The influence of alkali treatment on banana fibre's mechanical properties

Influencia del tratamiento alcalino sobre las propiedades mecánicas de la fibra de plátano

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RESUMEN

Este trabajo estudia el efecto del tratamiento alcalino sobre las propiedades mecánicas de la fibra de plátano (*Musa paradisiaca*). Las fibras fueron extraídas del seudotallo del plátano empleando una maquina desfibradora. Seguidamente fueron mercerizadas y modificadas mediante tratamiento alcalino con NaOH al 5% (m/v). El análisis morfológico evidenció que la rugosidad de la superficie es mayor en las fibras tratadas que en las no tratadas. La caracterización mecánica reveló que el módulo de Young, la resistencia máxima a la tracción y la deformación disminuyen con el aumento del diámetro de la fibra, tanto para las fibras tratadas como para las no tratadas.

Palabras clave: Fibra de plátano, Tratamiento alcalino, Propiedades mecánicas.

ABSTRACT

This work analyses the effect of alkali treatment on the mechanical properties of banana fibre (*Musa Paradisiaca*). Fibres were extracted from the pseudostem by a defibring machine; they were mercerised and modified by 5% NaOH (w/v) alkali treatment. Morphological characterisation showed that treated fibres' surface was rougher than that of untreated fibres. Mechanical characterisation indicated that Young's modulus, ultimate tensile strength and strain became decreased by increasing both treated and untreated fibres' diameter.

Keywords: Banana fibre, alkali treatment, mechanical property.

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Introduction

Natural fibres have appeared to be the materials which will become a feasible replacement for non-renewable, abrasive and expensive synthetic fibres during recent years, mainly due to their availability in large quantities, biodegradability, low cost, low density, recyclability and ease of manufacturing them (Oladele *et al*., 2010; Rout *et al*., 2001; Saheb and Jog, 1999). It is well known that natural fibres (i.e. banana fibres) have become an important reinforcement material over the last few decades because of their advantages over conventional materials. They supply to composite high specific stiffness, strength and biodegradability (Charlet *et al*., 2009; Herrera-Franco and Valadez-González, 2004; Ibrahim *et al*., 2010).

Banana fibre's lignocellulose nature means that it consists of several cells; such cells consist of cellulose, hemicellulose, lignin, pectin, wax and water-soluble components (Barreto *et al*., 2010; Bledzki and Gassan, 1999). The crystalline matrix consists of cellulose fibrils spirally wound in an amorphous matrix composed by hemicellulose, lignin and, in some cases, pectin (Zuluaga *et al*., 2009). The microstructure is extremely complex due to its hierarchical organisation and these materials' variable proportions in layers. They are distributed in two cell walls; the outer cell wall is made up of a single layer whilst the second one consists of three layers arranged as concentric cylinders with a small channel in the centre, called the lumen. The first wall is deposited during cell growth, thereby encircling the secondary wall which is a compound of three layers (Jayaraman 2003; Maya and Rajesh, 2008).

Fibre properties are determined by the physical, mechanical and chemical properties of its morphological constituents and their interfaces. Several studies have stated that fibre's mechanical properties are determined by the secondary wall's middle layer. Mechanical behaviour depends on factors such as the fibrils' spiral angle, the degree of cellulose polymerisation, porosity content and the size of the lumen (Baley 2002). Agricultural variables, age, specie, plant variety and fibre processing parameters also influence mechanical behaviour (Kulkarny *et al*., 1982).

Chemical reactions in cell walls generate biodegradability, thermoplasticity and dimensional instability regarding moisture due to their lignocellulose-associated hygroscopic nature (Rowell *et al*., 1993). This lignocellulose contains strongly-polarised hydroxyl groups causing water affinity; such hydrophilic nature makes it incompatible with a hydrophobic matrix (Alix *et al*., 2009; Cuellar

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and Muñoz, 2010). These shortcomings have been resolved by suitable physical, chemical or enzyme treatment. The treatment used can reduce moisture absorption, clean the surface and enhance fibre roughness, mainly reducing hydrophilic behaviour (Mohd *et al*., 2007; Wang *et al*., 2003). The structural changes occurring on the fibre surface and cell wall structure after treatment influence mechanical properties (Rong *et al*., 2001). Lignocellulose fibre surface modification has been reported, involving alkali treatment, silane treatment, acetylation treatment and benzoylation treatment; these have been discussed by Li *et al*., (2007). The simplest chemical modification is alkali treatment of fibres which has been used to treat almost all natural fibres with successful results (Bisanda, 2000; Valadez-Gonzalez *et al*., 1999).

