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Effect of alkali treatment on the mechanical, physical, and thermal properties of sweet sorghum [*Sorghum bicolor* (L.) *Moench*] fibers

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Abstract

A parametric study of alkali treatment was conducted to determine the effects of base concentration, reaction time, and temperature on the mechanical, physical and thermal properties of sweet sorghum fibers using a 2^3 factorial design. Fibers were extracted from sweet sorghum stalks using sodium hydroxide. Results showed that the tensile strength of extracted fiber bundles were directly proportional to reaction time and inversely related to concentration. Fibers with base concentration, temperature and reaction time of 0.5-N NaOH, 100˚C, and 60 minutes, respectively, exhibited the highest tensile strength of 505.845 MPa. Scanning Electron Microscopy (SEM) images showed that sweet sorghum fibers exhibiting the highest tensile strength have smooth, convex-shaped fibrils. Also, thermogravimetric analysis has shown three stages of weight loss with increasing temperature: 30°C to 185.73°C, 185.73°C to 588.88°C, and 588.88°C to 750.86°C. These weight losses were associated with the vaporization of water from the sample and the decomposition of cellulosic components of the fiber. The study concludes that sweet sorghum fiber has high potential as reinforcement for composite production.

Keywords: alkali treatment, base concentration, natural fibers, reaction time, sweet sorghum, tensile strength

Introduction

Global efforts to address environment and climate change issues through green chemistry have led to the search for alternative materials for manufacturing industries. Among the most sought after materials are those that may be obtained directly from natural biological sources, as these are usually renewable, biodegradable, and carbon neutral. One offshoot of this paradigm shift is the increased utilization of natural fibers that possess desirable mechanical properties. Consequently, a surge of studies on the extraction and characterization of natural fibers from various sources have been conducted in recent years.

The properties of natural fibers have been found to be intimately influenced by the extraction procedure and process parameters. Most fiber extraction procedures employ alkali treatment at various base concentrations and process conditions that depend on the fiber source. In a comparative study on the effect of extraction procedure on the morphology of kenaf fibers, Amel and Sudin (2012) found out that alkali treatment resulted in higher fiber density than other extraction methods such as crude extraction, water-retting, and benzoate-retting. Applying alkaline treatment with optimum conditions of 0.5 N NaOH at 80 ˚C for 30 minutes, Reddy and Yang (2009) were able to extract fibers from milkweed that have high

cellulose content and with properties suitable for high-value textile applications. In their work on the extraction of fibers from leafiran leaves, Mortazavi and Moghaddam (2010) found out that fibers obtained from prolonged alkali treatment at low alkali concentrations have higher tensile strength than those obtained from shorter reaction periods. Prolonged alkali treatment was also found to produce smoother fibers. In a study utilizing sugar cane bagasse, high alkali concentrations have been found to result in fibers that exhibit bending hysteresis, low tensile strength, and high bending rigidity (Michel, Bachelier, Drean, and Harzallah, 2013).

With the onset of the Philippine Biofuels Act of 2006, various energy crops have been extensively studied as potential energy source. Among these, sweet sorghum is considered to be the most promising and socially acceptable. Sweet sorghum is a perennial crop that grows from 10 to 15 feet tall in temperate and tropical climates. Because its stem has high sugar content, the plant is being cultivated for fodder, syrup, molasses, sugars, and small-scale ethanol production (Rooney, Blumenthal, Bean, and Mullet, 2007). Sweet sorghum can also be an excellent source of natural fiber. Sweet sorghum bagasse, which is usually thrown away or burned as fuel for heating, can still find use as raw material for high-value products. Sweet sorghum fiber and pulp can be used for the manufacture of fine-quality writing and printing paper as well as corrugated and solid particle boards due to its flexibility and tensile strength (Kumar and Marithmu, 2012). However, information on its fiber properties remains minimal.

Given the absence of data characterizing sweet sorghum fiber, this study aims to determine the effects of base concentration, temperature, and reaction time on the mechanical (tensile strength), physical (surface morphology), and thermal (thermal behavior at elevated temperatures) properties of sweet sorghum fiber obtained by alkali treatment. The availability and low cost of sweet sorghum stalk make it a potential source of fiber for various industrial applications. Data on the extraction conditions and fiber characteristics can be used for further studies especially for its application in the fabrication of improved biodegradable polymer composites.

