



Experimental study of physical, chemical and mechanical properties of enset and sisal fibers

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ABSTRACT

The properties of composites depend on type and properties of the fiber, fiber structure and treatment. In this study, effects of NaOH treatment on the physical, chemical and mechanical properties of fibers collected from South-West Ethiopia were investigated using untreated, 5% and 10% alkali treated enset and sisal fibers. The effects of NaOH on diameter, moisture absorption, density, lignin and hemicellulose was experimentally studied. In addition, tenacity of both fibers was investigated by using optical microscopy, Fourier transform infrared spectroscopy and single fibers tensile strength tester. The 5% and 10% NaOH treatment significantly improved the moisture absorption and diameters. The moisture absorption of 5% and 10% NaOH treated enset fibers improved by 33% and 34%, while the same treatment on sisal fibers improved the moisture absorption by 30% and 40% respectively. The 5% NaOH treated enset and sisal fiber showed better tenacity, which is an indication of the tensile strength of single fiber, than untreated and 10% NaOH treated fibers. Improving moisture content, diameter, lignin and hemicellulose contents of the fibers contributes to better bonding as interlock with the matrix materials enhancing the mechanical properties of composites.

1. Introduction

Nowadays, natural fibers as alternative reinforcements in polymer composites have got high attention in research and industrial applications due to their advantages over glass and carbon fibers. These fibers have lower environment impact, lower cost, lower density, low energy as well as higher availability (abundance) and wider range of applications [1–3]. Natural fiber sources are classified into three main categories as (1) plant, (2) animal and (3) mineral-based. Plant fibers contain larger quantities of cellulose, while animal fibers contain protein. In general, plant fibers give higher strength and stiffness than animal and mineral [4].

Plant fibers include bast, leaf, grass and seed/fruit fibers, where bast consists of a wood core surrounded by a stem. Examples include flax, hemp, jute, kenaf, wood, sun hemp and ramie. Leaf fibers, on the other hand, include sisal, abaca, banana, date palm, piassava, pineapple and henequen and they are coarser than bast fibers. Grass fibers are also available from plants such as bagasse, bamboo, while examples of fruit fibers are like coir, kapok, oil palm, sponge gourd and cotton is the most common seed fiber [5]. Chemical composition differs from plant to plant, and within different parts of the same plant, from different

geographic locations, ages, climate, and soil conditions. Sisal fiber extracted from leaf parts of sisal plants can be used for making the variety of products [6]. The chemical composition of the fiber influences its properties and the fibers themselves could be considered as fibrous composite materials [7]. Cellulose is the main component in the fiber. Hemicellulose is composed of cellulose chains, but it is much shorter in length and is built up of a mixture of polysaccharides of relatively low molecular weight. It is also responsible for biodegradation, moisture absorption, and thermal degradation of the fibers. The lignin content influences the fiber structure, properties, morphology, strength and moisture absorption when compared with hemicellulose [8]. Also lignin is thermally stable, but responsible for the ultraviolet degradation of the fibers [9].

The properties of fibers mainly depend on their chemical composition and their structure, which are related to the type of fibers used, method of extraction, harvesting period, growing conditions and chemical treatment [4,10]. Natural fibers are rich of cellulose, hemicelluloses, lignin, and pectins, all of which are hydroxyl groups, which are usually hydrophilic sources and strong polar whilst polymers show considerable hydrophobicity. Fiber alkali treatment reduces hemicellulose, lignin, and wax content and increasing surface roughness for

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better interlocking with the matrix materials to enhance the properties of the composites [11–15]. The physical, chemical and mechanical properties of natural fibers are essential and the main criteria for designing and developing industrial products [16]. To develop better property products, studying the effects of alkali treatment on physical, chemical and mechanical properties of natural fibers is very important [17]. Alkaline treatment increases the density of fibers by removing non cellulose components (hemicellulose and lignin), which are less dense [18].

