Fibers and Polymers 2012, Vol.13, No.5, 632-640 DOI 10.1007/s12221-012-0632-8

Fique Fibers: Enhancement of the Tensile Strength of Alkali Treated Fibers During Tensile Load Application

Catalina Gómez Hoyos, Vera A. Alvarez[1], Piedad Gañán Rojo[2], and Analia Vázquez*

Polymer and Composite Material Group-Technology and Engineer Science Institute (INTECIN)-CONICET, Buenos Aires University, Buenos Aires, Argentina
[1]*Research Institute of Materials Science and Technology (INTEMA)-CONICET, Mar del Plata University, Mar del Plata, Argentina*
[2]*School of Engineering, Mechanical Engineering Program, New Materials Research Group, Pontificia Bolivariana University, Medellín, Colombia*
(Received May 5, 2011; Revised October 28, 2011; Accepted November 5, 2011)

Abstract: Fique fibers were treated using Na(OH) solution at 5 w/v%, slack and under 1 N of tension, at room temperature, for 4 and 24 h respectively. Changes in their structure and composition were monitored using Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA) and X-ray diffraction (XRD). Additionally their mechanical properties were evaluated and analyzed. Results showed that tensile load application during alkali treatment improves their tensile strength and modulus. The most important change in mechanical properties was achieved in fibers treated for 24 h under tension. However, these fibers presented a high standard deviation; due to this treatment causing an important defibrillation. Moreover, fibers treated for 4 hours under tension, enhance their tensile strength around 56 %, while slack treated fibers improve only 38 %. When fibers were treated under tension, cellulose microfibrills were rearranged in the direction of tensile application and the spiral angle decreased, increasing the molecular orientation.

Keywords: Fique fibers, Mercerization, Spiral angle, Mechanical properties

Introduction

Fique is a Colombian native natural fiber -*Agave furcraea*-. It is a hard fiber, extracted by mechanical means from the leaves of the fique plant. It is considered to be the Colombian national fiber and is used in fabrication of sacks and packages for agriculture, resistant ropes and strings. Also it is used in the transportation, construction and sailing industries. Current research, with the aim to exploiting this natural resource, has been developing applications for the new material; i.e. Delvasto *et al.* [1] developed corrugated roof sheets manufactured by a vacuum forming process, using Portland cement reinforced with fique. Additionally, its use as polymer reinforcement and the effects of fiber surface treatments on mechanical behavior of composites have been studied [2-4].

Fique is mainly constituted of three polymers; cellulose, hemicellulose and lignin. Cellulose is the principal component which confers strength and stability to the plant's cell walls. This linear glucan consists of cellobiose units connected by β-(1 $\rightarrow$ 4) glycosidic linkages, which allows cellulose to form a flat and ribbon like long straight chain. This molecular linearity makes cellulose highly anisotropic with a theoretical strength of around 10 GPa in the chain direction [5,6].

Natural fibers could be described as having a hierarchical structure: (i) vascular bundles formed of elementary fibers, (ii) cellulose microfibril bundles, which consist of densely packed bundles of cellulose chains, embedded in a network of hemicellulose and lignin [5,7]. Microfibrils are helically wound along the fiber axis to form ultimate hollow cells, with a spiral angle (angle between fibrils and fibre axis) that varies for each type of plant [8,9].

Cellulose microfibrils are the main contributors to the tensile strength of natural fibers, and the spiral angle affects the mechanical properties of natural fibers due to cellulose's highly anisotropic behavior [10]. Several studies have shown when cellulose microfibrils are oriented along the axis of tension; the mechanical properties of natural fibers become higher [11-15]. Then when natural fibers, under tension, are treated with alkali cellulose microfibrills rearrange in the direction of tensile application, decreasing the spiral angle, and increasing the molecular orientation. This in turn would lead to better load sharing and higher stress development in the fibre. As reported by Goda *et al*. [15] Ramie fibres treated under 0.098 N of tension with 15 wt.% NaOH solution for 2 h increased their tensile strength by 18 %, while slack alkali treated fibres decrease in 1.8 % in respect to untreated fibres.

Liu and Hu [16] treated bamboo fibers with NaOH solutions at several concentrations from 4 to 24 wt.% for 20 minutes at 20 ^{o}C. They found that each condition had different effects and that the greatest effectiveness of crystal lattice conversion from cellulose I to cellulose II started at mercerization condition 12 wt.% of alkaline solution and could be completely achieved at alkaline concentration of 16 wt. %, 10 minutes at 20 ^{o}C. Tensile strength of cellulose II group are superior to those of cellulose I one because in

*Corresponding author: anvazque@yahoo.com

the crystal lattice transformation, the parallel chain arrangement of the cellulose I group changes into more stable antiparallel one of the cellulose II group. However several authors have reported that the Young's modulus of cellulose II is lower than that of cellulose I [17,18]. In this work we pretend to avoid the crystal lattice transformation from cellulose I to cellulose II with the aim to reach higher Young's modulus, so that we chose alkaline concentration lower than 12 wt.%. In addition, several authors have reported that alkaline concentrations near to 4-5 wt.% are the optimal concentration to improve the mechanical properties of different natural fibers [19-21].

The purpose of this research was to study the effect of time and tensile load application during alkali treatment on the composition, structure and mechanical properties of fique fibers. It was expected that tensile load application during alkaline treatments would produce an improvement in structural and mechanical properties of fique fibers due to its effect on microfibril orientation. Changes in mechanical properties were evaluated using a single filament test following the ASTM D 3379-75. The results were interpreted in terms of their structure, which were evaluated using: FTIR, DRX, and TGA. Moreover variations in morphology were evaluated using field emission scanning electron microscopy technique. The originality of this work lies in the study of alkaline treatment under tension for fique fibers and its effect on mechanical properties analyzed from changes in structure and morphology of the fibers. This study is of great importance for the development of new applications of natural fibers, and especially for fique fibers, due to the lack of published results currently available.

