of *purun tikus* fibers after treatment with NaOH and Ca(OH)₂ at different immersion times. The results indicate that alkali treatment affected the tensile strength depending on the type of alkaline solution and treatment duration. The 3 h treatment did not substantially increase the tensile strength compared with the untreated fiber; instead, it mainly maintained the tensile strength at a comparable level. In the case of NaOH treatment, the tensile strength at 3 h was 0.681 MPa, which was very close to that of the untreated fiber, 0.685 MPa. This suggests that short-duration NaOH treatment was able to modify the fiber surface without causing significant degradation of the load-bearing structure. A similar tendency has been reported by Roy et al., who found that mild alkali treatment can improve the surface condition and mechanical reliability of natural fibers when the treatment is properly controlled [9].

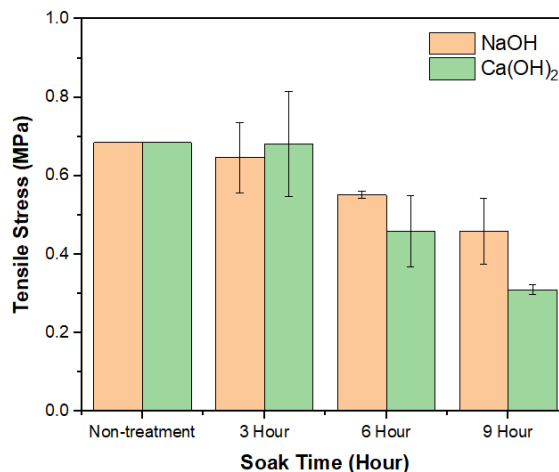


Figure 2. Tensile strength of *purun tikus* fibers treated with NaOH and Ca(OH)₂ at different immersion times.

When the immersion time was extended to 6 and 9 h, the tensile strength decreased for both NaOH and Ca(OH)₂ treatments. This reduction indicates that prolonged alkaline exposure may have caused excessive removal of non-cellulosic components and partial damage to the fiber structure. Although alkali treatment can improve surface cleanliness and roughness, excessive treatment may weaken the cellulose-rich region that contributes to tensile load resistance. This interpretation is consistent with previous studies reporting that moderate alkalization can improve fiber properties, whereas excessive treatment may lead to fiber degradation and reduced mechanical performance [10], [18], [19]

Compared with Ca(OH)₂, NaOH produced a stronger modification effect because of its higher alkalinity and greater ability to remove hemicellulose, lignin, waxes, and residual surface impurities. However, this stronger effect was only favorable at short immersion time. At prolonged immersion times, NaOH treatment tended to cause a more pronounced reduction in tensile strength, indicating that aggressive alkalization can damage the fiber structure if the treatment duration is not properly controlled. Similar observations have been reported in alkali-treated natural fibers, where excessive NaOH exposure caused surface deterioration, fibrillation, and weakening of the fiber load-bearing structure [20], [21].

In contrast, Ca(OH)₂ showed a milder effect on tensile behavior. This may be related to its lower alkaline strength compared with NaOH, which results in less aggressive removal of non-cellulosic components. However, the reduction in tensile strength at longer Ca(OH)₂ immersion times indicates that prolonged exposure to a milder alkaline solution can still affect the structural integrity of the fiber. Therefore, the effectiveness of alkali treatment should be understood as a balance between sufficient surface modification and the preservation of fiber strength.

Overall, the tensile strength results show that the 3 h treatment, particularly using NaOH, should not be interpreted as producing the highest tensile strength improvement. Rather, it can be considered the most favorable condition because it maintained the tensile strength close to that of the untreated fiber while providing surface modification that contributed to improved interfacial performance. This interpretation is supported by FESEM observations, where short-duration alkali treatment produced a cleaner and rougher surface, whereas longer immersion times caused cracks and structural damage.

2. Strain

Fig. 3 shows the strain behavior of *purun tikus* fibers after treatment with NaOH and Ca(OH)₂ at different immersion times. In general, alkali treatment affected the deformation capability of the fibers, but the strain response did not show a completely linear trend for all treatment conditions. Compared with the untreated fiber, most treated fibers showed lower strain, indicating that alkalization tended to reduce fiber deformability. This behavior may be associated with the partial removal of amorphous constituents, such as hemicellulose, lignin, waxes, and other residual surface impurities, which can make the fiber structure stiffer and less able to elongate under tensile loading [12].

