



# EFFECTS OF ALKALINE TREATMENT ON THE PROPERTIES OF PINEAPPLE (*ANANAS COMOSUS*) LEAF FIBER (PALF) REINFORCED WITH TAPIOCA-BASED BIO RESIN (CASSAVA STARCH)

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## ABSTRACT

The poor compatibility of natural fibers with hydrophobic matrices due to their hydrophilic nature has led researchers to improve their properties to enable better compatibility. Pineapple leaf fiber is one of the abundantly available natural fibers obtained from pineapple leaf. It has good chemical properties and strong admirable mechanical properties and can be used as a replacement for synthetic fibers despite the same deficiencies as other natural fibers. In this study, Pineapple leaf fiber was treated with Alkali at varying concentrations, temperatures, and times. Treated pineapple leaf fiber was reinforced with tapioca-based bio resin (cassava starch). This fiber was subjected to tensile testing and Fourier transform infrared (FTIR) spectroscopy. The results of FTIR indicated the various peaks in the absorbance versus wave number relation. The FTIR analysis of untreated pineapple leaf fiber indicated the presence of O-H stretch, N-H stretch, C  $\equiv$  stretch, C=O stretch, and H-C-H bond. The modification of fibers achieved by disruption of hydrogen bonding in the network structure was possible due to its treatment with alkali. Mechanical testing (tensile test) and FTIR were also used to know the effects of chemical treatment on the fibers. Alkali treatment improved the fiber properties as the concentration, temperature, and treatment time increased.

**Keywords:** alkali, cassava starch, fiber, pineapple-leaf, FTIR.

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## 1. INTRODUCTION

Natural fibers are obtained from plants, animals, or minerals which can serve as replacements for synthetic fibers due to their abundance availability, cheapness, and good chemical and mechanical properties. Pineapple (*Ananas comosus*) is one of the most essential tropical fruits after banana and citrus fruit [1, 2, 4]. Hence, the leaves of pineapple can be used for producing natural fibers. Natural fibers possess valuable properties such as low density, high specific strength, and stiffness [3]. Pineapple-leaf fiber (PALF) consists of about 80% cellulose, 6-12% hemi-cellulose, and 5-12% lignin [3, 4, 5, 6]. Natural fibers can be used in the fabrication of composite due to their biodegradability nature and their acceptable mechanical strength. Polypropylene has been used extensively as a matrix material with natural fiber in the preparation of composite [6, 7, 8]. Natural fibers and polymer composites have great limitations, which is their inability to compact with hydrophobic thermoplastic matrices and hydrophilic natural fibers [9, 10, 11].

Researchers have been studying composites based on natural and synthetic fibers which are the two known basic types of fibers [12, 13, 14]. However, when comparing the advantages of using fibers in composites over synthetic fibers, we look at their low cost, low

density, biodegradability, availability which are limitless, and their recyclability and renewability [15-20]. Natural fibers are so useful that some studies have suggested that they have all it takes to replace glass fibers in polymer composites [21, 22, 23]. It has been shown that the chemical treatment of natural fibers improves both their surface morphology and their mechanical properties [24, 25]. Researchers in the bio-fibers space have been exploring different treatment methods to improve the fiber-matrix bonding ability to achieve a better composite reinforcement application [26]. This is due to the treatment of natural fibers which is related to their hydrophilic nature that discourages good fiber-matrix bonding [27]. Researchers also studied the effect of alkali treatment on PALF reinforced composite's degradation, mechanical, and water intake properties [28, 29].

Alkali treatment disrupts the hydrogen bonding in the network structure thereby increasing surface roughness. It removes wax and oils, and some amount of lignin covering the external surface of the fiber cell wall, depolymerizes cellulose, and exposes short-length crystallites. It also promotes the ionization of the hydroxyl group of the alkoxide. Alkali treatment directly influences cellulosic fibril, the degree of polymerization, and the



extraction of lignin and hemi-cellulosic compounds [30, 31, 32, 33].

This work aims to investigate the mechanical properties of pineapple leaf fibers, both untreated and treated, with alkali reinforced with tapioca-based bio resin (cassava starch).

## 2. MATERIALS AND METHODS

### 2.1 Pineapple Leaf Fiber

The fibers were extracted from the pineapple leaf from a pineapple plantation at Naze, Owerri. The manual method was used during the extraction of fibers. The leaves were immersed in water for 48hr to aid the easy and quick scraping of the surface while removing the outer layer. This scraping was done using a broken plate and the fibers were pulled and washed severally to remove any trace of dirt before sun-drying for 4 days.

