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# Mechanical, morphological, structural and dynamic mechanical properties of alkali treated Ensete stem fibers reinforced unsaturated polyester composites

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## ABSTRACT

Present study deals the surface morphology and structural composition analysis of alkali (NaOH) treated 2.5% 5.0% and 7.5 wt% Ensete stem fiber obtained from the Ethiopian *Ensete ventricosum* plant. Treated Ensete fibers reinforced unsaturated polyester (UP) composites were characterized in terms of tensile, flexural, surface morphology and dynamic mechanical properties. Mechanical test results revealed that 5.0 wt% alkali treated Ensete fibers/UP composites showed 14.5% and 43.5% increase in flexural strength and Young's modulus respectively, with relative to untreated Ensete fibers/UP composites. Storage and loss modulus value also highest for 5.0 wt% alkali treated Ensete fibers/UP composites. Moreover, a positive shift in glass transition temperature ( $T_g$ ) of composites after alkali treatment and tensile fracture surface morphology indicates better interfacial interaction in treated Ensete fibers/UP composites. Overall we concluded that 5.0 wt% treated Ensete fibers satisfactorily and effectively improved mechanical, morphological and dynamic properties of UP for various engineered and hi-tech applications.

## 1. Introduction

The use of renewable resources in fiber-reinforced polymeric composites has gained significant importance due to their advantage from environmental and economic perspectives [1]. Plant based natural fibers are regarded as green reinforcement in polymer composites and usually grouped as stem fibers (flax, hemp, jute, ramie, kenaf, etc.), leaf fibers (sisal, banana, pineapple etc.) and seed fibers (coir, oil palm, etc.). These fibers are chemically composed of cellulose, hemicelluloses, lignin and pectin with a minor amount of extractives, and have mechanical and thermal properties quite comparable to glass fibers [2]. Moreover, commercialization of natural fiber reinforced polymer composites has increased in the last few years as its application in emerging industry grows such as in furniture's, packaging goods, automotive and as construction materials, based on their performance [3]. As the higher performance of fibers was achieved with varieties having higher cellulose content and with cellulose microfibrils aligned more in the fiber direction which tends to occur in stem fibers [4]. Based on evaluations after 2011, these industries estimated to grow 10% globally

[5]. Several research findings are reported in literature where the reinforcements of natural fibers either as fiber or filler improve the mechanical properties of polymer composites [6]. In one study researchers revealed that banana/phenol formaldehyde composites possess (tensile strength of 26 MPa) comparable mechanical properties with glass/phenol formaldehyde composites (tensile strength 42 MPa) at 40% fiber loading [7]. In other study, flexural modulus of glass fiber reinforced epoxy composites with untreated and treated (alkali, silane) kenaf and hemp fibers were compared [8]. For the fabrication of natural fiber reinforced polymer composites higher performance is achieved with varieties having higher cellulose content and with cellulose microfibrils aligned more in the fiber direction which tends to occur in stem fibers [4]. Remarkably, the core (stem) part of the plant has higher cellulose composition than the leaf veins, thus can be used as an economic source of cellulosic fiber where mechanical performance is major requirement. Ensete is one of the promising substitutes to commonly used kenaf, abaca and sisal fibers. Ensete is widely cultivated in East Africa and mostly used for food crops. Though, fiber extraction from Ensete stem is economical and as a byproduct it is abundant, however, no studies were

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**Table 1**  
Mechanical, physical and chemical composition of Ensete fiber.

Properties of Ensete fiber	
<i>Mechanical properties</i>	
Tensile strength	513 ± 57.7 MPa
Tensile modulus	26.7 ± 3 GPa
<i>Physical properties</i>	
Length	1–3 m
Diameter	115–185 μm
Density	1.53 g/cm <sup>3</sup>
Fineness	8–16 tex
<i>Chemical composition</i>	
Cellulose	56.05%
Hemicellulose	24.04%
Lignin	2.21%
Extractives, wax, ash, etc.	17.7%

yet performed to investigate its use in the fabrication of green polymer composites. Similar to other cellulosic natural fibers, Ensete fiber is chemically composed of cellulose, hemicellulose, lignin, pectin, moisture, wax and oils. The percentages of these contents for raw Ensete fiber are displayed in Table 1. Although the proportion of these components depends on the age, source of the fiber and the extraction conditions as like other natural fibers. Moreover, researchers already reported that the alkali treatment results in an increase of cellulose components and a decrease of lignin and hemicellulose components [9]. Ensete fiber also holds suitable physical and mechanical properties to be used as an alternative and promising candidate for manufacturing natural fiber reinforced polymer composites similar to other stem fibers, as shown in Table 1.

The mechanical properties and performance of natural fibers reinforced thermoplastic and thermoset polymers composites are mainly influenced by the critical selection of fibers and their interfacial adhesion that often improved through surface treatments (either physical or chemical) [11–13]. Hu and Lim [13] showed that alkali treatment significantly improved the tensile properties (tensile strength and modulus of 54.6 MPa and 85 GPa, respectively) of hemp fiber reinforced polylactic acid (PLA). In another work, alkali scouring and silane grafting increases the delamination resistance of jute/epoxy composites by 10%, more precisely due to enhanced interfacial bonding [14]. Researchers reported that the increase of interfacial adhesion due to surface treatments of fibers results an increase of tensile, flexural properties and fracture toughness of composites [15,14,16,17]. The effects of alkali surface treatment on stem fibers that have comparable properties with Ensete fibers have been analyzed and reported in many findings. Asumani et al. [18] have studied polypropylene composites reinforced with nonwoven form of untreated kenaf and NaOH treated kenaf fibers. NaOH solutions with concentrations ranging from 1% to 8% in intervals of 1% by mass were used for the alkali treatment. They observed that composite properties such as tensile strength, tensile modulus, flexural strength and modulus exhibited their maximum values at 5% alkali treated kenaf fibers. Cai et al. [19] have also studied the effects of immersing abaca fiber in 5, 10 or 15 wt% NaOH solutions for 2 h. They reported that abaca fibers treated in 5 wt% NaOH exhibited increased crystallinity, tensile strength and Young's modulus compared to untreated fibers and also enhanced interfacial shear strength with an epoxy. However, higher NaOH concentrations (10% and 15%) results in complete removal of fiber binding materials and fiber breakdown or fibrillation of the abaca fiber; which results with poor adhesion with the epoxy matrix. Similarly, the effects of alkaline treatment (4%, 6% and 8%) on mechanical properties of bamboo fibre reinforced polyester composites were investigated by Manalo et al. [20]. The mechanical properties of composites treated with 6% of NaOH solutions exhibited the maximum results. The result analysis for bending, tensile, compressive strength and stiffness of the composites

showed 7, 10, 81 and 25% respectively higher compared to the untreated composites. In other study kenaf fibers immersed in 6% NaOH solution for 3 h also showed higher flexural strength and modulus of 12% and 13%, respectively as compared to untreated kenaf polyester composites [21]. All this positive findings motivate us to carry out alkali treatment of Ensete fiber and to investigate its effect on mechanical and dynamic mechanical properties of developed composites.

