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Characterization and Optimization Techniques for the Extraction of Fibers from *Ficus Thonningii*

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ABSTRACT The investigation of natural fibers as viable substitutes for synthetic materials has intensified due to environmental and financial considerations. It has been acknowledged that *Ficus thonningii* is a viable source of natural fibers due to its resistance and capabilities. The current study aimed to extract and characterize *F. thonningii* fibers from *F. thonningii* plant found in Ethiopia. The extraction methods used included water and chemical retting with sodium hydroxide. The extraction was optimized by DOE. Mechanical, physical, and chemical properties of the extracted fibers, such as tensile strength, elongation, fiber length and diameter, fiber fineness, moisture content, moisture regain, fiber's cellulose, hemicellulose, lignin, and ash contents were characterized. Results showed that *F. thonningii* fibers have comparable fiber strength (39.65cN by chemical extraction and 37.83cN by water extraction), elongation (3.02% by water extraction and 2.6% by chemical extraction), fiber length (101.5mm by both methods), moisture content (10.35% by water extraction and 10.78% by chemical extraction), and moisture regain (11.02% by water extraction and 11.98% by chemical extraction) with jute, sisal, and flax. The chemical composition was found to be 52.35% of cellulose, 19.2% of hemicellulose, 17.2% of lignin, and 1.2% of ash for water extraction and 63.57% of cellulose, 16.1% of hemicellulose, 12.1% of lignin, and 0.83% of ash for chemical extraction. This proves that *F. thonningii* plant found in Ethiopia is a source of coarse fibers, thus, can be utilized for technical textiles application.

INDEX TERMS chemical composition, chemical extraction, *Ficus thonningii,* fiber, FTIR, physical properties, water extraction

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I. INTRODUCTION

At present, the world's textile industries are dominated by synthetic materials, such as polyester, rayon, nylon, and acrylic [1]. These synthetic materials are designed with particular qualities that make them appropriate for a range of uses. These fibers are produced by the process of polymerization, in which small molecules (monomers) are chemically bonded together to form long chains (polymers). These synthetic fibers offer various advantages, such as cost, versatility, and performance qualities adapted to specific demands [2]. However, their manufacturing and disposal may have environmental implications, leading to efforts to develop more sustainable alternatives and recycling methods. Whereas, natural fibers offer a host of advantages over man-made fibers, ranging from sustainability and comfort to biocompatibility and longevity. Many researchers have highly prioritized studying natural fibers due to their effectiveness, abundance, low cost, eco-friendliness, biocompatibility, and biodegradable nature [3]. *Ficus thonningii*, sometimes referred to as the forest fig or wild fig, is an important botanical species with varied ecological, cultural, and commercial significance [4].

It is a member of the *Moraceae* family and is indigenous to parts of East Africa, Central Africa, and West Africa. It is about 6-21 m, with a rounded to spreading and dense crown, and is abundantly available. Moreover, it does not need any cultivated land, insecticides, and pesticides, and is a fast-growing species. The tree is mainly distributed in the upland forests of tropical and subtropical Africa, at altitudes between 1,000–2,500 m, and grows best in light, deep, and well-drained soil [5]. The bark on young branches is hairy, with a stipules cap covering the growth tip, however, smooth and gray on older branches and stems, lenticel late, often with aerial roots hanging down from the branches. The whole plant exudes copious, milky latex, often turning pinkish. The species is widely distributed in upland forests, open grasslands, riverine and rocky areas, and sometimes in Savannah. It grows naturally in the Democratic Republic of Congo and Tanzania in the north to the Eastern Cape in South Africa. Trees are relatively drought resistant [6].

The bark of this tree is used in local medicine and is also used in treating cold, sore throat, dysentery, wounds, constipation, and nosebleeds and to stimulate lactation. Latex is used for wound fever, while an infusion of root and fiber is taken orally to help prevent abortion. Powdered root is taken in

porridge to stop nosebleeds and the milky latex is dropped into the eye to treat cataracts [7]. As a keystone species that maintains ecosystem stability and biodiversity, the *F. thonningii* plant is essential to its ecology [8]. Beyond its cultural and ecological value, the *F. thonningii* plant has economic potential which offers chances for resource utilization and revenue creation. Many researchers have extracted fibers from different plants. Moreover, there are trends to use and utilize the *F. thonningii* plant for different applications, such as for medicine, fodder, fire wood, and others in different literature reviews, and it is available in abundance, not seasonal, and is a fast-growing species [9], [10]. However, very few studies have been conducted on the extraction and characterization of fibers derived from *F. thonningii* plant and research on these fibers is yet dispersed. Asmare et,al. (0219) [11] examined the application of *F. thonningii* as animal feed in Ethiopia, however, did not investigate the fiber content of the plant. Studies on plant extracts have been conducted using phytochemical screening as well as qualitative methods; nevertheless, their primary focus remained on the presence or lack of secondary chemicals rather than fibers. By developing and optimizing methods to extract fibers from *F. thonningii* plant and to assess the mechanical, chemical, and physical properties of those fibers, the proposed study sought to close these gaps. The findings would be helpful to increase our understanding of the various applications for *F. thonningii* fibers in various fields.

FIGURE 1. *Ficus thonningii* plant

The current study focused on the extraction and characterization of natural fiber from the *F. thonningii* plant. It primarily focused on *F. thonningii* fiber extraction by using chemical (alkali) and water retting methods. After the extraction, characterizations of chemical and physical properties were analyzed.

II. MATERIALS AND METHOD

A. MATERIALS

Well-ripened *F. thonningii* plants were collected around Zegie, Bahir Dar, Ethiopia. All the chemicals used in the extraction process including sodium hydroxide (NaOH), 72% sulfuric acid (H2SO4), acetone, 95% ethanol, benzene, 80% acetic acid, nitric acid, and pure water were reagents from the Ethiopian Institute of Textile and Fashion Technology Laboratory.

B. METHODOLOGY

1) FIBER EXTRACTION

The *F. thonningii* plant was collected and cut. Afterwards, the bark of the plant which contains the fibrous portion was separated from the stem of the plant manually. After separating the bark from stem, it was dried for extraction. The dried bark of the plant which contains the fibrous portion was taken and fiber was extracted by water retting and chemical (alkali) retting extraction methods.

WATER RETTING EXTRACTION

Water was used to extract fiber from the bark of the *F. thonningii* plant. The prepared sample was immersed in a container of water until it covered the bark in its entirety at room temperature in an open system with different extraction times (7, 9, 14, 17, and 21 days). The barks were straightened on a flat plane and the bark skin and other substances covering the fibers were scoured until the fibers began to separate to be taken out. The fibers were then cleaned with water to remove the bad odor from the retting process. Afterwards, the fibers were dried up in shade to avoid direct sun exposure. The retted *F. thonningii* fiber can be seen in (Figure 2(a).

CHEMICAL RETTING EXTRACTION

In this extraction method (Figure 3(b)), sodium hydroxide (NaOH) was used with different MLR, temperature, and time of extraction. The peeled bark was dipped in sodium hydroxide solution ((3-10g/l)) with MLR of 10,

15, 20, 25, and 30 at 50, 60, 70, and 80 ℃ temperatures for 30, 45, 50, 60, 75, and 90 minutes. The extracted components were then drained and the fibers formed were thoroughly washed first in warm and later in cold water, neutralized in dilute acetic acid solution to remove any remaining alkali and were air dried afterwards. Using the design of an experiment, the most optimum conditions for fiber extraction were developed based on the yield and strength of fibers obtained. The chemical extraction of *F. thonningii* fiber was done by controlling four factors (temperature, MLR, concentration, and time of extraction) and evaluating the fibers with two responsive variables (fiber yield and strength). The extraction was done in 30 runs with a different combination of these four factors.