Alkali treatment's main purpose is to disrupt hydrogen bonding in the network structure and remove some hemicellulose, lignin, wax and oils, thereby increasing surface roughness and reducing its hydrophilic nature. This treatment influences fibre's mechanical behaviour, especially regarding strength and stiffness (Li *et al*., 2007).

The effect of alkali treatment on treated and untreated banana fibres' mechanical properties is analysed in this paper to identify composite behaviour by understanding the properties of reinforcement before becoming combined into the matrix. Treatment parameters such as fibre immersion time and NaOH concentration were selected from references. Structural changes regarding treated and untreated banana fibres' morphology and mechanical properties were evaluated by a tensile test and scanning electron microscopy (SEM).

Materials and methods

The stems from banana plants (*Musa paradisiaca*) were selected from an 11-month-old plantation. The plantation is located 1,050 meters above sea level, has 22.5ºC average temperature, 76% relative humidity, 2.100 mm annual rainfall, 6.1 PH and 2,010 hours annual sunshine (Aristizábal *et al*., 2008).

The banana fibres were drawn from the banana plants' pseudostems (helically surrounding the stem). The pseudostems were first air-dried at 18ºC average temperature for 72 hours (i.e. natural drying). Banana fibres were extracted from the pseudostems by a defibring machine; this consists of a framework and a paddle wheel. The stems were fed to the paddle wheel; the pulp was separated from the stem and the fibres extracted. Bundles of fibres were then placed in water for 12 hours at room temperature to remove impurities and facilitate their separation before being left at room temperature for 6 hours.

The following experimental procedure was based on the work of Wang *et al*., (2003) and Jafferjee *et al*., (2003). The treated fibres were first washed with 2% detergent-water prior to alkali treatment and dried at 70ºC for 24h to remove external wax. They were then mercerised to remove fibre surface impurities, causing changes in the crystalline cellulose, and preparing the fibre for the effects of chemical treatment. Sequential extraction was used for mercerisation with 1:2 mixture of ethanol and benzene for 6h, followed by washing with distilled water and air drying to eliminate water-soluble polymers and waxes. The fibres were then chemically-treated to remove lignin and hemicellulose to improve fibre surface roughness and compatibility when composite material was formed. Alkali treatment induced such modification where fibres were immersed in 5% NaOH aqueous solution (w/v) for 1h at room temperature (1:15 fibre-to-solution weight ratio). The treated fibre was then washed thoroughly with distilled water to remove excess NaOH from the surface and oven dried at 110ºC.

As suggested by Hu *et al*., (2010), fibres were carefully manually separated from the bundles. Single banana fibres were randomly chosen; fibres having apparent defects were discarded and those having greater uniform length were selected for the tensile test. It is well-known that natural fibres' cross-section is not perfectly round and that diameter size varies along a fibre's length (Mukhopadhyay *et al*., 2008). All fibres' diameters were measured at different locations along their length to overcome such problem, using an optical microscopy before tensile test (average value being taken as parameter).

A universal testing machine was used for assessing treated and untreated fibres' mechanical behaviour; 50 mm gauge and constant 4 mm/min crosshead speed were used for all tests. A paper frame with a window was designed, constructed and used with the testing machine's fixtures to hold the fibre during the tensile test. The fibres were carefully positioned on the paper frame to prevent slippage and misalignment looking for axial forces during the test. SEM was used for characterising the morphology of the structural changes occurring on the surface of treated and untreated banana fibres and also the tested specimens' fracture surface.

Results and Discussion

Fibre surface morphology analysis is important to ascertain the structural changes occurring in a fibre upon alkali treatment. This knowledge offers fundamental information regarding interfacial adhesion between a fibre and the matrix when developing the composite.

Figure 1(a) shows the untreated fibre; it has an irregular surface having variable roughness where the microfibrils appear to be parallel to the fibre's axis. The fibre has some impurities on its surface. Figure 1(b) shows treated fibre having a rougher surface produced by lignin and hemicellulose removal. It may also be noticed that most impurities have been removed from the fibre's surface. This cleaner and rougher surface would increase the number of possible interaction sites, thereby improving mechanical fibre-matrix adhesion.