Methodology

Chemicals used in the study were reagent-grade sodium hydroxide pellets (99.9% purity), glacial acetic acid (99.9% purity), hydrogen peroxide (99.9% purity), and distilled water. Milled sweet sorghum stalks were collected from the Institute of Plant Breeding, University of the Philippines Los Baños. Sweet sorghum stalks underwent alkali treatment and the extracted fibers were subjected to tensile tests. Surface morphologies of the fibers that yielded the highest and lowest tensile strengths were observed using scanning electron microscopy (SEM). Finally, the fiber sample that yielded the highest tensile strength was subjected to thermogravimetric analysis (TGA) to determine its thermal behavior at elevated temperatures.

Preparation of sweet sorghum stalks

Sweet sorghum stalks were washed with water and air dried. Prior to alkali treatment, the pith was scraped off to facilitate fiber extraction from the rind. The stalks were cut with length ranging from 5 to 8 inches.

Fiber Extraction

Preliminary experiments were performed to determine the suitable fiber extraction conditions and establish the lower and upper limits of the process parameters. The set of lower limit parameters, below which fibers remain in the stalk, were found to be 0.5 N NaOH, 80 ºC and 30 minutes. The set of upper limit parameters, above which fibers extracted are very short and difficult to subject to tensile tests, was found to be 2.0 N, 100 ºC and 60 minutes. With these findings, a 2^3 factorial experimental design that takes into account NaOH concentration, temperature, and reaction time was developed. The summary of the experimental design that consists of eight experimental runs is shown in Table 1. Each experimental run was done in duplicates.

The alkali treatment experiments were started by placing fifteen grams of sweet sorghum stalks in a 600-mL beaker. Three-hundred mL of NaOH solution with the corresponding concentration indicated in the experimental design was poured onto the stalks to make a mass-per-volume ratio of 1:20. Thereafter, the mixture was allowed to react by steaming for a specified period of time in a pressure cooker equipped with a thermostat for temperature

control at 103.42 kPa. The stalks were then washed thoroughly with water to arrest the alkali reactions, and rolled using a glass bottle to facilitate the separation of the fibers. The fibers were collected manually and allowed to dry at 105°C using an air-circulating oven for 24 hours. Dried fibers were then stored in a desiccator until ready for characterization.

Table 1. Experimental design for the extraction of sweet sorghum fibers.

Run Number	NaOH Concentration (N)	Temperature (°C)	Time (Minutes)
1	0.5	100	60
2	0.5	100	30
3	2.0	100	30
4	2.0	100	60
5	0.5	80	60
6	2.0	80	30
7	0.5	80	30
8	2.0	80	60

Characterization of sweet sorghum fibers

Mechanical Property

Single-fiber tensile strengths of sweet sorghum fibers were determined using a Jinan Testing Equipment with 1-kN scale load. Individual fiber strands were randomly chosen and mounted on a cardboard frame following the dimensions indicated by ASTM D3379. Epoxy was used to fix a fiber onto a paperboard jig and produce a test specimen with a gauge length of 25 mm. The diameter in three different parts of each fiber was measured using a Mitutoyo Digimatic Micrometer. The average diameter was calculated and used to get the equivalent fiber strength in MPa. Tensile load was applied at a crosshead speed of 0.25 mm/min. A total of 35 samples of fibers were tested. This was done for two trials. Q-test was performed as a first-level statistical tool to remove outlier values. The tensile strength was determined as the average of the acceptable values.

Scanning Electron Microscopy

SEM images of the fibers that yielded the highest and lowest tensile strength were taken using a Hitachi S-510 scanning electron microscope operating at an accelerating voltage of 5 kV. Prior to imaging, the samples were sputter-coated with Au-Pd for five minutes. The longitudinal and transverse cross sections of the fibers were examined at 1000x and 1200x magnification, respectively.

Thermogravimetric Analysis

Thermogravimetric analysis was performed using a Perkin Elmer Sta 6000 lab system. TGA was carried out from room temperature up to 850°C using an aluminum pan. The heating rate was 10°C/min under a nitrogen flow of 20 mL/min. The percentage of mass loss and decomposition temperature of each component was plotted.