Many researchers have used alkali concentration, immersing time and drying time as main parameters of their study. The used concentrations range from 1 to 15 wt%, immersing time ½ hr to 3 h and dry time from 1 day to 5 days [19,20]. At low alkali concentration, the impurities are not sufficiently removed, while higher alkali concentration above 10% can lead to damage of fibers [21]. The effects of immersion (soaking) time were also studied for coir fibers. The results of previous studies show that 2 h immersion time gives better tensile strength than immersing for less hours (1 h) or longer hours (3–5 h) [22]. The effects of alkali on diameter of sugarcane fiber was studied and the results confirm that alkali treated fibers have less diameter than untreated fibers. This implies that alkali treatment removes the outer (lignin) and the inner (hemicellulose) contents from the fibers [23]. Commonly, natural fibers have hydrophilic properties that are vulnerable to moisture. Fiber moisture absorption properties were improved by alkali treatment by reducing the hydrophilic hydroxyl groups of the fibers [24]. If the treatment parameters are not optimized, the NaOH can cause fiber defibrillation, pore formation, and fiber embrittlement [25].

As the overview of the research in the area indicates, various researchers have conducted study of the effects of alkali treatment on different natural fibers. Among others, the properties of plant fibers naturally depend on the climate in which the plant grows. The study of enset and sisal fibers in this region of Ethiopia that this study targets is so far not reported. Therefore, this study aims to characterize the fibers of enset and sisal from South-West Ethiopia by experimental investigation of the effects of alkali treatment on the physical, chemical composition and mechanical properties as a probe for reinforced polymer composites.

2. Materials and methods

2.1. Materials

Enset and sisal fibers for this study were collected from Oromia region around Woliso, South-West Ethiopia. Alkali (NaOH), sulfuric acid (H₂SO₄), ethanol, Toluene, and HCl are purchased from local market. To determine the fiber chemical properties, laboratory equipment such as soxhlet apparatus, sensitive balance, oven, furnace and water bath were used. In addition, optical microscopy (OM) and Fourier transform infrared spectroscopy (FTIR) were used to determine the diameter and chemical compositions of fibers.

2.2. Extraction and alkali treatment of fibers

The enset and sisal plant and their traditional fiber extraction methods are shown in Fig. 1 and Fig. 2 respectively.

For this study, the matured enset pseudostem and agava sisilana leaf was cut and the fiber was extracted by manual decortication methods and washed by fresh water for several times then dried in sunlight for 8 h, as illustrated in Fig. 3 (a) and (b). The enset and sisal fibers were immersed in 5 wt% and 10 wt% NaOH concentrations for about 2 h at room temperature. Then, fibers were washed several times with fresh water then followed by distilled water to take away any NaOH sticking on the fibers surface and dried at room temperature for 24 h. The 5% and 10% NaOH alkali treated enset and sisal fibers are shown in Fig. 3 (c)–(f) respectively.

2.3. Physical properties of enset and sisal fibers

2.3.1. Densities (ρ_F)

The sample, i.e. a bundle of treated and untreated enset and sisal fibres, was weighed in air and then in a distilled water, having lower density than the sample, to wet the sample. The difference between the two media was the buoyancy force. The weighing process was conducted on an analytical balance with a resolution of 0.001 g and was adapted for suspension weighing through a stainless steel suspension wire approximately 0.4 mm in diameter. The buoyancy force was converted to sample volume by dividing the liquid density. The sample density ρ_f was acquired by dividing sample weight in air with sample volume [26].

$$\rho_F = \left(\frac{m_3 - m_1}{(m_3 - m_1)(m_4 - m_2)} \right) \rho_w \quad (1)$$

where ρ_F is density of fiber (g/cm³), m_1 = weight of suspension wire in air, m_2 = weight of suspension wire in liquid, m_3 = weight of suspension wire plus sample weight in air, m_4 = weight of suspension wire plus sample weight in liquid (gram) and ρ_w is standard density of distilled water i.e 0.998 g/cm³. All densities were measured at 23 ± 0.2 °C using Mettler Toledo density measurement kit and a known standard density block [27].

2.3.2. Moisture absorption of fibers

Water absorption affects the physical properties of composites and the fiber-matrix interface [28]. The fiber moisture absorption properties were improved as a result of the reduced hydrophilic hydroxyl groups [24]. The moisture of Mendong fibers were determined by cutting into lengths of 5–10 mm and drying in an oven at temperature of 103 °C ± 2 °C for 4 h then placing in the desiccator for 24 h [29]. Similar to the calculation of the moisture contents of enset and sisal fibers, normal weight loss methods were used. Weighed enset and sisal samples were kept and allowed to dry in an oven at 105 ± 2 °C for 4 h and cooled in desiccator for 10–15 min and waited till the weight of fibers become constant. The following expression was used to determine the moisture contents in enset and sisal fibers.

