

## The Influence of Sodium Chloride Treatment on the Sisal Fiber Bundle's Properties

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### ABSTRACT

Currently, composite board manufacturing using natural fiber has the potential to be expanded due to environmental awareness. To produce high-performance natural fiber, treatment is needed to improve natural fiber's mechanical and physical properties. One of chemical treatments is by using sodium chloride (NaCl). This study aimed to investigate the characteristics of sisal fiber after NaCl treatment. The concentrations of NaCl treatment were 1, 3, and 5 (wt.%) at room and boiling temperature and the soaking duration was 1 hour. Meanwhile, tensile strength, strain, and Young's modulus were tested to evaluate the mechanical properties. Fiber bundle diameter, weight change due to treatment, and contact angle were tested to assess the effect of NaCl treatment. Sisal fiber bundle was treated with 5 wt.% NaCl for 1 h exhibited the highest value of tensile strength, Young's modulus, reduction of fiber bundle diameter, percentage of weight change, and decrement of contact angle in comparison with untreated fiber bundle. Treatment with 5 wt.% NaCl at boiling temperature successfully increased the tensile strength and Young's modulus by 48.39% and 76.8%, respectively, compared to untreated fibers. NaCl treatment was shown to be an effective method to improve the mechanical properties and wettability of fibers, which has potential for application in high-performance plant fiber composites. The surface of sisal fiber treated with 5 wt.% NaCl at boiling temperature appeared rougher than that of the untreated one. In addition, this treatment also reduced the contact angle between the fiber and the adhesive by 38.71% compared to the untreated.

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**Keywords:** *Mechanical properties, NaCl treatment, physical properties, sisal fiber*

## I. Introduction

Plant fibers have great potential to be utilized in material engineering, along with increasing public awareness of environmental conservation [1], [2]. There are various types of natural plant fibers, such as kapok, hemp, kenaf, banana, coconut, bamboo, and sisal. Recently, sisal fiber has become one of the most popular [3], [4] and has been widely researched and developed [5]. Sisal is a general term for fibers derived from the bark of plants of the genus *Agave*, family *Agavaceae*, especially the species *Agave sisalana*, which generally grows in tropical and subtropical regions such as Indonesia. From an environmental perspective, sisal has the ability to absorb nitrogen and phosphorus from the soil and carbon dioxide from the air in large quantities. Sisal plants produce fibers with superior mechanical and chemical properties among other natural fibers [2], [6] and are capable of forming composites with good mechanical characteristics [5], [7].

Several studies have examined the development of sisal fiber composites with various types of adhesives. Epoxy, for example, has a low molecular weight, and its monomers show



minimal shrinkage during the curing process [8], [9]. Previous studies have shown that natural fiber composites using epoxy have good mechanical strength and adequate dimensional stability [7], [10], [11]. The strength of natural fibers is the main factor influencing the properties of these composite boards [12]. Treatment of fibers is recommended to improve the quality of fibers [13], [14], [15] and their composites [7], [14], [16]. High fiber properties can produce mechanical properties such as flexural and tensile strength in composite board products. The mechanical strength of fiber is also an important factor that can affect the properties of the resulting composite board [15]. Improving the properties of sisal fiber can be obtained by providing several modifications such as physical, mechanical, and chemical modifications so that they can improve the properties of sisal fiber and the resulting composite board [17].

Various studies have reported several different fiber treatment methods. Chemical treatments are used to prepare fibers to be more optimal for composite applications, producing high-quality single fibers [1], [18], [19]. Natural fibers contain cellulose, hemicellulose, pectin, and lignin and are rich in hydroxyl groups. They are strongly polar and hydrophilic materials whilst polymer materials, which exhibit hydrophobicity. Therefore, the chemical modification of the natural fiber surface utilizing treatment is one of the largest fields of the latest study to increase the compatibility and strength of the interface bond [20]. Chemical treatment for bleaching, acetylation and alkali increases the adhesion of fibers by increasing roughness through the surface from dirt and by disrupting the process of absorption of moisture through the OH-group layer [5], [21]. However, the chemicals used usually have a pH far from neutral, which can cause dissolution of metal oxides in production equipment [22]. Therefore, the use of materials with a pH close to neutral for fiber treatment is a potential solution, for example by utilizing seawater which has a pH between 8.08 and 8.33 [23].

