



Research Article

Characterization and optimization of alkali-treated *yushania alpina* bamboo fiber properties: case study of ethiopia species

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Abstract

The mechanical properties of single *Yushania alpina* bamboo fibers have not been explored. This is a serious limitation on their application. The main purpose of this work is to fill up information gaps to prepare for the growing usage of Ethiopian bamboo in a variety of applications. This study looks at the characterization and optimization of *Y.alpina* bamboo fiber properties extracted both chemically and mechanically. Using response surface methodology (RSM) the mechanical properties were optimized and linear, quadratic and interaction of independent variables were determined. Samples of length 25–30 cm were harvested at various ages from the middle of the stem which was then soaked in different NaOH concentrations weight by volume for different times. Using a rolling machine that has three rollers, the fiber is mechanically extracted. The optimal mechanical properties were observed at plant age of 1.8 years, alkali concentration of 10%, and a soaking duration of 2.0 days. The model is significant ($P \leq 0.005$) with a 95% confidence level for predicted values that were closer to the measured values, indicating that the model's fit to the measured properties was strong at the optimized values. The optimized points of age and soaking duration were subjected to chemical, thermal and morphological analysis for each corresponding NaOH Concentration (6, 12, and 18%) levels. Scanning electron microscopy (SEM) was employed to examine the microstructure of the fibers and discovered that the 18% NaOH treated fiber resulted in more wrinkles in the surface of bamboo fibers when compared with the 6 and 12%NaOH Bamboo fiber. Using thermogravimetric analysis (TGA) and differential thermal gravimetric (DTG), the study investigated weight loss increased as alkali concentration increased but the scenario functioned for proper concentration.

Keywords *Yushania alpina* bamboo fiber · Alkali treatment · Characterization · Optimization · Mechanical properties

1 Introduction

Society's increased awareness of pollution in its many forms has led to increased use of natural instead of synthetic fibers for different applications in the energy, automotive, and textile industries. Other applications include construction materials, and home appliances [1]. Among

these natural fibers, bamboo possesses a high strength-to-weight ratio because of its longitudinally aligned fibers [2]. Chemically, bamboo is similar to wood with lignin, cellulose, and hemicelluloses accounting for more than 90% of the total bamboo mass [3]. The majority of previous studies considered bamboo fiber applications rather than their mechanical properties. Low-cellulose refined bamboo

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fiber can be used in textiles [4]. The possibility of developing economical bamboo Thailand textiles, motivated by the lack of weaving materials and the pollutants produced in their manufacture, and to replace imported weaving materials [5]. Due to the low alkali index, ash content, and hemicelluloses fiber properties, bamboo is suitable for bioenergy products including charcoal, biofuels, pyrolysis, gasification, briquettes, pellets, biomass, and bioethanol, through various production processes [6–8]. Bamboo is hard, lightweight, and has a high specific strength, tensile strength, and tensile modulus. Due to these properties, it is used for versatile construction materials, concrete and composite reinforcement, fencing, and housing [9–13]. Bamboo is suitable for pulverizing due to its form, chemical content, and structure. Paper produced from bamboo reduces the demand for wood and can cause less pollution in the manufacture [14]. Bamboo fiber pulp can be used to make newsprint, stickers, toilet paper, cardboard, cement bags, and coffee filters in the paper industry [15]. *Y. alpina*, which is Ethiopian indigenous bamboo species, suggested employing in the making of paper and suitable raw materials in the pulp industries [16]. The investigation on low-cost bamboo towers for small wind turbines explore that the characteristic properties of untreated bamboo and showed the potential for cost reduction for small wind towers [17]. The cellulose content of the fiber determines its mechanical properties, which is controlled by a variety of elements including fiber volume ratio, fiber length, yarn ratio, fiber-matrix adhesion or fiber orientation, plant age, fiber species type, and extraction procedures [18].

Bamboo species highly affect the mechanical properties as shown in Table 1. The study on the mechanical properties of different bamboo species: *Bambusa balcooa*, *Bambusa tulda*, *Bambusa salarkhanii*, and *Melocanna baccifera*, grown in southwestern Bangladesh was carried out at different ages and heights of sample collection [19]. The modulus of elasticity between bamboo species varies extensively and the modulus of elasticity values of the

Bambusa balcooa species changed significantly with age and height of collection [19]. The mechanical properties of bamboos at different ages (1, 3, and 5 years) were investigated by previous studies and explored that the bending and compressive strength improved as age increased [20].

Alkali treatment (NaOH) caused gaps in the cell wall by reducing the diameter, lumen, and cross-sectional area of the bamboo fiber. With alkali treatment, individual bamboo fiber tensile strength and modulus of elasticity (MOE) decreased [23]. The mechanical characteristics of different species and bamboo plant ages were explored and it was shown that Lv and Ma bamboo fibers reach their maximum tensile strength after one year [26]. At different NaOH concentration weight by volume (6, 8, 10, 15, and 25%), the tensile strength was 0.64, 0.68, 0.59 and 0.61 Gpa respectively. The concentration also affected the modulus of elasticity and elongation at failure of bamboo fiber, different methods have been used to extract bamboo fibers depending on their application [27]. These processes are classified as chemical and mechanical. In chemical extraction, the mechanical properties of bamboo fiber were affected by the soaking time of the fiber. The tensile strength at the different soaking times was 13.64, 15.75, 15.58 Nm/g, and 15.36 Nm/g, attained by soaking for 5 min, 10 min, 15 min, and 20 min respectively [28, 29]. An alkaline (e.g. NaOH) or an acid (e.g. HCl) is used to remove amorphous regions and reduce the lignin content of the fiber, In addition, this chemical treatment affects other components of the bamboo microstructure such as pectin and hemicelluloses. Mechanical methods involve various mechanical procedures such as; steam or autoclave sandblasting, high-pressure refining, grinding, rolling, and crushing [30]. These mechanical and other extraction methods have a significant effect on the mechanical properties as shown in Table 2.

Ethiopia has over 1.3 million hectares of bamboo, accounting for 67% of Africa’s bamboo growing area and over 7% of the global bamboo area. Ethiopia has two

Table 1 the mechanical properties of different bamboo species

Species	Breaking force N	Elongation %	Tensile strength Mpa	Work done cNmm	Modulus Gpa	References
<i>Moso</i>	4.1	–	150.1	–	9.6	[21]
<i>Thorny</i>	5.1	–	195.5	–	7.8	
<i>Makino</i>	3.5	–	137.76	–	10.2	
<i>G.S.Gamble</i>	–	–	368.3	–	22	[22]
<i>B.vulgaris</i>	–	7.7	366.8	–	3.8	[3]
<i>N.affinis</i>	–	8.21	590	–	8.9	[23]
<i>Bambusa vulgaris</i>	–	–	339	–	–	[24]
<i>Vulgaris</i>	3.58	3.98	212		5.3	[25]
<i>Y.alpina</i>	7.8	2	411.7	516.3	17.7	Measured

1 N = 100cN, 1Mpa = 0.111cN/tex

Table 2 Effect of extraction technique on the fiber properties [31–34]

Type of treatment	Parameter of treatment	Strength(Mpa)	Modulus (Gpa)	Elongation (%)
Mechanical	First, a roller crusher, then a pin roller, and last, a 10-h boil at 90° C	420 ± 170	38.2 ± 16	9.8 ± 2.5
Steam explosion	heated for 9 times at 175 °C, 0.8 MPa	383	28	2.82
Chemical	NaOH solution of higher concentration	329	22	2.35
Alkali + mechanical	1.5 N NaOH, heated at 70 °C for 5 h, pressed machine	419	30	2.67

common bamboo species, *Y.alpina* (highland bamboo) and the *Abyssinica Oxytenanthera* (lowland bamboo) [35]. Highland bamboo grows in Ethiopia's southern, south-western, central, and northwestern highlands and covers an estimated 300,000 hectares [36].

