



Original Research Article

Characterisation of Untreated and Natural Latex Treated *Borassus aethiopum* Palm Leaf Stalk Fibre

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ABSTRACT

The use of lignocellulosic fibres as reinforcement in polymer matrices is increasing daily because they address ecological and environmental issues such as recyclability, renewability and non-toxicity. In this work, fibres were extracted from Borassus aethiopum leaf stalk through the combination of water retting and mechanical method followed by natural plant latex treatment. The fibre was characterised using Fourier transform infrared (FTIR) spectroscopy, single fibre strength test and scanning electron microscopy (SEM). The SEM revealed striated grooves on the treated fibre surface, while FTIR showed improved reactivity of fibres due to the introduction of some active functional groups from natural plant latex onto the fibre surface. It was concluded that latex treatment improved fibre–matrix interfacial bonding through mechanical keying of the matrix on striated grooves and fibre matrix reaction at active sites on the fibre surface.

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1. INTRODUCTION

Borassus aethiopum (Figure 1a), with English names Ron palm, Borassus palm, African fan palm, fan palm, deleb palm, is known in Nigeria among the Hausa, Igbo and Yoruba as *giginya*, *ubiri* and *agbon-eye*, respectively (Orwa *et al.*, 2009). It is highly rich in lignocellulosic content, and there are many reports on the chemical composition of various parts of this palm from base to crown. The chemical composition of various parts of the tree has shown that the palm is rich in cellulose, hemicellulose and lignin (Ali *et al.*, 2010; Acheampong, 2014; Ngargueudedjim *et al.*, 2015a; Ngargueudedjim *et al.*, 2015b). These components are considered when lignocellulosic fibres are used as reinforcement in polymer matrices. Hence, their modifications or interactions with different polymers require the identification of their corresponding absorption bands by the use of Fourier transform infrared (FTIR) spectroscopy (Zafeiropoulos, 2011).

Cellulose is the major framework component of natural fibre structure that provides strength, stiffness and stability (Kabir *et al.*, 2012). Its molecular structure (Figure 2a) consists of three hydroxyl groups, two of which form hydrogen bonds within the cellulose (intramolecular), and the other forms hydrogen bond with

other cellulose (intermolecular) (Leonard and Martin 2002; Kabir *et al.*, 2012). Hemicellulose (Figure 2b) is a polysaccharide that occurs mainly in the primary cell wall, with branched polymers containing five and six carbon sugars (Bledzki and Gassan, 1999; Kabir *et al.*, 2012). Aromatic ring structure is the primary chemical constituent in lignin, and these compounds are extremely diverse and present in many forms within the plants and their cell walls. Lignin is a polyphenylpropanoid complex that arises from one or more of the following alcohols (Figure 2c): p-coumaryl (no methoxyl groups ($-O-CH_3$) on the aromatic ring), coniferyl (one methoxyl group at the 3' position) or sinapyl (two methoxyl groups at 3' and 5' positions) (Mussig, 2010).

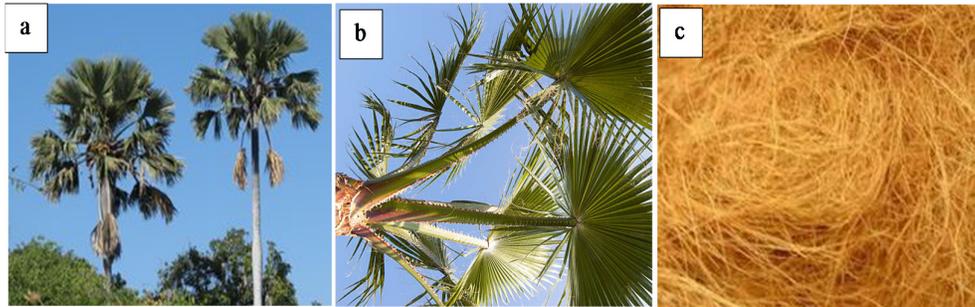


Figure 1: *Borassus aethiopum* (a) trees, (b) leave stalks and (c) extracted fibres