Seawater has been shown to increase the tensile strength of fibers and the ability of fibers to interlock with adhesives [14]. Seawater contains 89.86-92.24% NaCl components [24], which is higher than other components such as sulfur and carbonate. Mardin et al. [14] stated that NaCl in seawater can affect the characteristics of natural fibers. In addition, the duration of immersion in seawater also affects fiber properties. Research by Setyayunita et al. [15] revealed that composite boards from kenaf fibers treated with NaCl have better properties compared to untreated fibers. Based on this study, the characteristics of natural fibers play an important role in determining the quality of composite boards. In previous research, increasing NaCl from 1 wt.% to 3 wt.% and even up to 5 wt.% improved the properties of the kenaf fiber and composite boards produced. The use of pure NaCl in kenaf fiber can increase the tensile strength of the fiber and Young's modulus up to 16.06% and 24.29% respectively at room temperature conditions and 5% NaCl for 1 hour at room temperature. The use of higher levels of chemicals can cause damage to the fiber and reduce the fiber's properties. However, until now there has been no study on the effect of NaCl treatment temperature on the characteristics of natural fibers. Therefore, this study will examine the effect of different NaCl treatment conditions on the characteristics of sisal fibers, such as tensile strength, fiber diameter, mass changes, and contact angles between fibers and adhesives. The fiber surface will also be observed to compare the characteristics of the fiber after treatment with the untreated fiber.

## II. Material and Methods

### 1. Materials

Sisal fiber from Blitar, East Java, Indonesia, was used as raw material. For the chemical treatment of sisal fiber, NaCl with 99% purity from Sigma Aldrich (manufactured in Darmstadt, Germany) was used. Meanwhile, epoxy and hardener (oil-based adhesive) produced by Alfatama Inticipita (Indonesia) were used as adhesives in contact angle testing.

### 2. NaCl Treatment

Sisal fibers were collected and soaked in NaCl solution with concentrations of 1%, 3%, and 5% weight (wt.%) for one hour at room temperature and boiling temperature ( $96\pm 2^\circ\text{C}$ ). After soaking, the fibers were washed with water using a solution to fiber ratio of 20:1 (w/w). After that, the fibers were rinsed with water until the pH reached 7 and then dried.

### 3. Tensile Properties

Tensile testing of fiber bundles was performed using a universal testing machine (Instron 3360 Series Dual Column Table Frames, USA) with a cross-head speed of 5 mm/min. Typically, the samples were placed between two rectangular cardboard sheets, each with a rectangular hole in the center measuring 15 x 90 mm. At least three samples from each sisal fiber bundle were tested, and the testing machine recorded the failure force and cross-head displacement. Untreated fiber bundles were also tested for comparison.

### 4. Fiber Bundle Diameter and Weight Change

The fiber bundle diameter was measured from three random fiber bundles, with fiber images obtained using a Dino-Lite Digital Microscope (AM413ZT, Dino-Lite, AnMo Electronics Corporation, Taiwan). Diameter measurements were then performed using ImageJ software. The diameter of untreated fiber bundles was also measured for comparison. The weight change of the NaCl-treated fiber bundles was calculated based on the difference in weight before and after treatment. At least three samples were tested for each treatment condition.

### 5. Morphology Observation

Untreated fibers and those treated with 5 wt.% NaCl solution using room and boiling temperature were observed using a field emission scanning electron microscope (FE-SEM, JEOL JSM 650 LA, Japan). Before observation, the samples were coated with a thin layer of platinum. Observations were carried out in electron mode with a beam current of 10 mA and an accelerating voltage of 1.5 kV. The morphology of sisal fibers before and after 5 wt.% NaCl treatment for 1 h was analyzed to see the differences in their surface structures.

### 6. Contact Angle of Sisal Fiber Bundle

The fiber bundle specimens were conditioned at room temperature and humidity for one week. The epoxy solution was then dropped onto the fiber surface using a micropipette (100 $\mu\text{m}$ ), and photographs were taken 10 seconds after dropping with a Dino-Lite Digital Microscope (AM413ZT, Taiwan). The contact angle was calculated based on the height and length of the drop string, with five measurements taken for each sample. Contact angle measurements were performed for both untreated and treated fiber bundles.

### 7. Data Analysis

The measurement data from the study were then analyzed using analysis of variance (ANOVA) to evaluate the effect of the treatments given. This analysis of variance was

applied to each observed parameter, including the physical properties of the sisal fiber such as diameter and weight change, as well as the mechanical properties of the sisal fiber, which include tensile strength and Young's modulus.