For NaOH treatment, the strain behavior showed a non-uniform trend. The strain decreased after 3 h of treatment, increased again at 6 h, and then decreased at 9 h. This anomaly suggests that the effect of NaOH treatment on fiber deformation was not only controlled by immersion time, but also by the natural variability of *purun tikus* fibers, non-uniform alkali penetration, and inconsistent removal of amorphous components along the fiber structure. Natural fibers commonly exhibit variation in diameter, cross-sectional geometry,

lumen size, microfibril orientation, and local defects; therefore, their strain response can vary even under the same treatment condition. Similar considerations have been discussed in previous studies on alkali-treated natural fibers, where mechanical response was influenced by both chemical modification and inherent fiber variability [10], [12], [19].

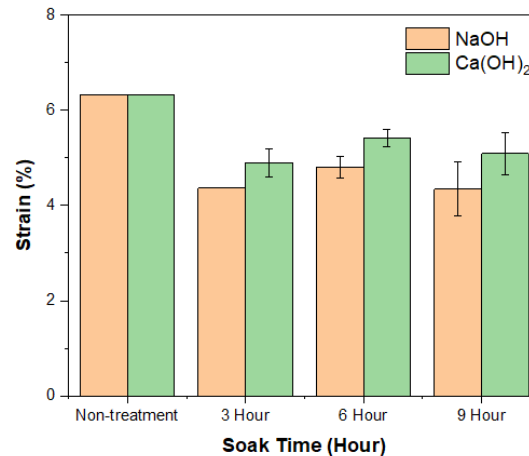


Figure 3. Strain of *purun tikus* fibers treated with NaOH and Ca(OH)₂ at different immersion times.

The increase in strain at 6 h for NaOH-treated fibers may indicate that partial fibrillation or localized surface opening occurred before severe structural degradation dominated the response. In this condition, the fiber may still retain some deformability because alkali penetration and removal of amorphous constituents were not completely uniform. However, at 9 h, the decrease in strain suggests that prolonged NaOH exposure caused more severe structural deterioration. This interpretation is supported by FESEM observations, which showed that NaOH treatment at longer immersion times produced surface cracks, local damage, and more severe fiber degradation. Such morphological damage can reduce the ability of the fiber to deform and may promote premature failure under tensile loading [20], [21].

In contrast, Ca(OH)₂-treated fibers showed a more gradual change in strain. This trend may be related to the milder alkaline character of Ca(OH)₂ compared with NaOH, which results in less aggressive modification of the fiber surface and slower removal of amorphous components [3], [14]. Nevertheless, longer immersion times still tended to reduce strain, indicating that prolonged exposure to a milder alkaline solution can also affect fiber flexibility. FESEM observations of Ca(OH)₂-treated fibers showed that surface cracks and gradual surface degradation became more visible as immersion time increased, supporting the reduction in strain at longer treatment durations.

Overall, the strain results indicate that alkali treatment altered the balance between fiber stiffness and deformability. The non-uniform strain trend, especially in the NaOH-treated fibers, should not be interpreted only as a direct function of immersion time. Instead, it reflects the combined influence of alkali-induced surface modification, natural fiber variability, non-uniform alkali penetration, and structural changes observed in FESEM images. Therefore, the strain behavior supports the need to control alkali treatment duration to obtain surface modification without excessive loss of fiber deformability.

3. Modulus Young

Fig. 4 shows the Young's modulus of *purun tikus* fibers after treatment with NaOH and Ca(OH)₂ at different immersion times. In general, alkali treatment influenced the stiffness of the fibers, although the interpretation should be made carefully because the relatively large error bars indicate variability among individual natural fiber specimens. Such variability is common in lignocellulosic fibers because their mechanical response can be affected by differences in diameter, cross-sectional shape, lumen size, microfibril orientation, and local defects.

For NaOH treatment, the Young's modulus tended to increase at 3 h compared with the untreated fiber. This tendency may be associated with the partial removal of amorphous components, such as hemicellulose, lignin, waxes, and residual surface impurities, which can make the fiber structure relatively stiffer and less deformable [10], [12]. This interpretation is also consistent with the strain results, where lower strain generally indicates reduced deformability after alkali treatment. However, because of the data variation shown by the error bars, the increase in modulus should be interpreted as a tendency rather than a definitive improvement. When the immersion time was extended from 3 h to 6 h and 9 h, the Young's modulus of NaOH-treated fibers

decreased from 14.789 MPa to approximately 11.48 MPa and 11.58 MPa, corresponding to reductions of about 22.4% and 21.7%, respectively. This reduction may indicate that prolonged alkaline exposure no longer only modified the surface but also caused structural deterioration. FESEM observations support this interpretation, as longer NaOH immersion produced surface cracks, local damage, and more severe fiber degradation. These defects can act as stress concentration points and reduce the ability of the fiber to maintain stiffness under tensile loading [11], [22], [23].