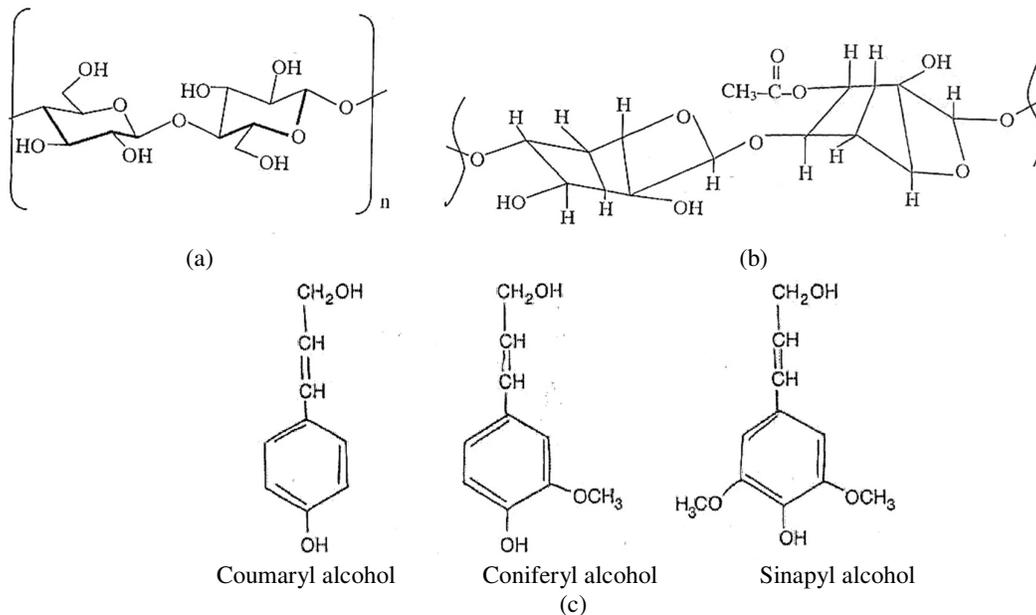


Figure 2: Chemical structure of (a) Cellulose (b) Hemicellulose and (c) Lignin

Insight into the physico-chemical properties and mechanical behaviour of lignocellulosic fibres is required to optimise their usage as reinforcements in polymer matrices (Alawar *et al.*, 2008). The performance of lignocellulosic fibres as reinforcement in polymeric materials depends on fibre properties such as strength, stiffness and the interfacial bonding between fibre and polymer matrix. The quality of the fibre–matrix interface is significant for the application of lignocellulosic fibres as reinforcement for polymers. Therefore, lignocellulosic fibre modifications are considered in modifying the fibre surface properties to improve their adhesion with different matrices (Faruk *et al.*, 2012). Since stress is transferred between matrix and fibres across the interface, good interfacial bonding is required to achieve optimum reinforcement and composite