#### 2.1.1 Tapioca bio resin (cassava starch)

Fresh tubers of cassava were purchased from farmland in Naze, Owerri West, Imo state. They were peeled, scraped, slashed into smaller sizes, immersed in water, ground, and sieved, using a chiffon fabric to separate starch from its chaff. The starch was put in a bag and squeezed to remove its water content and the dry cassava was left in the bag.

### 2.2 Chemicals (Reagents) Used

Sodium hydroxide (NaOH) pellets used were purchased from Chemi-sciences Nigeria Limited, in Owerri, Imo state. The chemical used was of analytical grade.

### 2.3 Equipment

Water baths, Beakers, an Electronic weighing balance, Spatula/Stirrer, Distilled Water, Paper Tape, Gloves, Scissors, Sample foil, and Yarn Textile Testing Machine were used.

### 2.4 Characterization

#### 2.4.1 Fourier transform infrared (FTIR) spectroscopy

The functional groups of the fiber were determined with the aid of an FTIR spectroscope (Shimadzu, Model: IR affinity-1; A2137470136 SI) at the Energy Center (UNN, Nsukka). It collects high special resolution data over the spectra range.

### 2.5 Alkali Treatment

Alkali treatment, (a reinforcement with bio resin and tensile test) was carried out in Polymer and textile engineering laboratory at the Federal University of Technology Owerri (FUTO). Twenty-five solutions were prepared which consist of five different concentrations. A solution of the same concentration was made in five different beakers as follows: Five different solutions were prepared by dissolving 1.3g, 2.0g, 3.7g, 4.5g, and 5.5g of sodium hydroxide in 500ml of distilled water and each containing one of the following weights of fibers: 1.4g,

2.0g, 2.5g, 3.2g, and 4.0g. They were treated for 30, 45, 60, 90, and 120 minutes and at temperatures of 27°C, 30°C, 45°C, 60°C, and 90°C respectively, using a water bath. After the treatments, fibers were washed carefully with distilled water to remove any trace of dirt in them and dried in air for about 24-48 hours. They were then reweighed to ascertain the change in weight if any. The samples were labeled and packed for further use.

### 2.6 Tensile Testing

The Tensile strength of the PALF was obtained with the yarn textile testing machine Fig1; in which the fiber is subjected to a controlled tension until it fails.



Figure-1. Yarn textile testing machine.

### 2.7 Reinforcement of the Tapioca based Bio Resin TBR (Cassava Starch) and Pineapple Leaf Fiber (PALF)

Dry Cassava starch (Tapioca based bio resin) was prepared by mixing with water and heating till the mixture becomes slightly viscous. 32.5g of cassava starch was used to reinforce 2.0g each, of untreated fiber (as control sample) Similar reinforcement was done with alkaline treated fiber at different temperatures of 30°C, 45°C, 60°C and, 90°C were used. This reinforcement was achieved by immersing 2.0g of the fibers into 32.5g of starch in a beaker. The mixture was left for 24hrs for proper absorption and then dried in air for 3-5 days. After drying, they were re-weighed to ascertain the level of absorption, and packed properly.

## 3. RESULTS AND DISCUSSIONS

### 3.1 FTIR Analysis

The FTIR of the fibers is presented in Figures (2-5) below. The spectrum of each of the graphs shows various peaks in the absorbance versus wave number. It is a graph of infrared light absorbance on the vertical versus wave number. The unit of the wave number is  $\text{cm}^{-1}$ . The peaks and their corresponding intensities represent the functional groups of the fiber [24-26].

The FTIR analysis of untreated pineapple leaf fiber is presented in Figure-2; and it showed the presence of O-H stretch, N-H stretch,  $\text{C}\equiv\text{C}$  stretch,  $\text{C}=\text{O}$  stretch, and H-C-H bond. The FTIR Analysis of alkali-treated pineapple leaf fiber is also presented in Figure-3; and it showed the presence of hydrogen-bonded O-H stretch,  $\text{C}\equiv\text{C}$  stretch,  $\text{C}-\text{C}=\text{C}$  asymmetric stretch, C-O stretch, C-H bond. Similarly, the FTIR results of untreated pineapple leaf fiber reinforced with cassava starch are presented in



Figure-4; and indicated the presence of hydrogen-bonded -OH stretch, C≡N stretch, C≡C stretch, N=O stretch and N=O bond. Figure-5; is the FTIR analysis of alkali-

reinforced pineapple leaf fiber which indicated the presence of hydrogen-bonded O-H stretch, C≡C stretch, and (C-O stretch).