In this work an attempt were made to extract the Ensete fiber from pseudo stem of Ensete plant. Extracted fibers were treated with NaOH alkali solutions in an aimed to improve single fiber strength and fiber-polymer interfacial bonding and their mechanical, thermal and morphological properties were investigated and compared with untreated fibers. Treated fibers are then reinforced to fabricate Ensete fibers/UP composites through VARTM and their mechanical (tensile and flexural strength) and dynamic properties are characterized. VARTM does not need high temperature and suitable to produce complex and large-scale components in one shot [18].

## 2. Materials and methods

### 2.1. Materials

In this study, the Ensete fiber obtained from the Southern region of Ethiopia. The standard unsaturated polyester (UP) resin with cobalt was supplied from Boytek Company, Turkey. Methyl ethyl ketone peroxide (MEKP) the curing of UP, used as an initiator. Sodium hydroxide (NaOH) was procured from Merck Company, Germany and its different weight percentage was used as a surface treating chemical.

### 2.2. Methods

#### 2.2.1. Alkali treatment

Ensete fiber was treated with a different weight fraction of NaOH concentration (2.5%, 5.0% and 7.5 wt%), by adding a measured amount of NaOH to the distilled water as per the ratio and allowed to heat the aqueous solution to 30 °C. The Ensete fibers were then immersed and treated for 2 h, until temperature rises to 35 °C. Fibers were rinsed with tap water until pH value set to 7 and allowed to dry at room temperature.

#### 2.2.2. Ensete fiber characterization

**2.2.2.1. Tensile tests.** The changes in uniaxial tensile strength due to alkali treatment of individual Ensete fiber (separated manually from fiber bundles) were studied to investigate the optimum surface treatment conditions and to study the physicochemical changes were made by Titan-Universal Strength Tester as per standard EN ISO 2062. The tensile tests carried out by using maximum load cell of 120 N with rate of extension of 250 mm/min. Results data are then analyzed to determine fiber tensile strength, modulus and strain at break.

**2.2.2.2. Scanning electron microscopy (SEM).** The morphologies of the untreated and alkali treated Ensete fibers were studied by using Carl Zeiss ultra plus Gemini FESEM. Prior the investigation, the fiber samples were coated with gold layer by Q150r rotary-pumped sputter coater/carbon coater.

**2.2.2.3. Fourier transforms infrared spectroscopy (FTIR).** FTIR spectra of untreated and alkali treated Ensete fibers were carried out through Vertex 70 ATR FTIR Spectrometer model of Bruker, with rapid scan > 70 spectra/sec at 16 cm<sup>-1</sup> spectral resolution and wave number ranging from 500 to 4000 cm<sup>-1</sup>.

#### 2.2.3. Fabrication of composites

In this study, the VARTM which is a derivative of the resin transfer molding (RTM) process, used to fabricate treated and untreated Ensete fiber/PU composites, providing good resin distribution and high

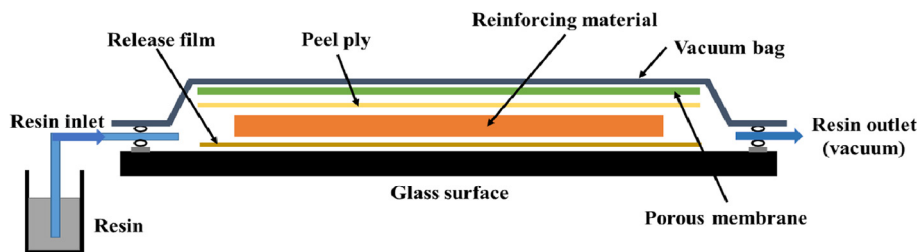


Fig. 1. Schematic process of VARTM process.

uniformity [22], displayed in Fig. 1. From the general point of view VARTM found very effective in impregnation, does not required high temperature and it is suitable to produce complex and large-scale components in one shot. However, the steps such as mold preparation, lay-up, and sealing the mold and creating a vacuum, resin preparation and degassing should be performed carefully for better end-product. The resin flow through the thickness of reinforcer will be controlled with the help of a vacuum pump where a suitable resin viscosity should be provided for better impregnation. Most generally fiber content were kept constant, if the fiber content increases there might be less impregnations relatively and thus amendments should be taken for uniform wetness of the filler.

As the uniformity of the nonwoven reinforcer is quite important for the mechanical properties of the developed composites. Thus in this study, considerable attention was paid for the carded Ensete webs by using lab scale SDL Atlas carder, which is capable to produce uniform nonwovens with  $250 \times 500$  mm. The carder is a standard for the uniform distribution of fibers in such nonwoven structures. The carding procedure was applied after the alkaline treatment to prevent fiber bundle formation and reduce the fiber adhesions. During the alkaline treatment, the fibers exhibited some color change (Fig. 2). Moreover, the discoloration could not be conducted to thermal processes, because the fibers were not exposed to high temperature. As during the alkaline treatment maximum temperature was  $35^\circ\text{C}$  where the fibers undergoes some color change (Fig. 2), which is due to removal of some left bark from extraction, waxes and other impurities.