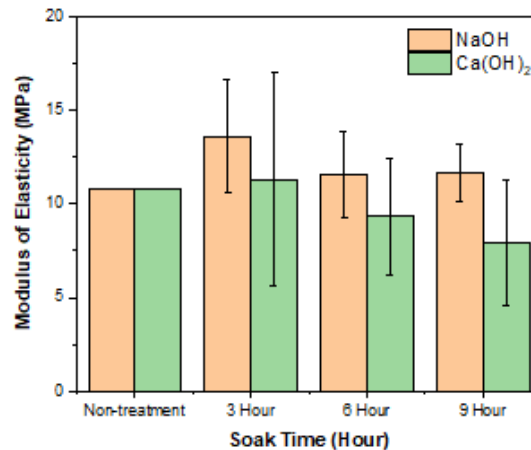


Figure 4. Young's modulus of *purun tikus* fibers treated with NaOH and Ca(OH)₂ at different immersion times.

For Ca(OH)₂ treatment, the change in Young's modulus appeared more gradual and less pronounced than that of NaOH. This behavior may be related to the milder alkaline character of Ca(OH)₂, which can result in slower and less aggressive removal of non-cellulosic components [14], [16]. Nevertheless, the presence of error bars suggests that the difference between treatment conditions should not be overinterpreted. FESEM observations showed that longer Ca(OH)₂ immersion also led to visible surface cracks and gradual surface degradation, indicating that prolonged treatment can still affect fiber integrity even when a milder alkaline solution is used.

Overall, the Young's modulus results indicate that short-duration alkali treatment, especially NaOH treatment for 3 h, tended to increase fiber stiffness without causing severe surface damage. However, this result should be interpreted together with tensile strength, strain, and FESEM observations. The modulus trend alone is not sufficient to conclude structural improvement because natural fiber variability and surface defects strongly affect the mechanical response. Therefore, the most favorable treatment condition should be determined based on the balance between maintained tensile strength, controlled stiffness, improved interfacial behavior, and limited morphological damage.

4. Pull-Out

Fig. 5 shows that alkali treatment affected the interfacial shear strength of *purun tikus* fiber. In general, short-duration treatment improved the interfacial response, whereas prolonged immersion reduced the bonding performance. This indicates that moderate alkalization was sufficient to remove part of the non-cellulosic substances from the fiber surface and increase surface roughness, thereby promoting better mechanical interlocking and stronger fiber–matrix adhesion. A similar trend was reported by Liu et al., who found that appropriate alkali treatment removed non-structural surface substances and improved bonding performance in pull-out behavior, while maintaining the initial mechanical properties of the fiber [13].

For NaOH treatment, the highest interfacial shear strength was obtained at 3 h, followed by a decrease at 6 h and 9 h. This result suggests that short-duration treatment produced sufficient surface modification without causing severe structural damage. However, when the immersion time was prolonged, the fiber surface was likely over-treated, leading to degradation of the load-bearing structure and weaker interfacial bonding. Widodo et al. also reported that alkali treatment improved adhesivity and pull-out performance by enhancing surface roughness and interfacial bonding, confirming the beneficial effect of controlled alkali treatment on composite performance [15]. A similar tendency was also observed for Ca(OH)₂ treatment, where the interfacial performance improved at the early treatment stage but declined with longer immersion time. Compared with NaOH, however, Ca(OH)₂ showed lower effectiveness in enhancing the pull-out response in the present study. The reduction in interfacial performance at longer treatment duration indicates that excessive alkalization can weaken fiber–matrix adhesion rather than improve it. This interpretation is supported by Istana

et al., who showed that increasing alkali-treatment duration could reduce internal bonding strength, indicating deterioration of interfacial stability under over-treatment conditions [24].

