Chemical and Mechanical Characterization of Two South-American Plant Fibers for Polymer Reinforcement: Caranday Palm and Phormium

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ABSTRACT: Two South-American plants, native Caranday Palm and Phormium, were characterized to investigate their potential as a source of fibers for the preparation of polymer composites. The plant leaves were subjected to different chemical characterizations, whose results were further corroborated by Fourier transformed infrared spectroscopy and Thermogravimetric analysis techniques. The results showed that leaves from Caranday Palm have higher cellulose content than Phormium leaves. The tensile testing of the technical fibers showed that they may be suitable for use in the production of polymer composites, as the properties are comparable to those of other vegetable technical fibers. Preliminary results on the effect of the incorporation of Caranday Palm fibers into polypropylene lead to a composite of improved modulus and relatively low density. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 115: 2236–2245, 2010

Key words: natural fibers; chemical characterization; thermal and mechanical properties; South American fibers

INTRODUCTION

The use of polymer products in general and their composites in particular, is increasing constantly and equally does the impact that they produce on the environment. A large proportion of the commercial polymer composites use glass fibers as reinforcement. These fibers and also their fabrics are nondegradable, and disposal of their derived composites is a growing concern around the world. During the last decade, natural fiber composites have partially replaced glass fiber composites, in nonstructural applications. So far, a good number of automotive components previously made with glass fiber composites are presently manufactured using these more environmentally friendly composites. Similarly, a large body of scientific literature is being devoted to the investigation or demonstration of the potential of cellulose based fibers as reinforcement for plastics.[1,2]

The composite properties are influenced by the fiber properties, which in the case of natural fibers are highly variable and depend on the conditions of the plant growth. Therefore, results repeatability, in particular regarding to the mechanical properties, is still a drawback for many applications. Their low density makes them attractive (natural fibers are about 50% lighter than glass) and in addition they are cheaper than synthetic fibers. Moreover, the incorporation of regional natural fibers into composites, can contribute to the development of local industries and new labor sources, and at the same time add a higher value to a low cost raw material.[3–5]

Caranday Palm is the common name for the palm *Trithrinax Campestris*, native of extended areas in northeast of Argentina. Previous studies, focused on improving fiber quality for traditional applications (cordage, bagging) and for its use in paper pulping, have demonstrated that besides the possibility of obtaining good quality fibers, Caranday leaves were suitable for the production of packaging and printing paper. Caranday Palm is a medium height palm, 2–4 m, exceptionally reaching 9 m, with very rigid and pricking leaves whose limb measures between 0.45 and 0.75 m long [Fig. 1(A)].[6] The leaves cross-section shows a large fibrous zone that spreads all across the leaf and represents about 50% of the total leaf volume.[7] Figure 1(C) shows the cross-section of a leaf and the zone corresponding to the technical fiber.

Phormium is the common name for *Phormium Tenax*, native of New Zealand, Argentina, and Chile. It is commonly called New Zealand flax or hemp, though it has no bast fiber characteristics. Phormium and Istle from north Mexico are the only hard fibers

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produced in template weather areas. In Argentina, Phormium grows spontaneously in the Delta of the Paraná River. It is a perennial plant with a fan-shaped cluster of leaves 1.6–4.3 m long, and 6–10 cm wide [Fig. 1(B)]. Leaves are green with red midrib and margins. Its main uses are coarse bagging and cordages.[7] The residues from Phormium primary product manufacturing are used to make multi-walled bags for industrial packaging of flour, cement and fertilizers.[8] Figure 1(D) shows the cross-section of a leaf and the zone corresponding to the technical fiber.

One of the goals, of this work was to characterize local vegetable fibers extracted from local plants, Caranday Palm and Phormium, both of which require minimal care conditions for their development. Additionally, the potential use of these types of fibers as reinforcement in thermoplastic composites was also addressed.