The thermal stability or decomposition behavior of alkali-treated *Y.alpina* bamboo fiber at different concentrations was investigated by thermogravimetric analysis (TGA). All of the alkali-treated bamboo fibers exhibited distinct weight loss (decomposition stages). Three thermal decomposition stages characterized the total degradation profile of bamboo fiber, namely moisture evaporation, cellulose decomposition, and lignin decomposition [37]. The first stage of weight loss is referred to as the dehydration stage, which mentioned moisture evaporation from the fiber structure. Whereas the rest stages are linked to the degradation of polysaccharides such as cellulose, hemicelluloses, and lignin [38, 39]. Hemicelluloses start to degrade earlier and decompose in the range 220–350 °C. The low thermal stability of hemicelluloses is assumed to be due to a lower degree of polymerization compared to cellulose and lignin [40, 41].

The mechanical properties of bamboo are mainly provided by the cellulose content, which is influenced by many factors such as fiber volume fraction, fiber length, fiber aspect ratio, fiber-matrix adhesion or fiber orientation, plant age, plant height, species type, and extraction methods [42–46]. However, the research focuses on the characterization and optimization of mechanical properties of alkali-treated *Y.alpina* single fiber properties. Because the remainder of the parameters is in the composite case, the study used plant age, alkali content, and soaking duration as factors for the RSM design.

The mechanical properties of *Y.alpina* single fibers have not been examined. This is a serious limitation on their use. The main aim of this study is to remedy this lack of information in anticipation of the increasing use of Ethiopian bamboo in a variety of applications. In the stage of surface-modified fibers, alkali treatment is currently applied. The material is immersed in an alkaline solution at various concentrations during the process.

In this experimental study, bamboo fiber (*Y.alpina*) was obtained through alkali treatment at different plant ages

(1, 2, and 3 years), concentrations weight by volume (6, 12, and 18%), and soaking durations (1, 2, and 3 days) were tested using a universal testing machine and optimized the best mechanical properties using Design Expert® 11 (Stat-Ease Inc, Minneapolis, USA) by compared with the interaction results of different variables.

2 Materials and methods

2.1 Materials

2.1.1 Bamboo sample

The bamboo species used in the experiments is *Y.alpina* (Fig. 1). Bamboo stem samples were obtained from 1 to 3 years-old plantations in Enjibara (latitude: 10,057'N, longitude: 36,056'E, and elevation 2560 m above sea level), Awi region, West Gojam, Amhara region, Ethiopia.

2.1.2 Chemicals

sodium hydroxide (NaOH, manufacturer: RESEARCH-LAB FINE CHEM INDUSTRIES, Assay: min98%) at a different



Fig. 1 *Y.alpina* bamboo plant in Ethiopia

concentration weight by volume of 6, 12, and 18% and soaking duration of 1, 2, and 3 days.

2.2 Methods

2.2.1 Raw material preparation

On collection, the bamboo sections were a wet mass of various residues. They were cleaved with a slicer at the middle stem around 25–30 cm in length, and 2.0–2.5 mm thickness to remove residual parts and prepare bamboo strips.

2.2.2 Chemical analysis

The following methods were used to determine the chemical composition of *Y.alpina*. The chemical compositions of lignin and hemicellulose content were determined using ASTM standards. The cellulose content was determined using the Kurschner-Hoffer method. The experiment was repeated three times.

2.2.3 Proximate analysis

The proximate chemical analysis was performed to estimate moisture content, volatile matter content, ash content, and fixed carbon content. In porcelain crucibles, 1.0 g samples were used. All of the tests were carried out in triplicate.

2.2.4 Moisture content

At 105 °C for 1 h, samples were placed in an oven (KEMI Model: KOA.6.F, INDIA) until they reached a consistent mass. Equation 1 was used to determine the moisture (Mc) content of the samples using the ASTM E 871 standard and wet basis moisture content.

$$Mc = \frac{m_1 - m_2}{m_1} \times 100 \quad (1)$$

Mc: Moistur content(%), m_1 : Wet mass(g), m_2 : Dry mass(g).

2.2.5 Volatile matter content

Dry samples were placed in a Box-type resistance furnace (Model: BK-5-12GJ) for seven minutes at 950 °C. The samples were weighed after cooling in a desiccator and determined the volatile matter content using the ASTM E872 standard using Eq. 2.

$$Mvm = \frac{m_2 - m_3}{m_1} \times 100 \quad (2)$$

Mvm: Volatile matter content (%), m_3 : Dry sample weight(g) after placed in an oven at 950 °C for 7 min.

2.2.6 Ash content

The identical material used in the analysis of ash content was taken to a Box-type resistance furnace (Model: BK-5-12GJ) at 750 °C for 30 min until it reached constant mass, according to the ASTM E1755 standard. It is assumed that only ashes remain inside the crucible at the end of this process.

$$Ma = \frac{m_4}{m_1} \times 100 \quad (3)$$

Ma: Ash content (%), m_4 : Ash weight(g).

2.2.7 Fixed carbon content

For moisture, ash, and volatile matter, conducted a proximate analysis based on ASTM standards E 871, E 1755, and E 872, respectively. fixed carbon content calculated using Eq. 4.

$$F_{CC} = 100 - [Mc + Mvm + Ma] \quad (4)$$

F_{CC} : Fixed carbon content (%).

2.2.8 Ultimate analysis

An elemental analyzer (Model: EA 1112 Flash CHNS/O-analyzer) was used to determine the contents of carbon, hydrogen, nitrogen, and sulfur with a carrier gas flow rate of 120 ml/min, a reference flow rate of 100 ml/min, and an oxygen flow rate of 250 ml/min; a furnace temperature of 900 °C, and an oven temperature of 75 °C. the oxygen content was calculated by subtracting the sum of carbon, hydrogen, nitrogen, and sulfur components.

2.2.9 Extraction

The following combinations of chemical and mechanical methods fiber extraction were used; soaked the bamboo strips with NaOH concentrations (6–18% w/v), plant age (1–3 years), and soaking duration (1–3 days). After soaking, the fibers were washed with tap water until the solution was neutralized. Then a roller crusher machine (PHOENIX, Belgium Products) was utilized to extract small fibers from the alkali-treated bamboo strips. the extracted fibers (Fig. 2) were dried at 105 °C oven for 1 h.

3 Experimental design

The subset of the response surface methodology (RSM) design known as the Box-Benhken design (BBD) was used to determine the main effects and interactions of



Fig. 2 *Y.alpina* bamboo fiber **a** Untreated **b** Treated **c** Treated fiber testing sample hold on a plastic holder

Table 3 Design layout of independent variables and coded values

Factors	Symbol	Unit	Range and level		
			Low	Middle/ Central	High
			-1	0	1
Plant age	A	Year	1	2	3
NaOH Concentration	B	%	6	12	18
Soaking duration	C	Day	1	2	3

the variables of independent processes as well as the model equations for each dependent variable to extend the application of the proposed process and optimize the process parameters [47–49]. Using BBD can strongly decrease the number of experimental sets without diminishing the exactness of the optimization compared with conventional factorial design methods [50]. The

study presents by focusing on the effect of plant age, alkali concentration, and soaking time on the mechanical properties because in the case of alkali-treated single fibers the cellulose content is highly affected by these factors [26, 46, 51, 52]. All variables (plant age, NaOH concentration, and soaking time) can be coded as, low (– 1) medium (0), and high (+ 1). The extent and extent of each independent factor based on BBD were presented in Table 3.