VARTM system includes laying fiber preform over a smooth open

mold surface. Peel ply and a porous membrane are placed to ease the flow of resin. The samples are covered with a vacuum bag and leaks of the bag were eliminated. Inlet and outlet feed hoses are placed in the vacuum bag. In this study, after carding process of Ensete fibers, four samples were fabricated over a glass surface with a polyurethane film to remove the final composites easily. The weight fraction of nonwoven Ensete webs was kept constant for all samples. The steps such as lay-up, sealing mold sealing, vacuum application, resin preparation and degassing were performed carefully for better and uniform sample production. Peel ply, porous membrane and a vacuum bag were placed over the samples respectively, and the system was sealed as shown in Fig. 1. The UP (including cobalt) and 1 wt% of MEKP as an initiator were mixed before the resin absorption/immersion process made. When the resin arrive the outlet, the vacuum pump was turned off. Then samples were allowed to cure at room temperature for 24 h. Fabricated Ensete fibers/UP composites are presented in Fig. 2 having total fiber weight fraction about 28%.

#### 2.2.4. Characterizations of composites

**2.2.4.1. Tensile and flexural properties.** All the composite sample specimens were cut into required dimensions using a CNC cutter and the edges and surfaces were finished by using emery paper for tensile (ASTM D3039) and flexural tests through Shimadzu universal testing with capacity of 50 kN, strain rate of 1 mm/min and cross head movement of 2.5 mm/min at room temperature. The three-point bending flexural test was done on each rectangular composite samples specimen having dimension of the order of 60 mm x

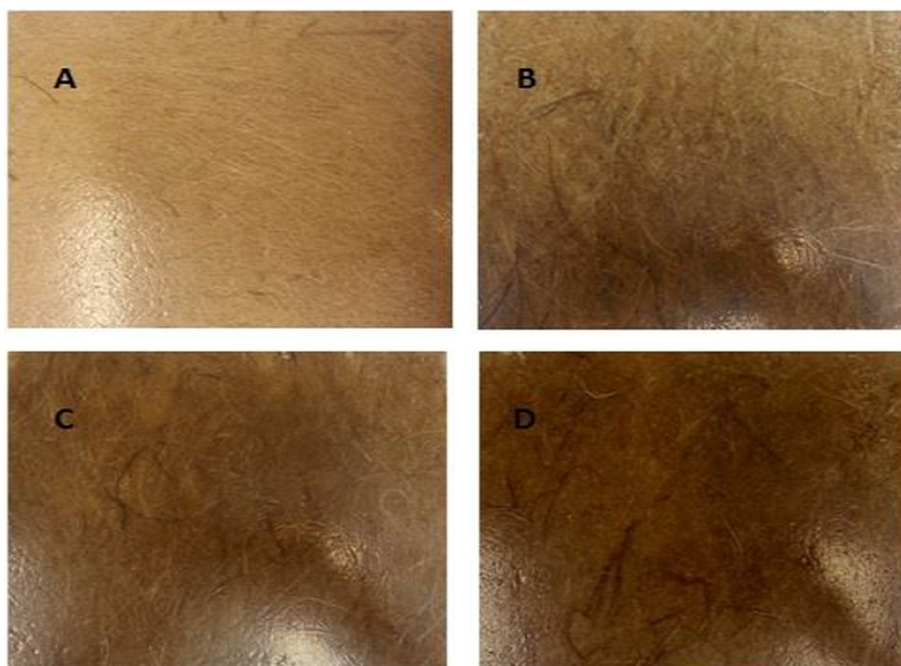


Fig. 2. Fabricated Ensete fibers/UP composites of different alkali treatment ratio (A: untreated, B: 2.5% NaOH tr, C: 5.0% NaOH tr, D: 7.5% NaOH tr).

12.7 mm × 3 ± 0.5 mm according to ASTM D790-10 with a crosshead speed of 2 mm/min.

**2.2.4.2. Scanning electron microscope (SEM).** The morphology and microstructure of tensile fractured specimens of treated Ensete fibers/UP and untreated Ensete fibers/UP composites were carried out through SEM to understand the effect of treatment on interfacial bonding.

**2.2.4.3. Dynamic mechanical analysis (DMA).** Storage modulus ( $E'$ ), loss modulus ( $E''$ ) and  $\tan \delta$  value of untreated and treated Ensete fibers/PU were evaluated through DMA Q800 model of TA instruments with single cantilever under multi frequency-strain module having the dimensions of 17.5 mm × 13.5 mm × 3 mm and temperature was range from 30 °C to 150 °C with 5 °C/min rate.

### 3. Results and discussions

#### 3.1. Evaluation of fiber properties

##### 3.1.1. Surface morphology of alkali treated Ensete fibers

SEM images of the surface and cross-sectional view of untreated and alkali treated Ensete fibers are presented in Fig. 3. From the Fig. 3 it is evident that the surface of untreated Ensete fibers is smooth resulting weak interfacial bonding with UP matrix, however the alkali treatment increase the roughness of fiber surface. From the cross-sectional images it was also observed that the alkali treatment decreased the fiber lumen size due to swelling of the fibers. Moreover, the surface morphologies of alkali treated Ensete fiber shows that the pectin, lignin and hemicellulose (binder) were removed leading to fibrillation and breakdown of the fiber bundle into elementary fibrils. Nakano et al. [23] declared the mechanism in longitudinal contraction of microfibrils related with the reduction of cellulose chain segments in the amorphous phase due to due to alkali penetration into the cellulose structure in the

longitudinal direction of fiber.

##### 3.1.2. Fiber tensile tests

In this subsection, the single fiber linear density and tensile strength was measured for untreated and treated Ensete fibers as shown in Table 2. The weight loss of Ensete fiber after treatment with different alkali concentrations was measured and linear density was given for comparison. Linear density is merely the weight of the fixed length of the fiber. It is used to avoid the ambiguity of variations in diameter and thickness of Ensete fibers observed due to the removal of waxes and other impurities. From the results, there is a decrease in linear density and fineness of Ensete fiber after alkali treatment when compared with untreated fiber. This is due to removal of waxes and other impurities and also reduction of cementing materials such as pectins, lignin and hemicelluloses in cell wall of fibers. Eventhough the variations of alkali concentrations from 2.5% to 7.5% resulted in slight changes in linear density, the fibrillation of single Ensete fiber at higher concentrations have its own effects on composite properties. Alkalinization of natural fibers leads to the fibrillation effect, which is splitting of a single-fiber bundle into smaller ones, that increase the effective area for mechanical interlocking between fibers and matrix, thus leading to improved interfacial bonding [9]. For the natural fiber reinforced composites, there shall be an optimum NaOH concentration level and fiber loading percentage for better tensile and flexural strength. The untreated Ensete fiber had a tensile strength of 552 MPa and Young's modulus of 28.75 GPa. The alkali treatment provides higher mechanical performance in all concentrations. After 5.0 wt% NaOH treatment, the tensile strength of Ensete fiber increased by 88% and Young's modulus by 64% with respect to the untreated Ensete fiber.