EXPERIMENTAL

Materials and methods

Leaves from the two plants, obtained from local sources, were cleaned, washed, and dried. Caranday Palm leaves, already soaked in water and cut into strips (0.5 cm wide), were provided by the División de Cultura from La Paz (Entre Ríos, Argentina). Phormium leaves were provided by INTA (National Institute of Agronomical Technology, Buenos Aires, Argentina). Filter paper (Schleicher & Schuell, 589 Black Ribbon, Ashless) was used as a cellulose reference material, to be compared with the residues remaining after different solvent extractions of the leaves. Solvents and reagents were analytical grade and were used as received.

A preliminary investigation on the feasibility of using this type of fibers as reinforcement for thermoplastic composites was undertaken. In this case, short fibers from one of them, Caranday Palm, were incorporated into a polypropylene matrix (PP R6100, Petroken, Argentina, melt index = 8 g/10 min at 230°C and 2.16 kg/cm^2) at a percentage of 50 wt %. A maleic anhydride-graft-polypropylene obtained in our laboratories (melt index = 19.5 g/10 min at 230°C and 2.16 kg/cm^2, percentage of maleic anhydride grafted = 0.46%) was used as a coupling agent.[9]

Fibers preparation

Palm-leaves strips and phormium leaves were cut to a length of 0.1 m, then they were dried at 60°C for 24 h, and milled (Fritsch GmbH, cutting mill) to break the structure of the leaves and to liberate the technical fibers. After the mechanical treatment most of the tissues surrounding the technical fibers were eliminated. Finally, the fibers were sieved (sieve series, Retsch GmbH), while the powders passing the 45 mesh were discarded.

Microscopy

The lengths and diameters of the technical fibers were measured from optical microscopy images (Nikon SMZ-2T, 20X magnification and Leitz Wetzlar, 100X magnification, respectively). The average length from 50 measurements is reported. The average diameter of the fibers utilized in the mechanical characterization was calculated from the average of at least five measurements taken along the fiber. The reported value implicitly assumes that the fibers have a circular cross section.

Leaves cross section surfaces were examined using a Scanning Electron Microscope (Philips SEM 505). The samples were included in epoxy resin, cut, polished, and coated with gold before examination.

Chemical composition of the leaves

To establish their chemical composition, both materials underwent different treatments, which are summarized in Figure 2. Starting from the leaves, one of the routes (Method 1) made possible to independently determine the percentages of: ashes (calcination at 560°C); total proteins, through the determination of total nitrogen (Kjeldahl method); moisture content (drying at 105°C); cellulose and lignin (Goering and Van Soest method).[10]

The other characterization route (Method 2) consisted on subjecting the leaves to a series of successive extractions that allowed to find out the contents of: fats and waxes (extraction with toluene–ethanol); pectins (extraction with water, ammonium oxalate and EDTA) and hemicellulose (extraction with sodium hydroxide) (Fig. 2).[11,12] Finally, the reflux extraction with glacial acetic acid and hydrogen peroxide (50 : 50 by volume) was used to obtain elementary fibers in a single step (Method 3).[13]

Infrared analysis

Milled fibers were analyzed by Fourier Transformed Infrared Spectroscopy (FTIR, Nicolet 5SXC) in the transmission mode, preparing KBr pellets. The absorption spectra were obtained as the average of 32 scans at a resolution of 4 cm^{-1}.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) was performed on a TGA (Mettler TG 50). The measurements were carried out in the 30–700°C range at a heating rate of 10°C/min and under N_2 flow. The sample weight