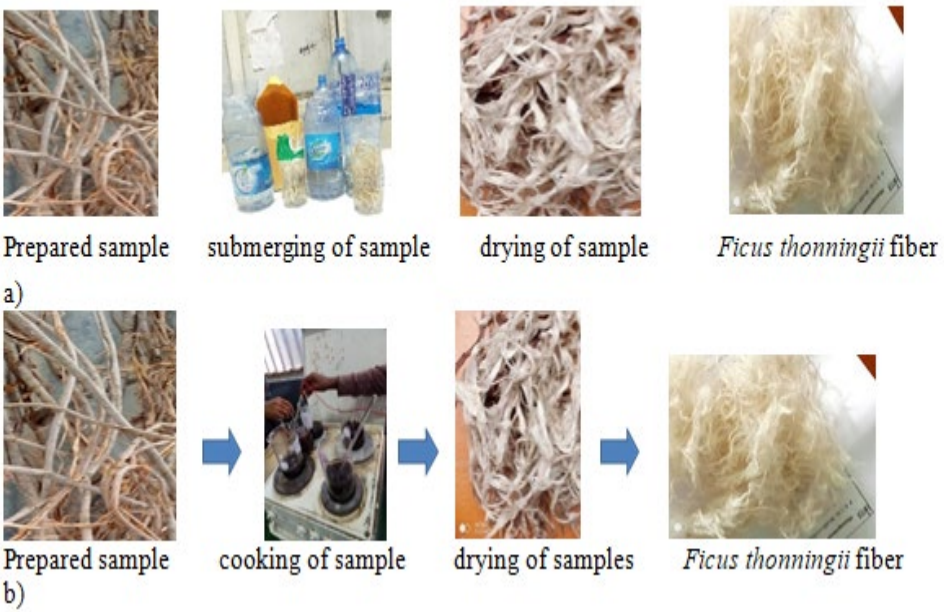

FIGURE 2. a) Water retting extraction b) *Chemical extraction of F. thonningii fiber*

2) DETERMINING THE RESPONSE VARIABLES

FIBER YIELD

The yield of extracted fiber (R %) was measured by the percentage of ratio between the final mass of the extracted fiber after the extraction process and that of the mass of plant before the extraction process, which is given in Equation 1 [12], where *Wi* is the weight of the plant before extraction and

Wf is the weight of fiber after the extraction process.

$$\text{Percentage yield(R \%)} = \frac{Wf}{Wi} * 100\% \quad (1)$$

TENSILE STRENGTH

The tensile properties of the *F. thonningii* fiber were determined using universal tensile testing machine at a crosshead speed of 10 mm/min, maintaining the gauge length of 80mm. The strength of each sample was tested by taking a specimen from each sample and the average value was calculated.

3) CHARACTERIZATION OF F*ICUS THONNINGII* FIBER

DETERMINATION OF FIBER DIAMETER, LENGTH, AND LINEAR DENSITY

An image analyzer was used to measure the diameter of a single fiber using an optical microscope. For both extractions, samples were taken and in each sample, some random points were taken along the fiber length. Finally, the average diameter of the fiber was calculated.

Fiber lengths are measured manually with a basic ruler. To measure fiber length, 10 samples were obtained from the extracted fibers. Each type and the average were utilized to compare the fibers extracted using different procedures. Finally, the average length of fiber was calculated and taken as the length of *F. thonningii* fiber. The fiber linear density was determined by the single-fiber weighing method according to the ASTM D-1577:07 test standard. The length of a single fiber was measured, and the fiber was weighted and finally, the linear density was calculated as shown in Equation 2 [13]. Ten (10) samples were taken for each extraction and the average was taken as the linear density of the *F. thonningii* fiber.

$$\text{Tex} = \frac{m(g)}{L(m)} * 1000 \quad (2)$$

DETERMINATION MOISTURE CONTENT AND MOISTURE REGAIN

There are two ways to describe the amount of moisture in the textile fibers. Moisture regain is defined as the weight of water in a material expressed as a percentage of the oven dry weight. Moisture content is the weight of water in a material expressed as a percentage of the total weight (Das, 2009 #56). The moisture regain and moisture content of the F. thonningii fiber were measured by the Etadry machine according to ASTM 2654. The mass of

sample fibers was first measured and dried in an oven at 105±3℃ until a constant mass was reached. The moisture content and moisture regain of the F. thonningii fiber were calculated by Equation 3 and Equation 4, respectively [13].

$$\text{Moisture regain (\%)} = \frac{Original\ weight\ of\ fiber - oven\ dry\ weight\ of\ fiber}{Oven\ dry\ weight\ of\ fiber} * 100 \quad (3)$$

$$\text{Moisture content (\%)} = \frac{Original\ weight\ of\ fiber - oven\ dry\ weight\ of\ fiber}{Oven\ dry\ weight\ of\ fiber} * 100 \quad (4)$$

DETERMINATION OF CHEMICAL COMPOSITION

Determination of cellulose content: Fiber samples were treated with 40 ml of 80% acetic acid and 2 ml of concentrated nitric acid and were refluxed for 30 min. The samples were then centrifuged at 15,000 rpm for 5 min in 95% hot ethanol and filtered by suction. Washing was done in hot benzene followed by 95% ethanol and finally with petroleum ether. The sample was dried for 1 hour in a 105℃ oven and weighed in a closed container. Afterwards, the sample was placed in a furnace at 500℃ for 3 hours and the resulting ash was weighed. The cellulose content in the percentage was evaluated using Equation 5 [14], where, W1 is the weight of crucible + sample after washing, W2 is the weight of crucible + sample after drying, and Ws is the weight of sample.

$$\%\ \text{Cellulose} = \frac{W2 - W1}{Ws} * 100 \quad (5)$$

DETERMINATION OF HEMICELLULOSE CONTENT

A sample weighing 1gm of extracted fibers was placed into a conical flask in which 5% KOH was added, and then allowed for 2 hours. The mixtures were filtered, washed with their corresponding KOH solution, and the filtrate was collected. Ethanol was added to precipitate the hemicellulose, which was isolated by centrifuging for 15 minutes. The isolated hemicellulose was washed with ethanol and ether and was transferred into a crucible. This was followed by oven drying for 1 hour at 105°C, thereafter, transferred into a desiccator and allowed to cool for 30 min and weighed. The fiber sample was also placed in a furnace maintained at 500°C for 3 hours, after which it was cooled inside a desiccator and weighed. The

weight of precipitated hemicellulose (W1) was measured. The percentage hemicellulose composition of the fiber sample was calculated using Equation 6 [15], where, W1 is the dried weight of precipitated hemicellulose and W2 is the dried weight of the fiber sample.