##### 3.1.3. FTIR spectroscopy of treated and untreated fibers

The FTIR spectra of untreated and treated Ensete fibers are shown in Fig. 4. The Figure shows that the main peaks are originated from the backbone of cellulose, i.e. cellulose, hemicellulose, and lignin. The FTIR

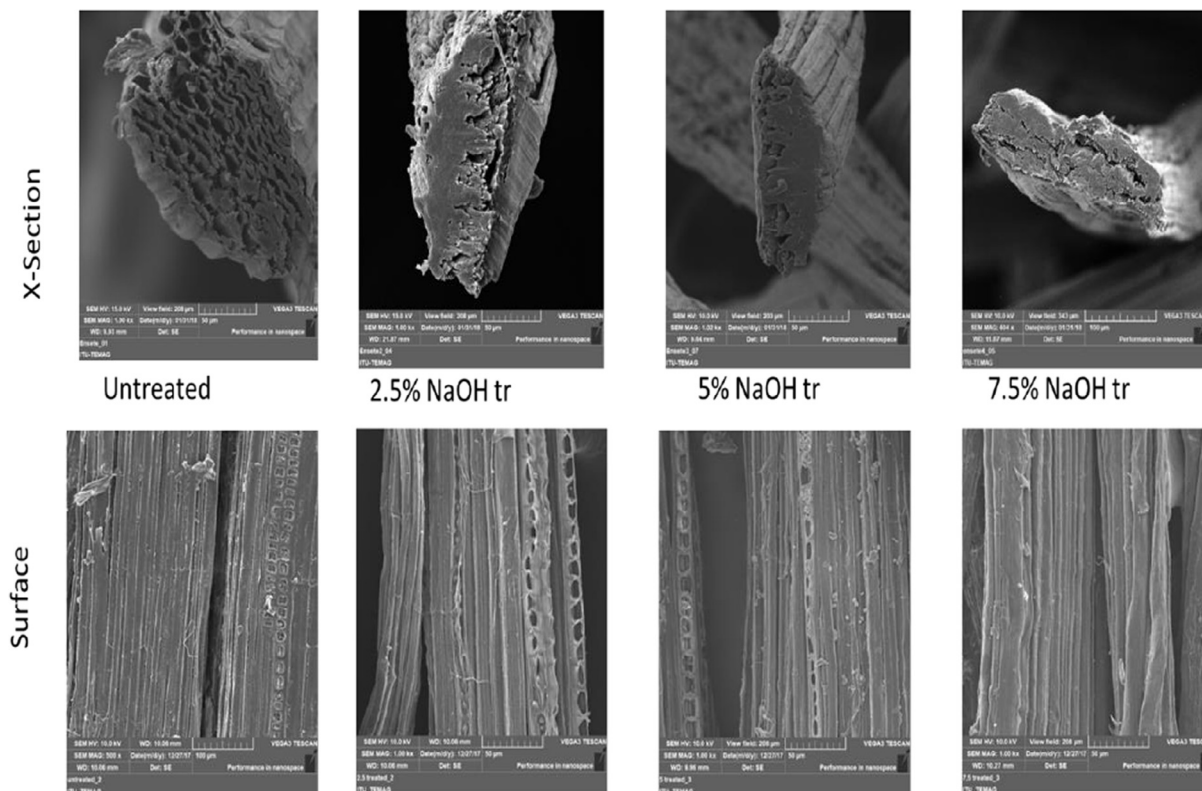


Fig. 3. SEM images of untreated and treated Ensete fibers.

**Table 2**  
Untreated and treated Ensete fiber mechanical properties.

Composites	Linear Density (tex) $\pm$ SD	Tensile Strength (cN/tex) $\pm$ SD	Young's Modulus (N/tex)	Extension (%)
Untreated Ensete fiber	16 $\pm$ 1.2	56.5 $\pm$ 6.5	29.42	1.92
2.5% NaOH treated Ensete fiber	11 $\pm$ 0.7	76 $\pm$ 12	46.62	1.63
5.0% NaOH treated Ensete fiber	10 $\pm$ 0.8	105 $\pm$ 9	68.18	1.54
7.5% NaOH treated Ensete fiber	7.6 $\pm$ 1	139 $\pm$ 19	63.47	2.19

$\pm$  Standard deviation.

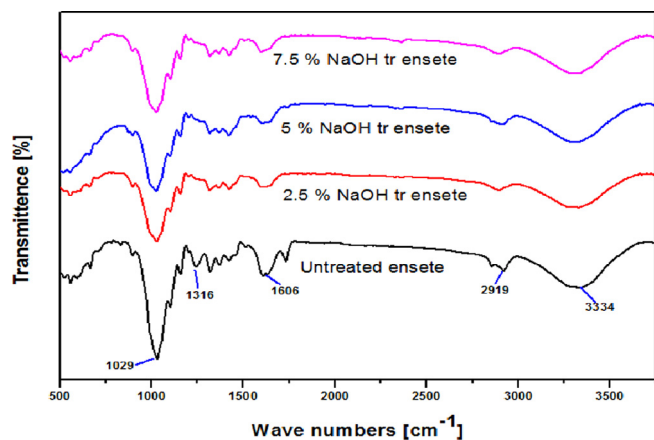


Fig. 4. The FTIR spectra of the untreated and treated Ensete fibers.