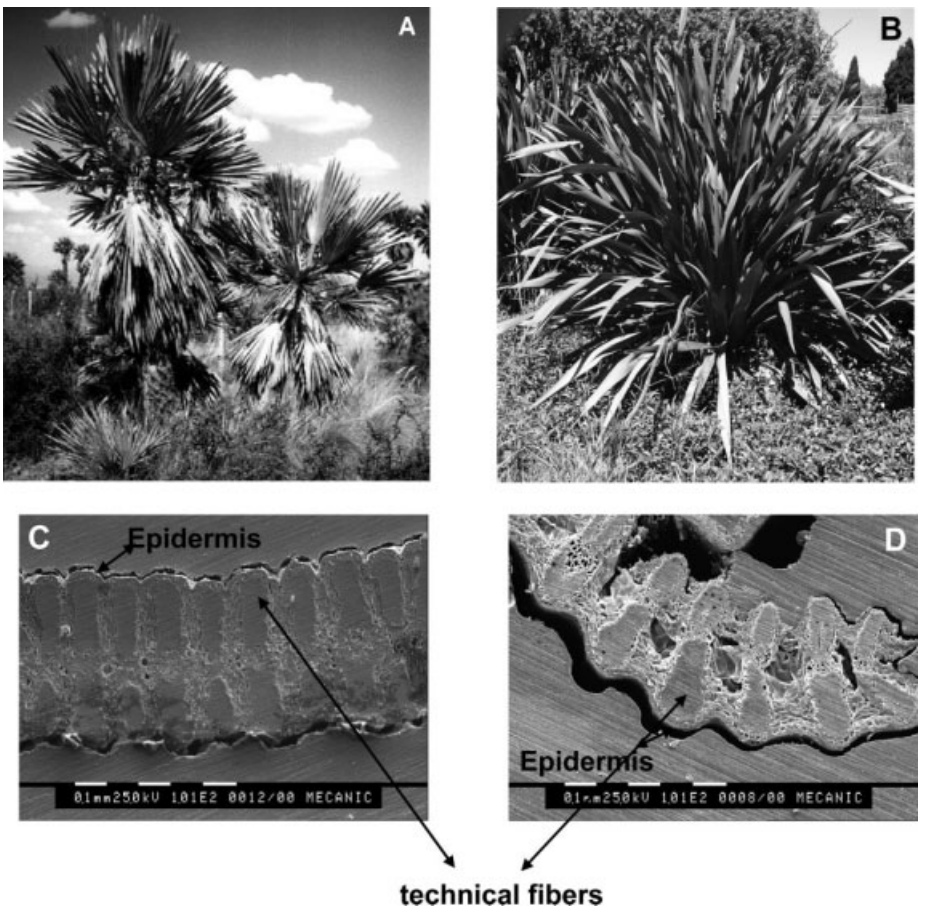

Figure 1 Caranday Palm (A) and Phormium (B) plants and cross-sections of the leaves of Caranday Palm (C) and Phormium (D).

loss versus temperature was measured after each of the chemical treatments corresponding to Method 2. Samples in their powder form were used (approximately 10 mg each).

Tensile mechanical properties of the technical fibers

Single fiber tests were performed on technical fibers longer than 2 cm long. The tests were carried out using a Universal Testing Machine (INSTRON 1125) at room temperature at a crosshead rate of 0.5 mm per minute, according to ASTM D 3379-75. The elastic modulus, strain at failure and tensile strength were calculated from the stress–strain curve. Twenty samples were tested for each type of fiber.

Composite preparation and mechanical characterization

Polypropylene and technical fibers were mixed by extrusion (twin screw extruder, Werner & Pfleiderer) at a 50 : 50 weight proportion. A 5 wt % (with respect to the fiber weight) of the coupling agent was also incorporated to the mixture during extrusion. Composite sample specimens were further obtained by injection molding.

Finally, the tensile properties of the composites were determined at room temperature following the norm, ASTM D- 638, at a crosshead speed of 5 mm/min.

RESULTS AND DISCUSSION

Chemical composition of the leaves

As summarized in the scheme shown in Figure 2, the right route of chemical characterization (Method 1) allows estimating the total protein content by the Kjeldahl method and the cellulose and lignin contents. Table I summarizes the results, indicating a similar composition of the leaves, with the exception of larger cellulose content in the Caranday Palm leaves.