### III. Results and Discussions

The treatment of sisal fiber with different NaCl content and temperature affected the mechanical strength of the fiber. The tensile strength and Young's modulus of the treated fiber bundles are shown in Figure 1. Sisal fiber treated with 5 wt.% NaCl at boiling temperature showed the highest average tensile strength of  $488.64 \pm 7.22$  MPa and Young's modulus of  $46.41 \pm 3.31$  GPa. In contrast, fiber treated with 1 wt.% NaCl at room temperature produced the lowest tensile strength and Young's modulus of  $354.81 \pm 9.26$  MPa and  $28.16 \pm 1.90$  GPa, respectively. Meanwhile, untreated sisal fiber had a tensile strength of  $329.29 \pm 6.29$  MPa and Young's modulus of  $26.25 \pm 2.53$  GPa. In general, increasing NaCl concentration and soaking temperature was followed by increasing tensile strength and Young's modulus. The increase of NaCl and soaking temperature significantly increased the tensile strength and Young's modulus. This increase is likely due to sodium infiltration into the fiber, which can reduce lignin content and increase cellulose content, thereby strengthening the treated fiber, as explained by Mardin et al. [14].

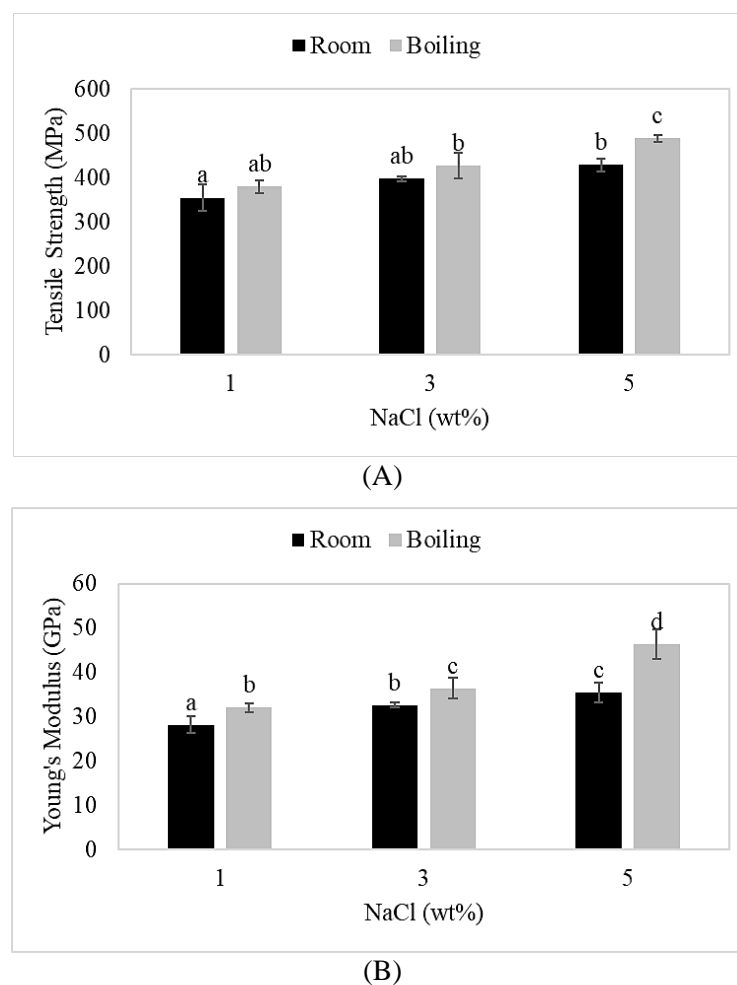


Fig.1. Tensile strength (A) and Young's Modulus (B) of NaCl-treated sisal fiber. Vertical line through the bar represents standard deviation from the mean. Different letters in the graph are significantly different ( $P < 0.05$ ).

Treatment with 5 wt.% NaCl at boiling temperature successfully increased the tensile strength and Young's modulus by 48.39% and 76.8%, respectively, compared to untreated fibers. NaCl treatment on sisal fibers had a positive impact on increasing the tensile strength and Young's modulus. The percentage increase in sisal fibers treated at boiling temperature was higher compared to the results of other studies. In another study, treatment using 5 wt.% NaCl for 1 hour at room temperature was only able to increase the tensile strength and Young's modulus by 16.06% and 24.29%, respectively, when compared to untreated fibers [15]. The increase in tensile strength and Young's modulus was likely owing to the infiltration of sodium into the fiber, which may cause a decrease in lignin and an increase in cellulose contents, which strengthens the treated fiber, as reported by Mardin et al. (2016). This is also attributed to the viscoelastic shearing of the fiber, indicating that recovery is linked to an induced crystallization of amorphous cellulose components and a microfibril alignment with the loading axis. NaCl, which is the most abundant salt found in seawater, can affect the strain of fibers under different treatment conditions [14].