spectra of Ensete fibers indicate the broad transmittance peak at 3200–3600  $\text{cm}^{-1}$  range representing O–H stretching of hydrogen bond network [21], while C–H stretching vibration of methyl and methylene groups in cellulose and hemicellulose corresponds to the peaks at 2910  $\text{cm}^{-1}$ . Peaks at wavelength number 1739  $\text{cm}^{-1}$  shows the stretching of C=O bonds in carboxylic acid and ester components of hemicellulose. This peak clearly observed in raw Ensete, but vanished upon treatment. For all treated Ensete fibers, the C=O stretching vibration of hemicelluloses disappeared which might be because of dissolution of hemicelluloses in alkaline solution. Similar observation was reported for alkali treated cellulosic natural fibers by other researchers [24–26]. In addition to the main peaks, there are distinct peaks obtained for the surface-modified Ensete fibers, which vary in its transmittance intensity from untreated fibers. Consequently, the transmittance bands at 1464  $\text{cm}^{-1}$  and 1376  $\text{cm}^{-1}$  assigned to  $-\text{CH}_3$  asymmetric and C–H symmetric deformations of lignin are also weakened when compared with untreated Ensete fiber. The absorption peaks of lignin which showed lower intensities were due to the reduction of lignin by alkali treatment. The absorption peak in the region of 1030–1150  $\text{cm}^{-1}$  was attributed mainly to the C–O–C and C–O stretching of primary and secondary hydroxyl group in the cellulose, lignin and glycoside linkage [27]. The decrease in intensity of absorbance upon alkali treatment of fiber indicates a reduction in bond stretching of C–O in cellulose and lignin of Ensete fiber. However, the characteristic peak at 1420  $\text{cm}^{-1}$  is for  $-\text{CH}_2$  symmetric bending of cellulose which remained unaffected after alkali treatment and it might be playing an important role in the increase of Ensete fibre tensile strength.

### 3.2. Evaluation of untreated and treated Ensete reinforced UP composites

#### 3.2.1. Tensile properties of the composites

The alkali treated Ensete fiber composites showed a higher tensile strength when compared with untreated are shown in Table 3. The composite with untreated Ensete fiber having a tensile strength of  $18.97 \pm 1.59$  MPa was increased by 20, 18 and 14% when the alkali concentration of fiber treatment is 2.5, 5.0 and 7.5% respectively.

Alkali concentration of 2.5% resulted in the highest tensile strength of  $22.81 \pm 2.67$  MPa but less tensile modulus of  $597.37 \pm 33.25$  MPa, when compared with 5.0% alkali treated composite having  $758.4 \pm 12.09$  MPa (Fig. 5). It can be explained by the fact that the treatment improves the fiber surface roughness which in turn improves the interfacial interaction between the Ensete fiber and UP matrix.

However; further conclusions can be developed based on the fiber orientation in the composites. The Ensete fibers are randomly oriented by carding of chopped fibers and only the fibers which are oriented along the axis of tensile loading provided reinforcement to the composites. Myslamsy and Rajendran [28] also observed dependency of fiber matrix adhesion and fiber length and orientation, which determines the tensile properties of composites.

#### 3.2.2. Flexural properties of the composites

Table 4 shows that alkali treatment improves both flexural strength and modulus of Ensete fiber/UP composites. The flexural strength of untreated Ensete fiber composites increased from 56.60 MPa to  $59.53 \pm 4.39$  MPa,  $64.89 \pm 8.91$  MPa, and  $62.80 \pm 8.27$  MPa when treated by 2.5%, 5.0%, and 7.5% NaOH concentration respectively (Fig. 6). Similar works have been reported where alkali treatment of cellulosic natural fibers improved tensile strength of composites with thermoset resins (epoxy, polyester).

Furthermore, the flexural strength and modulus of 5.0% NaOH treated composite are 64.89 MPa and 2823.33 MPa respectively which is the optimum treatment based on its better performance. The possible reason for this specific concentration of NaOH is the improved interfacial adhesion of the fiber and matrix by allowing the resin to penetrate into the fibers bundles and fibrils. This resulted from a better contact and the increase in the area of contact between the fiber and the PU. Haque et al. [29] also reported in literature that increased fiber–matrix adhesion chiefly due to effective stress transfer between them. The micrographs from SEM also supported that fibers have well adhered to the matrix. In this study we assumed that the reinforcing fiber distribution and networking is similar in all samples since Ensete fiber carding and webs lay up was performed carefully where the weight fractions of the fibers was also kept constant. However, there are factors such as filler networks that affects analyzing of the mechanical performances of composites in addition to stress transfer from fiber matrix adhesion. Aspect ratio of composite fillers and its shape are also reported as one of the key factors which play an important role in determining the properties of the composites specially nanocomposites [30,31].

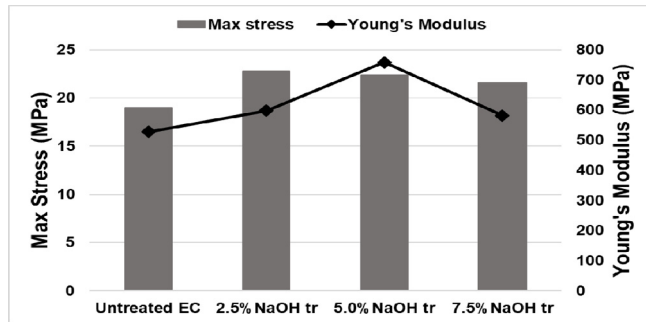
#### 3.2.3. Fracture surface morphology of composites

Fig. 7(a) and (b–d) displayed the fractured surface morphologies of untreated and treated Ensete fibers/PU composites. From the Fig. 7(a) it is evident that SEM shows large number of long fiber pullouts without matrix adhered to its surface, indicating a poor fiber–matrix interfacial adhesion. Hence the occurrence of tensile failures in composites was ascribed on the basis of fiber/matrix de-bonding and matrix fracture. SEM micrographs of each alkali treated Ensete fiber composites showed an improvement in fiber–matrix adhesion. Though, even at 2.5% NaOH concentration there were changes on fiber surface, we still observed short single fiber pullouts with some matrix adhered to its surfaces and

**Table 3**  
Tensile properties of untreated and treated Ensete composites.

Samples	Max. Force (N)	Max. Stress (MPa)	Max. Stroke (mm)	Strain (%)	Young's Modulus (MPa)
Untreated EC	486.43 ± 84.61	18.97 ± 1.59	2.28 ± 0.52	6.71 ± 1.29	528 ± 23.43
2.5%NaOH tr	698.59 ± 72.69	22.81 ± 2.67	3.22 ± 0.43	8.05 ± 1.07	597.37 ± 33.25
5.0%NaOH tr	573.19 ± 72.25	22.39 ± 1.08	2.27 ± 0.22	5.69 ± 0.58	758.4 ± 12.09
7.5%NaOH tr	583.17 ± 93.42	21.61 ± 3.33	2.26 ± 0.42	5.63 ± 1.05	582.1 ± 61.67

Where, ± = standard deviation.