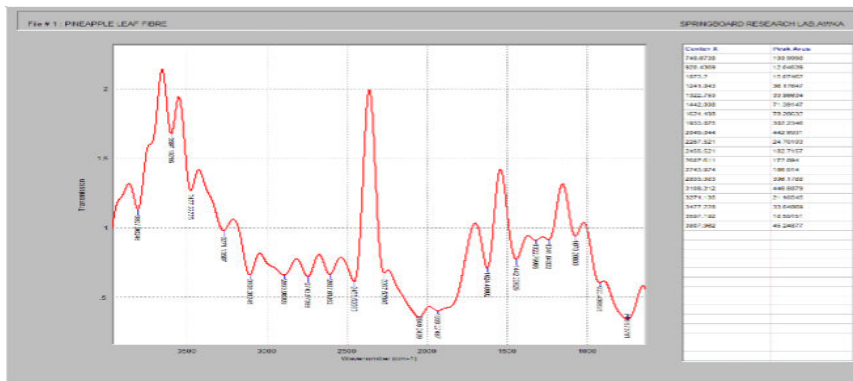


Figure-2. FTIR of untreated pineapple leaf fiber.

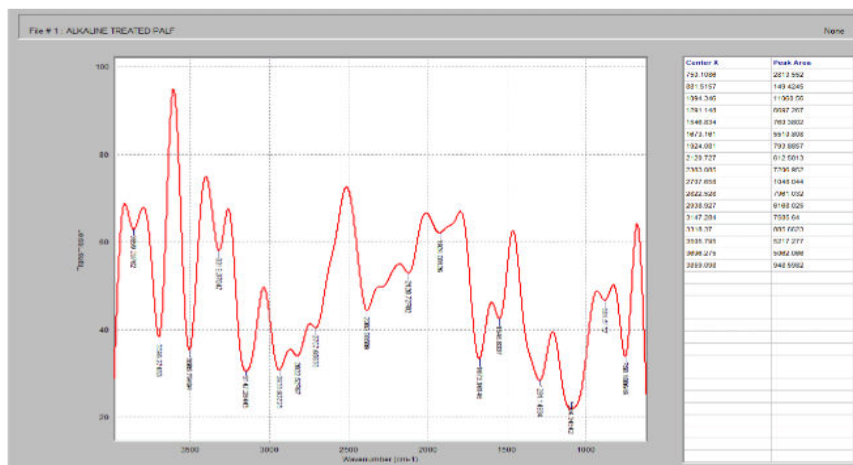


Figure-3. Alkali-treated pineapple leaf fiber.

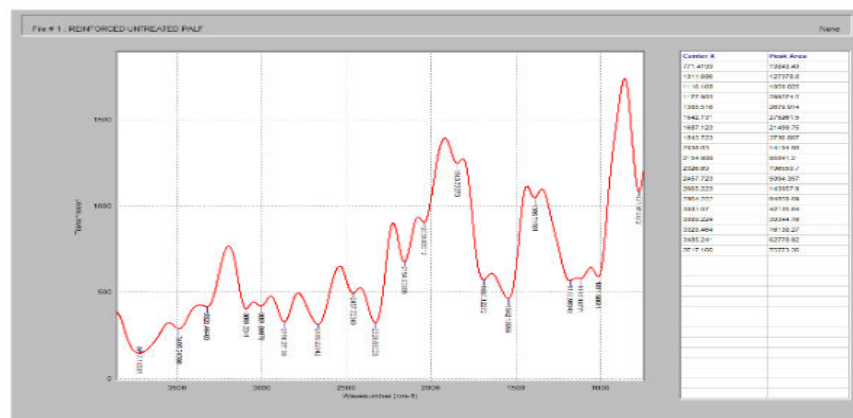


Figure-4. FTIR of untreated PALF reinforced with TBR.