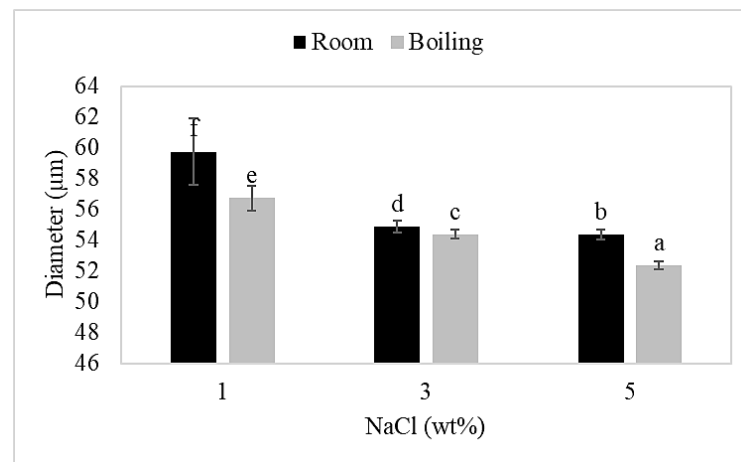


Fig.2. Diameter of NaCl-treated sisal fiber. Vertical line through the bar represents standard deviation from the mean. Different letters in the graph are significantly different ( $P < 0.05$ ).

NaCl treatment affects the reduction of fiber bundle diameter. Figure 2 shows the diameter of fiber bundles treated under various conditions, while Figure 4 shows a photo of sisal fiber bundles. The results showed that the diameter of the fiber bundles decreased due to NaCl treatment. The use of 1 wt.% NaCl at room temperature was able to reduce the diameter of the fiber bundle by 2.27% compared to untreated fibers. In contrast, the diameter of the untreated fiber bundle, which reached  $61.14 \pm 1.93 \mu\text{m}$ , was larger than that of the fiber bundles treated under all conditions. The most significant decrease in the diameter of the sisal fiber bundle, which was 14.38%, occurred in the fiber bundle treated with 5 wt.% NaCl at boiling temperature. The increase of NaCl and soaking temperature significantly decreased the diameter of sisal fiber. Variations in treatment with NaCl and temperature resulted in a significant decrease in the diameter of the fiber bundle. This decrease is thought to be caused by the removal of substances on the fiber surface. According to Setyayunita et al. [15], NaCl in the fiber processing process with seawater plays a role in attracting impurity molecules that have  $\text{OH}^-$  groups, which interact with the  $\text{Na}^+$  component.

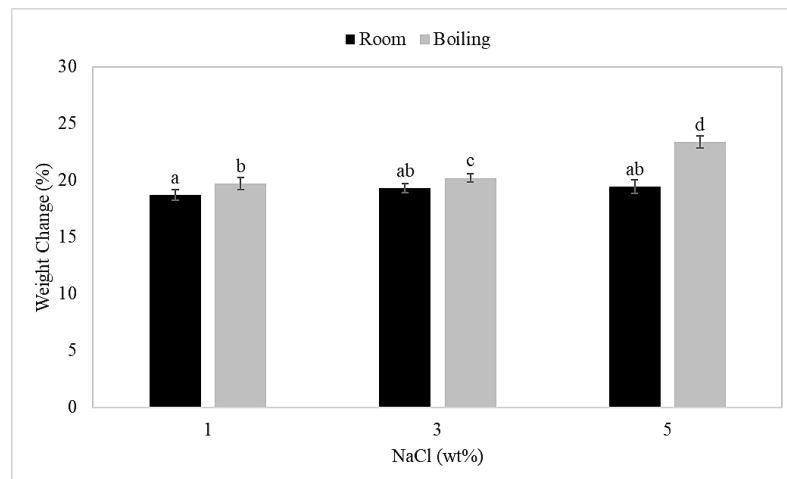


Fig.3. Weight change of NaCl-treated sisal fiber. Vertical line through the bar represents standard deviation from the mean. Different letters in the graph are significantly different ( $P < 0.05$ ).