$$\% \text{ Hemicellulose} = \frac{w1}{W2} * 100 \quad (6)$$

DETERMINATION OF LIGNIN CONTENT

The lignin content was determined by gravimetric method according to the ASTM D1106 standard. Roughly, 2gm of fiber sample was placed inside a beaker and 72% of H2SO4 was added and allowed for 2 hours. Eight percent (8%) H2SO4 was added later and the solution was refluxed for 3 hours. The residue was filtered and washed several times with hot water. The sample was oven dried at 105°C for 1 hour and weighed (w2). The sample was then placed in a furnace at 500°C for 3 hours, after which it was cooled and weighed in desiccators (w) [15]. The percentage of lignin was calculated using equation 7, where, W1 is the weight of the ash sample, W2 and Ws are the weights of the oven and the initial weight of the dried sample, respectively.

$$\% \, Lignin = \frac{W2 - W1}{Ws} * 100 \quad (7)$$

DETERMINATION OF ASH CONTENT

The ash content of the extracted *F. thonningii* fiber was evaluated based on the ASTM D1106 standard. Firstly, the weight of a clean dry crucible was determined. About 2 gm of sample was placed and weighed to determine the accurate weight of the sample taken. The weighed crucible was carefully placed over the electric burner. The crucible was partially opened. The sample got blackened with the initial expulsion of smoke. The crucible furnace was placed, heated up to 600ºC, and kept for 2 hours. At this temperature, all the organic matter was burnt and minerals were left behind. The crucible was carefully removed from the furnace and was allowed to be cooled in desiccators at room temperature and weighed again. Finally, the ash content of the fiber was calculated by Equation 8 [16], where; X, Y, and X are the weights of empty crucible, crucible + sample, and crucible + ash, respectively.

$$\text{Ash content } (\%) = \frac{Z - X}{Y - X} * 100 \quad (8)$$

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

FTIR was used to determine the presence of functional groups of the *F. thonningii* fiber. The IR spectra of samples was recorded using the Perkin Elmer FTIR instrument in the frequency range 4000-500 cm-1 with a scanning speed of 2mm/sec and recorded in the transmittance mode as a function of wave number.

C. DATA ANALYSIS

The response surface methodology (RSM) was used to analyze the experiment. The research design was used to study the relationship between variables (NaOH concentration, time of extraction, MLR, and temperature) and responses (fiber yields and strength of fibers). Analysis of variance (ANOVA) was used to test whether the variables (NaOH concentration, time of extraction, MLR, and temperature) were significantly affecting the response values (fiber yields and strength).

III. RESULTS AND DISCUSSION

A. CHEMICAL EXTRACTION METHOD ANALYSIS

In the chemical extraction method, the factors, such as NaOH concentration, time of extraction, temperature, and material liquor ratio were considered during the extraction process. Each factor plays a significant role in the extraction of fiber. Tensile strength and the yield of *F. thonningii* fibers were determined and analyzed based on each factor. The effect of different extraction factors and the results obtained from the chemical method are reported in Table I.

TABLE I
EFFECT OF FACTORS AND RESPONSE VALUE

Run	Factors				Responses	
	A:NaOH concentration gpl	B:time Min	C:MLR	Effect of factors and response value ^{0}C	Fiber yield %	Strength cN/tex
1	4.75	45	25	50	58.71	39.6
2	6.5	60	20	60	50.36	34.5
3	8.25	45	25	70	50.5	31
4	8.25	75	15	50	46.14	31.9
5	6.5	60	20	40	39.61	35.55
6	3	60	20	60	49.1	39

Run	Factors				Responses	
	A:NaOH concentration gpl	B:time Min	C:MLR	Effect of factors and response value ^{0}C	Fiber yield %	Strength cN/tex
7	6.5	60	20	50	49	36.1
8	8.25	45	25	70	54	33.33
9	4.75	45	25	60	55.03	37.6
10	10	60	20	60	42	26.5
11	6.5	60	20	70	51.65	33.53
12	4.75	75	15	50	40.18	36.83
13	8.25	75	25	50	45.11	31.6
14	4.75	75	25	60	47	36.8
15	6.5	60	20	50	48	33.9
16	8.25	45	15	70	53	33.4
17	8.25	45	15	80	38	32.8
18	6.5	60	20	80	34.97	33
19	6.5	60	20	60	49	35.9
20	6.5	60	30	60	59.2	41.5
21	4.75	45	15	50	56	40
22	6.5	60	10	60	46.46	42.5
23	4.75	75	15	50	41	36
24	4.75	75	25	70	50.69	35.4
25	6.5	30	20	60	60.65	36.3
26	4.75	45	15	70	48	38.5
27	8.25	75	25	70	54.32	30.4
28	6.5	60	20	60	50.36	35.7
29	8.25	75	15	70	45	29.6
30	6.5	90	20	60	55	30.7

Table II presents the ANOVA analysis of chemical extraction for fiber yield. The model F-value of 22.06 and P-values less than 0.0500 indicate the significance of model. There was only a 0.01% chance that an F-value this large could occur due to noise. In this case, A (NaOH concentration), B (time), C (MLR), D (temperature), AB (product of NaOH concentration and time), BD (product of time and temperature), CD (product of MLR and temperature), A^2 (multiple effects of NaOH concentration), B^2 (multiple effects of time), C^2 (multiple effects of MLR), and D^2 (multiple effects of temperature) were significant model terms, affecting the fiber yield.

TABLE II
ANOVA ANALYSIS OF CHEMICAL EXTRACTION FOR FIBER YIELD

Source	Sum of Squares	*df*	Mean Square	*F*-value	*p*-value	
Model	1133.91	14	80.99	22.06	0.0001	significant
A-conc.	25.50	1	25.50	6.95	0.0187	
B-time	126.50	1	126.50	34.45	0.0001	
C-MLR	225.22	1	225.22	61.34	0.0001	
D-te	33.89	1	33.89	9.23	0.0083	
AB	72.00	1	72.00	19.61	0.0005	
AC	1.24	1	1.24	0.3386	0.5693	
AD	0.1640	1	0.1640	0.0447	0.8355	
BC	0.1521	1	0.1521	0.0414	0.8415	
BD	105.68	1	105.68	28.78	0.0001	
CD	58.83	1	58.83	16.02	0.0012	
A^2	24.96	1	24.96	6.80	0.0198	
B^2	122.67	1	122.67	33.41	0.0001	
C^2	20.57	1	20.57	5.60	0.0318	
D^2	249.99	1	249.99	68.08	0.0001	
Residual	55.08	15	3.67			
Lack of Fit	46.54	10	4.65	2.73	0.1400	not significant
Pure Error	8.54	5	1.71			
Cor Total	1188.99	29				

The relationship between the actual ranging from the minimum 34.9% to the maximum 60.65% and the predicted yield percentage was close to straight line such that A (NaOH concentration), B (time), C (MLR), D (temperature), AB (product o-f NaOH concentration and time), BD (product of time and temperature), CD (product of MLR and temperature), A^2 (multiple effect of NaOH concentration), B^2 (multiple effect of time), C^2 (multiple effect of MLR), and D^2 (multiple effect of temperature) were

significant effects for this model and assumption of the analysis was satisfied. The relationship between the actual and the predicted fiber yield of the experiment is shown in Figure 3.