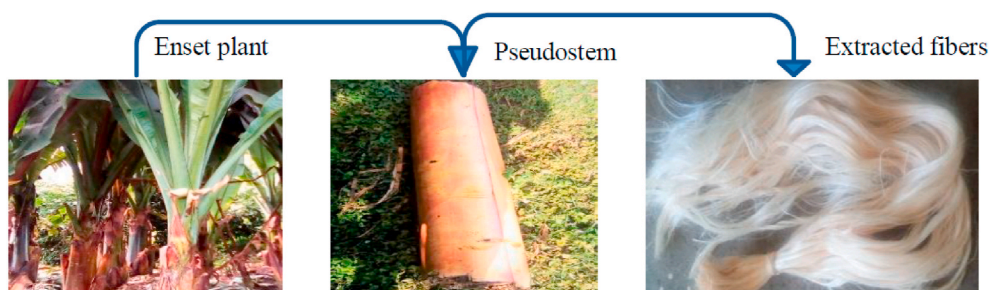


Fig. 1. Enset plants and fiber extraction.

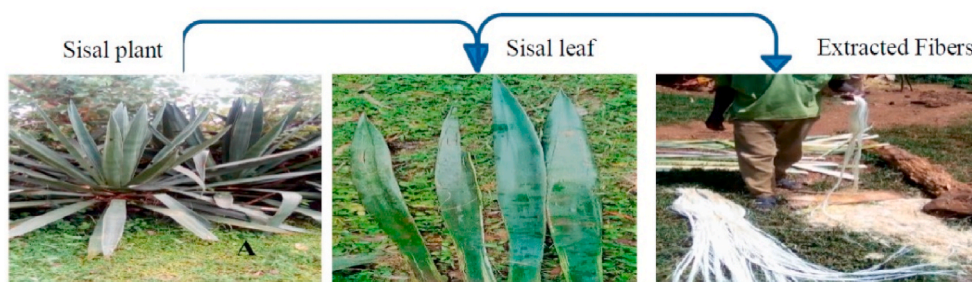


Fig. 2. Sisal plants and fiber extraction.



(a) Untreated Enset (E_0) fiber

(b) Untreated Sisal (S_0) fiber



(c) 5% NaOH treated Enset ($E_{5\%}$) fiber

(d) 10% NaOH treated Enset ($E_{10\%}$) fiber



(e) 5% NaOH treated Sisal ($S_{5\%}$) fiber

(f) 10% NaOH treated Sisal ($E_{10\%}$) fiber

Fig. 3. Untreated and NaOH treated enset and sisal fibers.

$$\%Mc = \frac{W_{fb} - W_{fa}}{W_{fb}} * 100 \quad (2)$$

where Mc is moisture content of fiber, W_{fb} (gram) is weight of fiber before putting in oven and W_{fa} is weight of fiber after putting in oven.

2.3.3. Measuring fiber diameter

The sample of single fiber is cut into small splice, placed on to a microscopy slide and projected into screen through a microscopy. The enset and sisal fibers' diameter was measured using the optical microscopy. Randomly selected 10 samples were measured at five locations along the length and the average diameter value was reported.

2.4. Chemical compositions of enset and sisal fibers

2.4.1. Wax, cellulose, hemicellulose and lignin

The chemical composition of the fiber influences its properties, and the fibers themselves could be considered as fibrous composite materials. Both sisal and enset, untreated and NaOH treated, were determined by TAPPI Standard T 264 method [7]. The wax content was measured with the help of soxhlet apparatus. The ethanol-toluene solution was prepared with a ration of ratio 1:2. Two grams of treated and untreated enset and sisal fibers were extracted for 8 h. Then, after refluxing for 1 h, the fiber sample was dried in an oven and it was weighed and the weight difference confirmed the wax content.