Design Expert® 11 was utilized within the plan lattice and examination of the test information. 13 experiments with 5 central points per block were carried out, as shown in Table 4.

The relationship between the independent variables of plant age (A), concentration (B), and soaking time (C), and responses 'y' (breaking force, elongation, tenacity, work done, and modulus) were fit to the 3-factor polynomial model:

$$y = \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_{12}AB + \beta_{11}A^2 + \beta_{22}B^2 + \beta_{33}C^2 + \beta_{13}AC + \beta_{23}BC + \beta_{123}ABC \tag{5}$$

Table 4 Experimental design factors and responses values from design expert 11

Run	A Year	B %	C Day	Breaking Force cN	Elongation %	Tenacity cNtex ⁻¹	Work done cNmm	Modulus cNtex ⁻¹
1	2	12	2	780.8	2.0	45.9	508.2	1876
2	1	6	2	574.8	1.7	33.8	414.6	2463
3	2	6	1	460.1	1.3	27.1	139.4	1588
4	1	12	1	417.9	0.8	24.6	95.6	1748
5	3	12	1	491.8	1.8	28.9	166.3	911.7
6	1	18	2	460.7	1.3	27.1	161.8	1416
7	3	18	2	569.6	1.3	33.5	238.7	1748
8	3	6	2	449.3	1.1	26.4	146.9	1449
9	2	6	3	616.7	1.2	36.2	276.6	1943
10	1	12	3	374.5	1.0	22.0	99.5	1319
11	2	18	1	326.1	0.7	19.2	54.2	1516
12	2	18	3	484.3	0.9	28.5	125.4	1978
13	3	12	3	363.3	0.8	21.4	57.3	1582

where β_0 is the constant term, β_i , β_{ij} , and β_{ijk} are the coefficients of the various order terms.

3.1 Characterization

Mechanical properties (breaking force, elongation, tenacity, work done, and modulus) of 13 samples were examined based on design expert software recommendations. The tests were carried out according to ISO 13934–1: 2013 testing standard by a semi-automatic tensile testing machine. The available load cells were 20, 100, 500, 1000 N, and 5000 N. At stable conditions, the speed and force measurement accuracy of the machine are $\pm 0.01\%$ and $\pm 0.03\%$ respectively. The capacity of the load cell used was 20 N with a force maximum resolution of 0.0002 cN. The gauge length of the measurement is 50 mm and the testing speed range is 0.001–1000 mm/min. The samples were aligned and an axial tensile load was then applied. No rotation or translation of the sample was allowed. The mechanical properties (responses) were recorded up to failure which often started with a small notch-like crack or splitting in the sample.

The chemical and thermal characterization were carried out on the optimized points of age and soaking duration (1.8 years ago, 2.0 days soaking duration) that generated the optimal mechanical properties were subjected to FTIR, TGA, DTG, and SEM analysis for each corresponding NaOH Concentration (6, 12, and 18%) levels.

The functional groups and other impurities were identified using Fourier transform infrared spectroscopy (JASCO MODEL FT-IR 6660) analysis in the wave range of 4000–400 cm^{-1} . The thermal stability studied was done by thermogravimetric (BJHENVEN HCT-1) Analysis with the temperature ranging from 20 to 700 °C at a rate of 20 °C per minute in an air atmosphere. Scanning electron microscopy (JCM_6000 PLUS) illustrated the morphological variations due to the alkali treatment of fibers with a random orientation at different concentrations with various magnifications and an acceleration voltage of 10 kV.

4 Results and discussion

4.1 Raw materials chemical analysis

For improved mechanical properties, the bamboo plant which has high hemicellulose and cellulose content and low lignin content is preferred [53]. Table 5 shows the main chemical composition of the *Y. alpina* bamboo and its comparison to other bamboo species. The table exhibited that the chemical composition ranged cellulose 37.21–53.50%, hemicellulose 12.18–21.6%, and lignin 21.3–25.27%. The findings of this study indicated that cellulose (51.06%),

Table 5 Main chemical composition analysis of *Y. alpina* compared with other different bamboo species

Species	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	Reference
<i>Haur hejo</i>	52.3	19.15	20.01	[54]
<i>Tali</i>	46.91	17.29	21.3	
<i>P.heterocycla</i>	37.21	21.6	24.29	[55]
<i>O.abysinia</i>	52.06	16.90	22.47	[56]
<i>Y.alpina</i>	46.76	12.18	25.27	[57]
<i>Y.alpina</i>	53.50	18.10	24.20	[16]
<i>Y.alpina</i>	51.06	20.19	23.79	Measured

hemicellulose (20.19%), and lignin (23.79%) in *Y. alpina* bamboo species, had good cellulose and hemicellulose content so had good mechanical qualities.

The proximate and ultimate chemical analyses of the bamboo species *Y. alpina* as well as comparisons to other bamboo species are shown in Table 6. The moisture content of all species ranged from 7.1 to 18%. From the previous studies' results, Malaysia Bamboo showed higher moisture content (15.3%), while the *Betung* bamboo presented a lower value (7.1%). The bamboo species used in this study exhibited the highest value of moisture content (18%). This is a reasonable result considering the result of observing the high thickness of the woody part and growing weather. As seen in Table 6, the ash content ranged from 1.02 to 3.29%, with *Y. alpina* having the lowest value and *G.atrovio* having the highest value. Therefore *Y. alpina* is suitable for thermal use because it has low ash content. A species with a higher ash concentration has a lower calorific value. The bamboo species are considered in this study to have a fixed carbon content of 7.28% and volatile matter of 73.70%. As the temperature increased, the fixed carbon content increased but the volatile carbon content declined. In the combustion process, nitrogen and sulfur in fuel can produce polluting gases, such as NO_x and SO_x. As shown in Table 6, all bamboo species including *Y. alpina*, contain less than 1% nitrogen and sulfur respectively, indicating that using bamboo as a source of energy does not result in significant air pollution. The amount of carbon and hydrogen content in the bamboo culm is very satisfactory as they contribute immensely to the combustibility of any substance in which they are found.

4.2 FT-IR analysis

The chemical structures of 2 years age *Y. alpina* bamboo fibers treated with 6–18% NaOH soaking for 2 days were investigated using FTIR. For clarity, the FTIR spectra were shifted vertically using Software Origin, as shown in Fig. 2 All of the samples' results showed typical spectra

Table 6 Proximate and ultimate analysis of *Y.alpina* compared with other different bamboo species

Species	Proximate analysis (%)				Ultimate analysis (%)					Reference
	Moisture	VM	Ash	FC	C	N	O	H	S	
Malaysia Bamboo	15.30	70.12	1.76	11.32	38.40	–	–	–	0.07	[58]
<i>G.apus</i>	8.89	70.13	2.45	18.35	44.29	0.53	46.47	6.16	0.10	[59]
<i>G.levis</i>	8.76	72.71	2.46	16.07	44.65	0.48	45.97	6.35	0.09	
<i>G.atrovio</i>	8.13	71.70	3.29	16.88	44.11	0.47	45.8	6.26	0.07	
<i>Andong</i>	8.40	78.00	2.08	11.5	49.00	0.40	44.40	6.09	0.05	[60]
<i>Hitam</i>	7.80	72.40	1.36	18.4	50.30	0.46	42.90	6.21	0.05	
<i>Tali</i>	7.30	80.00	1.89	10.8	50.90	0.24	42.30	6.44	0.07	
<i>Kuning</i>	7.60	75.40	2.68	14.3	48.20	0.39	45.20	6.08	0.05	
<i>Ampel</i>	10.20	72.00	1.15	16.70	49.50	0.43	43.70	6.30	0.04	
<i>Betung</i>	7.10	75.40	2.44	15.10	48.70	0.33	44.90	6.00	0.05	
<i>Y.alpina</i>	18.00	73.70	1.02	7.28	43.21	0.37	49.83	6.50	0.09	Measured