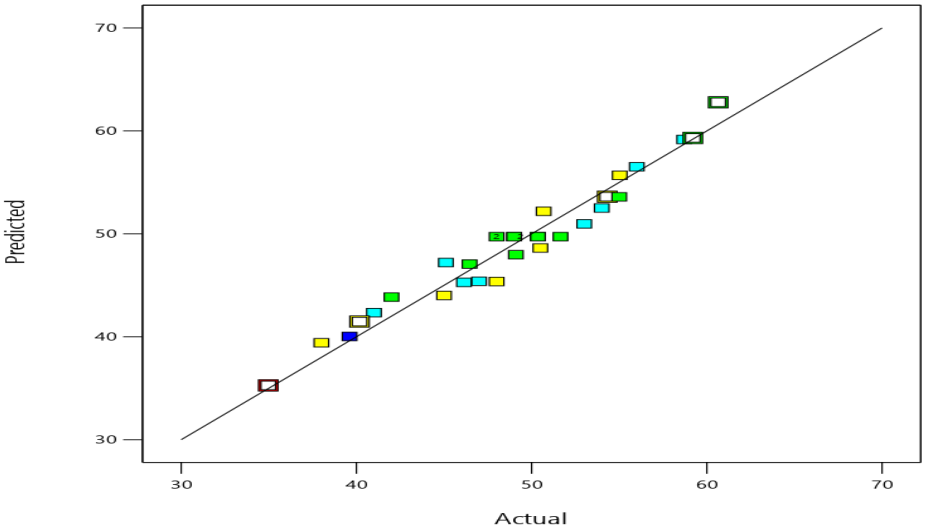

FIGURE 3. Actual versus predicted fiber yield

From the ANOVA Table 2, it is clear that the fiber yield was affected by all factors. As the concentration, time, and temperature increase from 3-10 gpl, 30-90 min, 40-80°C respectively, the fiber yield decreases from 60.65% to 34.9%. This is because, as the concentration, time, and temperature increase, the activity of the NaOH on the surface of the sample increases and damages the fibers. Finally, the fiber yield is reduced due to the decrement of the final weight of the sample after extraction. As the amount of NaOH in the bath increased, the yield went down and the gum decomposition increased. This is due to the solubility of lignin from the cellulosic fiber [17], [18]. When the temperature and time of extraction increase, the yield of the fiber decreases, accelerating the lignin removal process. This is because the ester bonds between lignin and hemicellulose hydrolyze more quickly at greater NaOH concentrations and under more harsh extraction conditions (higher temperature and longer time), which loosens the lignocellulosic matrix and makes lignin removal easier [19]. The fiber yield is directly affected by the MLR value (when MLR increases, the fiber yield increases).

As seen from the combination effect of each factor on fiber yield according to Figure 4 (a), when the concentration and temperature increase, the fiber yield is decreased and, as shown in Figure 4 (b), the combination of MLR and time of extraction and the fiber yield have an inverse relationship.

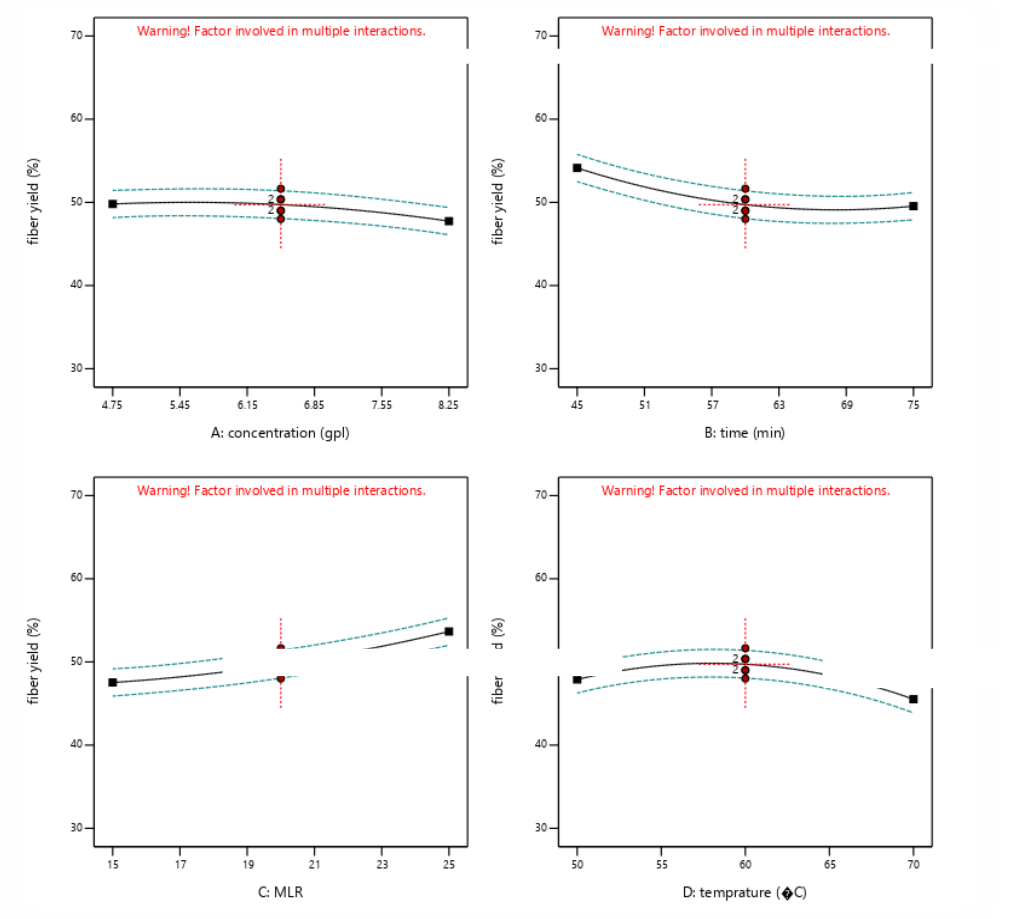

FIGURE 4. Effect of Each Factor on Yield of *Ficus thonningii* Fiber

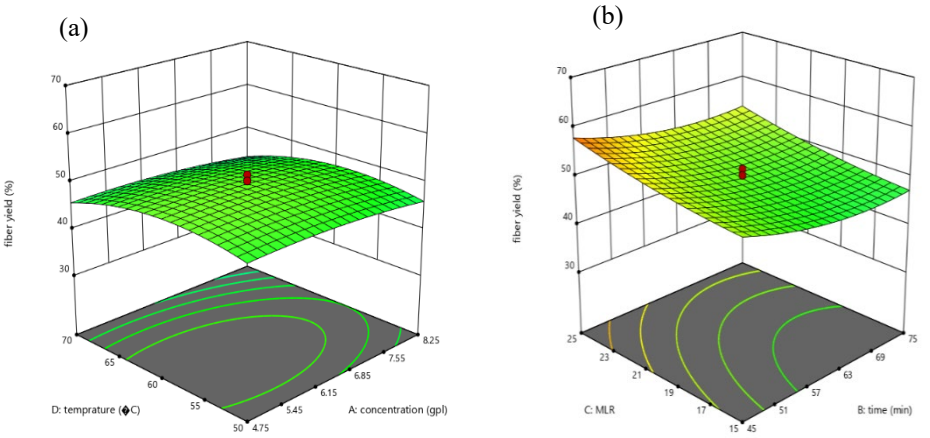

FIGURE 5. Effect of combination of factors on fiber yield

Table 3 presents the ANOVA analysis for fiber strength using chemical extraction method. The model F-value of 27.98 implies that the model was significant, as shown in Table 3. There was only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate the significance of model terms. In this case, A (concentration), B (time), D (temperature), A^2 (multiple effect of concentration), B^2 (multiple effect of time), and C^2 (multiple effect of MLR) were significant model terms and other factors. P-Values greater than 0.05 indicate that the model terms were not significant. The lack of a Fit F-value of 0.67 implies the lack of Fit was not significant relative to the pure error. There is a 72.39% chance that a lack of Fit F-value this large could occur due to noise. Non-significant lack of fit was good.