Experimental

Materials

The properties of the fique fibers determined in this investigation, are listed in Table 1. Fique fibers were washed and dried before alkaline treatment or tests to eliminate impurities and waxes of its surface. Fique or *furcraea andina* is extracted from the fique plant, which is the most cultivated natural fiber plant of Colombia. Fique is similar to the sisal fibers, and the fique plant is often confused with agave plant. The differences are that the agave leaves are yellowish and stiff, with a strong spike in the tips, while the fique plant leaves are droopy and greenish without a spike [1].

Table 1. Characteristics of the fique fibers

Characteristic	Value
Equivalent diameter (mm)	0.20±0.04
Tensile strength (MPa)	197±65
Young's modulus (GPa)	5.7±1.8
Cellulose (%)	57±3
Hemicellulose (%)	29±5
Lignin (%)	13±2

The content of cellulose, hemicellulose and Lignin reported in Table 1 was determined as follows [17]. The holocellulose content (cellulose + hemicellulose) was determined according to TAPPI T19m-54, which consists of a selective degradation of the lignin. Sodium hypochlorite and glacial acetic acid were added to an aqueous suspension of previously dried and milled fique fibers, at 75 °C, under magnetic stirring for 2 h. Then after 2 h the hollocellulose was filtered and washed with hot water, nearly 100 ^{o}C and acetone and then dried at 60 °C, in an oven, until constant weight. For cellulose content determination, sodium hydroxide solution 17.5 wt.% was added to holocellulose previously dried at room temperature, under magnetic stirring for 30 minutes. Then after 30 minutes the cellulose was filtered and washed until neutral pH and then dried at 60 °C, in an oven, until constant weight. The hemicellulose content is the subtraction between hollocelluloce and cellulose content. Finally lignin content was determined as the subtraction of the initial weight of fiber and hollocellulose content.

Alkali treatments were carried out slack and with an initial load of 1 N in order to investigate their effects on mechanical and chemical properties. The fique fibers were treated with NaOH solution 5 w/v% for both 4 and 24 h. The ratio of fiber weight to alkaline solution volume corresponds to 1:4 for all treatments. The apparatus presented in Figure 1 was used to develop the treatment under tension. Different treatments were designed using time as the variable.

Methods

Attenuated Total Reflection Fourier Transforms Infrared Spectroscopy (FTIR-ATR)

Infrared spectroscopy experiments were conducted using a FTIR spectrometer (Nicolet 6700 Series) equipped with a single-reflection ATR and a type IIA diamond crystal mounted in tungsten carbide. The diamond ATR had sampling area of approximately 0.5 mm^2, in which a consistent reproducible pressure was applied to every sample. Infrared spectra were collected at 4 cm^{-1} resolution and 64 scans from wave number 4000 to 450 cm^{-1}, were carried out.

Thermogravimetric Analysis (TGA)

Thermo gravimetric analysis were developed using a

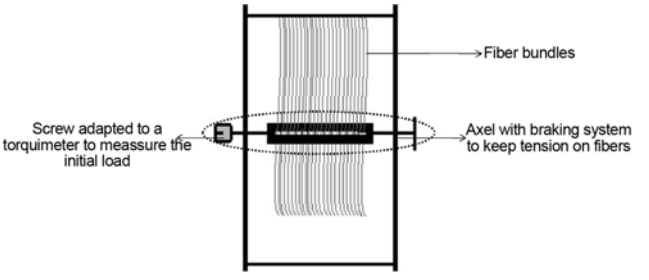

Figure 1. Scheme of the designed device for fiber alkali treatments under tensile load.

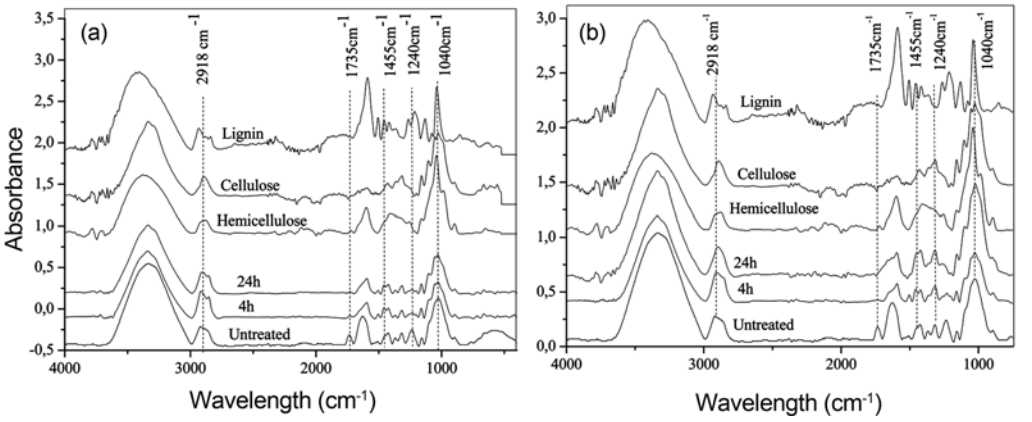

Figure 2. FTIR spectra of fique fibers alkali treated (a) slack alkali treated and (b) alkali treated under tension.

Mettler STARe TGA/sDTA851e analyzer. The thermograms were run from 25 to 800 °C under nitrogen atmosphere at a heating rate of 10 °C/min using around 14 mg of sample.

X-ray Diffraction (XRD)

DRX patterns were recorded by a PW1710 diffractometer equipped with an X-ray generator ($\lambda = 0.179$ nm).

Field Emission Scanning Electron Microscopy (FE-SEM)

SEM was used to observe the morphology of fibers and also the surface after fiber fracture. Fiber microstructure was analyzed using FE-SEM (Zeiss Supra 40) The Fe-SEM was operated in secondary electron mode, under an accelerating voltage of 1 kV.