properties. The success of the bonding depends on the modification method efficiency, which could be either physical or chemical. The surface of the lignocellulosic fibre is influenced by fibre composition, fibre modification method (chemical or physical) and processing conditions. Various types of chemical and physical fibre surface modification have been used to enhance natural fibre surface properties. Shukor *et al.* (2013) investigated the effect of NaOH concentration (3%, 6% and 9%) for treating kenaf fibre used as reinforcement in poly lactic acid matrix bio-composite. They found that 6% alkali treatment at ambient temperature is the optimum condition to obtain maximum flexural modulus and impact strength of the composite. Haque *et al.* (2015) investigated the effects of acetylation, acrylation, silanisation, alkalisation and permanganate treatment on physical, chemical and mechanical parameters of sisal/polyester composite. From their findings, they concluded that treatments such as acetylation, acrylation and silanization can increase interfacial strength, wetting and compatibility between fibre and matrix. Amirou *et al.* (2013) modified date palm fibre by using corona discharge treatment (a physical modification method), which results in surface oxidation. They showed a significant increase in tensile strength and young modulus compared to the composites with untreated fibres, and concluded that the interfacial contact between both components improved because of a higher mechanical anchorage enhanced as a consequence of the etching effect. Han *et al.* (2006) irradiated natural fibres with an electron beam to improve adhesion between the fibres and thermoplastics and demonstrated that electron beam irradiation is effective in both impurity removal and functional group development on the surface of natural fibres for better bonding between natural fibre and the polymer matrix. The main disadvantage of natural fibres as reinforcements in composites is the poor compatibility between fibre and matrix due to their relatively high moisture absorption property as a result of hydroxyl (-OH) group that is present between the macromolecules in the fibre cell wall. When moisture from the atmosphere comes in contact with the fibre, the hydrogen bond breaks and hydroxyl groups form new hydrogen bonds with water molecules. The cross-section of the fibre becomes the main access to water penetration. The interaction between hydrophilic fibre and hydrophobic matrix causes fibre swelling within the matrix. This results in weakening the bond strength at the interface, which leads to dimensional instability, matrix cracking and poor mechanical properties of the composites. The quality of fibre-matrix interface is significant for application of natural fibres as reinforcement fibres for plastics. Therefore, natural fibre modifications are considered in order to improve their adhesion with different matrices (Faruk *et al.*, 2012). Additionally, most natural fibres have low degradation temperatures (~200°C), which make them incompatible with polymers of higher melting temperatures. This also restricts natural fibre composites to relatively low-temperature applications (Sgriecia *et al.*, 2008). Physical and chemical methods can be used to optimise this interface. These modification methods are of different efficiency for adhesion between the matrix and the fibre (Bledzki and Gassan, 1999).

The fibre-polymer interface serves as the means of stress transfer in a polymer matrix composite. The extent at which fibre and polymer bind together is influenced by the morphology and composition of the fibre, reactive sites available and the polarity of the matrix resin. Therefore, it is pertinent to study ways of improving the interfacial bonding in fibre/polymer composites. To achieve this, numerous coupling agents have been used. This work uses the natural plant latex to accomplish improvement in the interfacial bonding of epoxy/Borassus palm leaf stalk fibre composite. The aim of the present study was to characterise the natural latex treated and untreated *Borassus aethiopum* palm leaf stalk fibre by Fourier infrared spectroscopy, single fibre tensile strength and morphological examination.

2. MATERIALS AND METHODS

2.1. Materials and Equipment

The materials and equipment used in this work include: Borassus palm leaf stalk fibre extracted from Borassus palm leaf stalk, obtained from a Borassus palm (*Borassus aethiopum*) tree in Dorayi area of Panhauya (11° 7' N, 7° 37' E) Giwa Local Government, Kaduna State, Nigeria, natural plant latex, tap and distilled water, digital weighing balance (Mettler, AE 160 Model: 8606, Zurich, Switzerland), oven (Model:

60628 Cole and Palmer, London, UK.), sodium hydroxide pellets (Fishers Scientific Company, Fair Lawn, New Jersey, U.S.A.), glacial acetic acid (99% American Chemical Society), attenuated total reflectance infrared spectrometer (Agilent Technologies, Santa Clara, California, U.S.A) optical microscope (NJF – 120A, Zhejiang, China), Zwick/Roell fibre testing machine (BDO – FBO-2005/s, Ulm, Germany).

2.2. Methods

2.2.1. Fibre extraction

A combination of water retting and mechanical method was used to extract *Borassus aethiopum* palm leaf stalk fibre (Figure 1c) from the leaf stalks (Figure 1b). This was achieved by submerging bundles of leaf stalks in water for 30 days after the thorns at the edges and the bark of the leaf stalk had been shaved. The retted stalk was dried in open air and stored for 7 days to dry. Finally, fibre was separated by breaking process in which the brittle woody portions of the retted stalks were broken by beating and scraping.

2.2.2. Fibre surface treatment

Surface pretreatment with sodium hydroxide (NaOH) was carried out by immersing the fibre in 6 wt.% solution of NaOH for an hour at room temperature (25 - 26°C), after which it was washed with distilled water and the effect of NaOH was neutralised with 2 % glacial acetic acid solution according to Ansell and Aziz (2004) and Thomas *et al.* (2004). The fibre was oven-dried for 3 hours at 110°C. The NaOH treated fibre was then given latex coating by dipping into a natural plant latex solution having 10% dry latex content, dissolved in water (Sreekala *et al.*, 2002). Thereafter, the fibre was oven-dried at 110°C for 3 h.