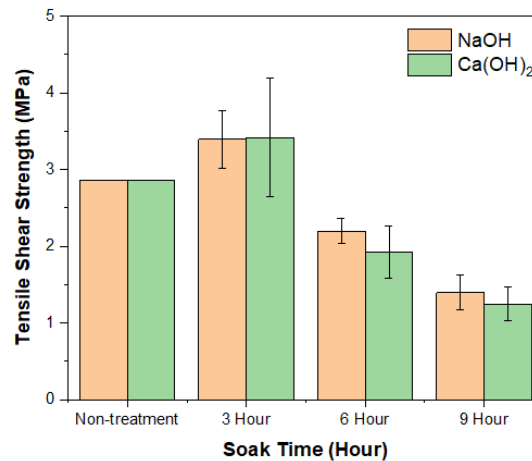


Figure 5. Interfacial shear strength of *purun tikus* fibers treated with NaOH and Ca(OH)₂ at different immersion times.

Overall, the pull-out results indicate that the optimum interfacial performance was achieved under short-duration alkali treatment, particularly with NaOH. These findings confirm that treatment effectiveness depends on achieving a balance between improving surface characteristics and avoiding excessive degradation of the fiber structure.

5. Surface Morphology of NaOH-Treated Fibers

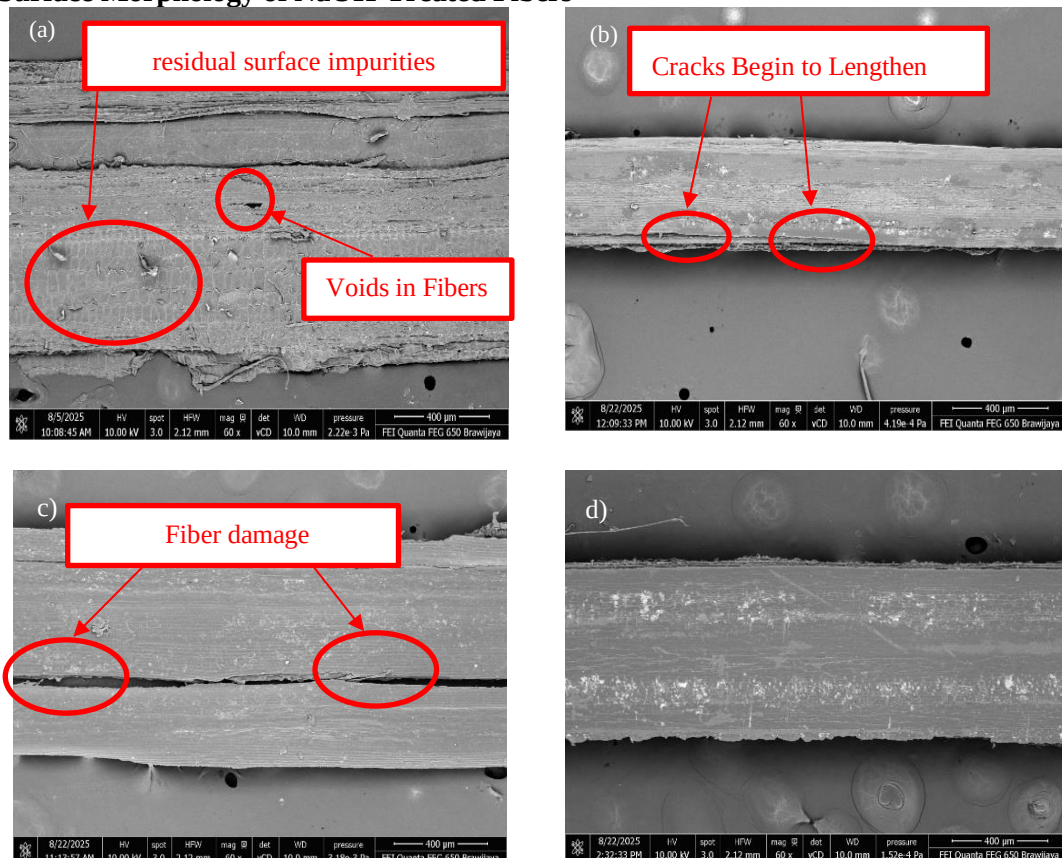


Figure 6. Surface morphology of *purun tikus* fibers after NaOH treatment at different immersion times: (a) 3 h, (b) 6 h, (c) 9 h, and (d) untreated fiber.

Fig. 6 shows the surface morphology of *purun tikus* fibers after NaOH treatment at different immersion times. The untreated fiber exhibited a relatively compact surface with residual surface impurities, indicating that non-cellulosic components and natural surface layers were still present on the fiber surface. After 3 h of NaOH treatment, the fiber surface became cleaner and rougher. This suggests that short-duration NaOH treatment partially removed residual surface impurities and non-cellulosic constituents, such as hemicellulose, lignin, waxes, and extractives. The formation of a rougher surface is beneficial because it can increase surface irregularity and promote mechanical interlocking between the fiber and matrix [12], [20].