TABLE III

ANOVA ANALYSIS OF CHEMICAL EXTRACTION FOR FIBER STRENGTH (CN/TEX)

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	373.64	14	26.69	27.98	0.0001
A-concentration	163.57	1	163.57	171.50	0.0001
B-time	22.30	1	22.30	23.38	0.0002
C-MLR	1.85	1	1.85	1.94	0.1837
D-temperature	12.18	1	12.18	12.77	0.0028
AB	0.8281	1	0.8281	0.8682	0.3662

Source	Sum of Squares	df	Mean Square	F-value	p-value
AC	0.0196	1	0.0196	0.0205	0.8879
AD	0.3481	1	0.3481	0.3650	0.5548
BC	0.5776	1	0.5776	0.6056	0.4486
BD	0.3481	1	0.3481	0.3650	0.5548
CD	0.7056	1	0.7056	0.7398	0.4033
A^2	13.71	1	13.71	14.37	0.0018
B^2	7.40	1	7.40	7.76	0.0139
C^2	70.71	1	70.71	74.14	0.0001
D^2	2.91	1	2.91	3.05	0.1012
Residual	14.31	15	0.9538		
Lack of Fit	8.20	10	0.8199	0.6711	0.7238
Pure Error	6.11	5	1.22		
Total	387.95	29			

Figure 6 shows the relationship between the actual and predicted fiber strength of the experiment extracted by chemical method. The relationship between the actual a ranging from the minimum 26.5cN/tex to the maximum 42.5cN/tex and the predicted strength of the fiber was close to a straight line, such that A (NaOH concentration), B (time), D (temperature), A^2 (multiple effect of NaOH concentration), B^2 (multiple effect of time), C^2 (multiple effect of MLR) were significant effect for this model and assumption of the analysis was satisfied.

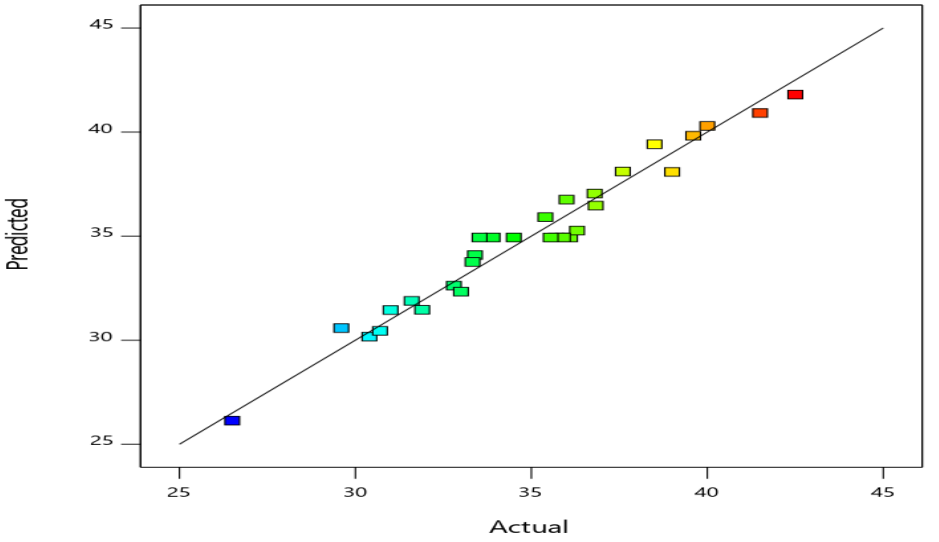

FIGURE 6. Actual versus predicted strength

The tensile strength of *F. thonningii* fiber was significantly affected by sodium hydroxide concentration, time, and temperature, and MLR did not have any significant effect on the strength of the fiber, since the P-value was greater than 0.05 according to the ANOVA Table 3 and Figure 7. This is because, as the concentration, time, and temperature increase, the activity of the NaOH on the surface of the sample increases and damages the fibers. Finally, the strength of the fiber is reduced.