Each fibre's mechanical properties were analysed. Banana fibres were chosen at random from the bundle fibre, ensuring a wide diameter range. The banana fibres were classified into 4 classes based on a 0.035 mm class interval, considering lower and higher diameters between treated and untreated fibres. The fibre selected for tensile test was the most representative of each interval (i.e. closest to the class mark) to facilitate analysis of the information and its subsequent comparison with other fibres.

Figure 2(a) and 2(b) shows stress-strain curves for untreated and treated fibres, respectively. The stress-strain curve for 0.175 mm-0.210 mm diameter treated fibres was not displayed due to lack of fibres in this interval. Treated and untreated banana fibres had brittle behaviour, characterised by a lineal relationship between deformation and stress until failure, without noticeable plastic deformation. The fibre's Young's modulus was evaluated by the slope of the curve in the linear region. The stress-strain curve had fluctuation activity in specific sections; this phenomenon may have been associated with constant rearrangement of microfibrils in the direction of the fibre axis. Mukhopadhyay *et al*., (2008) also ob-

served this behaviour in banana fibre. Table 1 shows the results of the tensile test for untreated banana fibres and Table 2 shows that for treated banana fibres.

Figure 1. SEM micrograph of (a) an untreated banana fiber and (b) a treated banana fiber

Young's modulus experimental value for untreated fibres was 6.6 to 25.6 GPa, ultimate tensile strength was 222.3 to 780.3 MPa and strain was 1.79% to 3.27%. The resulting values for untreated banana fibre mechanical behaviour agreed with data presented in the literature (Guimarães *et al*., 2009; Lilholt and Lawter, 2000; Mahji *et al*., 2010). Value variation amongst authors can be explained by three factors: defects such as cross-marks, kink bands or dislocations that become stress concentrators (Baley *et al*., 2006), the fibre's internal structure causing a non-constant cross-section along fibre length and variability in fibre composition. Table 1 shows that Young's modulus, ultimate tensile strength and strain decreased with increased fibre diameter in the range being investigated. This may have been due to the presence of a hollow fibril in the centre of each fibre cell modifying the real cross-section and therefore the mechanical properties. Baley (2002) has reported similar results for flax fibre. These results motivated a deeper study of the effect of the real cross-section on mechanical properties.

Table 1. Different diameter untreated banana fibres' mechanical properties

Diameter range (mm)	Diameter (mm)	Young's modulus (Gpa)	Ultimate strength (Mpa)	Strain (%)
0,07-0,105	0,0874	25,6	780,3	2,68
0,105-0,140	0,1328	13,7	300	1.93
0,140-0,175	0,1563	11,3	198,9	1,79
0,175-0,210	0,1925	6,6	222,3	3,27

Young's modulus experimental value for treated fibres was 9.73 to 21.6 Gpa, ultimate tensile strength was 148.1 to 536.2 MPa and strain was 1.38% to 2.57%. Young's modulus and tensile strength decreased with increasing fibre diameter in the range investigated due to cross-section variation explained above for untreated fibres. Comparing untreated fibres' Young's modulus, ultimate tensile strength and strain values to those for treated ones showed that all decreased in the ranges investigated here. This may have been due to cellulose delignification and degradation during alkali treatment thereby disrupting bonding and leading to morphological changes, like increased surface roughness. Similar findings have been reported by Arifuzzaman *et al*., (2009) in okra fibre. Such increased roughness acts as stress concentrator for decreasing fibre's mechanical behaviour.