To determine the cellulose percentage, 1-g sample was placed into 250 ml round bottom flask and reflux for an average of 1 h in a mixture of concentrated nitric acid in ethyl alcohol. It was refluxed with three successive portions of a mixture of concentrated nitric acid (20% volume

by volume) in ethyl alcohol. At the end of the extraction process, which lasts 1 h each, the flask containing the extractive nitric acid solution is transferred into known weight of gauche crucible to proceed to filtration process using under vacuum suction. The extractive materials were washed several times by distilled water. Then the acid mixture extractives were dried in oven at 105 °C for an average of 1 h, cooled in desiccators, and weighed until a constant weight is obtain.

$$\text{Cellulose}(\%) = \frac{W_2}{W_1} * 100\% \quad (3)$$

where w_1 is the amount of extract free samples taken for analysis and w_2 is the residual mass of cellulose.

Furthermore, to determine ligning contents, 1 g of treated and untreated enset and sisal fibers were placed in beaker, 15 ml of 72% H_2SO_4 was added slowly and stirred for 2 h and then after diluting the H_2SO_4 to 3% and refluxed for 3 h. The filter residue was washed by hot water several times and then after oven dried at 105 °C for 1 h and weighed (w_2). The residue weight was noted as the lignin content [30]. In the same way, to determine the hemicellulose content of fibers, the treated and untreated enset and sisal fiber of weighed quantity was immersed in a mixture of 5% NaOH solution at room temperature for ½ hrs and neutralized by HCl and the residue was dried and weighed. The weight difference accounts for the presence of hemicellulose content.

2.4.2. Ash contents of fibers

To determine the fiber ash contents, about 2 g of sample was put on pre-weighted crucibles and placed in the furnace at 575 °C for 3 h and cooled. The crucibles were placed in desiccator and weighed until the difference of two successive weights are less than 1 mg, as recommended in Ref. [31]. The percentage of ash contents is calculated using Equation (4).

$$\% \text{ of Ash content} = \frac{\text{Initial sample weight}}{\text{Final ash weight}} \times 100 \quad (4)$$

2.5. Mechanical properties of enset and sisal fibers

The tensile strength of untreated, 5% and 10% NaOH treated sisal and enset fibers were investigated using Mantis single fiber tensile tester. This tester is capable of testing single fiber strength using a pair of clamps that grip the fiber without damaging the fibers. The test was done at textile industry development institute in Addis Ababa, Ethiopia, according to ASTM-D4605 standard recommendations. Ten specimens of untreated and treated fibers were tested with 100 N load cell having gauge length of 200 mm at room temperature. To avoid the diameter variation effects, the specimens were compared by indirect counting using the ratio of breaking force to length per unit mass, which is referred to as “tex” [24].

2.6. Fourier transform infrared spectroscopy

The FTIR analysis method was used to detect the characteristic functional groups in the fibers [29,32] by passing the infrared light through the sample fibers. NaOH was used to remove the hemicellulose, lignin, and other impurities from the fiber surface and change of fiber chemical composition was analyzed using FTIR. Each spectrum was recorded by co-adding 32 scans at 8 cm^{-1} resolutions within the range of 4000–600 cm^{-1} range [33]. The FTIR spectrum was made for untreated, 5% and 10% NaOH treated of sisal and enset fibers.

3. Results and discussion

3.1. Physical properties of enset and sisal fibers

3.1.1. Moisture content

The measured fiber density and diameter of enset and sisal fibers in

this study are given in Table 1. As the results in the table show, the moisture content of enset and sisal fiber improved by NaOH treatment. It has been noticed that there is higher moisture absorption of untreated enset and sisal fibers than that of treated fibers. In the structure of fibers, the hydrogen bond is broken down due to alkali treatment and improves the fiber moisture absorption properties by reducing the hydrophilic hydroxyl groups of the fibers for better interlocking with the matrix.

3.1.2. Fiber density and diameter

The measured results given in Table 1 show that the diameter reduced by 45.2% and 33.3% for 5% NaOH treated and 62.8% and 52.1% for 10% NaOH treated of enset and sisal fibers than untreated fibers respectively. This is due to removal of impurities from the inner and outer fiber surface. The density of both untreated fibers is found to be lower than the treated fibers. Increase of the densities is due to the removal of the lower denser regions such as lignin and hemicellulose. Higher density indicates that the fibers have better cellulose contents.