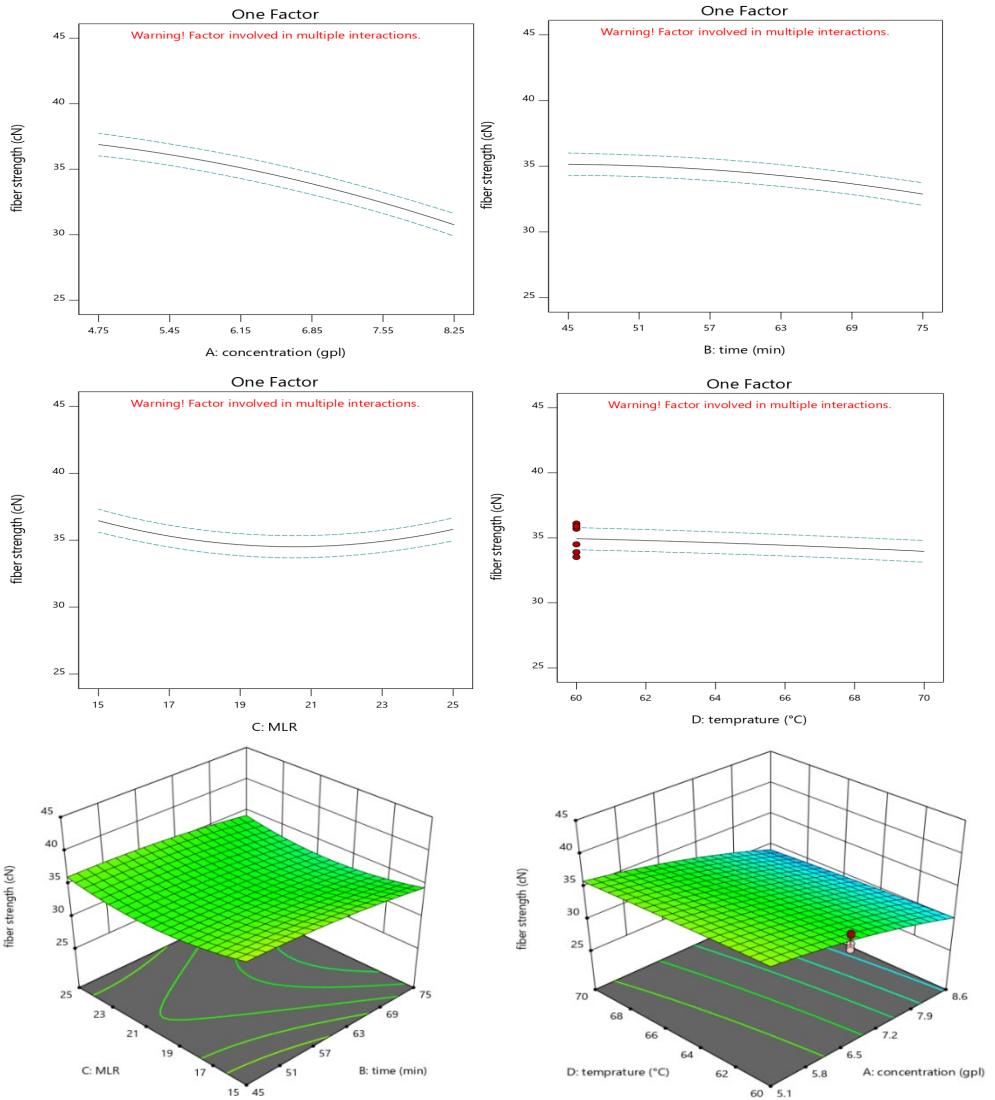

FIGURE 7. Effect of combination of factors on fiber strength

The combinatory effect of each factor on the composite strength of the fibers is shown in Figure 7 (b). The figure depicts that when the concentration and temperature increase, the strength of the fiber is decreased and, as shown in Figure 7 (a), the combinatory effect of MLR, time of extraction, and fiber strength have an inverse relationship. Therefore, based on the above information, following optimized *F. thonningii* fiber extraction parameters were generated in the chemical extraction process.

TABLE IV

OPTIMIZED CONDITIONS FOR CHEMICAL EXTRACTION OF *FICUS THONNINGII* FIBER

No.	A	B	C	D	Fiber yield (%)	fiber strength (cN/tex)	Desirability	
1	4.750	45.000	25.000	55.256	60.593	39.65	0.905	Selected
2	4.750	45.000	25.000	55.573	60.626	39.6	0.905	

The model equations that are used to predict the yield and tensile strength of fibers are presented in equations 9 and 10, where, A is concentration, B is time, C is MLR, and D is temperature. In these equations, the negative coefficient of variables indicates that there is an inverse relationship with the response. Whereas, the positive sign of the coefficient of the variable indicates that there is a direct relationship with the response value.

$$\text{Fiber yield}=49.73-1.03A-2.30B+3.06C-1.19D+2.12AB-0.2788AC-0.1013AD+0.0975BC+2.57BD+1.92CD-0.9540A^2+2.11B^2+0.8660C^2-3.02D^2 \quad (9)$$

$$\text{Fiber strength}=34.5-3.06A-1.13B-0.3258C-0.4878D+0.2275AB+0.0350AC-0.0738AD+0.1900BC+0.0737BD-0.10550CD-0.7069A^2-0.5194B^2+1.61C^2-0.0814D^2 \quad (10)$$

B. WATER EXTRACTION METHOD ANALYSIS

In this extraction method, the dried barks of the *F. thonningii* plant were immersed in a container of water with different extraction timings (7, 9, 14, 17, and 21 days) to extract the *F. thonningii* fiber from the bark of *F. thonningii* plant. The tensile strength and yield of the *F. thonningii* fibers were determined and analyzed based on the variation of extraction time. The effect of extraction of time on yield and strength of fiber are reported

in Table V.

TABLE V

EFFECT OF EXTRACTION OF TIME AND YIELD AND STRENGTH OF FIBER

No.	Time(day)	Yield (%)	Strength (cN/tex)
1	7	63.53	37.83
2	9	58.98	37.43
3	14	53.35	36.78
4	17	50.56	36.36
5	21	39.67	35.61

As indicated in Table 5, the time of extraction and yield and strength values have indirect relationships. It means that, when the time of extraction increases, the value of fiber yield and strength decreases due to the increment of bacteria's activity as the time increases and the fiber part of the sample is damaged by the bacteria [20]. Finally, the yield and strength of the fiber is decreased. According to Table 5, the value of yield and strength of *F. thonningii* fiber were maximum during the extraction time of 7 days.

C. LENGTH, DIAMETER, AND FINAL DENSITY OF THE *F. THONNINGII* FIBER ANALYSIS

The properties of *F. thonningii* fiber are summarized in Table 6. The diameter of a single fiber is a key parameter for textile fibers' characterization. This is because it determines different properties of fibers. The average value of diameter was obtained to be 103µm for chemical retting and 107 µm for water retting method (Figure 8). These results show that the alkaline extraction removes more non-fibrous parts than water retting. Water extraction gives greater diameter due to the presence of non-cellulosic components on the surface of the fibers. The average length of the fiber is 101.5mm for both methods. The length remained the same for both water-retted and chemical-retted methods.

FIGURE 8. Microscopic View of ficus thonningii Fiber at 100x

Magnification

Fineness determines the number of fibers present in the cross-section of a given thickness. Fiber fineness affects the materials' surface area, porosity, and strength, indirectly determining the performance and applications of the fiber. The mean linear density of *F. thonningii* fiber was found to be 2.8 Tex for water retting and 1.92 for chemical extraction. There is a small difference between chemical and water extracted. The main reason is that, in chemical extraction, the fiber bundles have the probability of separating into a single fiber and their fiber linear density is less than water retting. This constancy in fiber length indicates that the length of the fibers is not greatly impacted by the retting process, and the separation of fiber bundles and removal of non-cellulosic components during chemical extraction are the main causes of the observed difference in linear density [21].

D. MOISTURE CONTENT AND MOISTURE REGAIN OF THE FICUS THONNINGII FIBER ANALYSIS

Most natural fibers tend to absorb moisture (water vapor) when in contact with the atmosphere. The amount of water absorbed by textile fiber depends on the chemical and physical structure plus properties of the fibers, as well as the temperature and humidity of the surroundings. Table 6 shows that the moisture content and moisture regain of chemicals extracted (11.98% and 10.76%, respectively) were greater than water extracted (11.02% and 10.35% respectively). This is due to surface treatment of the fiber during extraction and more hydrogen bond formation. Generally, the *F. thonningii* fibers have good moisture absorption in both chemical and water extraction.

E. CHEMICAL COMPOSITION OF THE FICUS THONNINGII FIBER ANALYSIS

The chemical composition (cellulose, hemicellulose, lignin, and ash content) of *F. thonningii* fiber was measured, and the results have been presented in Table 6. The cellulose content was higher (63.57%). The hemicellulose content of water-retted fiber (19.20%) was higher than chemically-retted fiber (16.10%). The higher hemicellulose content was given to the fiber's good contribution to biodegradation, moisture absorption, and thermal decay of the fiber. The lignin content of the fiber was higher in water which (17.20%) was better than in the chemical-rated method (12.10%). This is due to lignin being affected by alkali. The higher range of lignin helps to retain water within the fiber, which safeguards the

fiber from biological attacks. The high amount of lignin makes the fiber dark. The ash content of water-retted fiber (1.20%) was also higher than chemical-retted fiber (0.83%). In general, the F. thonningii fiber has a high amount of cellulose that can be used for biopolymers, such as for agro-polymers. Additionally, the low ash content value of this fiber and moderate value of lignin content indicate that the fiber can be used for producing pulp and paper [22].