2.2.3. Single fibre tensile test

Single fibres were manually separated from the fibre bundles and cut to the length of 100 mm. The strength of the fibres were determined according to ASTM D – 3379 – 75. A gauge length of 50 mm and crosshead speed of 1 mm/min were employed. The machine is equipped with 500 N standard load cell. Specimens were gripped at both end on a Zwick/Roell fabric testing machine and pulled until failure, and load at fracture was then read on a screen (Alawar *et al.*, 2009). Five readings were taken for each specimen and the average value was recorded.

2.2.4. Single fibre diameter measurement

Optical microscope (model: NJF – 120A) at the Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria, Nigeria in conjunction with a computer software “Digimizer” at the Department of Anatomy, Faculty of Medicine, Ahmadu Bello University, Zaria, Nigeria was used to measure fibre diameter. Prior to the measurement, calibration was done using a laboratory slide. Each sample of fibre consisted of five specimens on which three adjacent readings were taken and averages recorded to the nearest 10 µm.

2.3. Fourier Transform Infrared (FTIR) Spectroscopy

The Fourier transform infrared (FTIR) spectra of untreated and treated fibres were obtained using the Agilent Technology attenuated total reflection spectrophotometer at the Multi-user Laboratory, Department of Chemistry, Faculty of Physical Sciences, Ahmadu Bello University, Zaria, Nigeria. This was done by clamping the sample tightly on the spectrophotometer and transmitting the IR through the probe. Spectra that corresponded to functional group stretching or vibration were captured on an attached computer and printed out for further analysis.

2.4. Surface Morphology

Scanning electron microscopy (SEM) was carried out on the surfaces of the treated and the untreated fibre. The test was carried out using Phenom Pro – X Scanning electron microscope Eindhoven, Netherlands. The samples were sputter coated with a thin layer of gold (5 nm) prior to being exposed to the electron beam to make it conductive for proper imaging. The image was captured on an attached computer monitor.

3. RESULTS AND DISCUSSION

3.1. Single Fibre Tensile Strength and Diameter

Figure 3 presents the results of single fibre tensile strengths and average fibre diameters of untreated and latex treated fibres. A comparison of the untreated and the treated fibres indicated that untreated fibre had the highest average fibre strength (σ_f) of 1250 MPa with an average diameter of 350 μm . Latex treated fibre exhibited a lower average strength at fracture of 820 MPa with an average diameter of 150 μm . Reduction in strength of treated fibre as compared to the untreated may be attributed to the loss of intracellular hemicellulose and cell-wall lignin between microfibrils during NaOH surface pretreatment, thereby destroying the packing in cellulose chain and causing disorder in the crystalline pattern (Zhu *et al.*, 2013). Reduction in the average diameter in treated fibre as compared to the untreated was as a result of the removal of a certain portion of alkali soluble amorphous matters such as impurities, waxes, oil, hemicellulose and lignin (Kabir *et al.*, 2012).