3.2. Mechanical properties of enset and sisal fibers

3.2.1. Tensile strength

The tensile force of 5% NaOH treated enset and sisal fibers was increased by 51.5% and 31.67% respectively compared with untreated fibers, as shown on Fig. 4 (a). Tenacity measures the strength of a fiber expressed by maximum tensile force (breaking force) divided by the linear density of the fibers and it is expressed by cN/tex. Tenacity of 5% NaOH treated single fiber is higher than untreated and 10% NaOH treatment. Higher concentration of NaOH damages the fibers and decreases tenacity value as shown on Fig. 4 (b). Similar observation is reported in Refs. [34,35].

3.3. Chemical properties of fiber

The cellulose, lignin, hemicellulose and wax are the most common fiber chemical compositions that affect the physical, chemical and mechanical properties of the fibers. Table 2 shows the result of the chemical compositions for untreated and 5% and 10% NaOH treated enset and sisal fibers. The change in fiber chemical properties was observed due to NaOH treatment. The NaOH treatment caused the cellulosic fiber to swell and it removed the hemicellulose, lignin and other impurities from the fiber surface. A higher cellulose content ensures good mechanical properties, while high levels of hemicellulose decreases the fiber strength by causing disintegration of cellulosic microfibrils. Fiber treatment by NaOH removes most of the lignin content, which affects the fiber structure, properties and morphology. The lignin contents of 5% NaOH treated fiber reduced by 71.1% and 54.1% than untreated enset and sisal fibers respectively. The chemical composition values in Table 2 indicate that both enset and sisal fibers have very high cellulose content, much higher than the other compositions. This huge variation is not unexpected because natural fibers in nature have more amount of cellulose than hemicellulose, lignin and others. In general, it is also observed that NaOH treatment increases cellulose contents and ash contents as the values in Table 2 demonstrate.

3.4. Fourier transform infrared Spectrometry (FTIR)

The fibers are mixtures of cellulose, hemicellulose, lignin, wax and other ingredient, where the NaOH treatment was used to remove the hemicellulose, lignin, and other impurities from the fiber surface. The effects NaOH treatment on fiber chemical compositions was analyzed using FTIR. The infrared whole pattern of the untreated, 5% and 10% NaOH treated sisal fibers are shown in Fig. 5 and enset fibers are showed on Fig. 6.

All spectra were recorded in the range from 4000 cm^{-1} to 400 cm^{-1} . The broadband at around 3000–4000 cm^{-1} was referred to alcohol compound or hydrogen-bonded OH stretching from cellulose,

Table 1
Physical and mechanical properties of untreated and treated enset and sisal fibers.

Properties	Untreated fibers		NaOH treated fibers			
	Sisal	Enset	Sisal		Enset	
	S ₀	E ₀	S _{5%}	S _{10%}	E _{5%}	E _{10%}
Diameter (μ m)	474.4 ± 0.065	334.97 ± 0.11	316.8 ± 0.03	227.2 ± 0.07	183.37 ± 0.17	124.25 ± 0.12
Moisture (%)	12.32 ± 0.02	13.91 ± 0.16	8.61 ± 0.10	7.64 ± 0.05	9.32 ± 0.10	9.13 ± 0.15
Density (g/cm ³)	1.66 ± 0.128	1.52 ± 0.09	2.31 ± 0.14	2.51 ± 0.20	2.02 ± 0.05	2.23 ± 0.13
Tensile strength (cN/tex)	24.78 ± 0.22	15.81 ± 0.16	20.23 ± 0.20	8.26 ± 0.21	13.96 ± 0.15	13.01 ± 0.21
Tensile module (cN/tex)	1434.78 ± 0.25	2296.95 ± 0.17	1064.06 ± 0.11	1070.28 ± 0.23	1998.08 ± 0.14	1320.75 ± 0.12
Elongation (%)	1.74 ± 0.13	2.11 ± 0.29	1.29 ± 0.14	1.24 ± 0.04	1.92 ± 0.11	1.78 ± 0.42

where: E₀, S₀, are untreated enset and sisal fibers, while E_{5%}, S_{5%}, E_{10%}, S_{10%} NaOH treated enset and sisal fibers respectively.