TABLE VI

SUMMARIZED *FICUS THONNINGII* FIBER PROPERTIES

Fiber property	Value (mean), Alkaline extracted	Value (mean), Water extracted
Strength (cN/tex)	42.5	37.00
Elongation (%)	2.6	3.00
Length(mm)	101.5	101.50
Diameter (µm)	103	107.00
Linear density (tex)	1.95	2.94
Moisture content (%)	10.78	10.07
Moisture regain (%)	11.66	11.02
Cellulose content (%)	63.57	52.35
Hemicellulose content (%)	16.10	19.20
Lignin content (%)	12.10	17.20
Ash content (%)	0.83	1.20

Table VII summarizes different fiber properties compared with *F. thonningii* fiber. As compared to other known fibers, *F. thonningii* fiber has comparable properties in terms of strength, cellulose content, length, and diameter to flax, sisal, and hemp fibers, however, better than jute fiber. *F. thonningii* fiber has the highest extensibility property.

TABLE VII

COMPARISON OF *FICUS THONNINGII* FIBER WITH OTHER FIBERS

Fiber property	Fiber type				*Ficus thonningii*	
	Jute	Flax	Sisal	Hemp	Chemical	Water
Strength(cN/Tex)	27.41	42-59	40-49	41 - 74	39.53	37.832
Elongation (%)	1.51	1.50	1.50	1.3–4.7	2.60	3.00
Cellulose (wt%)	61.00	71.00		74.41	63.57	52.35
Hemicellulose(wt%)	20.40	18.60		7.90	16.10	19.20

Lignin (wt %)	13.00	2.20	3.70	12.10	17.20
Length(mm)	150–360	20–140	100–300	101.50	101.50
Diameter(µm)	30–140	40–620	16–50	103	107

F. FTIR OF THE FICUS THONNINGII FIBER ANALYSIS

FTIR is utilized to characterize lignocellulosic fiber to identify chemical compounds in a wide range of capacities, by considering the infrared features and transmittance bands of their constituents. The FTIR spectrum of *F. thonningii* fiber is presented in Figure 9 and the detailed has been shown in Table 8. The functional groups of the spectrum ranged from 4000 to 500 cm.

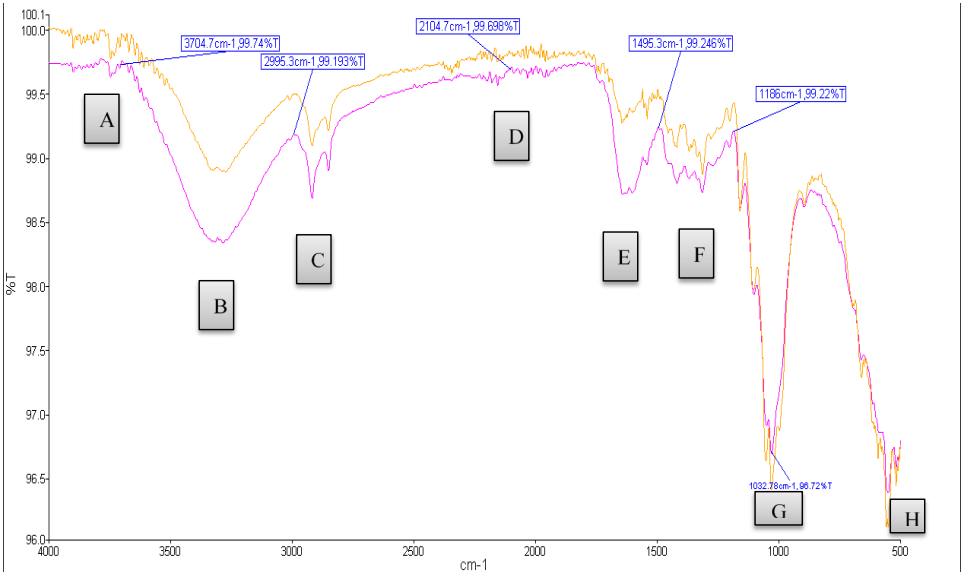

FIGURE 9. FTIR Spectrum of *Ficus thonningii* fiber in the frequency 500-4000cm^{-}

TABLE VIII

THE FUNCTIONAL GROUP ANALYSIS RESULT WITH FTIR

Peaks (cm^{-1})	Functional group	Composition	Reference
A (3854)	Strong O-H stretching	Cellulose and its structure	(Anderson, 1938 #1)
B (3300)	Vibration of methyl and methylene groups, C-H stretching vibration	Cellulose and hemi cellulose molecules	(Atalie, 2018 #66)

Peaks (cm^{-1})	Functional group	Composition	Reference
C (3000)	CH_2 symmetrical analysis	Wax	
D(2000)1495.3	CH_2 wagging and deformation and CH_3 bending	Lignin	
E (1700)	C-O-C groups asymmetric bridge stretching.	Cellulose and hemi cellulose molecules	(Bacci, 2011 #6)
F(1000)	C-O aromatic ring and skeletal vibration	Cellulose	
G (523)	Out of plane bonding of OH		

V. CONCLUSION

The current study examined the mechanical and physical properties, chemical composition, and functional groups of *F. thonningii* fiber. The new natural lignocellulosic fibers were successfully extracted from a *F. thonningii* plant using water and a chemical-retting process. The optimized conditions for the extraction of *F. thonningii* fiber were 4.75gpl of NaOH, 45 min of extraction time, 25 MLR, and 55.250c in chemical extraction and 7 days of extraction time in water extraction methods. The tensile strength and elongation were found to be 39.65 cN/tex and 2.6% by chemical extraction and 37.832 cN/tex and 3.02% by water extraction method, respectively. Moreover, it also had average moisture content and moisture regain of 10.35% and 11.02% for water extraction and 10.76% and 11.98% for chemical extraction, respectively. This fiber showed the linear density of 2.8 Tex for water extraction and 1.92 for chemical extraction.

The chemical composition analysis of *F. thonningii* fiber was found to be 52.35% of cellulose, 19.2% of hemicellulose, 17.2% of lignin, and 1.2% of ash for water extraction and 63.57% of cellulose, 16.1% of hemicellulose, 12.1% of lignin, and 0.83% of ash for chemical extraction. The FTIR curves of both water and chemical extraction showed the presence of functional groups of cellulose, hemicellulose, and lignin. The peaks from 3704.7 to 523cm-1 showed the total spectrum in which all functional groups occurred.

Generally, the *F. thonningii* plant is relatively easy to grow, available everywhere, and is easy to cultivate. Fiber from this plant has comparable

chemical composition with other natural fibers, such as hemp, jute, sisal, and flax. It may be considered as an alternative source for natural cellulosic fibers and, the fiber can be used to make products, such as packaging material (bag), carpet backing, rope, paper and pulp composite, carpet manufacturing, and biopolymer synthesis applications.

A. FUTURE WORK

The extraction methods used to conduct this research were done by water and alkaline extraction at which other extraction methods may be used further. The extracted fiber degree of crystallinity, morphological structure, and thermal stability can be further characterized.

CONFLICT OF INTEREST

The authors of the manuscript have no financial or non-financial conflict of interest in the subject matter or materials discussed in this manuscript.

DATA AVALIABILITY STATEMENT

The data associated with this study will be provided by the corresponding author upon request.

FUNDING DETAILS

No funding has been received for this research.

REFERENCES

[1] R. Prasad and E. Aranda, Eds. *Approaches in Bioremediation: The New Era of Environmental Microbiology and Nanobiotechnology*. Springer, 2018.