**Fig. 5.** Tensile strength and modulus of untreated and treated Ensete composites.

bundles of Ensete fibers adhered to each other. At 5.0% NaOH the compatibility between fiber–matrix was attained and showed better resin–matrix adhesion, resulting failure occurs after sufficient stress transfer between matrix and fiber. Even though better fiber–matrix adhesion was showed for 7.5% alkali concentration, it also resulted in some of the weak points on single fibers since the fiber fibrils were almost totally revealed. The tensile failure occurred at these weak points. The SEM micrographs are also in agreement with the experimental results discussed in other subsections. Comparatively similar findings are also reported in literature. In one study, Mahjoub et al. [32] have compared the effects of treating kenaf fibers at different alkali concentrations (5, 7, 10 and 15%). They concluded that 10% and 15% solution resulted in more twisted, much finer and brittle fiber than untreated fiber with tension on fiber texture and structure which in turn lead to easy damage. Reserachers in other work treated the abaca fiber with 5,10 and 15% alkali and its effect on fiber–matrix adhesion [19] were investigated. The SEM images revealed that at higher alkali concentration, more twisted and fibrillated abaca was observed. Due to this fibrillation, binding of epoxy resin to gaps between fibers was reported to be poor when compared to abaca fiber bundle showing weaker fiber–matrix adhesion at 10 and 15% NaOH treatments.

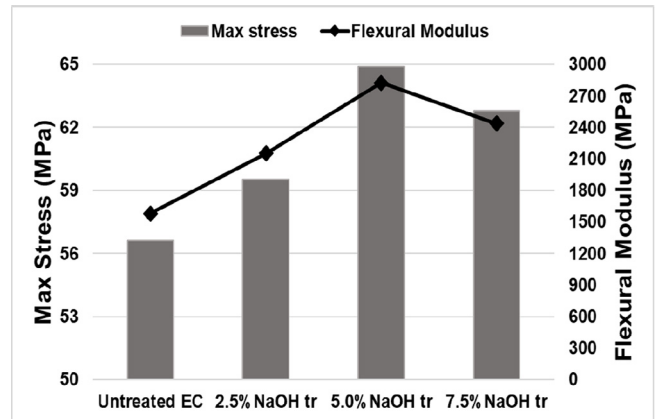
**3.2.4. Dynamic properties of composites**

**3.2.4.1. Storage modulus (E').** E' typically related to Young's modulus and are associated with "stiffness" of a material [33]. The variation of the E' as a function of temperature is given in Fig. 8. The DMA results showed an increase in E' indicating higher stiffness for treated Ensete fibers/UP as compared with untreated Ensete fibers/UP composites. The value of E' was found to be higher for treated fiber composites (i.e. 750 MPa, 1412 MPa and 874 MPa for 2.5%, 5.0% and 7.5% NaOH

**Table 4**  
The flexural properties of untreated and treated Ensete composites.

Samples	Max. Force (N)	Max. Stress (MPa)	Strain (%)	Flexural Modulus (MPa)
Untreated EC	170.21 ± 34.89	56.60 ± 9.65	3.59 ± 0.41	1579.66 ± 268.8
2.5%NaOH tr	147.34 ± 16.91	59.53 ± 4.39	2.81 ± 0.23	2152.66 ± 156.63
5.0%NaOH tr	126.67 ± 48.38	64.89 ± 8.91	2.35 ± 0.38	2823.33 ± 380.75
7.5%NaOH tr	94.67 ± 36.67	62.81 ± 8.27	2.66 ± 0.91	2438 ± 312.19

Where, ± = standard deviation.



**Fig. 6.** Flexural strength and modulus of untreated and treated Ensete composites.

treated fiber composites, respectively) than untreated fiber composites (i.e. 678 MPa) at low temperature. It can be explained on account of hydrogen bonding resulting from the hydroxy groups to make close packing of the cellulose chain in the treated Ensete fibers. Thus the reinforcement of treated fibers induced better stress transfer and fiber-UP interaction when compared with that of untreated fiber composite [34]. The E' of the composites reinforced with untreated/alkali treated Ensete fibers at 25 °C is also comparable with the flexural modulus, shown in Table 4.

The composite samples shows a considerable decreasing trend in the E' over temperature. This changes in E' showed considerable fall at temperatures between 75 and 100 °C for the composites studied. These temperatures are in the range of Tg where a sharp decrease in E' value was observed when material undergoes transition from glassy to rubbery stage [35]. Treated Ensete fiber composites with 5.0% NaOH showed the highest E' (1412 MPa), which is 108% higher than that of untreated Ensete fiber composites. However, a lower E' than 5.0% NaOH was obtained for the composites with 7.5% NaOH treated Ensete fibers (i.e. 874 MPa). At higher concentration, the decrease in E' can be described by the removal of hemicellulose and lignin at the inter-fibrillar region, making fiber less dense and brittle. Manalo et al. [20] investigated that substantial delignification and degradation of long-chain cellulose molecules at higher concentration (8% NaOH) resulted lower mechanical properties of composites due to damaged bamboo fiber weakening the load transfer and the bonding with the matrix. Thus, it can be concluded that 5.0% NaOH treatment with Ensete fibers reduces fiber breakage and provides optimal dynamic properties for the