The percentage of weight change showed an increase in all NaCl treatment conditions, with the highest change recorded in the 5 wt.% NaCl treatment for 1 hour, as seen in Figure 3. The average weight change for all treatment conditions was around  $20.12 \pm 1.62\%$ , with the highest value reaching  $23.37 \pm 0.54\%$  compared to the untreated bundle fibers. Treatment with 5 wt.% NaCl at boiling temperature resulted in a significant weight change compared to other conditions. On the other hand, treatments at room temperature with various NaCl levels showed relatively similar weight changes and did not show a significant increase. The pattern of increasing weight change in sisal bundle fibers was in line with the increase in NaCl concentration and soaking temperature, indicating that the increase in weight change was correlated with the decrease in bundle fiber diameter.

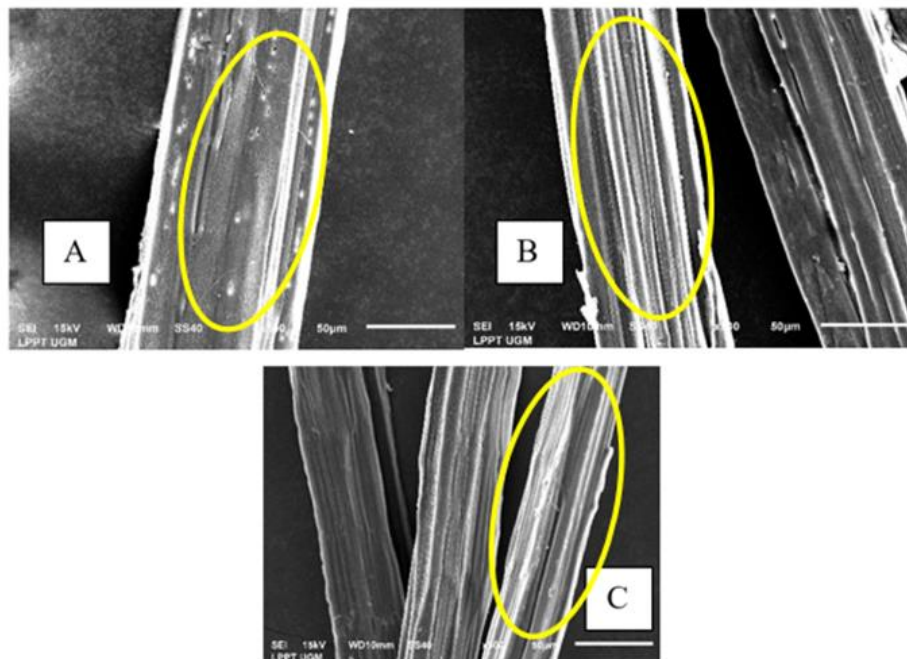


Fig. 4. The scanning electron microscope image of (A) untreated, (B) 5% NaCl/room temperature, and (C) 5% NaCl/boiling temperature sisal fiber.

The surface morphology of untreated and treated sisal fibers with 5 wt.% NaCl at room temperature and boiling temperature was observed using FE-SEM (Figure 4). The surface of sisal fibers treated with 5 wt.% NaCl for 1 h appeared rougher compared to untreated fibers. This image clearly shows the changes in the fiber surface after treatment. The treated fiber surface appeared rough, with elongated protrusions that are expected to improve the mechanical interlock with the adhesive. These morphological changes, caused by NaCl treatment, affected the weight and diameter of the fibers, as seen from the increase in the percentage change in weight and the decrease in the fiber diameter. These modifications also contributed to the improvement of the mechanical properties of sisal fibers, such as tensile strength, which can be related to the relationship between fiber diameter and tensile strength. In addition, the fiber surface morphology also affected the compatibility between the fibers and the adhesive, as seen from the contact angle between the fiber bundles and the adhesive.