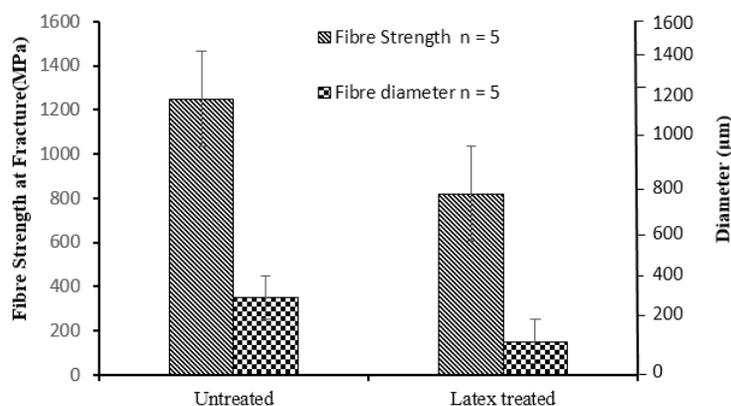


Figure 3: Average fibre strength at fracture and fibre diameter

The finding is in line with those of Singha and Rana (2012) and Zhu *et al.* (2013), that reported that aminopropyltriethoxysilane (APS) treatment destroys packing of the cellulose chain and cause disorder in the crystalline pattern. Thus, the result revealed that effect of surface modifications on lignocellulosic fibre strength to be used as reinforcements in composites is an important aspect that requires careful optimization. The fibre bulk structure may also be altered, especially when the treatment involves extended reaction times or aggressive chemicals. In such cases, increased interfacial strength may be observed when treated fibres are used as reinforcement in polymer matrix composites but a decreased in fibre strength are exhibited by fibres due to destroyed packing in cellulose chains that may result in crystalline pattern disorder in the fibre structure (Pickering, 2011).

Figures 4 and 5 and Tables 1 and 2 present the results obtained from attenuated total reflection (ATR) FTIR analysis. From Tables 1 and 2, it was observed that C – H stretch at 2922.2 cm^{-1} was present in all fibres. The carbonyl peak at 1714.6 cm^{-1} in the untreated fibre was seen to have been reconfigured in the treated

fibre. The removal of hemicellulose from the fibre surface caused this peak to disappear. The lignin peak at 1505.8 cm^{-1} in the untreated fibre was altered in the latex treated fibre. Latex treatment introduced amine and asymmetric and symmetric stretch = C – O – C, which could improve the reactivity of the fibre with the matrix. Sgriccia *et al.* (2008) reported that changes in peaks in the FTIR spectrum at 1730, 1625 and 1239 cm^{-1} indicated that the alkali treatment removes hemicellulose and lignin from natural fibre surfaces.