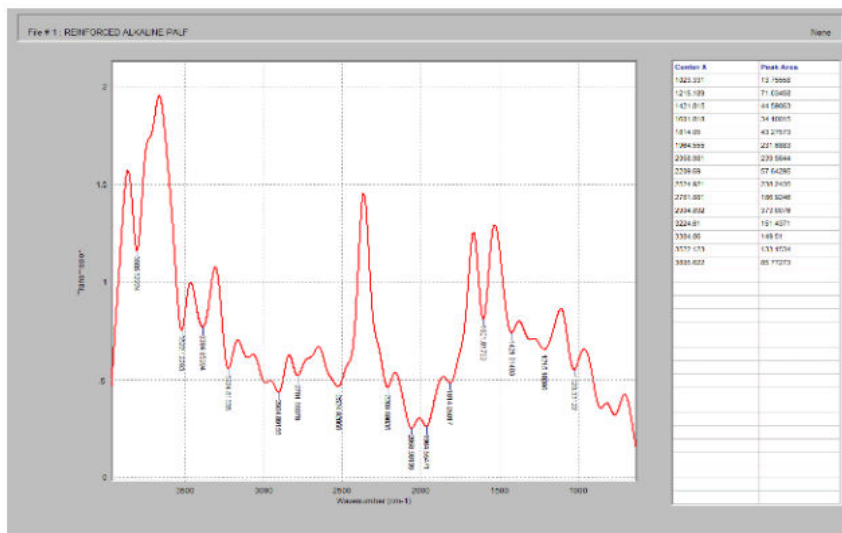


Figure-5. FTIR of alkaline treated PALF reinforced with TBR.

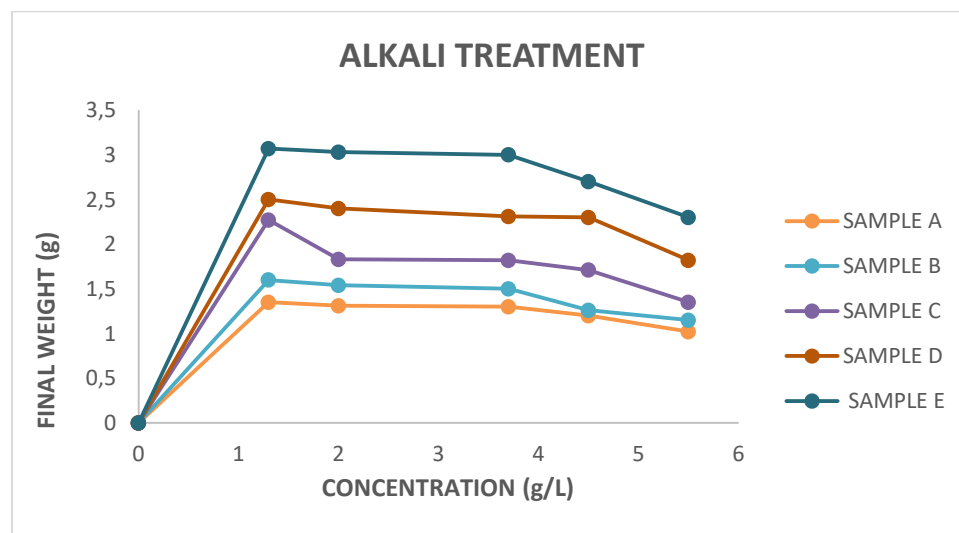
### 3.2 Alkali Treatment

From the alkali treatment of pineapple leaf fiber of different samples at varied concentrations, temperatures, and time, we observed a decrease in fiber weight with an increase in the concentrations of NaOH, temperature, and time. The highest weight losses of (0.38g, 0.85g, 1.38g, 1.38g, and 1.70g) on the fiber were got at the highest concentrations of alkali (5.5g/L, 4.5g/L, 3.7g/L, 2.0g/L, and 1.3g/L), temperatures of (90°C, 60°C,

45°C, 30°C, and 27°C), and time (120, 90, 60, 45 and 30 minutes) respectively, for all the samples investigated. Similarly, the lowest weight loss of (0.05g, 0.4g, 0.23g, 0.70g, and 0.93g) on the fiber was got from the lowest concentration of alkali (1.3g/L, 2.0g/L, 3.7g/L, 4.5g/L, and 5.5g/L), temperatures of (27°C, 30°C, 45°C, 60°C, and 90°C), and time (30, 45, 60, 90 and 120 minutes) respectively, for all the samples investigated. See Table-1.