VM Volatile matter, FC Fixed carbon, C Carbon, N Nitrogen, O Oxygen, H Hydrogen, S Sulphur

Table 7 Characteristic bands for a functional group of alkali-treated *Y.alpina* bamboo fiber at various concentrations [64–69]

Wavenumber (cm ⁻¹)	Functional group	Compounds
3434 – 3415	-OH	Alcohol(cellulose; hemi-cellulose; lignin), phenol (bond H), carboxylic acid
2926 – 2922	C-H	Alkanes
1621 – 1619	C=O	hemicelluloses and lignin
1387 – 1386	C-H	C-H deformation in cellulose and hemicelluloses
1110	C-O	Alcohol (cellulose; hemicellulose; lignin), ether, carboxylic acid, ester
618 – 616	O-H	out of plane bending

of cellulose were in line with earlier research [61–63]. The FTIR spectra of all samples were substantially identical, indicating that no new functional groups in the cellulose molecules were added. When the concentration of alkali is raised, however, the O–H stretching and H-bonded broad absorption band in the region is reduced. These are due to a decrease in the functional group of phenolic or aliphatic hydroxyl in the fiber due to reaction with sodium hydroxide that promotes free hydroxyl that caused the addition of extra peak in free hydroxyl bond structure [64]. The elimination of hemicelluloses and lignin could be linked to the reduction in these vibrational bands of the IR spectrum of alkali-treated fibers [37]. Table 7 shows the characterization of alkali-treated *Y.alpina* bamboo fibers at different concentrations of NaOH as extracted from Fig. 3.

4.3 Thermal decomposition analysis

The effect of increased temperature on the mass change of alkali-treated *Y.alpina* bamboo fiber was shown in Fig. 4 and Table 8. Four thermal decomposition stages characterized the total degradation profile of bamboo fiber, namely moisture evaporation, hemicelluloses decomposition, cellulose decomposition, and lignin decomposition. The

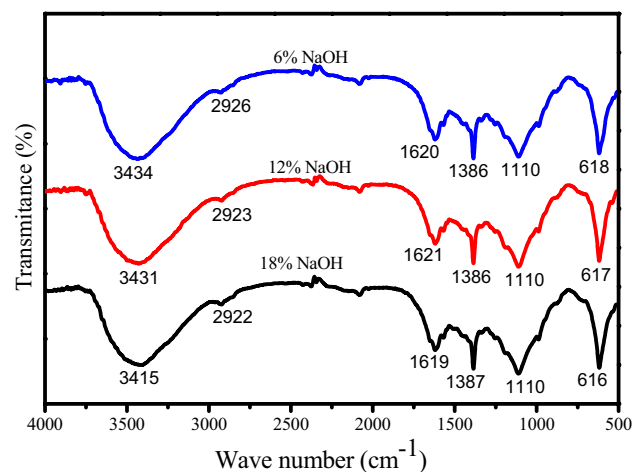


Fig. 3 FTIR analysis of alkali-treated *Y.alpina* bamboo fiber at different concentration

weight loss of *Y.alpina* bamboo fiber treated with 12% NaOH concentration during the dehydration stage is 8.8%, which is smaller than the 6% NaOH concentration (20% weight loss). The presence of water in the fiber was found to be reduced after an increase in the concentration

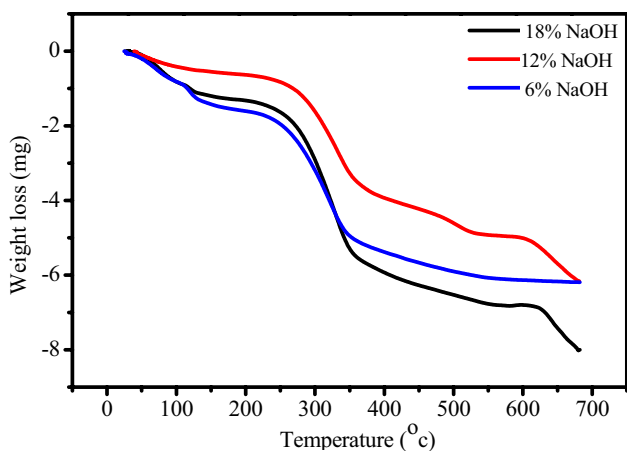


Fig. 4 TGA analysis of alkali-treated *Y.alpina* bamboo fiber with various NaOH concentrations

of alkali [70]. This phenomenon occurs as the alkali-sensitive components (active sites for moisture uptake) are eliminated by alkali treatment, resulting in a decrease in hydrophilicity and moisture absorption in the fiber [37]. even though this scenario happened as a result of proper concentration, weight loss increased to 17.5% when concentration was raised to 18%. The pattern shown above is similar to that described in the earlier report on bamboo fiber thermal properties. The decomposition of hemicelluloses, cellulose, and lignin correspondingly caused the weight loss of bamboo fibers in the first, second, and third degradation stages [63].

Maximum temperature (T_{max}) is the decomposition temperature that corresponds to the maximum weight loss and decomposition rate, as well as a key indicator of the materials' thermal stability [71]. As shown in Table 8, the maximal decomposition temperature of *Y.alpina* bamboo fibers treated with 6% NaOH is 355 °C. The maximal decomposition temperature climbed to 368 °C when the NaOH concentration was increased to 12%. It was discovered that alkali treatment with proper concentration can improve the thermal stability of individual bamboo fibers. However, the highest decomposition temperature was reduced to 353 °C when treated with alkali at a greater

concentration of 18%. This maximum temperature and weight loss discussion is also confirmed with DTG analysis as shown in Fig. 5. The onset temperature of decomposition shifted to a lower temperature as well [38]. This experimental investigation concluded that among the 6–18% NaOH treated *y.alphina* bamboo fibers, 12%NaOH concentration has optimal thermal stability properties.

5 Morphological analysis

Figures 6 and 7 exhibited the surface morphology of randomly arranged alkali-treated *Y.alpina* bamboo fibers with various NaOH concentrations. Figure 6 demonstrated the diameter of individual bamboo fibers decreased as the NaOH concentration increased. The dimension change of fiber possibly resulted from the loss of hemicelluloses and lignin matrix and also microfibrils aggregations with alkali treatment. Similar conduct was also observed in *Neosinocalamus affinis* bamboo fiber [23]. As shown in Fig. 7, the 18% NaOH treated fiber resulted in more wrinkles in the surface of bamboo fibers when compared with the 6 and 12%NaOH Bamboo fiber. This is due to the change

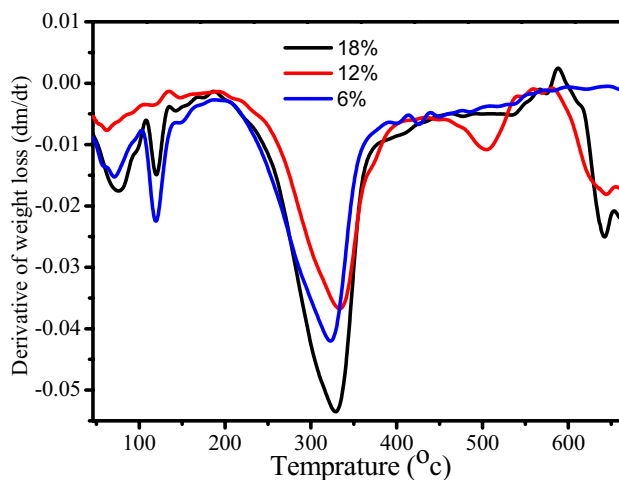


Fig. 5 DTG analysis of alkali-treated *Y.alpina* bamboo fiber with various NaOH concentrations