Data and Statistical Analysis

For statistical analysis and model fitting, one-way analysis of variance (ANOVA) at 95% level of significance was conducted to determine if the data obtained are statistically different. The significantly different parameters were also determined using the same statistical method.

Results and Discussion

Mechanical Property

Figure 1 shows the summary of the tensile strength of the extracted fibers. Fibers extracted using 0.5-N NaOH at 100°C and 60-min reaction time (Run 1) exhibited the highest tensile strength of 505.85 MPa.

Applying a three factor design and considering significant parameters, the regression model for the tensile strength was determined to be $y = 411.69 - 41.65A + 27.19C$ where y is tensile strength, A is alkali concentration, and C is reaction time. The high magnitude value of A indicates that NaOH concentration is the most important factor. Factorial analysis at 95% confidence level showed that the model is significant and, among the factors, only alkali concentration and reaction time register significant effects on tensile strength. The regression model has a coefficient of determination (R^2) of 0.7206, a standard error of 46.57%, and a coefficient of variation (CV) of 11.34 %. Table 2 shows the analysis of variance for tensile strength as response variable.

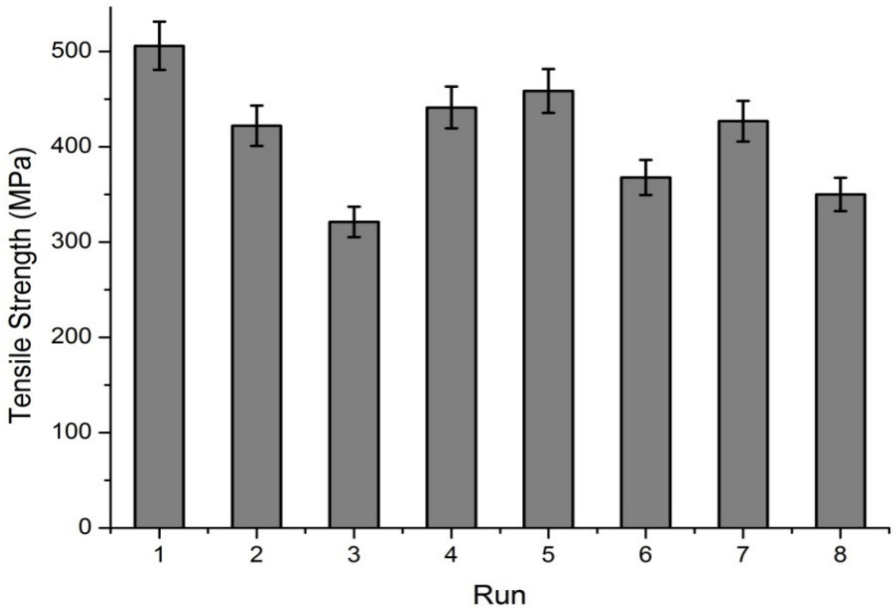


Figure 1. Tensile strength of sweet sorghum fibers.

Table 2. Analysis of variance for tensile strength as response variable.

SOURCE	SUM OF SQUARES	DF	MEAN SQUARE	F-VALUE	P-VALUE PROB > F	REMARKS
A-NaOH concentration	27753.89	1.00	27753.89	12.741	0.006	Significant
B-temperature	1897.04	1.00	1897.04	0.871	0.375	not significant
C-time	11825.48	1.00	11825.48	5.429	0.045	significant
AB	1.12	1.00	1.12	0.001	0.982	not significant
AC	45.43	1.00	45.43	0.021	0.888	not significant
BC	9042.11	1.00	9042.11	4.151	0.072	not significant
Residual	19605.50	9.00	2178.39			
Cor Total	70170.57	15.00				

Figure 2 shows a model graph depicting the effect of alkali concentration on the tensile strength of sweet sorghum fibers. The graph shows that tensile strength decreases with increasing alkali concentration. This happens because alkalization disrupts the hydrogen bonding in the network structure of fibers. In general, this reaction may be represented as follows:

$$\text{fiber-OH} + \text{NaOH} \rightarrow \text{fiber-O-Na} + H_2O$$

Alkalization removes a certain amount of hemicelluloses, lignin, wax, and oils covering the external surface of a fiber cell wall; depolymerizes celluloses; and, exposes short length crystallites (Mohanty et al., 2001). Gums which act as matrix that facilitates transfer of stress between fibrils are also removed, resulting in the separation of fibrils from the original fiber bundle. Taken together, these reactions result in a reduction in strength (Hagad, 2009).