Diameter Measurement

To determine fiber diameter an optic microscopy (Olympus SZH-10) was used.

Single Filament Test

A Universal Testing Machine, Instron 4467, was used for tensile tests. Tests were carried out by following ASTM 3379-75 recommendations. Around 20 fibers, with a length of 30 mm, were tested for each treatment at a crosshead speed of 2 mm/min.

Results and Discussion

FTIR Analysis

FTIR spectra alkali treated fibers slack and under tension are shown in Figures 2(a) and 2(b) respectively. FTIR spectroscopy is an appropriate technique to establish the variations introduced by both alkali treatments on the chemical structure of the fibers. Comparing Figures 2(a) and (b), tensile load application during alkali treatment does not introduce any changes to the fibers chemical structure, as slack treated fibers and fibers treated under tension present similar FTIR spectra. Moreover, the spectra of commercial cellulose, xylose (principal component of hemicellulose) and lignin were superposed to compare with fibers, with the aim of characterizing the chemical structure by identifying the functional groups present in each fiber sample. Peaks of around 2918 cm^{-1}, corresponding to O-H stretching in methyl and methylene groups in cellulose hemicelluloses and lignin, are present in all spectra. The vibration at 2850 cm^{-1} originating from C-H stretching in lignin and waxes became more visible after both treatments [23,24]. Absorption centered at 1735 cm^{-1}, assigned to vibrations of acetyl and uronic ester groups of hemicelluloses and ester linkage of carboxylic group of the ferulic and p-coumaric acids of lignin, was significantly reduced after both treatments [23,25,26]; also the band near to 1230 cm^{-1} originating from C-O-C stretch of ether groups present in lignin and hemicellulose was significantly decreased after alkali treatment [13-25]. Those facts indicate that both treatments appreciably remove the hemicellulose. In addition, the spectra of both treated and untreated fibers clearly show some characteristic vibrations of lignin: 1510 cm^{-1} (aromatic rings vibrations), 1460 cm^{-1} (C-H deformations) [24-26], thus the treatments remove partially lignin from fiber structure. Lignin cannot be totally removed by the alkali process because lignin degradation is very difficult by presence of strong C-C linkages and aromatic groups, which are very resistant to chemical attack [27].

Thermogravimetric Analysis

The TGA and DTGA analyses of fique fibers treated slack and under tension, presented in Figures 3(a) and 3(b) respectively, were used in order to characterize the presence of each compound in the fiber before and after the treatment. Below 100 °C weight loss was observed, attributed to water evaporation. Moreover, in agreement with the reported literature [2,13,28] three thermodegradation processes were identified: (i) hemicellulose degradation related with a shoulder around at 300 °C (ii) cellulose decomposition attributed to the peak at around 360 °C. During the degradation of those polysaccharides occurs, a rupture of bonds like -O-, C-H, C-O and C-C; as well reactions of dehydration, decarboxylation and decarbonylation, with the formation of

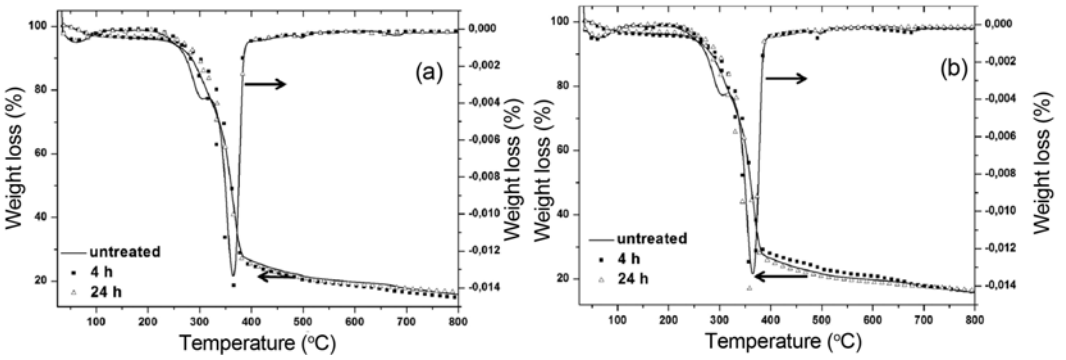

Figure 3. TGA and DTGA of fique fibers (a) slack alkali treated and (b) alkali treated under tension.

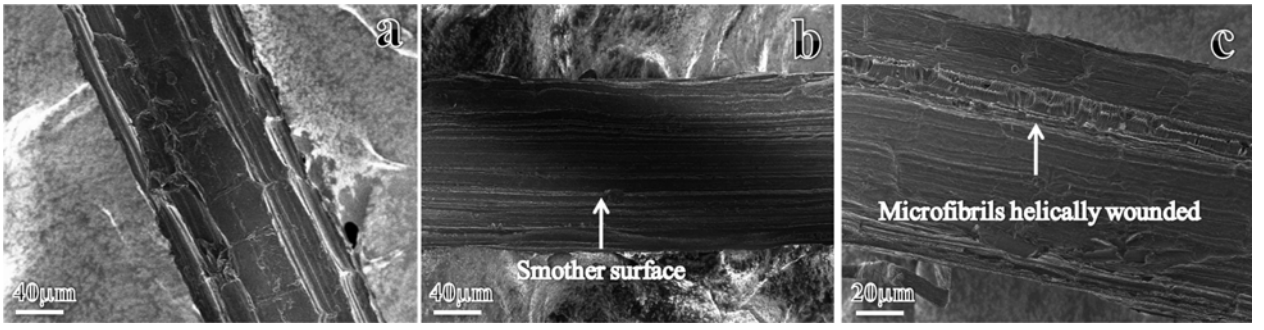

Figure 4. Fe-SEM micrographs of fiber (a) fique, (b) slack alkali treated 2 h and (c) slack alkali treated 24 h.

carbonyl and carboxyl groups. (iii) Lignin was the most difficult to decompose, its degradation occurred slowly. Due to it being related to dehydration, yielding derivatives structures, with lateral unsaturated chains releasing water, CO_2, CO and CH_4 [29,30].