Table 8 Thermal stability data of alkali-treated *Y.alpina* bamboo fiber with various NaOH concentrations

Stages	6% NaOH			12% NaOH			18% NaOH		
	T	Weight loss		T	Weight loss		T	Weight loss	
	°C	Mg	%	°C	mg	%	°C	mg	%
Dehydration	≤212	1.6	20	≤220	0.7	8.8	≤213	1.4	17.5
1st Degradation	212–355	3.4	42.5	220–368	2.9	36.3	213–353	4.0	50.0
2nd Degradation	355- 574	1.1	13.8	368–588	1.4	17.5	353–573	1.4	17.5
3rd Degradation	574–680	0.09	1.13	588–680	1.19	14.9	573–680	1.2	15.0

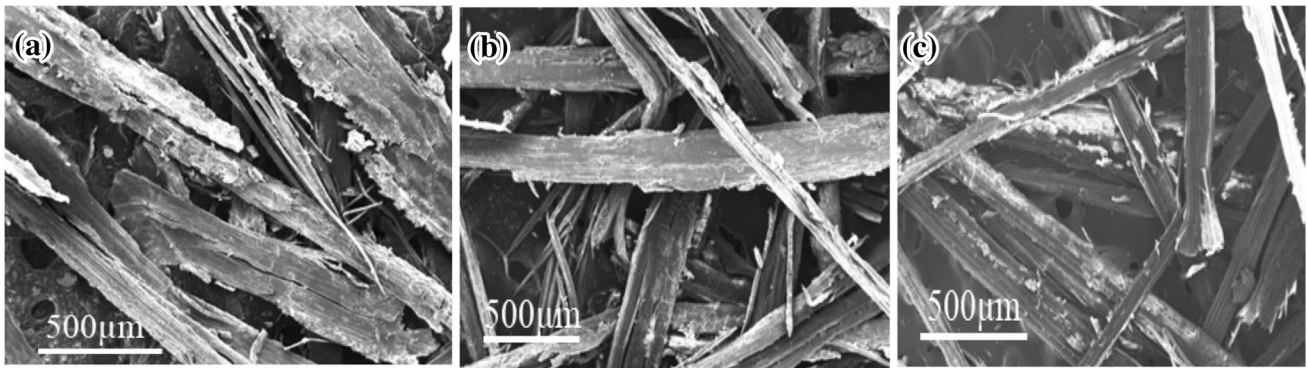


Fig. 6 SEM image of **a** 6% **b** 12% **c** 18% alkali treated *Y.alpina* bamboo fiber micromorphology

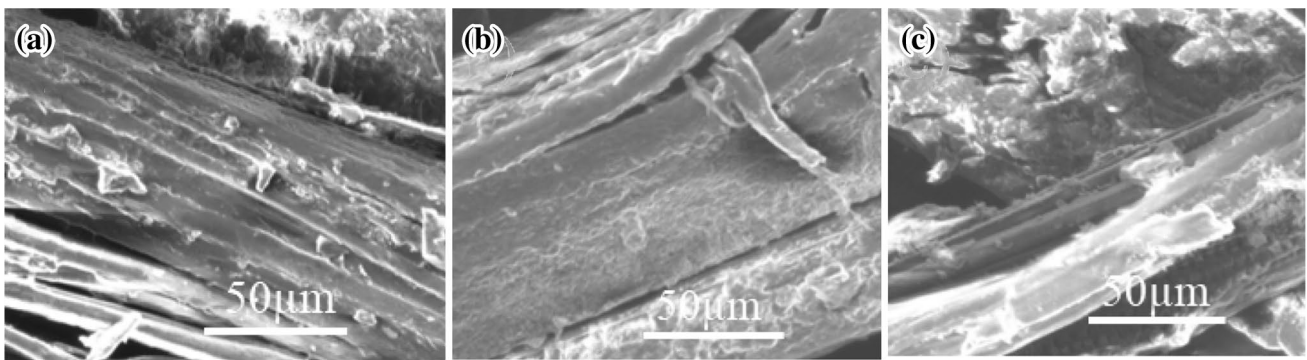


Fig. 7 SEM image of **a** 6% **b** 12% **c** 18% alkali treated *Y.alpina* bamboo fiber micromorphology

of the microfibril aggregates from a randomly interwoven structure to a granular structure. When the alkali concentration is increased, the surface morphological roughness increases due to the elimination of hemicelluloses lignin and other surface impurities Fig. 7. Because they provide good interfacial bonding between the polymer matrixes, these rough surfaces have more advantages in the fabrication of composite materials [72]. The partial loss of cellulose causes a decrease in fiber strength after treatment with high alkali concentrations. Hence an excessive concentration alkali treatment can damage the strength of single cellulosic bamboo strips [73]. There were also holes and grooves as shown in the figures, indicating that the alkali treatment was capable of removing considerable amounts of soluble elements from the layers.

6 Mechanical properties

The mechanical properties (breaking force, elongation, work done, modulus, and tenacity) of bamboo were affected by plant age. Elongation and modulus were not altered as much as breaking force, tenacity, and work

done. These mechanical properties increased with age until two years, after which they were reduced as shown in Fig. 8. This discovery is in line with the findings of a previous study of Moso bamboo (*Phyllostachys hetero-cyclavar. pubescens*) [46]. There was no significant difference in modulus with age, however, there were significant differences in tensile strengths of Lv and Ma (*Dendrocalamopsis oldhami* and *Dendrocalamus latiflorus Munro*) bamboo [26].

In this investigation, the alkali treatment was found to have a substantial impact on mechanical properties due to changes in fiber structure which is consistent with the previous study results [23]. The mechanical properties, except for modulus, increased when concentration was increased up to 12% and then decreased. The changes, however, were small as shown in Fig. 9.

With longer soaking, all the mechanical properties were increased. However, breaking force, elongation, tenacity, and work done declined quicker than modulus when the soaking duration was longer than 2 days as shown in Fig. 10. These findings are similar to those of a prior study [27].