Alkalization also affects fiber morphology, particularly the aspect ratio and fiber diameter. According to Saira et al. (2007), alkali treatment reduces fiber diameter and increases the aspect ratio, with both effects having desirable consequences in most fiber applications. High aspect ratio is associated with increased material strength in fiber-reinforced composites. On the other hand, small diameters result in fewer flaws that propagate during processing or when composites are placed under a load (Bagherpour, 2012).

Another important factor that affects the tensile strength of sweet sorghum fibers is reaction time. Tensile strength was found to increase with increasing reaction time (Figure 3). This happens because the spiral angle of cellulose microfibrils tends to decrease during alkali treatment, allowing for the rearrangement of cellulose chains to a more linear configuration (Amel et al., 2012). Prolonged alkali treatment ensures that the rearrangement of cellulose chains proceeds substantially, making the fibers more stable.

Scanning Electron Microscopy

The effect of alkali treatment on the physical properties of sweet sorghum fibers was observed

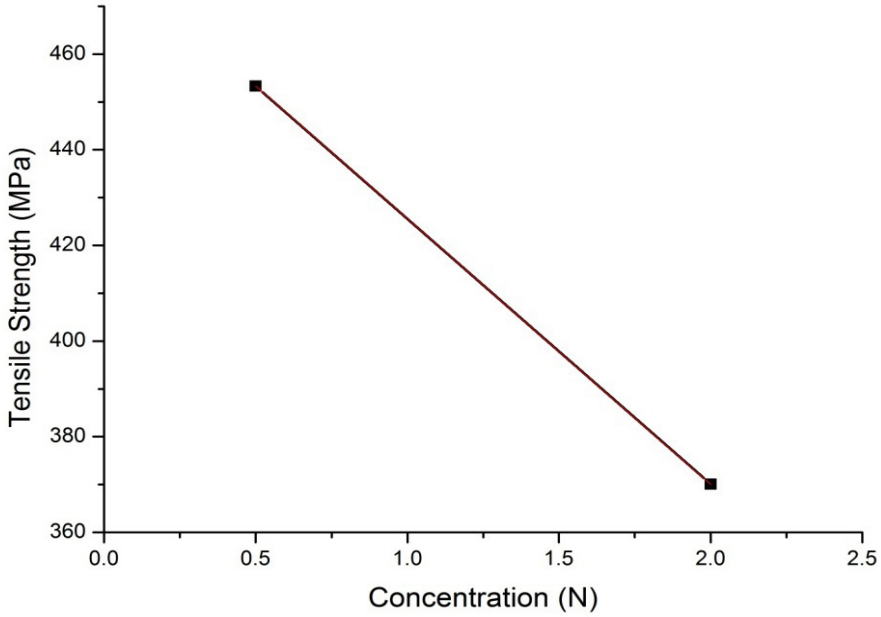


Figure 2. Model graph showing the significant effect of concentration.