SEM Micrographs

Figures 4(a)-(c) show fibers surface before and after treatment. It can be observed that after treatment fiber surfaces become smoother (Figure 4(b)). Figure 4(c) displays the images of fique fiber treated for 24 h it is evident that microfibrils helically wounded became exposed due to alkali treatment.

Fique fibers could be described as natural composite mainly constituted of cellulose microfibrils (reinforcement) wound helically in an amorphous matrix (lignin). The exact structural organization of the chemical constituents in the primary and secondary cell walls is still a subject for debate, however it is commonly accepted that cellulose, hemicellulose and lignin are arranged in separate entities and not homogeneously mixed [26]. In primary cell walls, the orientation of cellulose microfibrils is often dispersed. The secondary cell walls are made up of three layers with helicoidally arrangement (opposing microfibrils orientation), the middle layer is the most important, and its helicoidally arranged spiral angle plays an important role in determining the mechanical properties of the fiber [21].

X-Ray Diffraction

The XRD patterns of slack alkali treated fique fibers and alkali treated under tension are shown in Figure 5. In agreement with previous studies [16,32], all samples present patterns characteristics of cellulose I structure, three peaks were identified, at $2\theta = 16^o$, 22.9^o and 34.5^o. The peak at $2\theta = 16^o$ correspond to the (110) and (110) crystallographic planes and the peaks at $2\theta = 22.9^o$ and 34.5^o correspond to the (002), and (023) or (004) planes, respectively [13,25,29, 33]. The XRD was performed in order to identify if the treatment produced any change in the degree of crystallinity, or type of crystalline structure. The XDR patterns show changes in signal as a function of treatment time. However, alkali treatment slack and under tension does not produce a lattice transformation. These results are in agreement with those reported by Liu and Hu [16], only concentrations of NaOH higher than 16 wt.% at room temperature produced the lattice transformation of cellulose I to cellulose II for bamboo fiber, at lower concentrations, the hydroxide ions could be fully hydrated and they will not be able to penetrate and disrupt the cellulose lattice due to size restriction [5,32].

Mechanical Properties

The alkaline treatment influences not only the cellulosic components, but also the non-cellulosic components, hemicellulose and lignin. These compounds are situated between the cellulose regions that exist inside and between the elem-

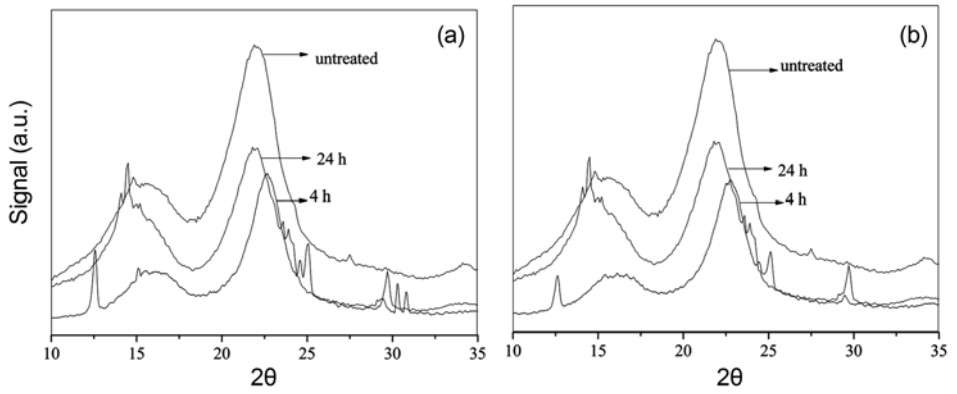

Figure 5. XRD patterns of fique fibers (a) slack alkali treated and (b) alkali treated under tension.

Table 2. Mechanical properties of treated fique fibers (average± standard deviation)

Time of treatment (hour)	Slack			
	Average diameter (mm)	Modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)
0	0.20±0.04	5.7±1.8	197±65	7.7±2.9
4	0.13±0.03	7.2±2.7 (↑26%)	272±106 (↑38%)	9.2±2.0
24	0.15±0.04	8.1±3.3 (↑46%)	218±104 (↑11%)	5.9±1.0
Time of treatment (hour)	Under tension			
	Average diameter (mm)	Modulus (GPa)	Tensile strength (MPa)	Elongation at break (%)
0	0.20±0.04	5.7±1.8	197±65	7.72±2.9
4	0.20±0.02	7.7±2.7 (↑35%)	308±93 (↑56%)	8.51±2.1
24	0.22±0.09	18.7±12.3 (↑228%)	391±315 (↑99%)	2.11±0.5

entary fibers. The removal of non-cellulosic materials makes the interfibrillar regions less dense and rigid, allowing fibrils to rearrange along the direction of tension [17,33]. In addition, tensile load application during alkali treatment stimulates the reorientation of microfibrils along the fibers axis resulting in important improvements of their mechanical properties [15, 35-37].

Table 2 summarizes mechanical properties of untreated and treated fique fibers. As it was expected, treatment under tension increases mechanical properties more than in slack treatment. The tensile strength of slack treated fibers for 4 and 24 h increased by 38 % and 11 % respectively; while fibers treated and under tension for 4 and 24 h improved their tensile strength by 56 % and 99 % respectively. In addition, the Young's modulus, for instance, are expected to increase with increasing levels of molecular orientation. Highly oriented cellulosic fibers have higher Young's modulus than fibers with lower orientation [5,6]. In fact, slack treated fibers for 4 and 24 h raise their modulus by 26 % and 46 % respectively while fibers treated and under tension for 4 and 24 h increase their modulus by 35 % and 228 %. The results obtained clearly show the superior effect achieved for tensile load application during alkali treatment for fique fibers, especially for fibers treated for 24 h. In addition the tensile load applied to fibers was 1 N, it is a higher load than applied by other authors [8,15] but is still inside the elastic range of fique fiber. Moreover Stocchi *et al*. [19], reported for woven jute/vinyl ester laminates, improvements in composite tensile strength and modulus around 43 % and 29 % respectively, after treated jute woven with sodium hydroxide solution at 5 w/v% and 1 N of tension for 4 h.