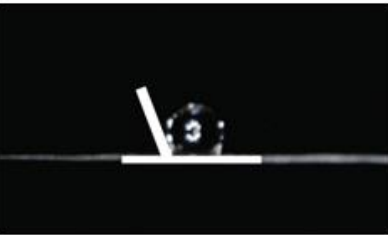

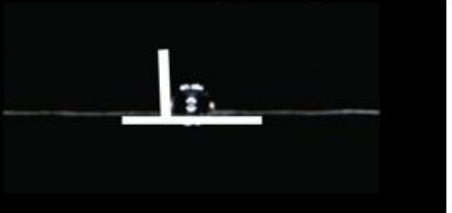


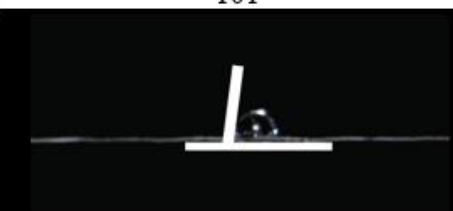
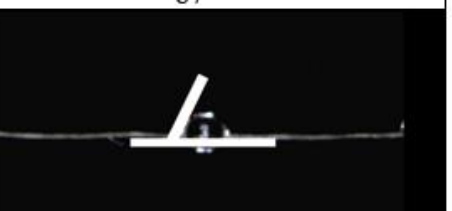
| Untreated  |   |  |
|--|---|--|
|  |   |  |
| 124°   |   |  |
| NaCl   | Temperature   |  |
|  | Room  | Boiling  |
| 1 wt%  |  |  |
|  | 105°  | 95°  |
| 3 wt%  |  |  |
|  | 101°  | 87°  |
| 5 wt%  |  |  |
|  | 79°   | 76°  |

Fig. 5. The contact angle image of untreated and NaCl-treated sisal fiber.

The contact angle of sisal fibers tends to decrease with increasing NaCl concentration and immersion temperature (Figure 5). Untreated sisal fiber bundles have a contact angle of  $124^\circ$ , which decreases to  $76^\circ$  at 5 wt.% NaCl conditions with boiling temperature. The results showed that NaCl treatment resulted in a smaller contact angle between epoxy and fiber bundles, indicating that the wettability of epoxy on the treated sisal fiber bundles was more optimal than the untreated ones. A smaller contact angle indicates that the surface can absorb adhesive more effectively to cover the surface of the fiber bundles. Mardin et al. (2016) reported that soaking natural fibers in water containing NaCl increased the internal bond strength. This indicates that the decrease in contact angle after treatment can have a positive impact on the internal bond strength of the corresponding composite board. Furthermore, immersion in seawater containing NaCl provided the highest internal bond strength on the composite board using a natural sago matrix as an adhesive [14]. NaCl treatment under various conditions also improves the physical and mechanical properties of composite boards by increasing the percentage of epoxy content [10]. Therefore, NaCl treatment of sisal fibers increases the cohesiveness between the fibers and the adhesive. chemical modification or treatment of natural fibers containing kenaf is carried out using reagents containing functional groups capable of binding to the hydroxyl groups of the natural fibers. A few types of chemical modifications reported in previous studies of the literature have achieved some level of success in improving the adhesion of fiber matrices of natural compounds. Chemical treatment creates multiple voids on the fiber surface, and mechanical bonding improves surface adhesion [14]. Chemical fiber treatment is very important for enhancing the adhesion between hydrophilic natural fibers and the hydrophobic polymer matrix at the interface. This study focuses on the effect of different levels of NaCl and temperature on fiber properties. The use of the same chemicals in other natural fiber treatments needs to be observed further. The combination of using other factors in fiber treatment methods also has great potential for changing the properties of natural fibers.

#### IV. Conclusions

The treatment with variations in NaCl levels and soaking temperatures on sisal had a significant influence on the properties of the sisal fibers. The results showed that sisal fiber treated with 5 wt.% NaCl at boiling temperature produced the highest tensile strength and Young's modulus values. This treatment was able to increase the tensile strength and Young's modulus of sisal fiber by 48.39% and 76.8%, respectively, compared to untreated sisal fiber. The highest percentage decrease in sisal fiber diameter, which was 14.38%, occurred in the 5 wt.% NaCl treatment at boiling temperature compared to untreated sisal fiber. Variations in treatment with NaCl and temperature resulted in a significant decrease in fiber bundle diameter. The average weight change value was recorded at 20.12%, with the highest value reaching 23.37%. The surface of sisal fiber treated with 5 wt.% NaCl at boiling temperature appeared rougher than that of the untreated one. In addition, this treatment also reduced the contact angle between the fiber and the adhesive by 38.71% compared to the untreated one. NaCl treatment was shown to be an effective method to improve the mechanical properties and wettability of fibers, which has potential for application in high-performance plant fiber composites. This study focuses on the effect of sodium chloride treatment on the properties of sisal fiber. However, the effect of treatment on the properties of modified fiber composite boards still needs to be studied further to determine the benefits of sodium chloride treatment. NaCl treatment has a significant effect on the properties of sisal fiber, but its effect on the development of composite board products still needs to be widely known.

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