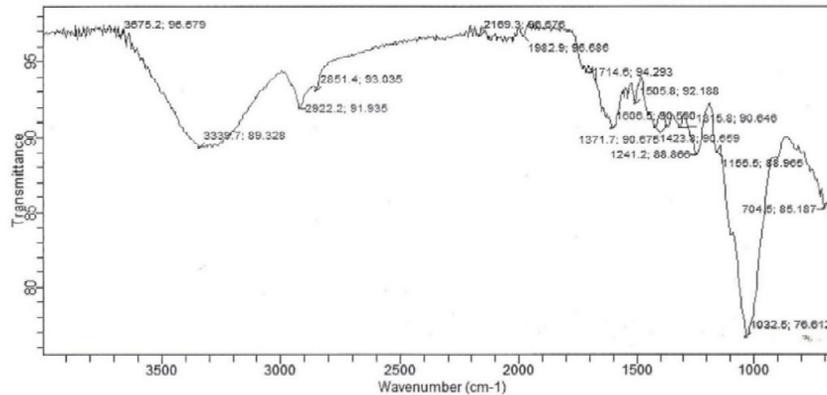


Figure 4: FTIR spectrum for untreated fibre

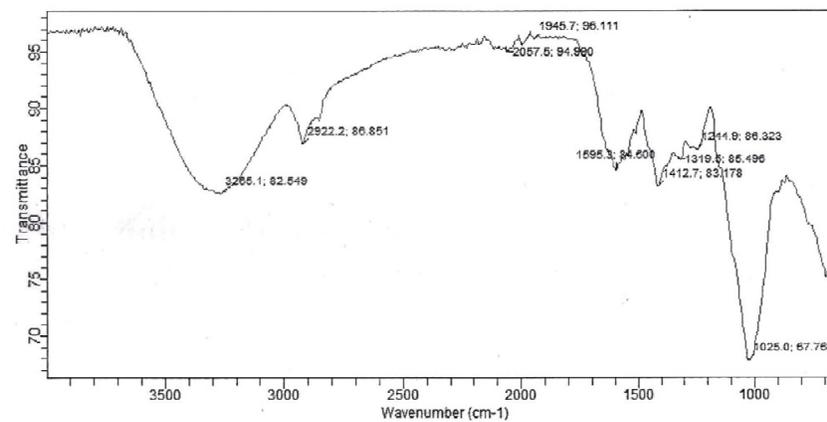


Figure 5: FTIR spectrum for latex treated fibre

Table 1: Untreated fibre functional groups, nature and types of vibration

Wave number (cm^{-1})	functional groups, nature and types of vibration
3675.2	Weakly bonded O – H stretch
3339.7	Hydrogen-bonded O – H stretch
2922.2	C – H Stretch
2851.4	C – H stretch off – C = O
2169.3	C \equiv C stretch HC \equiv C – CH ₃
1714.6	C = O stretch (R – C (O) – OH)
1606.5	C = C stretch
1505.8	N – H bend
1371.7	N – O stretch
1315.8	N = O bend

Table 2: Latex treated fibre functional groups, nature and types of vibration

Wave number (cm^{-1})	Functional groups, nature and types of vibration
3265.1	$\equiv \text{C} - \text{H}$ stretch ($\text{HC} \equiv \text{C} - \text{CH}_3$) propyne
2922.2	$\text{C} - \text{H}$ Stretch
2057.5	$\text{C} \equiv \text{C}$ ($\text{R} - \text{C} \equiv \text{C} - \text{H}$)
1595.3	$\text{C} - \text{C} = \text{C}$ symmetric stretch
1412.7	Aromatic ring OR $\text{N} - \text{H}$ bend
1319.5	$\text{C} - \text{N}$ Amines
1244.9	$= \text{C} - \text{O} - \text{C}$ asymmetric stretch
1025.0	$= \text{C} - \text{O} - \text{C}$ symmetric stretch
1099.6	$\text{N} = \text{O}$ stretch

3.2. Fibre Morphology

Figure 6 (a) and (b) is scanning electron microscopy of untreated and treated fibre respectively. Figure 6 shows that interfibrillar material, hemicellulose and lignin present in (a) were etched away in (b) by the NaOH treatment revealing striated grooves that serves as good sites for mechanical keying. This finding was in accordance with the result obtained by Sgriccia *et al.* (2008) who found that kenaf and hemp fibre morphology change after NaOH with SEM revealing the evidence of removal of pectin, waxy substances and natural oils covering the surface of fibre cell wall. They further stated that this makes the fibres surface rough by revealing the fibrils. Haque *et al.* (2015) reported that fibrillation occurs between fibre cells in alkali treated sisal fibre and that rougher surface was observed in SEM, of the treated sisal. Ansell and Aziz (2004) reported similar results by observing that SEM of hemp fibres showed a very clean surface after 6% NaOH treatment, and stated that wax, oil and surface impurities are observed on the SEM of the untreated fibre.

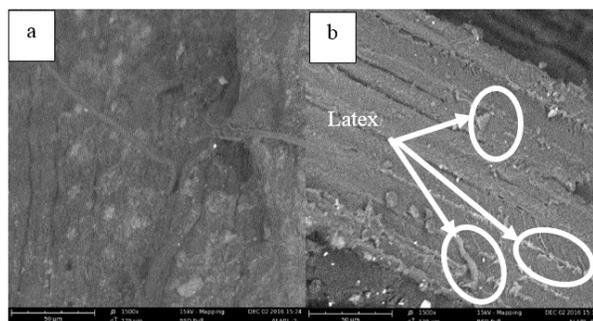


Figure 6: Scanning electron microscopy of fibre longitudinal surfaces morphology (a) untreated (b) latex treated

4. CONCLUSION

1. Latex treated fibre at 6 wt.% NaOH pretreatment for 1 hour showed a decrease in single fibre tensile strength and a corresponding reduction in fibre average diameter.
2. FTIR analysis revealed the removal of surface impurities, hemicellulose and lignin and the introduction of amine, which could improve the reactivity of the fibre with the matrix.
3. Fibre surface morphology revealed grooves and latex layers on the fibre surface after surface modification. Striations or grooves are good sites for mechanical keying, while latex layers serve as reactive sites with polymer matrices.

5. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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