[2] A. M. Manich, P. N. Marino, M. D. de Castellar, M. Saldivia, and R. M. Sauri, "Viscoelastic modeling of natural and synthetic," *J. Appl. Polym. Sci.*, vol. 76, pp. 2062–2067, June 2000, doi: https://doi.org/10.1002/(SICI)1097-4628(20000628)76:14%3C2062::AID-APP9%3E3.0.CO;2-T.

[3] M. Prithiviraj, R. Muralikannan, P. Senthamaraikannan, and S. S. Saravanakumar, "Characterization of new natural cellulosic fiber from the Perotis indica plant," *Int. J. Polym. Anal. Charact.*, vol. 21, no. 8, pp. 669–674, 2016, doi: https://doi.org/10.1080/1023666X.2016.1202466.

[4] S. F. Oyelere *et al.*, "Phytochemical analysis of Ficus thonningii: A qualitative study," *J. Med. Herbs Ethnomed.*, vol. 7, pp. 47–51, July 2021, doi: https://doi.org/10.25081/jmhe.2021.v7.7109.

[5] R. Dangarembizi, K. H. Erlwanger, D. Moyo, and E. Chivandi, "Phytochemistry, pharmacology and ethnomedicinal uses of Ficus thonningii (Blume Moraceae): a review," *Afr. J. Tradit. Complement. Altern. Med.*, vol. 10, no. 2, pp. 203–212, Jan. 2013, doi: https://doi.org/10.4314/ajtcam.v10i2.4.

[6] E. T. Fokunang *et al.*, "Phytochemical screening and in vivo evaluation of antiulcer properties of secondary metabolites in aqueous extracts of Ficus. thonningii Blume tested on Wistar rats," *Int. J. Biol. Chem. Sci.*, vol. 13, no. 1, pp. 475–492, May 2019, doi: https://doi.org/10.4314/ijbcs.v13i1.38.

[7] E. Taban *et al.*, "Morphological, acoustical, mechanical and thermal properties of sustainable green Yucca (Y. gloriosa) fibers: An exploratory investigation," *J. Environ. Heal. Sci. Eng.*, vol. 18, no. 2, pp. 883–896, July 2020, doi: https://doi.org/10.1007/s40201-020-00513-9.

[8] M. O. Falade *et al.*, "In vitro and in vivo antimalarial activity of ficus thonningii blume (Moraceae) and lophira alata banks (Ochnaceae), identified from the ethnomedicine of the nigerian middle belt," *J. Parasitol. Res.*, vol. 2014, 2014, doi: https://doi.org/10.1155/2014/972853.

[9] R. Dangarembizi, K. H. Erlwanger, D. Moyo, and E. Chivandi, "Phytochemistry , pharmacology and ethnomedicinal uses of ficus thonningII (Blume Moraceae): A review," *Afr. J. Tradit. Complement. Altern. Med.*, vol. 10, no. 2, pp. 203–212, Jan. 2013, doi: https://doi.org/10.4314/ajtcam.v10i2.4.

[10] A. Usman, F. I. Abdulrahman, and A. Usman, "Qualitative phytochemical screening and in vitro antimicrobial effects of methanol stem bark extract of Ficus thonningii (Moraceae)," *Afr. J. Tradit. Complement. Altern. Med.*, vol. 6, no. 3, pp. 289–295, 2009, doi: https://doi.org/10.4314/ajtcam.v6i3.57178.

[11] B. Asmare and Y. Mekuriaw, "Assessment of ficus thonningii tree production and utilization for livestock feed by smallholder farmers in

northwestern Ethiopia," *Agric. Food Secur.*, vol. 8, no. 1, Art. no. 8, Jan. 2019, doi: https://doi.org/10.1186/s40066-018-0247-4.

[12] M. Hasan, M. Rahman, Y. Chen, and N. Cicek, "Optimization of typha fibre extraction and properties for bio-composite applications using desirability function analysis," *Polymers*, vol. 14, no. 9, Art. no. 1685, 2022, doi: https://doi.org/10.3390/polym14091685.

[13] T. Radhakrishnan, P. Neelakantan, and T. Narasimham, "Airflow through fiber plugs," *Text. Res. J.*, vol. 61, no. 7, pp. 382–385, July 1991, doi: https://doi.org/10.1177/004051759106100703.

[14] G. N. Onyeagoro, "Effect of chemical treatment on the constituents and tensile properties of oil palm leaf fibre," *Acad. Res. Int.*, vol. 2, no. 3, pp. 88–98, 2012.

[15] D. Atalie and R. K. Gideon, "Extraction and characterization of Ethiopian palm leaf fibers," *Res. J. Text. Appar.*, vol. 22, no. 1, pp. 15–25, Mar. 2018, doi: https://doi.org/10.1108/RJTA-06-2017-0035.

[16] Z. Daud, H. Awang, A. S. M. Kassim, M. Z. M. Hatta, and A. M. Aripin, "Comparison of pineapple leaf and cassava peel by chemical properties and morphology characterization," *Adv. Mater. Res.*, vol. 974, pp. 384–388, 2014, doi: https://doi.org/10.4028/www.scientific.net/AMR.974.384.

[17] C. L. Gomes, E. Gonçalves, C. A. G. Suarez, D. de Sousa Rodrigues, and I. C. Montano, "Effect of reaction time and sodium hydroxide concentration on delignification and enzymatic hydrolysis of brewer's spent grain from two brazilian brewers," *Cellul. Chem. Technol.*, vol. 55, no. 1–2, pp. 101–112, 2021, doi: https://doi.org/10.35812/CelluloseChemTechnol.2021.55.10.

[18] L. Tan, Y. Q. Tang, H. Nishimura, S. Takei, S. Morimura, and K. Kida, "Efficient production of bioethanol from corn stover by pretreatment with a combination of sulfuric acid and sodium hydroxide," *Prep. Biochem. Biotechnol.*, vol. 43, no. 7, pp. 682–695, June 2013, doi: https://doi.org/10.1080/10826068.2013.773338.

[19] A. A. Modenbach and S. Nokes, "Effects of sodium hydroxide pretreatment on structural components of biomass," *Trans. ASABE*, vol. 57, pp. 1187–1198, Sep. 2014, doi: https://doi.org/10.13031/trans.57.10046.

[20] A. Lotfi, H. Li, D. V. Dao, and G. Prusty, "Natural fiber–reinforced composites: A review on material, manufacturing, and machinability," *J. Thermoplast. Compos. Mater.*, vol. 34, no. 2, pp. 238–284, 2021, doi: https://doi.org/10.1177/0892705719844546.

[21] M. M. Thrasybule, "Differentiated cntent: An expose on ceiba pentandra and ficus thonningii species as perceived in the haitian beliefs," *Saudi J. Humanit. Soc. Sci.*, vol. 7, no. 8, pp. 380–397, Aug. 2022, doi: https://doi.org/10.36348/sjhss.2022.v07i08.006.

[22] B. S. Kaith, H. Mittal, R. Jindal, M. Maiti, and S. Kali, "Cellulose fibers: Bio- and nano-polymer composites," in *Cellulose Fibers: Bio- And Nano-Polymer Composites: Green Chemistry and Technology*, S. Kalia, B. S. Kaith and I. Kaur, Eds. Springer, 2015, pp. 425–452.