The left route for the characterization of the leaves (Fig. 2) consisted in a series of solvent extractions (Method 2). The first extraction eliminates the fats and the waxes, leaving Residue 1. This residue is extracted with water, ammonium oxalate, and EDTA, which allowed solubilizing the pectins,

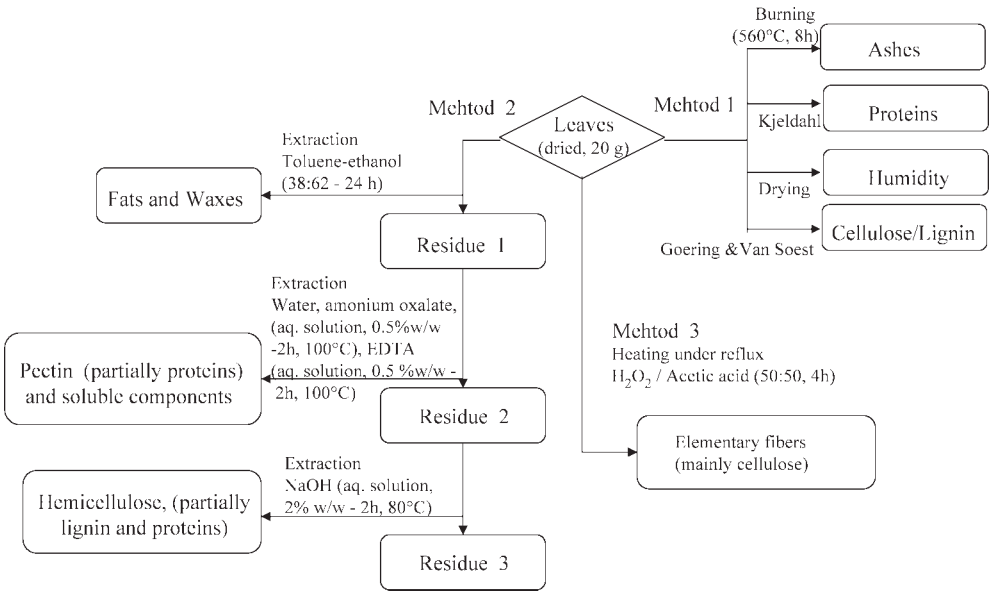

Figure 2 Schematic of the chemical methods used in the characterization of the leaves.

besides other soluble components, such as salts, low molecular weight carbohydrates, and part of the proteins, leaving Residue 2. In turn, when this residue was treated with NaOH, hemicelluloses were mostly eliminated, as well as part of lignin and some proteins. Therefore, the percentage of pectins and hemicellulose calculated by the simple difference between the weights before and after each extraction would be overestimated. With the object of diminishing this error, the content of the total nitrogen in each of the solid residues (Residues 2 and 3) was determined, to obtain an estimation of the mass of proteins extracted in each of these steps. Figure 3(A–D) shows the structure of the Caranday Pam leaves, original and after successive extractions and Table I summarizes the results obtained. The structure of the material is affected by the successive extractions. It looks spongier after extraction of the fats and waxes and a fibrous structure becomes visible [Fig. 3(B)]. In the following micrograph [Fig. 3(C)] the fiber separation becomes clearer and there is one elementary fiber separated at the right. Finally, Residue 3 [Fig. 3(D)] shows separated elementary fibers, although some remaining tissue is still attached. The series of micrographs reveals the intimate structure of the fibers, composed by an amorphous matrix of hemicellulose, lignin, and pectins that puts the ultimate fibers together. In the case of phormium, equivalent results have been obtained.

TABLE I
Chemical Composition of Caranday Palm and Phormium leaves

		Caranday Palm	Phormium
Method	Component	Wt % (total weight)	
Drying at 105°C	Humidity (water)	8.85%	12.21%
		wt % (dry basis)	
Method 1			
Kjeldahl	Total proteins	12.73	12.87
Goering y Van Soest	Cellulose	42.36	37.63
	Lignin	9.52	9.10
Calcination	Ashes	5.76	4.79
Method 2			
Extraction with toluene–ethanol	Fat and Waxes	3.83	4.45
Extraction with Water ammonium oxalate EDTA	Pectins and other soluble components	10.35	12.58
	Proteins	8.69	9.87
	Partial total	19.04	22.45
Extraction with NaOH	Hemicelluloses and partly lignin	19.85	14.07
	Proteins	3.27	1.89
	Partial total	23.12	15.96