**Table-1.** Alkali treatment of pineapple leaf fiber samples at varied concentrations, temperatures, and times.

Sample	Conc. of NaOH (g/L)	Volume of water(ml)	Temp (°C)	Time (min)	Initial weight (g)	Final weight (g)	Weight loss (g)
A (1.4g)	1.3	500	27	30	1.4	1.35	0.05
	2.0	500	30	45	1.4	1.31	0.09
	3.7	500	45	60	1.4	1.30	0.10
	4.5	500	60	90	1.4	1.20	0.20
	5.5	500	90	120	1.4	1.02	0.38
B (2.0g)	1.3	500	27	30	2.0	1.60	0.40
	2.0	500	30	45	2.0	1.54	0.46
	3.7	500	45	60	2.0	1.50	0.50
	4.5	500	60	90	2.0	1.26	0.74
	5.5	500	90	120	2.0	1.15	0.85
C (2.5g)	1.3	500	27	30	2.5	2.27	0.23
	2.0	500	30	45	2.5	1.83	0.67
	3.7	500	45	60	2.5	1.82	0.68
	4.5	500	60	90	2.5	1.71	0.79
	5.5	500	90	120	2.5	1.35	1.38
D (3.2g)	1.3	500	27	30	3.2	2.50	0.70
	2.0	500	30	45	3.2	2.40	0.80
	3.7	500	45	60	3.2	2.31	0.89
	4.5	500	60	90	3.2	2.30	0.90
	5.5	500	90	120	3.2	1.82	1.38
E (4.0g)	1.3	500	27	30	4.0	3.07	0.93
	2.0	500	30	45	4.0	3.03	0.97
	3.7	500	45	60	4.0	3.00	1.00
	4.5	500	60	90	4.0	2.70	1.30
	5.5	500	90	120	4.0	2.30	1.70

**Figure-6.** Graph of alkali treatment of samples at different temperatures and concentrations.



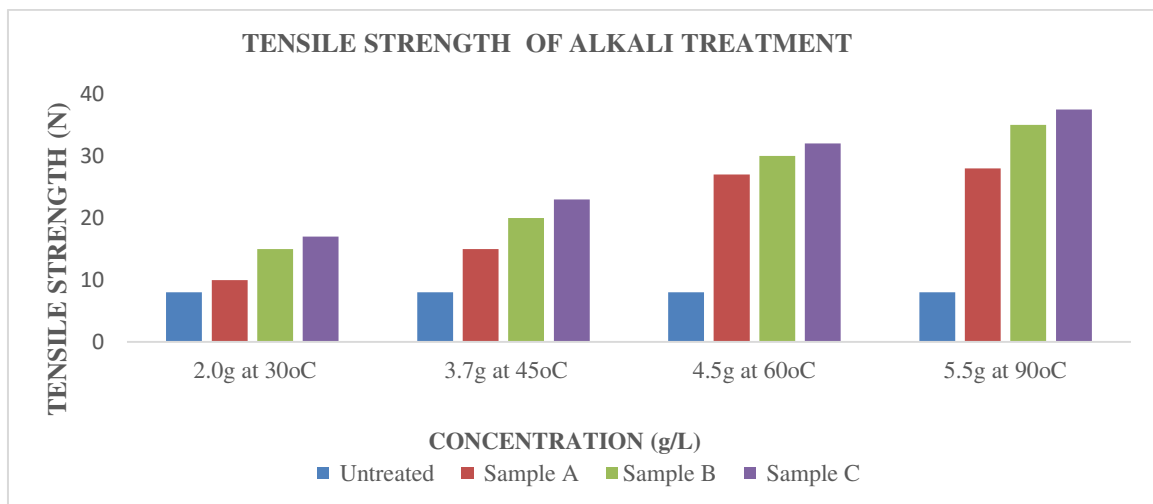
### 3.3 Tensile Strength

After the analysis of alkali-treated pineapple leaf fiber using a yarn textile testing machine, we observed that alkali treatment increased the strength of fibers with the increase in concentration. This was proved by the results obtained from all samples A, B, and C respectively. They had their highest tensile strengths of 28N, 35N, and 37.5N respectively, at their highest concentrations of NaOH of

5.5 g/l. They also had their lowest tensile strengths of 8N, 15N, and 17N respectively at their lowest concentrations of NaOH of 2.0 g/l. In general, fibers that were not subjected to any treatment possessed the lowest tensile strength of 8N. It then shows that alkali treatments on the fibers enhance their strength. It will be of great importance that all fibers be treated chemically especially treating with alkali. See Table 2 and Fig. 7 below.