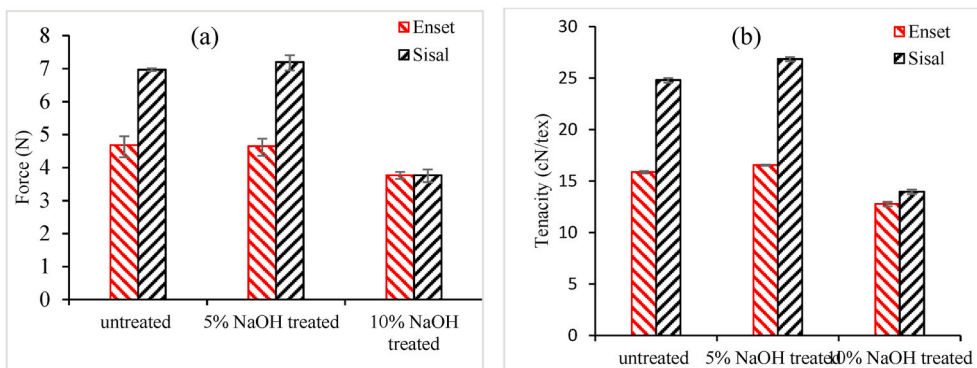


Fig. 4. (a) Tensile force and (b) Tenacity of treated and untreated enset and sisal.

Table 2
Chemical properties of untreated and NaOH treated enste and sisal fibers.

Sample	Wax	Cellulose	Hemicellulose	Lignin	Ash contents
E ₀	5.13 ± 0.09	80.88 ± 0.09	10.90 ± 0.09	10.43 ± 0.05	2.67 ± 0.1
E _{5%}	1.80 ± 0.69	82.97 ± 0.28	6.33 ± 0.08	3.00 ± 0.01	3.17 ± 0.05
E _{10%}	0.09 ± 0.11	85.19 ± 0.46	3.01 ± 0.46	3.33 ± 0.13	5.17 ± 14
S ₀	1.10 ± 0.77	73.53 ± 0.36	10.02 ± 0.46	8.00 ± 0.23	1.50 ± 0.13
S _{5%}	1.37 ± 0.73	85.58 ± 0.24	5.00 ± 0.34	5.20 ± 0.12	2.00 ± 0.15
S _{10%}	2.47 ± 0.48	90.66 ± 0.18	2.67 ± 0.18	3.67 ± 0.25	3.33 ± 0.21

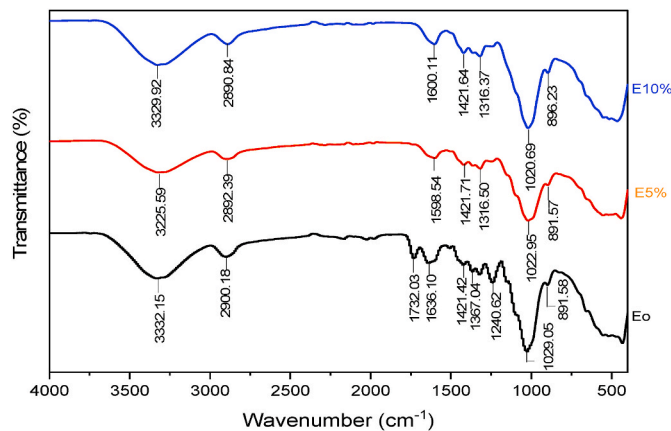


Fig. 6. FTIR Spectrum of enset fiber.

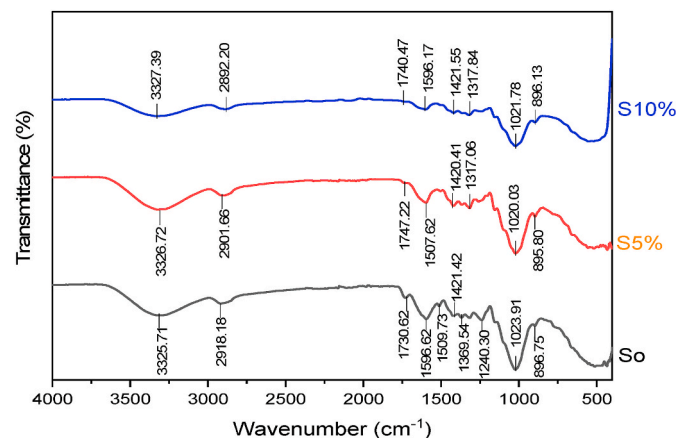


Fig. 5. FTIR Spectrum of sisal fiber.

hemicellulose, and lignin. The broadband at 3400–2850 cm⁻¹ was attributed to C–H bond frequently observed in alkane groups. The intense peaks for C=O groups in the ketone and carbonyl groups were shown as peaks at 1740–1720 cm⁻¹ and 1450–1650 cm⁻¹ referred to the hemicellulose and ligning compound [36].