Table 3 shows results reported for other authors, and is important to emphasize that reported improvements of this magnitude have not been found for other fibers under similar treatment conditions. Gañan and Mondragon [2] treated fique fibers without tension, with sodium hydroxide at 20 wt.%. for 1 h. Authors reported improvements in modulus and tensile strength comparable to our results for fibers treated under tension for 4 h with sodium hydroxide at 5 w/v%. Thus the application of a tensile load during treatment makes it environmentally friendly, due to reducing the alkali concentration about 4 times, comparable improvements were achieved.

Figure 6 shows the optical micrograph of untreated and treated fique fibers section, it could be observed that alkaline treatment causes defibrillation, increasing with both time of treatment and tensile load application. This defibrillation introduces many defects on fibers surfaces, causing a high distribution in mechanical properties, as can be observed in fibers treated under tension for 24 h. These fibers achieved the highest mechanical properties but they also displayed the highest standard deviation due to defibrillation.

On the other hand, Table 2 clearly shows that slack treated fibers change their diameter due to swelling, while fibers

Table 3. Improvement in mmechanical properties of treated natural fibers reported by other authors (↑, ↓ indicate that property increase or decrease respectively)

Fiber/Treatment	E (GPa)	ρ (MPa)	References
Ramie NaOH 15 % 2 h 0 N	↓ 41 %	↓ 1,8 %	[15]
Ramie NaOH 15 % 2 h 0.049 N	↓ 39 %	↑ 18 %	
Ramie NaOH 15 % 2 h 0.098 N	↓ 31%	↑ 8 %	
Sisal NaOH 2 % 4 h 60 °C	↓ 14 %	↑ 27 %	[33]
Jute NaOH 5 % 2 h	↓ 0.1 %	-	[31]
Jute NaOH 5 % 4 h	↑ 12 %	-	
Jute NaOH 5 % 6 h	↑ 68 %	-	
Jute NaOH 5 % 8 h	↑ 79 %	-	
Ramie Na(OH) 15% 2 h	-	↓ 31 %	[34]
Agave NaOH 5 % 30 min	-	↑ 24 %	[35]
Fique 20 % 1 h	↑ 37 %	↑ 57 %	[2]
Sisal NaOH 5 % 24 h	↑ 6 %	↑ 6 %	[36]
Coir alkali solution 5 % 28 °C 76 h	↑ 40 %	↑ 15 %	[37]

The values of mechanical properties are reported in the original papers. This Table only reports percentages of increments or decrements with the aim to illustrate the effect of alkaline treatments on mechanical properties.

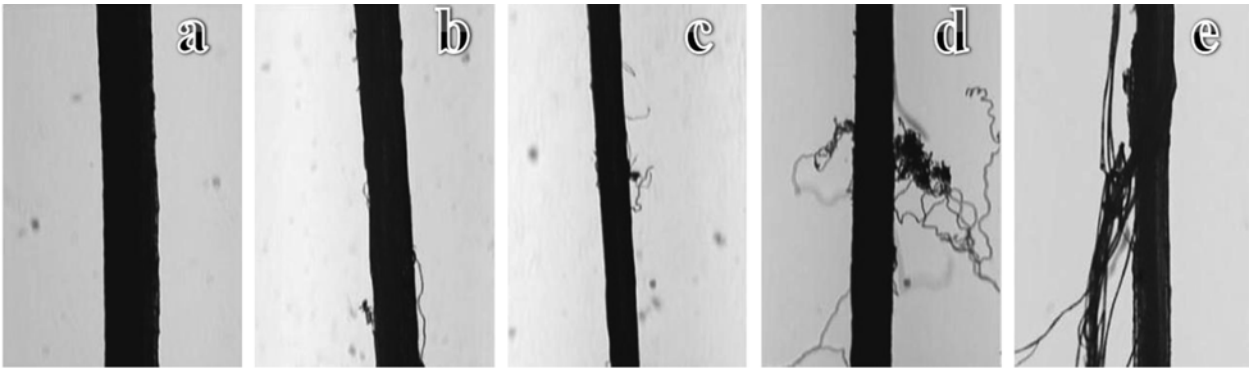

Figure 6. Optical micrographs of fibers (a) untreated, treated (b) 4 h slack, (c) 24 h under slack, (d) 4 h under tension, and (e) 24 h under tension.

treated under tension undergo a restricted swelling, insignificant changes in its diameter were observed. The fibers diameter directly influences its mechanical properties. Figure 7 presents Young's modulus, strength and elongation at breaking point for fique fibers treated for 24 h as function of diameter; similar behavior was displayed in the other treatments. It can be noted that both, tensile strength and the elastic modulus of fibers, could be adjusted to a decreasing exponential curve, which indicates that both properties decrease with increasing diameter [13,38,39], while the strain at breaking point is roughly constant between 6-8 %.

Figure 8(a) shows tensile stress tests of untreated fique fiber. When an untreated fique fiber is subjected to a tensile stress test, at early stages, in the elastic region, microfibrils tend to slip and rearrange their position; however the presence of lignin and hemicellulose between microfibrils acts as a bonding agent and restricts the slippage. When the stress applied on the fiber increases and the first stage is completed, the slope of the curve changes and a small scale irreversible slippage within the microfibrils occur, until reach its maximum tensile strength [15,33].