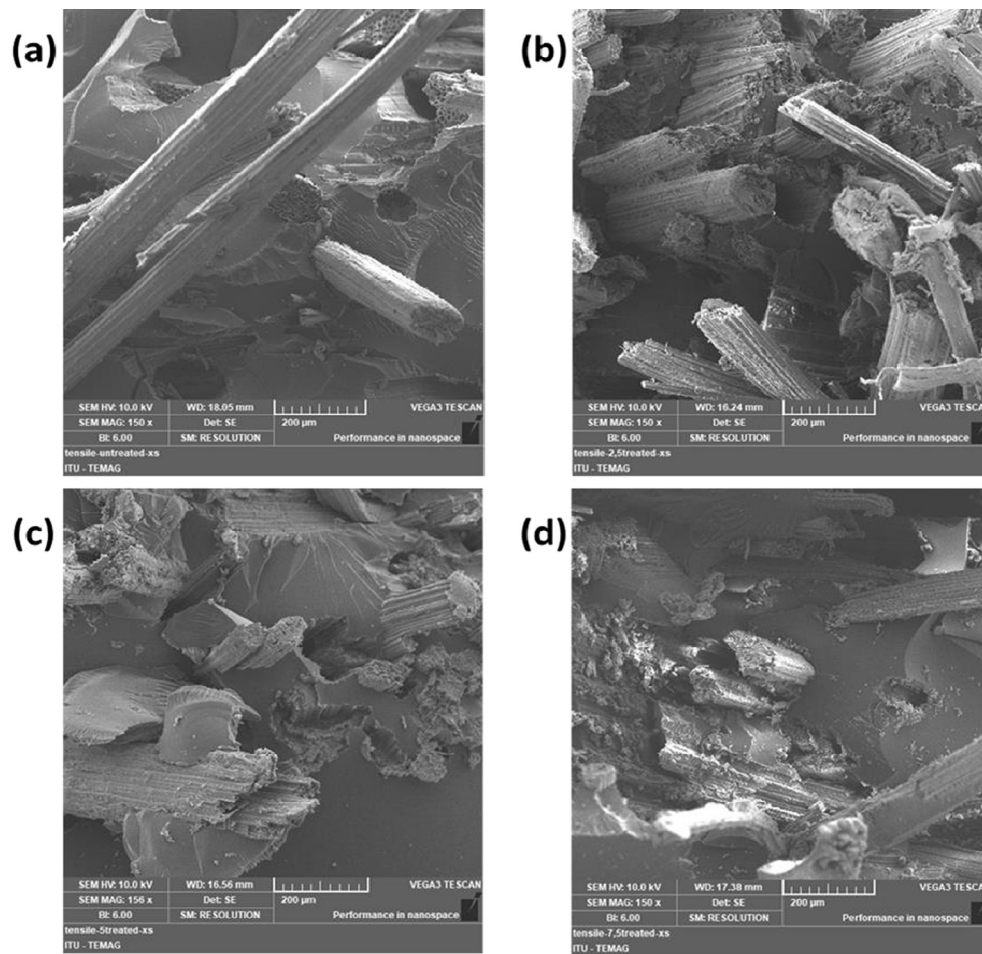


Fig. 7. SEM images of fractured surfaces of Ensete composites with different alkali treatment ratio (a) untreated, (b) 2.5%NaOH tr, (c) 5.0%NaOH tr (d) 7.5%NaOH tr.

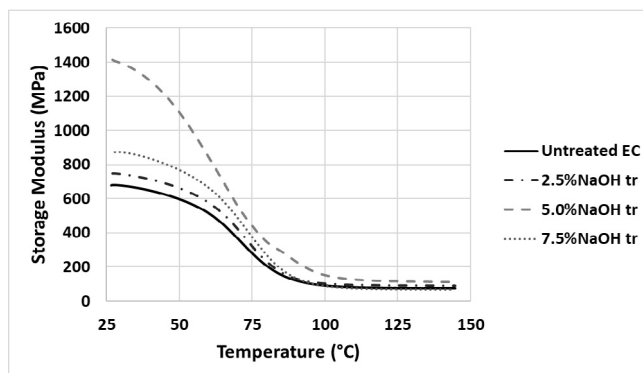


Fig. 8.  $E'$  of untreated and treated Ensete composites.

composites.

**3.2.4.2. Loss modulus ( $E''$ ).** Dynamic  $E''$  usually associated with “internal friction” and defined viscous response of the materials along with different types of molecular motions, transitions, relaxation processes, morphology and structural heterogeneities [33,36]. Table 5 presents the  $E''$  and  $\tan \delta$  data of untreated and treated Ensete composites. The variation of  $E''$  of Ensete fiber and its surface treated composites as a function of temperature is given in Fig. 9. The  $E''$  graph showed the gradual increase of  $E''$  value until its peak value at  $T_g$ . In the region of  $T_g$  molecular segmental motions are activated, however, motions occur with difficulty and these can be described as the

molecular friction that dissipates much of the force [37]. Corresponding to the  $E''$  peak of treated Ensete fibers/UP, which shift to higher temperature region with relative to the untreated reinforced UP composites, most probably due to the restricted segmental motion of the amorphous UP chains at the fiber matrix interface.

Fiore et al. [38] also found that mercerization of cellulosic natural fibers (kenaf) improved the  $E''$  of the composites material at higher temperature. Similarly, surface modification of Ensete fiber with NaOH resulted finer fiber with an increased number of  $-OH$  groups on their surface. The stronger fiber matrix bonding resulted in higher  $T_g$  of treated Ensete fiber composites. Hence the  $T_g$  of Ensete fiber composites showed an increasing trend (74.27 °C, 75.56 °C, 76.35 °C and 85 °C) for untreated, 7.5%, 2.5% and 5.0% NaOH treated fiber composites respectively (Table 5). The 5.0% NaOH treated Ensete fiber reinforced UP composites showed the maximum  $T_g$  value of 85 °C. Thus alkali concentration of 5.0% was found optimum and resulted in the best dynamic properties for Ensete fiber composites.

**3.2.4.3. Damping factor ( $\tan \delta$ ).**  $\tan \delta$  or mechanical damping factor is calculated from ratio of  $E''/E'$  [39]. Researchers reviewed that a high  $\tan \delta$  value indicates that material possess high non-elastic strain component while a lower value revealed high elasticity, as the molecular chains mobility get decreases at the fiber/matrix interface [39]. The variation of  $\tan \delta$  values measured over a range of temperatures for untreated and alkali treated Ensete fiber composites are shown in Fig. 10. From the graph, the  $T_g$  of untreated Ensete fiber composites attained early before others.  $T_g$  is the temperature at which the damping or the  $E''$  attain their maximum values and from the graph,

**Table 5**  
E'' and Tan  $\delta$  data of untreated and treated Ensete composites.

Composites	Peak E'' MPa	Peak E' MPa	Peak height of Tan $\delta$	Tg from Tan $\delta$ curve (°C)	Tg from E'' curve (°C)
Untreated EC	71.50 $\pm$ 2.33	678 $\pm$ 4.65	0.332 $\pm$ 0.005	85.03	74.27
2.5% NaOH tr	81.85 $\pm$ 3.06	750 $\pm$ 3.45	0.375 $\pm$ 0.008	85.78	76.35
5.0% NaOH tr	99.70 $\pm$ 1.26	1412 $\pm$ 4.17	0.370 $\pm$ 0.011	89.77	85
7.5% NaOH tr	89.45 $\pm$ 2.65	874 $\pm$ 3.56	0.372 $\pm$ 0.009	87.48	75.56

Where,  $\pm$  = standard deviation.