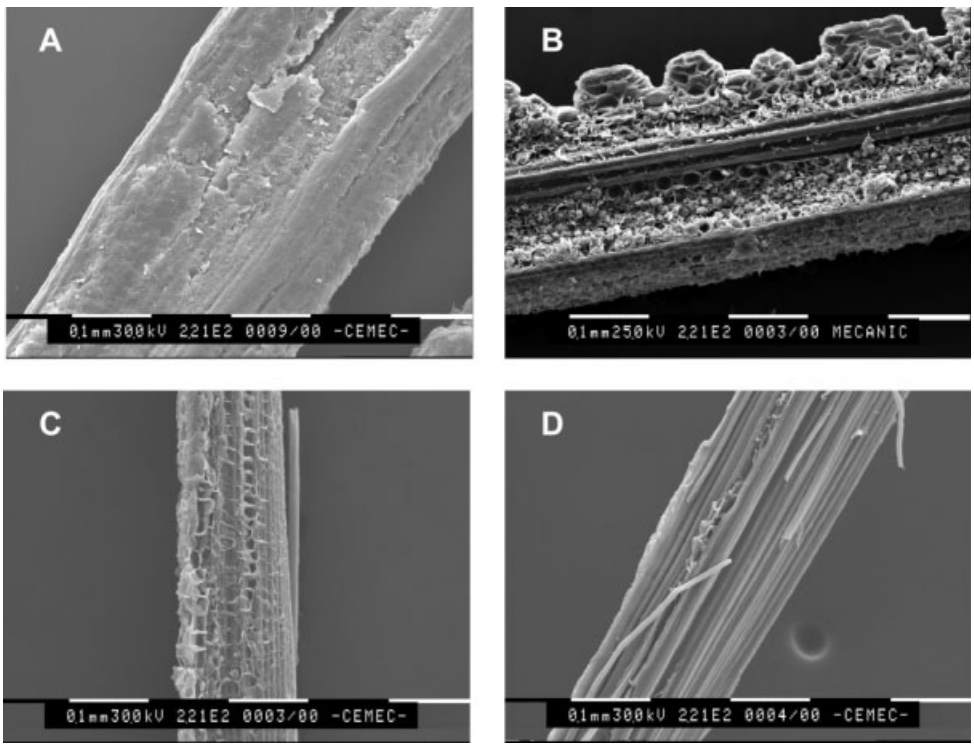

Figure 3 SEM images of the Caranday Palm strips before treatment (A), and after extractions: Residue 1 (B), Residue 2 (C), Residue 3 (D).

The characteristic solubilities of each component partially overlap, so they are not clearly separated and the series of extractions (Method 2) only allows for a rough estimation of the individual component compositions. Besides, the vegetable tissues can include compounds, other than protein, that also contain nitrogen and can contribute with as much as the 30% to the total nitrogen measured.[14] Nevertheless, the accounting for partial proteins extraction of proteins in Method 2 was in agreement with the total protein determination performed by direct measurement (Method 1). As an example, it can be noticed that for Caranday Palm the summation of proteins determined in the successive extractions steps equals 11.96 wt % (from Table I, method 2, total proteins: 8.69 + 3.27 = 11.96), which compares very well with the direct measurement of 12.73 wt % (Table I, method 1).

Table II summarizes the chemical composition of a few vegetable fibers as reported previously by other authors, including Caranday Palm and Phormium. Industrially, the production of plant fibers involves chemical and/or mechanical treatments that soften, dissolve, and break the pectin cements allowing the liberation of the technical fibers. In these processes, pectins and part of the fats that cover the fibers are eliminated, resulting in the lower relative contents reported in Table II. The comparison with the composition of the leaves reported in Table I, illustrates the changes occurring during fiber extraction. Leaves contain more waxes and pectins than the technical fibers.