Figures 8(b)-(d) shows, that fiber treatment introduces changes in mechanical behavior. As compared with untreated fibers, treated ones present (except in fibers treated for 24 h under tension see Figure 8(e)) more plastic and less elastic deformation due to: (i) when hemicelluloses and lignin are removed the interfibrillar region becomes less dense and more flexible, (ii) then elementary fibrils are able to rearrange themselves along the direction of tensile load, generating irreversible slippage and breakage of microfibrils, resulting in the plastic deformation of the fiber upon tensile load [13,40-42]. During alkaline reactions, fibers suffers welling and shrinkage [35], therefore fibers treated under tension were not under constant load due to shrinkage, fibers treated for 24 h support more charge during treatment than fibers treated during 4 h, for that reason fibers treated

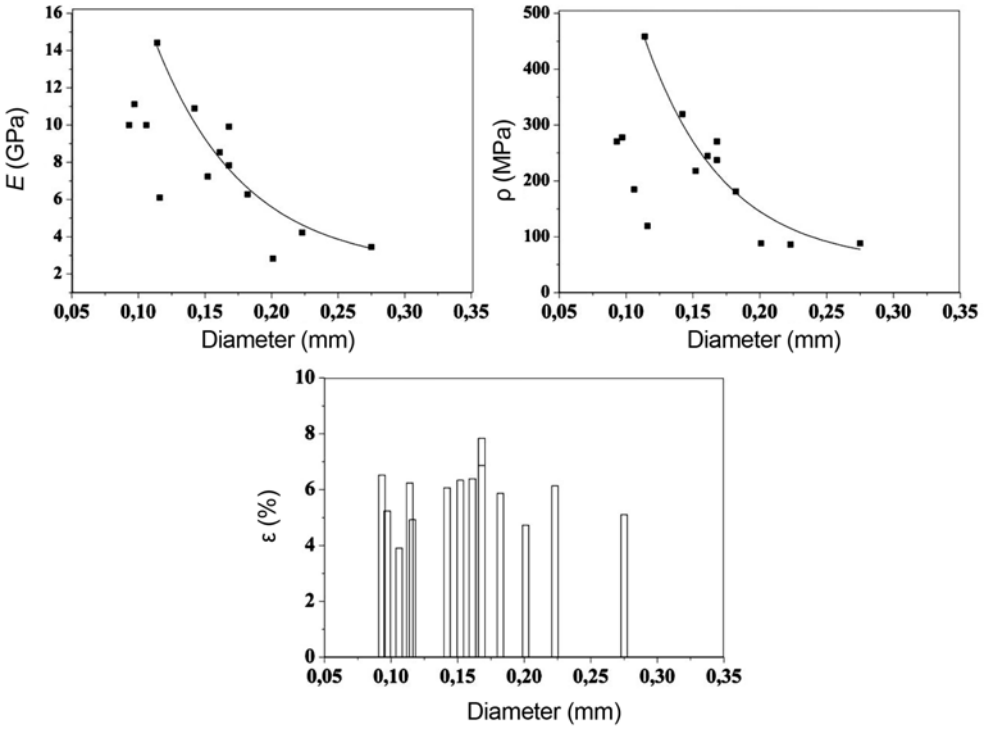

Figure 7. Influence of diameter on mechanical properties of fique fibers slack treated 24 h.

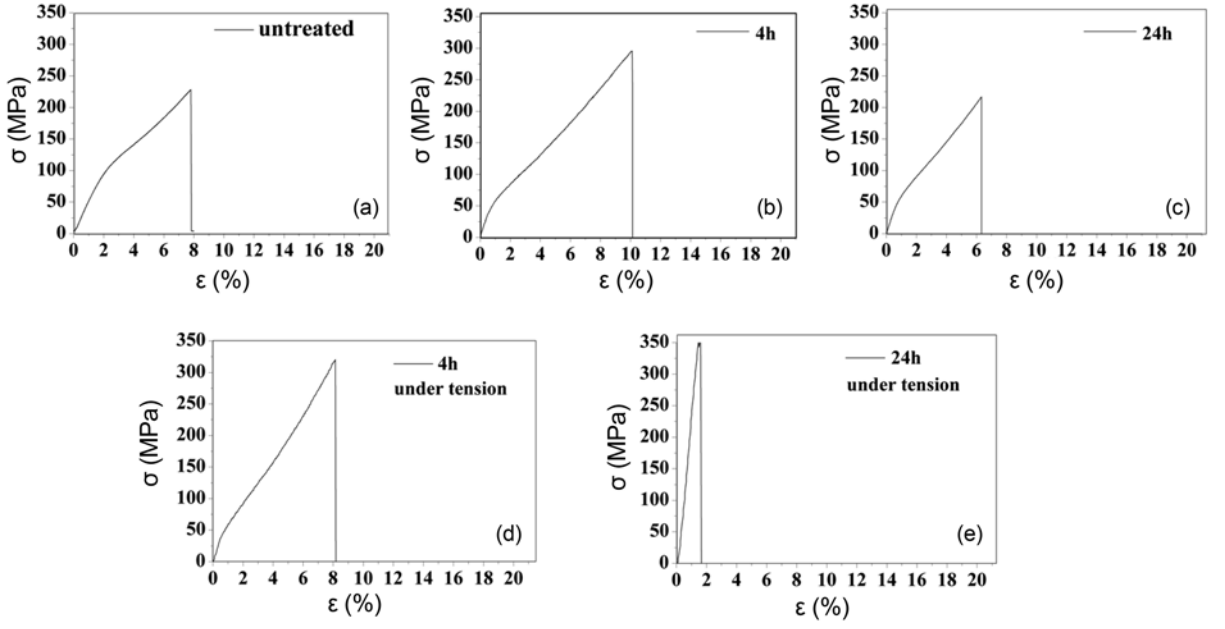

Figure 8. Mechanical test for: untreated and treated fique fibers.

under tension for 24 h, reach higher mechanical properties and lower elongation at breaking point.