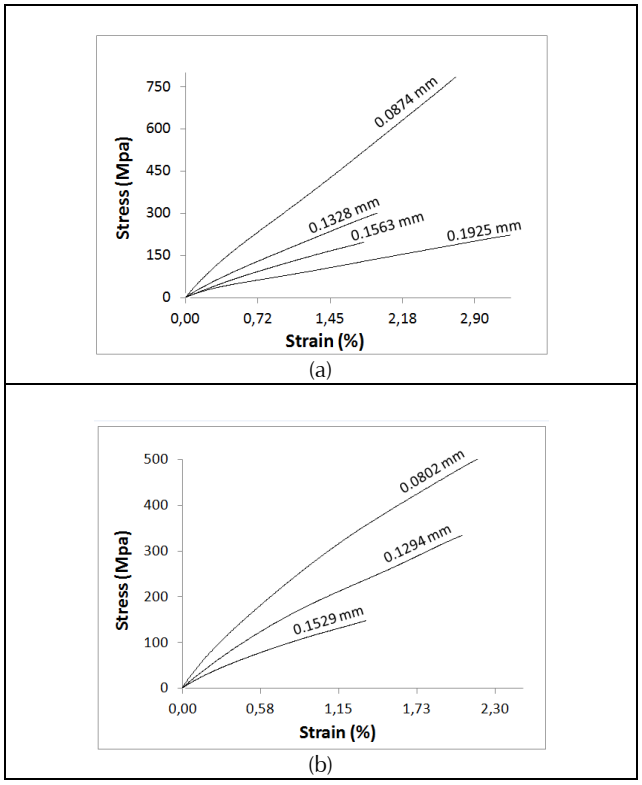

Figure 2. Stress–strain curves for (a) untreated banana fiber and (b) treated banana fiber

Table 2. Different diameter treated banana fibres' mechanical properties

Diameter range (mm)	Diameter (mm)	Young's modulus (Gpa)	Ultimate Strength (Mpa)	Strain (%)
0.07-0.105	**0.0802**	**21.6**	**536.2**	**2.37**
0.105-0.140	**0.1294**	**17.2**	**337.3**	**2.1**
0.140-0.175	**0.1529**	**9.73**	**148.1**	**1.38**

Fracture surface analysis studied internal structure behaviour regarding axial forces. It also provided better understanding of brittle fracture by analysing each fibril's behaviour. Figure 3 (a) shows that many fibre cross-sections had channels negatively influencing mechanical properties. Figure 3 (b) shows that cell wall thickness appeared to decrease due to loss of material compared to that of untreated fibres. Such loss of material changed the shape and size of the holes, making them highly variable. The microfibrils shared the load during tension test, causing a gradual brittle fracture.

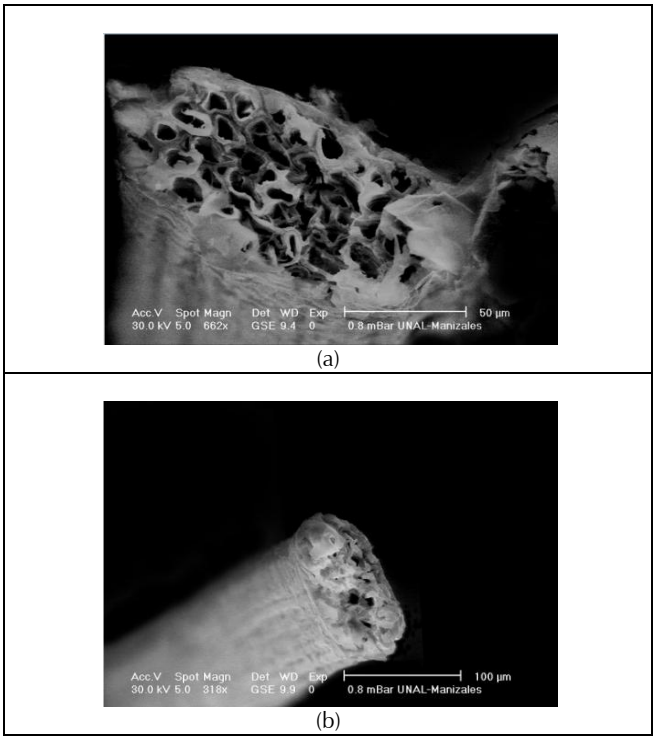

Figure 3. Angular view of surface fractured of (a) untreated banana fiber and (b) treated banana fiber.

Conclusions

Two types of behaviour were observed and analysed regarding applying loads to alkali-treated banana fibres before becoming a composite material. Young's modulus and ultimate tensile strength decreased as fibre diameter increased in treated and untreated banana fibres due to the presence of a hollow fibril in the centre of each fibre cell thereby modifying the fibre's real cross-section. The treated fibres showed decreased mechanical properties. It is thus suggested that such variations were related to the removal of lignin and hemicellulose from the surface of the fibres, possibly causing weakening in the fibre's outer wall. Studying banana fibres' behaviour before becoming combined into the matrix has led to understanding reinforcement's real contribution to the composite material.

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