At 6 h of NaOH treatment, the fiber surface showed more pronounced morphological changes, including surface cracks and local surface damage. This indicates that the effect of NaOH was no longer limited to surface cleaning, but had begun to affect the structural integrity of the fiber. At 9 h, the surface deterioration became more severe, as indicated by more visible damage and degradation on the fiber surface. This condition suggests that prolonged NaOH exposure caused excessive alkalization, which may weaken the cellulose-rich load-bearing structure of the fiber. Similar behavior has been reported in alkali-treated natural fibers, where excessive treatment can lead to microstructural degradation, fibrillation, and deterioration of mechanical performance [10], [18], [21].

These FESEM observations support the tensile, strain, and modulus results. The 3 h NaOH treatment mainly maintained the tensile strength close to that of the untreated fiber while producing a cleaner and rougher surface. This surface condition also supports the improvement in interfacial shear strength because a rougher surface can enhance mechanical interlocking with the matrix [13], [15]. However, the cracks and surface degradation observed after longer immersion times explain the decrease in tensile strength and interfacial performance at 6 and 9 h. Therefore, the FESEM results indicate that NaOH treatment is effective only when the immersion time is properly controlled. Short-duration treatment provides beneficial surface modification, whereas prolonged treatment causes structural damage that reduces fiber performance.

6. Surface Morphology of $\text{Ca}(\text{OH})_2$ -Treated Fibers.

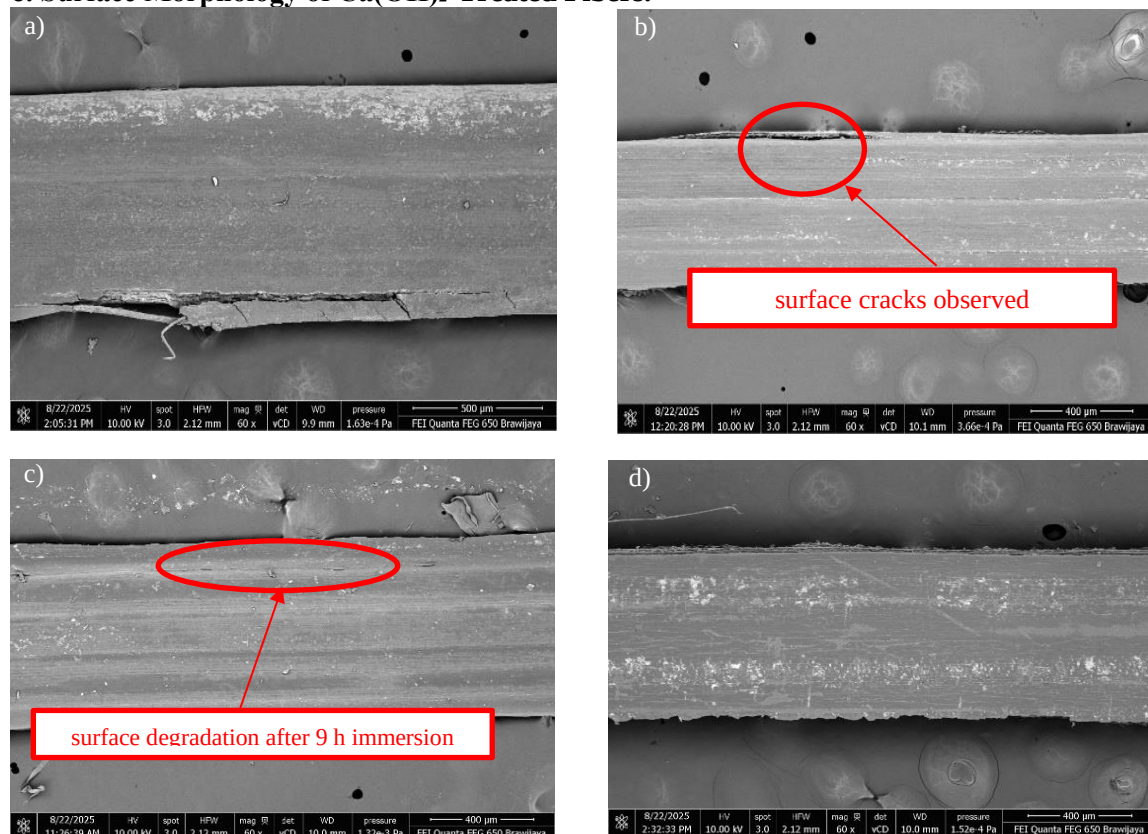


Figure 7. Surface morphology of *purun tikus* fibers after $\text{Ca}(\text{OH})_2$ treatment at different immersion times: (a) 3 h, (b) 6 h, (c) 9 h, and (d) untreated fiber.