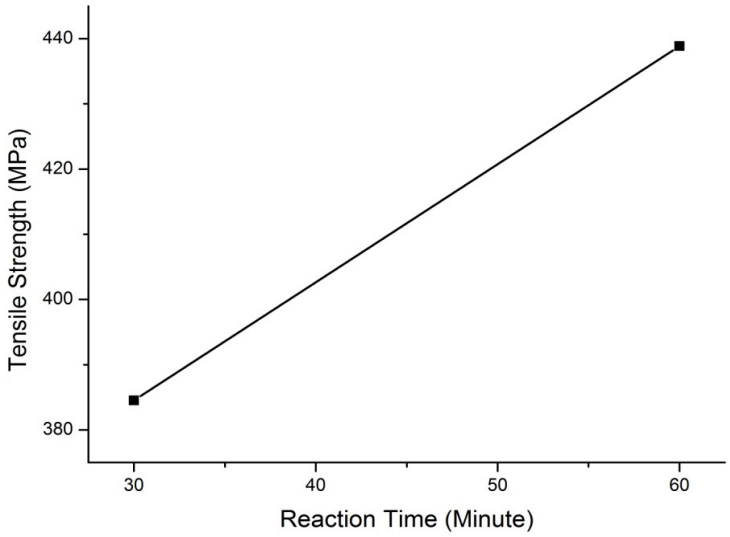
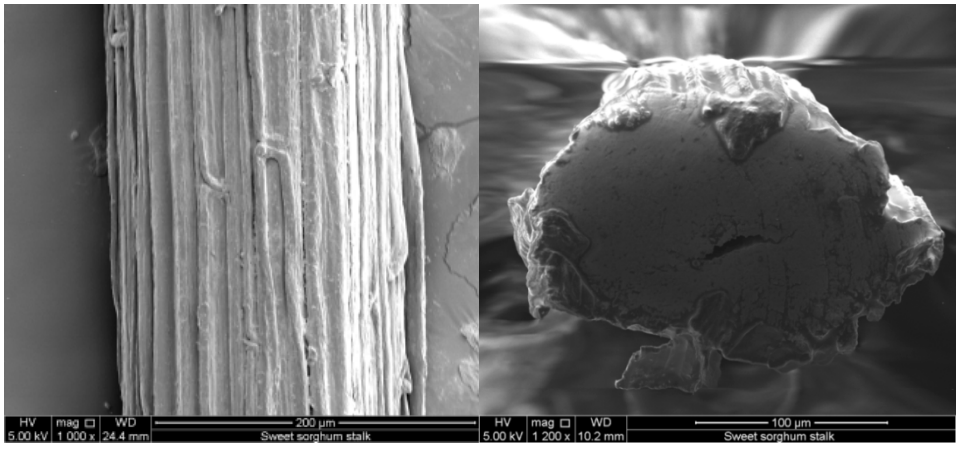


Figure 3. Model graph showing the significant effect of reaction time.

using scanning electron microscopy. Figures 4 and 5 show the SEM images of the longitudinal and transverse cross sections of the fibers that yielded the highest and lowest tensile strength, respectively.

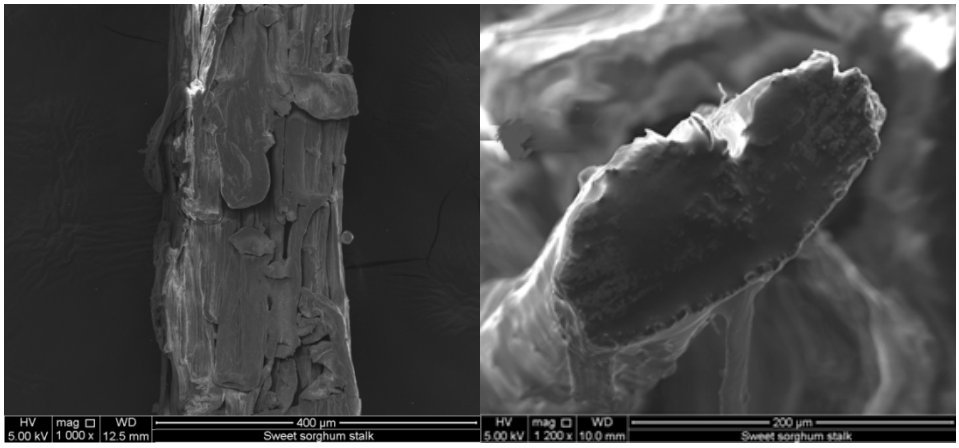
Figure 4a shows that high strength fiber has convex-shaped fibril strands that may have been the result of the partial removal of lignin and hemicelluloses. Moreover, as shown in Figure 4b, the fiber has retained its circular form. On the other hand, low strength fiber is characterized by the presence of cracks and lack of convex microfibrils (Figure 5a), and an ovate cross section (Figure 5b). These morphologies are similar to the observations made by Hagad (2009).

The images also show the development of a rough surface topography as a consequence of alkali treatment. The development of rough surfaces in fibers is a desired effect since this leads to better fiber-matrix interface adhesion that result in improved mechanical properties when fibers are used as reinforcements in composites (Li, Tabil, and Panigrahi, 2007).



(a) (b)

Figure 4. SEM images of high strength fiber extracted at 0.5 N NaOH, 100°C, 60 minutes: (a) longitudinal, and (b) cross section view.



(a) (b)

Figure 5. SEM images of low strength fiber extracted at 2 N NaOH, 100°C, 30 minutes: (a) longitudinal, and (b) cross-section view.