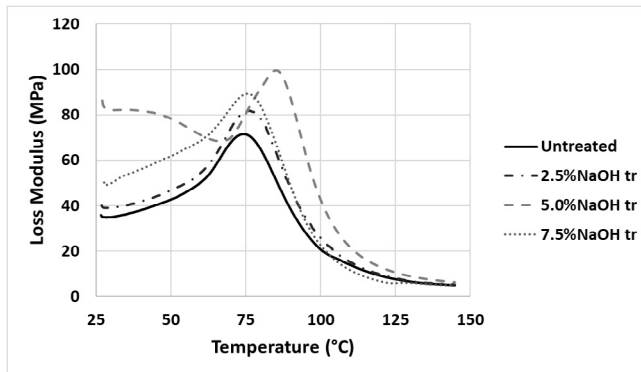


Fig. 9. E'' of the untreated and treated Ensete composites.

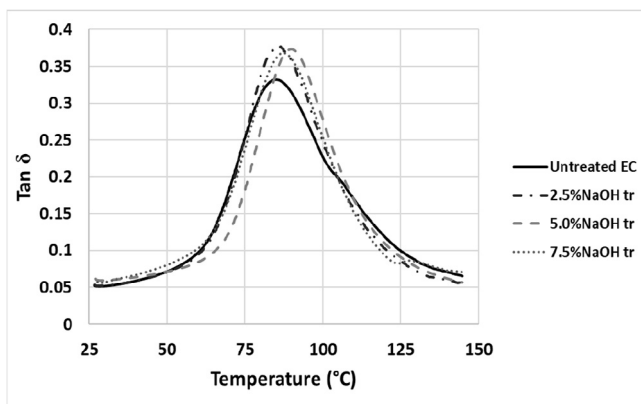


Fig. 10. Tan  $\delta$  of the untreated and treated Ensete composites.

untreated Ensete fiber composites attained it early and its damping factor started to decrease. At its rubbery state the molecular segmental motion are more resulting lower damping factor. However, alkali treated fibers attained their maximum damping when untreated one is already in the rubbery state. As shown in Table 5 maximum Tan  $\delta$  values of 2.5%, 5.0% and 7.5% NaOH treated Ensete fiber composites are (0.375, 0.370 and 0.372) respectively. Even though a little difference was observed in damping values among alkali concentration used to treat Ensete fiber the maximum decrease in damping was found for 5.0% NaOH. Additionally, shifting of Tg values to right revealed the better interfacial adhesion due to alkali treatment of the fibers [40]. For 5.0% NaOH treated Ensete fiber composites was observed at 89.77 °C (the highest Tg value) which indicates optimum alkali concentration for better fiber matrix interface adhesion.

#### 4. Promising applications of Ensete fibers/UP composites

Comparable, acceptable and satisfactory mechanical properties in terms of tensile strength, tensile modulus, flexural strength, flexural modulus and dynamic mechanical properties such as E', E'', Tg and damping factor of 5.0% alkali treated Ensete fibers/UP composites with respect to other natural fibers reinforced polymer composites like jute,

kenaf, oil palm and hemp composites governs certain promising and potential applications where dynamic mechanical performances along with the prime benefits of renewability, technical, environmental and economical issues. The most feasible industrial applications might include automotive, aerospace, constructional like hurricane-resistant housing and exterior designing together with an efficient and sustainable waste management with minimal repairing costs. Remarkably the 5.0% alkali treated Ensete fiber formulation can be used to fabricate automotive components like parcel shelves, dash boards, seat cushions, door trim panels, backrests and cabin linings leading to multi dollar business. They can also be used in non-structural applications like in packaging industries (egg shelves), consumer products and sports items (racket handle, bicycle frames).

#### 5. Conclusion

Present study investigates the effect of different concentration of alkali treatments (2.5%, 5.0%, and 7.5%) on Ensete fiber and its Ensete fibers reinforced UP composites. Mechanical and surface morphologies and FTIR spectroscopy of treated and untreated fibers and the Ensete fibers reinforced UP composites were carried out along with the dynamic properties of composites. FTIR of Ensete fiber revealed the variation of intensity in transmittance on the treated and untreated fiber. Tensile and flexural mechanical test indicate that the mechanical properties of Ensete fiber tend to increase as NaOH concentration increased. Additionally, the fineness of the alkali treated Ensete fiber was reduced due to the removal of some parts of wax, lignin, and hemicelluloses. SEM and cross-section analysis of the alkali treated fiber shows surface roughness and swelling of fiber lumen. Further alkali concentration shows fibril separation by removing binding components such as lignin and hemicelluloses. The tensile and flexural mechanical test shows that these properties were influenced positively by alkali treatment due to improved fiber–matrix interface. The flexural strength of 5.0% alkali treated Ensete fiber composites (~65 MPa) was improved by 14.5% compared to untreated composites. Similarly, a tensile modulus of 5.0% alkali treated Ensete fiber composite has ~758 MPa which is 43.5% higher than the untreated composites. The flexural modulus and E' of both untreated and alkali treated Ensete fiber reinforced UP composites showed a similar trend in increments and these results are good implications for conclusions. Moreover, composites from Ensete fibers treated with 5.0% NaOH showed the highest E' of 1412 MPa, which is 108% higher than that of untreated Ensete fibers UP composites. Thus both static and dynamic mechanical properties were found highest for 5.0% alkali treatment concentration. Overall it can be concluded that mechanical and dynamic properties of Ensete fibers reinforced UP composites have satisfactory and comparable properties to other lignocellulose fiber reinforced composites hence are suitable for further commercial fabrication as promising green composites.

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## Conflict of interest

None.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.compstruct.2018.09.043>.

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