Fig. 7 shows the surface morphology of *purun tikus* fibers after $\text{Ca}(\text{OH})_2$ treatment at different immersion times. Compared with the untreated fiber, the $\text{Ca}(\text{OH})_2$ -treated fibers showed changes in surface morphology, although the changes appeared more gradual than those observed in the NaOH-treated fibers.

After 3 h of treatment, the fiber surface became slightly cleaner and rougher, indicating partial removal of residual surface impurities and amorphous constituents. This type of surface modification can improve fiber–matrix interaction by increasing contact area and mechanical interlocking without causing severe surface damage [12], [20].

As the immersion time increased to 6 h, surface cracks became more visible on the $\text{Ca}(\text{OH})_2$ -treated fibers. At 9 h, surface degradation after prolonged immersion was observed, indicating that longer $\text{Ca}(\text{OH})_2$ treatment gradually affected the integrity of the fiber surface. However, compared with NaOH treatment, the morphological changes caused by $\text{Ca}(\text{OH})_2$ appeared less aggressive. This may be related to the milder alkaline character of $\text{Ca}(\text{OH})_2$ compared with NaOH, which can result in slower removal of non-cellulosic components and less severe structural damage under comparable treatment conditions [4], [14], [25].

The FESEM results of $\text{Ca}(\text{OH})_2$ -treated fibers are consistent with the mechanical behavior observed in this study. The moderate surface roughening at short immersion time may contribute to improved fiber–matrix interaction, but the development of surface cracks and gradual degradation at longer immersion times can reduce mechanical integrity and interfacial performance. Therefore, although $\text{Ca}(\text{OH})_2$ can modify the fiber surface, its effect is more limited than NaOH in producing strong surface modification. These observations confirm that the effectiveness of alkaline treatment depends not only on the type of alkali, but also on the balance between surface cleaning, roughness development, and preservation of fiber structural integrity [12], [20].

Conclusion

Alkali treatment affected the mechanical and morphological properties of single *purun tikus* fibers. Among the treatment conditions investigated, NaOH treatment for 3 h was identified as the most favorable condition because it improved the interfacial shear strength from 2.858 MPa to 3.394 MPa, corresponding to an increase of approximately 18.8%, while maintaining the tensile strength at 0.681 MPa, which was close to that of the untreated fiber at 0.685 MPa. The elastic modulus also increased from 10.821 MPa to 14.789 MPa, indicating an improvement of approximately 36.7%. Longer immersion times of 6 and 9 h reduced the tensile and interfacial performance of the fibers, indicating that prolonged alkali exposure caused over-treatment. FESEM observations supported these results, showing that short-duration treatment produced a cleaner and rougher fiber surface, whereas longer immersion caused cracks and surface degradation. Compared with $\text{Ca}(\text{OH})_2$, NaOH produced a more pronounced surface modification effect under short immersion time. Overall, controlled NaOH treatment for 3 h can improve the suitability of *purun tikus* fiber as a potential reinforcement material in polymer composites. Future studies are recommended to evaluate different alkali concentrations and validate the performance of treated *purun tikus* fibers in actual composite system.

Credit authorship

Akhmad Syarief: Conceptualization, Methodology, Data Curation, Formal Analysis, Investigation, Writing – Original Draft, Funding Acquisition, **Valentino Krisna Mukti:** Methodology, Data Curation, Formal Analysis, **Iphan Fitrian Radam:** Investigation, Writing – Original Draft, Writing – Review & Editing, **M. Nizar Ramadhan:** Writing – Original Draft, Writing – Review & Editing, Supervision, **Akhmad Ghiffary Budianto:** Writing – Original Draft, Writing – Review & Editing, Supervision, **Fadlyannur:** Methodology, Writing – Review & Editing, **Aulia Aufa Ramadasari:** Data Curation, Formal Analysis, **Rizqi Ilmal Yaqin:** Writing – Review & Editing, Supervision.

Declaration of Competing Interest

The authors declare no competing interests.

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