**Table-2.** Tensile strength of Alkali treated pineapple leaf fiber with varied concentrations.

Conc. of NaOH (g/L)	Sample A		Sample B		Sample C	
	Weight of fiber (g)	Force at break (N)	Weight of fiber (g)	Force at break (N)	Weight of fiber (g)	Force at break (N)
Untreated	1.40	8	3.20	8	4.00	8
2.0	1.40	10	3.20	15	4.00	17
3.7	1.40	15	3.20	20	4.00	23
4.5	1.40	27	3.20	30	4.00	32
5.5	1.40	28	3.20	35	4.00	37.5



**Figure-7.** Tensile strength of alkali treated pineapple leaf Fiber.

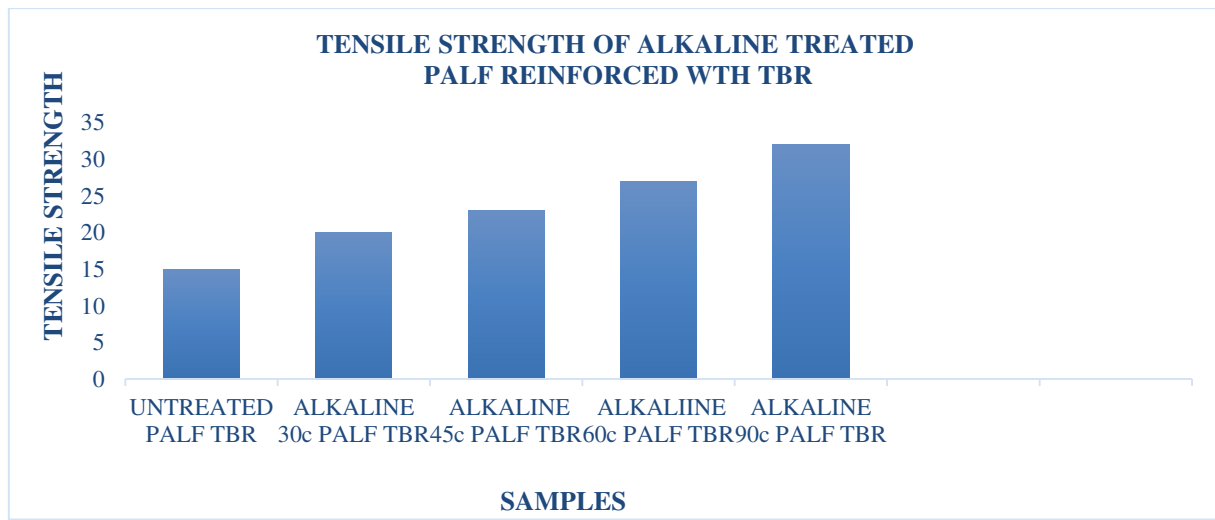
### 3.4 Effect of Reinforcing Pineapple Leaf Fibers with Cassava Starch Bio Resin

The weight of fibers increased after reinforcement, and the level of absorption decreased with an increase in temperature, the highest rate of absorption was achieved at the lowest temperature of 30°C, while the fibers treated at 90°C had the lowest rate of absorption.

There was an increase in the strength of the fibers after reinforcing them with bio resin. There was an increase in the strength of the fiber with an increase in temperature and an increase in the concentration of alkali. The strength of all the treated reinforced fibers was better than the untreated fiber. This showed that alkali treatment on the fiber enhances the fiber strength See Table-3 and Figure-8.

**Table-3.** Change in mass and tensile strength for alkaline treated PALF reinforced with TBR.

Sample	Conc of NaOH (g)	Conc. of TBR (g/L)	Weight before reinforcement (g)	Weight after reinforcement (g)	Weight Gain (g)	Tensile strength (N)
Untreated	–	32.5	2.00	2.45	0.45	15
Alkaline 30°C	2.0	32.5	1.54	2.86	1.32	20
Alkaline 45°C	3.7	32.5	1.50	2.67	1.17	23
Alkaline 60°C	4.5	32.5	1.26	2.34	1.08	27
Alkaline 90°C	5.5	32.5	1.51	2.15	1.00	32

**Figure-8.** Tensile strength of alkaline treated PALF reinforced with TBR.

#### 4. CONCLUSIONS

In conclusion, we observed that the chemical treatment of fibers enhanced the fiber's strength. Similarly, an increase in the concentration of alkali, treatment time, and temperatures enhanced the mechanical properties of fibers. Therefore, we are encouraged to treat our fibers before subjecting them to further use.

#### Conflict of interest:

On behalf of all the authors, the corresponding author(s) state that there is no conflict of interest.

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