Effect of Microfibrillar Angle in Mechanical Properties

In order to analyze the possible effects of treatment in microfibrillar angle and cellulose content the correlations proposed by Kulkarni *et al*. and Maclaughlin *et al*. [43,44] were used (see equations (3) and (4)).

$$\varepsilon = -2.78 + 7.28 \times 10^{-2}\theta + 7.7 \times 10^{-3}\theta^2 \quad (3)$$

$$\sigma = -334.005 + 2.83\,\theta + 12.22\,W \quad (4)$$

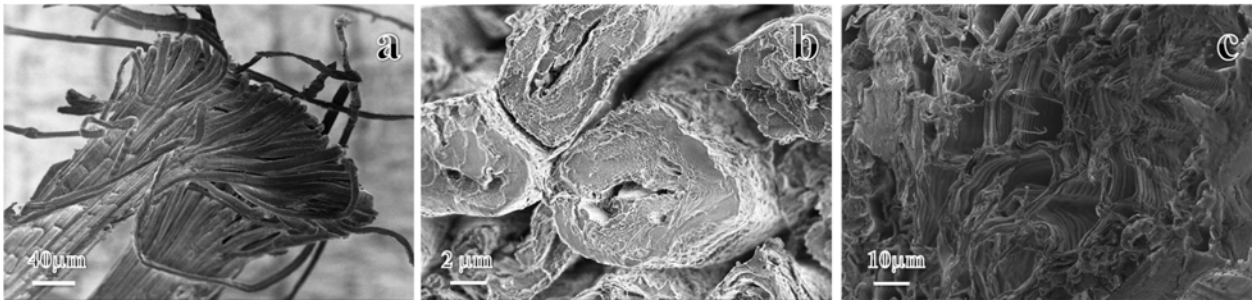

Figure 9. Fe-SEM images of fracture surface to fique fiber showing: (a) and (b) elementary fibers detached for fibers treated 4 h, (c) uncoiling of microfibrils for untreated fibers.

Table 4. Theoretical spiral angle and cellulose content for treated fique fibers

Time of treatment (h)	Slack		Tension	
	Theoretical spiral angle (°)	Theoretical cellulose content (%)	Theoretical spiral angle (°)	Theoretical cellulose content (%)
0	32.5±5.1	53.7±8.1	32.5±5.1	53.7±8.1
4	35.0±3.2	62.2±12.0	33.8±3.6	65.7±10.6
24	29.1±1.9	54.7±11.4	21.0±1.4	70.2±33.1

Where W represents cellulose content, θ the spiral angle and ε, σ, E, represent the elongation at breaking point, tensile strength and modulus respectively.

The overall results reported in Table 4 were calculated solving the equations (3)-(4) using Newton-Raphson method. As was expected spiral angle was affected by alkali treatment and tensile load application during treatment. Remarkable changes in spiral angle were achieved after 24 h of treatment especially for fibers treated under tension, although its standard deviation is proportionally to the observed in mechanical properties.

Conclusion

Special concern was taken studying the changes introduced in mechanical properties when fibers were treated with alkali solution under tension, being that this is a low cost treatment and without organic solvent application. The relationship between morphology and chemical composition to properties of untreated and alkaline treated fique fibers have been investigated.

The results showed that fibers treated for 24 h with 5 w/v% NaOH solution under 1 N of tension increased their modulus and tensile strength more than slack treated ones, but with a higher standard deviation. Furthermore fique fibers treated for 4 h with 5 w/v% NaOH solution under 1 N of tension achieved an improvement in mechanical properties with low dispersion, increasing their modulus 36 % and their tensile strength 56 % in comparison to untreated fiber.

A comparison between alkaline treatment with tension, developed in this research and alkaline treatment without tension developed by other authors for fique fibers; showed that tensile load application during treatment makes it environmentally friendly, due to reducing the alkali concentration about 4 times, comparable improvements were achieved.

Changes in mechanical properties were discussed from the viewpoint of morphological and structural changes. It was found that, hemicellulose and lignin were partially removed from fique fibers after alkali treatment. However alkaline treatment with tensile load application does not introduce significant changes to the chemical composition of fique fibers.

Alkali treated fibers present more plastic and less elastic deformation, because hemicelluloses and lignin were removed which makes the microfibrills more prone to irreversible slippage and even breakage, resulting in the plastic deformation of the fiber upon tensile load. However fibers treated for 24 h under tension behave differently due to the impact of alkali treatment. As a result the fibers suffer swelling and shrinkage and therefore they support more charge during treatment than fibers treated for 4 h.

Owing to its mechanical properties, fique fibers could became an alternative reinforcing agent for polymeric matrices. As demonstrated in the results, alkali treatment under tension optimized their mechanical behavior due to reduction in spiral and bundle angle, and hemicellulose and lignin content.

Acknowledgements

The authors acknowledged to CONICET and to the Agencia de Promoción Científica y Tecnológica (ANPCyT) from the Ministery of Science, Technology and Innovation (PICT 08-0223).

References

1. S. Delvasto, E. F. Toro, F. Perdomo, and R. Mejía de Gutiérrez, *Construction and Building Mater.*, **24**, 187 (2010).