The two-dimensional (2D) contour impacts of the input parameters on the mechanical behaviour were shown in

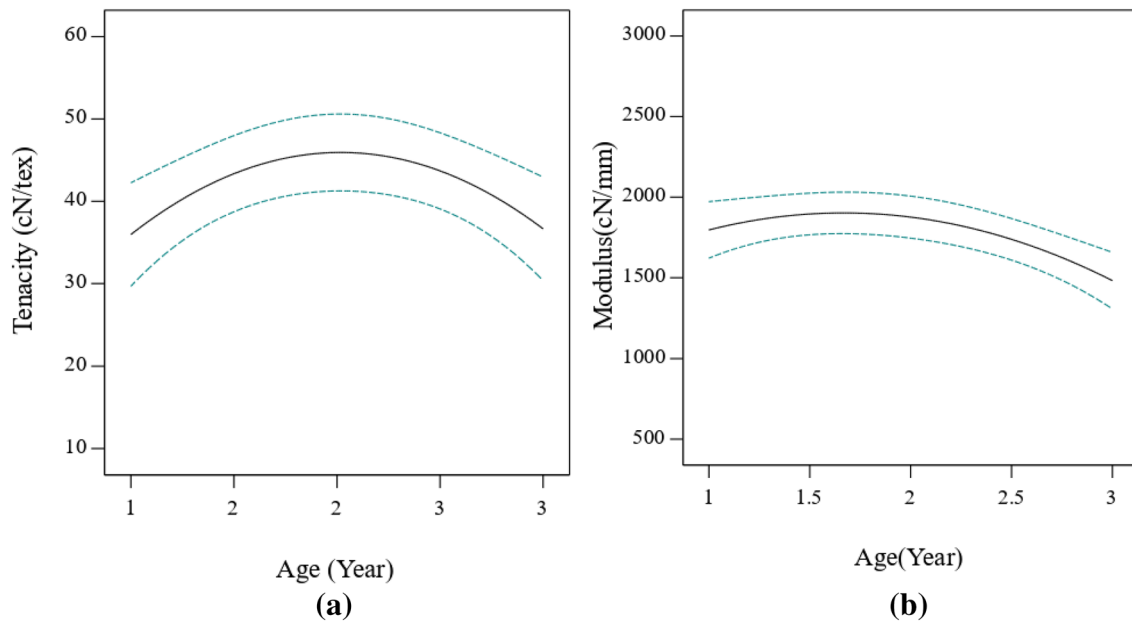


Fig. 8 Eq. (1) for concentration 12% and soaking time 2 days, solid line Dash lines show one standard deviation. **a** Age versus Tenacity **b** Age versus Modulus

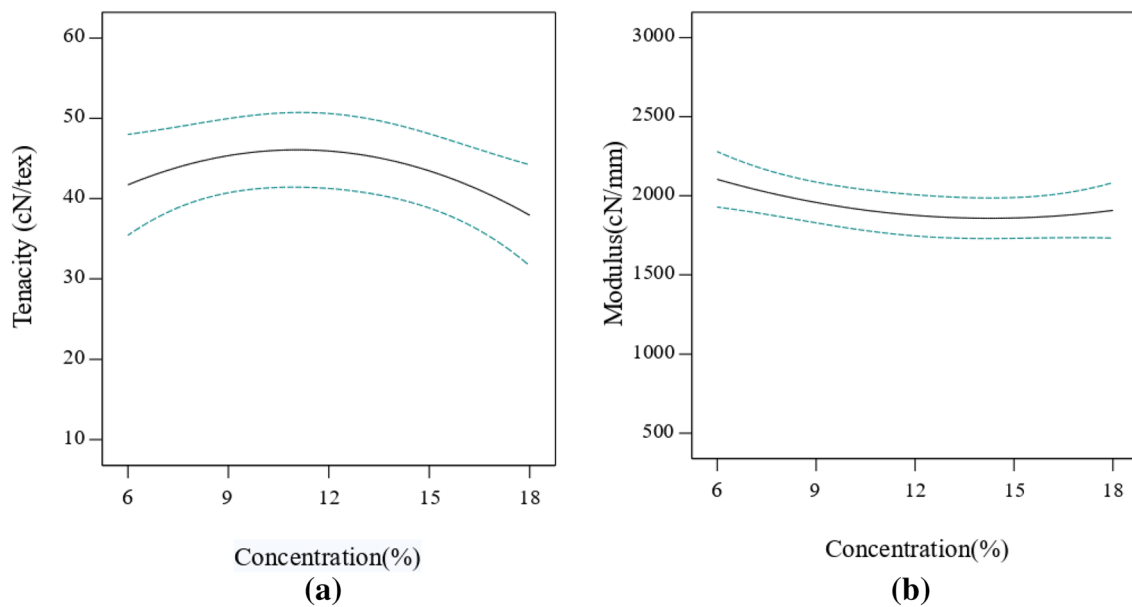


Fig. 9 Independent variables effect on the responses **a** Concentration versus Tenacity **b** Concentration versus Modulus

Fig. 11. The figure shows the breaking force, elongation, tenacity, work done, and modulus were all affected by the interaction of plant age, concentration, and soaking time parameters. In terms of plant age and soaking time, the mechanical properties (breaking force, elongation, tenacity, and work done) were maximized near 2.0 years, 2.0 days plant age, and soaking duration respectively. These mechanical properties reach their lowest point

near to 1 year and 3 years, except for modulus. The modulus had its highest value below 2 years, 2.5 days, and the lowest value near to 3 years, 3 days plant age, and soaking duration respectively. Except for modulus, the lowest values were near 1 year and 18% plant age and concentration respectively. The modulus was raised by reducing the plant age to approximately 1 year and the concentration to 6%. The lowest modulus occurred at 3 years and below 9% plant age and

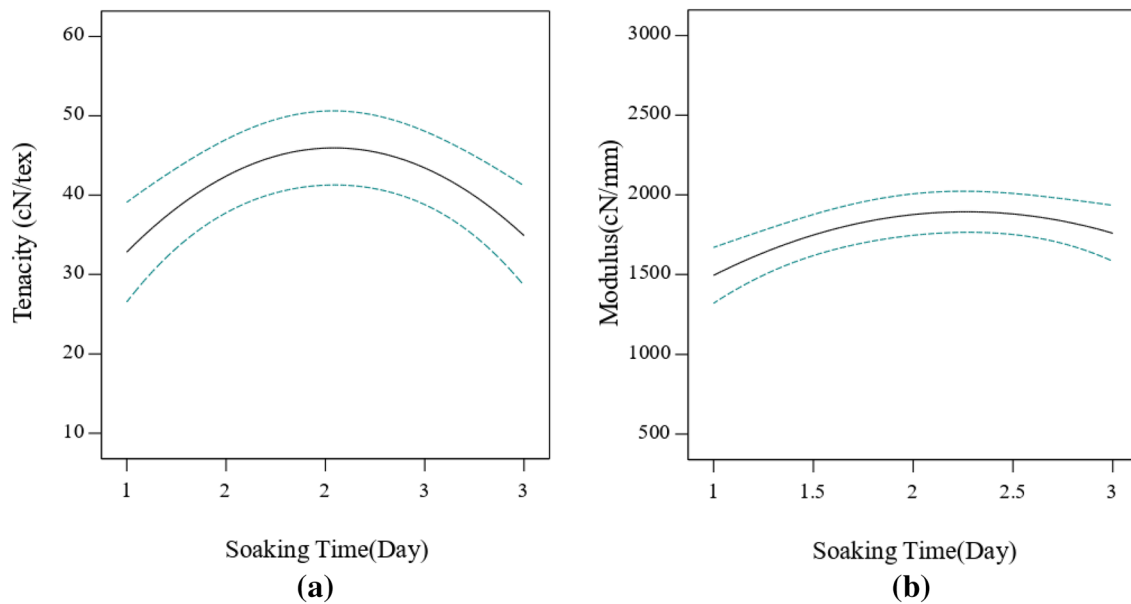


Fig. 10 independent variables effect on the responses **a** Soaking time versus Tenacity **b** Soaking time versus Modulus

concentration respectively. The interaction of soaking duration and concentration also affects the mechanical properties of highland bamboo. As seen in Fig. 5, the highest values occurred at around 2 days, 12%, soaking duration, and concentration respectively, the lowest values were at the lower and upper limits of the variables. The modulus, on the other hand, differed from the other mechanical properties because the maximum values were attained near to 6%, 2 days, and the lowest value was near to 14%, 1-day concentration, and soaking time respectively.