The spectrum display O–H absorption for enset is around 3332.15 cm⁻¹, 3325.59 cm⁻¹ and 3330.35 cm⁻¹ for untreated and 5% and 10% NaOH treated fibers respectively, while the corresponding values for sisal are 325.71 cm⁻¹, 3326.72 cm⁻¹ and 3329.75 cm⁻¹. Similarly, the C–H stretching of untreated, 5% and 10% NaOH treated enset are around 2900 cm⁻¹, 2892.32 cm⁻¹ and 2899.15 cm⁻¹, while the corresponding values for sisal are around 2918.18 cm⁻¹, 2901.66 cm⁻¹ and 2891.90 cm⁻¹ are respectively α-cellulose [37].

The peaks at 1732.32 cm⁻¹ and 1730.02 cm⁻¹ were attribute to C=O stretching vibration of acetyl groups in hemicellulose compounds for both untreated sisal and enset fibers as shown on Fig. 5 and 6. The

intensity of those peaks are not seen (removed) in the case of 5% and 10% NaOH treatment of sisal and enset fibers. This shows that NaOH treatment reduces or removes the hemicellulose compounds from the fibers.

The peak at 1450–1650 cm^{-1} refers to aromatic structure of lignin present in the fiber. The peak 1596.62 and 1636.1 cm^{-1} appear in untreated sisal and enset fiber respectively. In case of NaOH treatment, the decreased intensity of those peaks is a proof that lignin reduced by NaOH treatment as the concentration of treatment increases from 5% to 10%. The peak at 1300–1500 cm^{-1} shows that C–H bending of hemicellulose, O–H stretch of cellulose and C–O stretching of NaOH group. The peak at 1240.3 cm^{-1} and 1240.62 cm^{-1} is attributed to C–O stretching of acetyl (lignin) and its intensive removal by 5% and 10% respectively. The peak at 1369.54 cm^{-1} and 1367.04 cm^{-1} are attributed to C–H bending (deformation) of hemicellulose compound and their intensity were removed by NaOH treatment.

The peak at 1029.05 and 1023.91 cm^{-1} can be attributed to the C–H stretching of hydroxyl group in cellulose and 891.58 and 896.30 cm^{-1} existence of b-glycosidic linkage between the monosaccharides on untreated enset and sisal fibers. Similarly, those compounds do exist on treated fibers indicating that treatment does not minimize or remove those compounds from the fibers.

4. Conclusions

This article presented study on characterization of natural fibers extracted from pseudostem enset and leaf of sisal plant that were treated with 5% and 10% NaOH solution. The effects of NaOH treatment on physical, mechanical, and chemical properties of the fibers were investigated. The main findings of the present work are:

- Treated enset and sisal fiber showed less moisture absorbance than that of untreated fibers. As the percentage of NaOH concentration increased, its moisture absorbance further improved.
- As concentration of alkali treatment increased, the density of fibers also increased slightly for both enset and sisal fibers. This is due to removal of non cellulose components like hemicellulose and lignin.
- The chemical composition of enset and sisal fiber was improved by NaOH treatment for the better interfacial surface with the matrix materials.
- Increasing NaOH concentration removes the impurities from the fibers leading to reduced diameter of the fibers.
- FTIR spectroscopy data also indicates that there is a reduction of hemicellulose and lignin with 5% and 10% NaOH treatments.
- Tenacity results indicated that tensile strength of single fiber at 5% NaOH treated enset and sisal fiber got better tenacity than untreated and 10% NaOH treated fibers.

CRedit author statement

Abera E. Bekele: Data collection, Testing and Investigation, Methodology, Result analysis, Writing - Original draft; **Hirpa G. Lemu:** Conceptualization, Methodology, Supervision, Review & Editing and Validation; **Moera G. Jiru:** Conceptualization, Funding acquisition, Methodology, Supervision and Validation.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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