2. P. Gañán and I. Mondragon, *Polym. Composites*, **23**, 383 (2002).
3. P. Gañán and I. Mondragon, *J. Mater. Sci.,* **39**, 3121 (2004).
4. P. Gañán and I. Mondragon, *J. Composite Mater.*, **39**, 633 (2005).
5. D. Fengel and G. Wegener in "Wood: Chemistry, Ultrastructure Reactions" Mc Graw Hill Book, pp.67-101, Berlin, 2003.
6. H. Krassig in "Polymer Monographs" 2nd ed. (Malcom B. Huglin Eds.), Vol. 11, pp.6-10, Gordon and Brach Science Publishers, Amsterdan, 1996.
7. S. R. Karmakar in "Textile Science and Technology" 1st ed. (Sara Burgerhartstraat Eds.), Vol. 12, pp.1-44, Elsevier, Amsterdam, 1999.
8. A. K. Bledzki and J. Gassan, *Prog. Polym. Sci.*, **24**, 221 (1999).
9. K. Joseph, R. D. Tolêdo Filho, B. James, T. Sabu and L. Hecker de Carvalho, *Revista Brasileira de Engenharia Agrícola e Ambiental*, **3**, 367 (1999).
10. W. Gindl, M. Reifferscheid, R.-B. Adusumalli, H. Weber, T. Röder, H. Sixta, and. T. Schöberl, *Polymer*, **49**, 792 (2006).
11. K. G. Satyanarayana, C. K S. Pillai, K. Sukumaran, S. G. K. Pillai, P. K. Rohatgi, and V. Kalyani, *J. Mater. Sci.*, **17**, 2453 (1982).
12. C. Pavithran, P. S. Mukherjee, M. Brahmakumar, and A. D. Damodaran, *J. Mater. Sci. Lett.*, **6**, 882 (1987).
13. F. Tomczak, T. H. Demétrio Sydenstricker, and K. G. Satyanarayana, *Compos. Part A-Appl. S.*, **38**, 1710 (2007).
14. M. S. Sreekala, M. G. Kumaran, and T. Sabu, *J. Appl. Polym. Sci.*, **66**, 821 (1997).
15. K. Goda, M. S. Sreekala, A. Gomes, T. Kaji, and J. Ohgi, *Compos. Part A-Appl. S.*, **37**, 2213 (2006).
16. Y. Liu and H. Hu, *Fiber. Polym.*, **9**, 735 (2008).
17. T. Nishino, K. Takano, and K. Nakamae, *J. Polym. Sci. Poly. Phys.*, **33**, 1647 (1995).
18. A. Ishikawa, T. Okano, and J. Sugiyama, *Polymer*, **38**, 463 (1997).
19. A. Stocchi, B. Lauke, A.Vázquez, and C. Bernal, *Compo. Part A-Appl. Sci.,* **38**, 1337 (2007).
20. J. George, R. Janardhan, J. S. Anand, S. S. Bhagawan, and S. Thomas, *Polymer*, **37**, 5421 (1996).
21. J. Maya Jacob and T. Sabu, *Carbohyd. Polym.*, **71**, 343 (2008).
22. W. G. Trindade, W. Hoareau, J. D. Megiatto, I. A. T. Razera, A. Castellan, and E. Frollini, *Biomacromolecules,* **6**, 2485 (2005).
23. W. Liu, A. K. Mohanty, L. T. Drzal, P. Askel, and M. Misra, *J. Mater. Sci.*, **39**, 1051 (2004).
24. N. Sgriccia, M. C. Hawley, and M. Misra, *Compos. Part A-Appl. S.*, **39**, 1632 (2008).
25. E. Shina and S. K. Rout, *Bull. Mater. Sci.*, **32**, 65 (2009).
26. R. Zuluaga, J.-L. Putaux, J. Cruz, J. Velez, I. Mondragon, and P. Gañán, *Carbohyd. Polym.*, **76**, 51 (2009).
27. H. M. Wang, R. Postle, R. W. Kessler, and W. Kessler, *Text. Res. J.*, **73**, 664 (2003).
28. A. Valadez-Gonzalez, J. M. Cervantes-Uc, R.Olayo, and P. J. Herrera-Franco, *Compos. Part B-Eng.*, **30**, 309 (1999).
29. M. A. S. Spinacé, C. S. Lambert, K. K. G. Fermoselli, and M. A. de Paoli, *Carbohyd. Polym.*, **77**, 47 (2009).
30. H. Yang, R. Yan, H. Chen, D. H. Lee, and C. Zheng, *Fuel*, **86**, 1781 (2007).
31. L. Donaldson, *IAWA J.*, **29**, 345 (2008).
32. M. H. Lee, H. S. Park, K. J. Yoon, and P. J. Hauser, *Text. Res. J.*, **74**, 146 (2004).
33. K. L. Pickering, G. W. Beckermann, S. N. Alam, and N. J. Foreman, *Compos. Part A-Appl. S.*, **38**, 461 (2007).
34. J. Maya Jacob, F. Bejoy, K. T. Varughese, and T. Sabu, *Compos. Part A-Appl. S.*, **39**, 352 (2008).
35. I. Van de Weyenberg, T. Chi Truong, B. Vangrimde, and I. Verpoest, *Compos. Part A-Appl. S.*, **37**, 1368 (2006).
36. D. Ray and B. K. Sarkar, *J. Appl. Polym. Sci.*, **80**, 1013 (2000).
37. M. Kostic, B. Pejic, and P. Skundric, *Bioresource Technol.*, **99**, 94 (2008).
38. C. Baley, *Compos. Part A-Appl. S.*, **33**, 939 (2002).
39. W. Hu, M.-T. Ton-That, F. Perrin-Sarazin, and J. Denault, *Polym. Eng. Sci.*, **50**, 819 (2010).
40. J. Gassan and A. K. Bledzki, *Compos. Sci. Technol.*, **59**, 1303 (1999).
41. K. Charlet, S. Eve, J. P. Jernot, M. Gomina, and J. Breard, *Procedia Eng.*, **1**, 233 (2009).
42. F. A. Andrade Silva, N. Chawla, and R. D. Tolêdo Filho, *Compos. Sci. Technol.*, **68**, 3438 (2008).
43. G. Kulkarni, K. G. Satyanarayana, K. Sukumaran, and. P. K. J. Rohatgi, *J. Mater. Sci.*, **16**, 905 (1981).
44. E. C. McLaughlin, and G. A. Tait, *J. Mater. Sci.*, **15**, 89 (1980).