6.1 Model fitting and ANOVA analysis

As shown in Table 9, the p-values were used to determine the significant interaction between factors and responses. Values of $P \leq 0.05$ were considered to be significant. Breaking force Model F-value of 7.80 implies the model is significant. In other words, there is only a 0.65% chance that an F-value this large could occur due to noise. In this case, the quadratic terms of plant age, soaking time, and concentration were the only significant model terms ($P \leq 0.0256$). An F-value of elongation 6.93 has a 0.91% chance of occurring due to noise. The only significant model terms in the case of elongation ($P \leq 0.0496$) were the interaction of plant age and soaking time, quadratic values of plant age, soaking time, and concentration. The

tenacity model's F-value of 7.85 indicates that it was significant as this value has a 0.64% chance of occurring due to noise. The quadratic values of plant age, soaking duration, and concentration were the only model terms that were meaningful in the case of tenacity ($P \leq 0.0259$). The Model F-value of work done, 21.00, indicates that the model is significant as an F-value of this magnitude has a 0.03% chance of occurring due to noise. The p-values of the significant interactions between the factors and work done were used to assess their significance and the only significant model factors ($P \leq 0.0308$) were concentration, the interaction between plant age and concentration, quadratic values of plant age, soaking time, and concentration. The F-value of 12.75 for the modulus model indicates that the model is significant. Due to noise, this F-value has a 0.14% probability of occurring. The only significant model factors of modulus ($P \leq 0.0189$) were plant age, soaking time, plant age and concentration interaction, plant age and soaking time interaction, quadratic values of plant age, and soaking time.

$$Y_b = 780.76 - 163.54A^2 - 103.64B^2 - 205.33C^2 \quad (6)$$

$$Y_e = 2.04 - 0.2850AC - 0.2775A^2 - 0.3825B^2 - 0.6375C^2 \quad (7)$$

$$Y_t = 45.93 - 9.62A^2 - 6.11B^2 - 12.08C^2 \quad (8)$$

$$Y_w = 508.22 - 49.67B + 86.14AB - 155.95A^2 - 111.75B^2 - 247.57C^2 \quad (9)$$

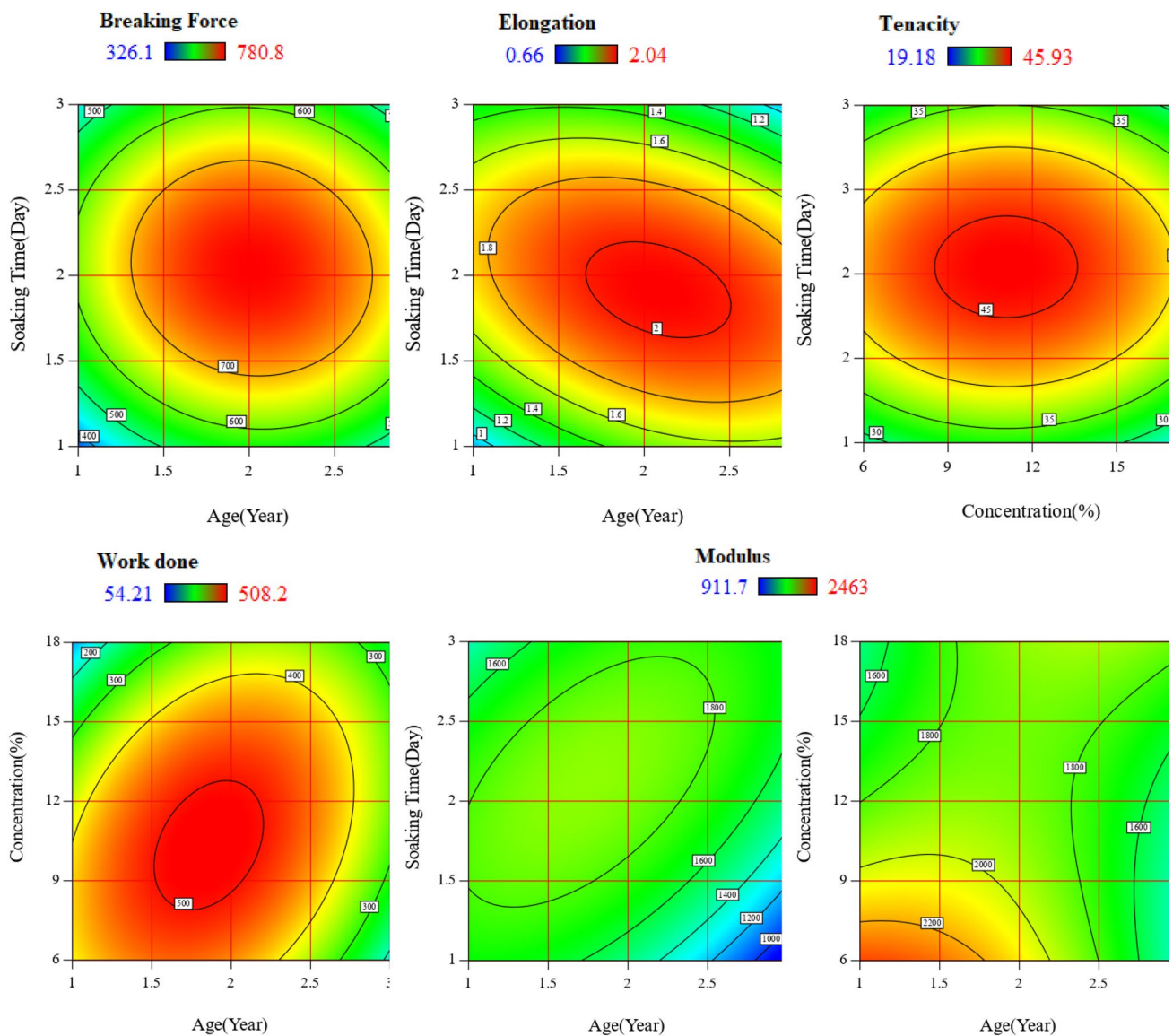


Fig. 11 2D plots of concentration versus plant age, soaking time versus plant age, and concentration versus soaking time on tested mechanical properties

$$Y_m = 1876.18 - 157.1A + 336.49AB + 274.71AC - 236.76A^2 - 249.40C^2 \tag{10}$$

where A, B, and C represent fiber Age (year), NaOH concentration (wt %), and soaking duration (day), respectively.

$Y_b, Y_e, Y_t, Y_w,$ and Y_m are breaking force, Elongation, Tenacity, Work done, and Modulus in terms of significant terms model equation respectively generated by RSM.

The quadratic model was used to fit the response value and detect which independent variables from Eq. 6 to Eq. 10 were affected for each of the responses through Analysis of Variance (ANOVA). Table 10 shows that quadratic coefficients of the soaking duration except modulus

have the highest values among the linear, quadratic, and interaction coefficients of significant independent variables. This shows that the quadratic values of the soaking duration have a significant effect on breaking force ($P \leq 0.0008$), elongation ($P \leq 0.001$), tenacity ($P \leq 0.0008$), and work done ($P \leq 0.0001$). The coefficient of AB has the highest value among the coefficients of the significant independent variables in the equation of modulus, indicating that the interaction of plant age and alkali concentration ($P \leq 0.0009$) has the most positive effect on the model equation.