Thermogravimetric Analysis

Thermogravimetric analysis was performed to determine the thermal behavior of the extracted fiber that yielded the highest tensile strength. The TG curve (Figure 6) shows three stages of weight loss with increasing temperature. The first, second, and third stages of weight loss were at 30°C to 185.73°C, 185.73°C to 588.88°C, and 588.88°C to 750.86°C, respectively.

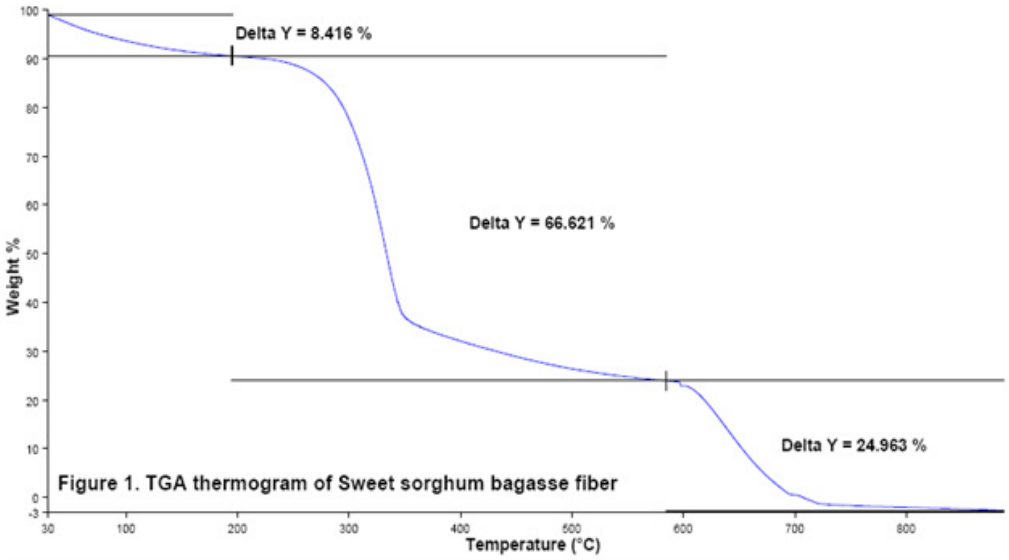


Figure 6. Thermogravimetric curve of sweet sorghum fiber.

The first stage of decomposition that occurred at 30°C to 185.73°C could have been due to the liberation of absorbed moisture or the vaporization of water from the sample. The second stage of decomposition that occurred at 185.73 °C to 588.88 °C registered a 66.621% weight loss. This phase may be attributed to the decomposition of the cellulosic components of the fiber (cellulose, hemicelluloses, and lignin). Hemicelluloses generally degrade more quickly than lignin and celluloses due to the presence of acetyl groups. Hemicelluloses tend to decompose between 180 °C and 350 °C while the decomposition of lignin could occur at the entire temperature range up to 900 °C (Alwani et al., 2014).

Conclusion

The study has extracted and characterized fibers from sweet sorghum stalk using alkali treatment. Fibers extracted using 0.5 N NaOH at 100 °C and 60-min reaction time exhibited the highest tensile strength of 505.85 MPa. Factorial analysis at 95% confidence level showed that concentration and reaction time have significant effects on the tensile strength of the extracted fibers. On one hand, increased concentration led to decreased tensile strength of fibers due to the removal of gums that act as matrix facilitating the transfer of stress between fibrils. On the other hand, increased reaction time led to increased tensile strength due to longer time for rearrangement of cellulose chains, which made the fibers more stable.

SEM images showed that the high tensile strength fiber bundle has convex-shaped fibrils. Alkali treatment disrupted the hydrogen bonding in the network structure of the fiber leading to the development of rough surface topography. Thermogravimetric analysis has shown three stages of weight loss with increasing temperature. These weight losses were associated with the release of absorbed moisture and the decomposition of cellulosic components of the fiber.

With the properties of sweet sorghum fibers having been determined, the study concludes that sweet sorghum fibers have high potential as reinforcement for different polymer matrices. This could help increase the industrial application of sweet sorghum fibers. However, more studies need to be conducted to determine further the effect of alkali treatment on the mechanical, physical and thermal properties of sweet sorghum fibers using wider ranges of temperature, base concentration, and longer reaction time.

Acknowledgments

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