Table 9 Analysis of variance for response surface quadratic model of alkali-treated *Y.alpina* bamboo fiber

Source	Breaking force		Elongation		Tenacity		Work done		Modulus	
	F-value	p-value	F-value	p-value	F-value	p-value	F-value	p-value	F-value	p-value
Model	7.8	0.0065*	6.93	0.0091*	7.85	0.0064*	21	0.0003*	12.75	0.0014*
A	0.047	0.835	0.0702	0.7987	0.0492	0.8308	1.21	0.3072	13.01	0.0087
B	1.49	0.2612	3.12	0.1208	1.47	0.2643	7.27	0.0308	5.08	0.0589
C	0.45	0.5237	0.9432	0.3638	0.4447	0.5262	0.4908	0.5062	9.23	0.0189
AB	2.43	0.1634	1.56	0.2519	2.41	0.1643	10.93	0.013	29.84	0.0009
AC	0.32	0.5894	5.63	0.0494	0.3217	0.5883	1.17	0.3148	19.89	0.0029
BC	1E-04	0.9921	0.5612	0.4782	0.0005	0.9826	0.4008	0.5468	0.191	0.6756
A ²	19.87	0.0029	5.62	0.0496	19.98	0.0029	37.71	0.0005	15.55	0.0056
B ²	7.98	0.0256	10.67	0.0137	8.07	0.025	19.36	0.0032	4.66	0.0678
C ²	31.33	0.0008	29.64	0.001	31.51	0.0008	95.03	<0.0001	17.25	0.0043
Lack of Fit	1.322	0.397**	0.1347	0.404**	0.4551	0.135**	0.6336	0.091**	0.354	0.1063**

A age(Year) B Concentration (%) C Soaking Time(Day) * Significant **Not Significant

Table 10 Coefficients of responses: Y_b , Y_e , Y_t , Y_w , and Y_m are the breaking force, Elongation, Tenacity, Work done, and Modulus, respectively

Coefficients	Mechanical properties (Responses)				
	Y_b	Y_e	Y_t	Y_w	Y_m
β_0	780.76	2.04	45.93	508.22	1876.18
β_1	5.75	0.0225	0.3463	-20.29	-157.10
β_2	-32.53	-0.15	-1.89	-49.67	-98.15
β_3	17.86	-0.0825	1.04	12.91	132.35
β_{12}	58.61	0.15	3.43	86.14	336.49
β_{13}	-21.28	-0.2850	-1.25	-28.22	274.71
β_{23}	0.3875	0.09	0.05	-16.49	26.89
β_{11}	-163.54	-0.2775	-9.62	-155.95	-236.76
β_{22}	-103.64	-0.3825	-6.11	-111.75	129.56
β_{33}	-205.33	-0.6375	-12.08	-247.57	-249.40
β_{123}	0.000	0.00	0.00	0.00	0.00

7 Optimization and confirmation test

The Design-Expert software's optimization tool was utilized to determine the best parameters for the alkali-treated *Y.alpina* bamboo single fibers. As shown in Table 11, the parameters that affect significantly the responses (breaking force, elongation tenacity, work done, and modulus) on the range between 326.1 and 780.8 cN, 0.665 to 2.04%, 19.18–45.93cNtex⁻¹, 54.21–508.2 cNmm, and 911.7–2463 cNtex⁻¹ was set as predicted values of breaking force, elongation, tenacity, work done, and modulus respectively with the three factors balanced within the ranges that were measured and Box-behnken design (BBD) analysis error (%) values of experimental with predicted values of responses (Table 12). The conditions

to optimize mechanical properties were a plant age of 1.8 years, a NaOH concentration of 10%, and a soaking duration of 2.0 days. The optimal values at these variables of breaking force, elongation, tenacity, work done, and modulus is 777.8cN, 2.0%, 45.7cNtex⁻¹, 516.3cNmm, and 1969cNtex⁻¹, respectively, according to the BBD results.

To validate the model, a confirmation of five experiments was performed. Samples were prepared by using the optimal variables obtained by BBD (1.8 years age, 10%NaOH concentration, and 2 days soaking days), and mean values of the experimental results (breaking force, Elongation, Tenacity, Work done, and Modulus) were, 779.8 cN, 3.0%, 48.01 cNtex⁻¹, 511.22 cNmm, and 2001.12 cNtex⁻¹ respectively. Results demonstrated that the percentage error between the measured and predicted value was well within the value of 5%. This demonstrated that the model's validity was verified and the existence of the optimal point was confirmed by the good agreement between predicted and experimental results.

8 Conclusions

This study was conducted to generate information about the mechanical (Breaking force, Elongation, Tenacity, Work done, and Modulus), thermal and chemical properties of the alkali-treated *Y.alpina* Ethiopia indigenous bamboo species single fiber. The response surface methodology Box–Behnken design was used to evaluate the effect of three independent variables (plant age, soaking duration, and NaOH concentration) on the mechanical properties of *Y.alpina* bamboo single fibers. Using this software optimization and showing linear, quadratic, and interaction effects of independent variables on the mechanical properties was carried out. Optimal

Table 11 Predicted values of responses (breaking force, elongation tenacity, work done, and modulus)

Run	A Year	B %	C Day	Breaking Force cN	Elongation %	Tenacity cNtex ⁻¹	Work done cNmm	Modulus cNtex ⁻¹
1	2	12	2	780.8	2.0	45.9	508.2	1876.2
2	1	6	2	599.0	1.7	35.2	396.6	2360.7
3	2	6	1	486.9	1.3	28.6	169.2	1749.0
4	1	12	1	367.0	0.9	21.6	83.9	1689.5
5	3	12	1	421.1	1.5	24.8	99.7	825.9
6	1	18	2	416.7	1.1	24.5	125.0	1491.5
7	3	18	2	545.4	1.4	32.1	256.7	1850.2
8	3	6	2	493.3	1.4	29.0	183.8	1373.5
9	2	6	3	521.8	1.0	30.6	228.0	1959.9
10	1	12	3	445.3	1.3	26.2	166.1	1404.8
11	2	18	1	421.0	0.9	24.8	102.8	1499.0
12	2	18	3	457.5	0.9	26.9	95.6	1817.4
13	3	12	3	414.2	0.8	24.4	69.1	1640.0

Table 12 Box-behnken design (BBD) analysis standard error (%) values of experimental with predicted values of responses

Run	Breaking force	Elongation	Tenacity	Work done	Modulus
1	0.0	0.0	0.0	0.0	0.0
2	-0.6	0.7	-0.6	0.7	1.7
3	-0.7	-0.2	-0.7	-1.1	-2.6
4	1.4	-0.5	1.4	0.5	0.9
5	1.9	2.4	1.9	2.6	1.4
6	1.2	2.2	1.2	1.4	-1.2
7	0.6	-0.7	0.6	-0.7	-1.7
8	-1.2	-2.1	-1.2	-1.4	1.2
9	2.5	1.7	2.5	1.9	-0.3
10	-1.9	-2.4	-1.9	-2.6	-1.4
11	-2.5	-1.7	-2.5	-1.9	0.3
12	0.7	0.2	0.7	1.1	2.6
13	-1.4	0.5	-1.4	-0.5	-0.9

mechanical properties were attained at optimal points of a plant age 1.8 years, NaOH content of 10% (wt/vol %), and soaking duration of 2.0 days. At these points (age 1.8 years and soaking duration 2.0 days) the thermal, morphological, and functional group identification was explored for the corresponding alkali concentration of 6, 12, and 18%. Increasing the alkali concentration promotes weight reduction by improving the removal of lignin and hemicellulose components. Thermal and mechanical properties, on the other hand, varied up to a point. Except for modulus, mechanical characteristics improved when concentration and age were increased to 12% and 2 years age respectively and then dropped. The 12% NaOH concentration exhibits the best thermal stability properties among the 6–18% NaOH treated *Y.alphina* bamboo fibers. The surface appearance and

microfibril aggregates in the cell wall were altered by alkali treatment.

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Declarations

Conflict of interest The author declare that they have no conflict of interest.

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