Additionally, the leaves of both materials were subjected to a reflux with a mixture of hydrogen peroxide and acetic acid in a 50 : 50 proportion (Method 3). This procedure solubilizes the

TABLE II
Chemical Composition of Different Vegetable Technical Fibers, in wt %

Fibers	Moisture	Cellulose	Lignin	Ash	Fats and waxes	Hemicellulose	Pectins	Ref.
Flax	10	56.5, 71.2	2.2	–	1.7	15, 18.6	2.0	4,15
Jute	9.9	63.2, 71.5	13.1, 23	0.7	0.6	13.4, 18	0.2	15,16
Hemp	8.8	67, 77.1	3.3	0.8	–	16.1	0.8	15,16
Kenaf	9.8	55, 65.7	6.8, 8	1.0	–	18, 20	5	15,16
Sisal	6.2	53, 77.2	11.0	1.0	0.3	13.3	0.9	15,16
Caranday palm	9.3	45	20	2.0	1.4 – 1.8	22, 24	–	7,17
Phormium	10–12	45.1, 63	11.2	0.7	–	–	–	16,18

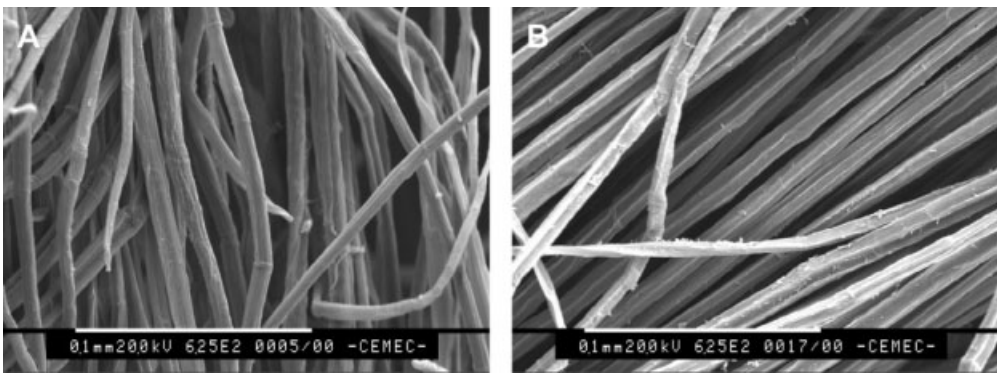

Figure 4 SEM images of elementary fibers obtained from Method 3. Caranday Palm (A), Phormium (B).

components that act as cement (pectins, hemicelluloses, and lignin), releasing the ultimate fibers (Fig. 4). The percentage of lignin remaining after the treatment was determined through the Kappa number (an indication of the grade of delignification of a cellulosic paste and linearly related with the content of Klason lignin).[19] The measured percentages were 0.5 and 1.3 wt %, for Caranday Palm and Phormium, respectively. This indicates that the treatment was effective in releasing the cellulosic elementary fibers, but some contribution from other components are still present after the treatment.[13]

Infrared analysis of the fiber treatments

Figure 5 shows three curves corresponding to the FTIR spectra of the raw fibers of Caranday Palm (A), Residue 3 obtained after extracting waxes and pectins plus hemicelluloses and most of the lignin (curve B) and finally, the spectra corresponding to the ultimate fibers (curve C), obtained by treatment with H_2O_2 and glacial acetic acid, as indicated in the previous section.

The main changes in the spectra appear in three regions 2950−2840 cm^{-1}, 1750−1500cm^{-1}, and 1220–1250 cm^{-1}. In the region of 2950−2840 cm^{-1}, corresponding to the stretch of C—H groups, at least two clear peaks were observed in the A and B curves. This is clear confirmation of the presence of lignin, which absorbs in 2904–2940 cm^{-1} and 2840 cm^{-1}. Cellulose also absorbs in that region as a single peak at 2880 cm^{-1}, as it is observed in curve C, suggesting that after that treatment very little lignin is present in the fibers (as it was also discussed in the previous section).[20]

In the ester carbonyl region (present in pectins and hemicellulose) 1710–1750 cm^{-1}, the used treatments lead to a reduction of the intensity of the peak centered at 1737 cm^{-1} (carbonyl from saturated esters). This is so for the B curve, where the fiber treatment includes the use of NaOH and thus, part of the lignin present in the fibers was extracted. The peak centered at 1516 cm^{-1}, assigned to the aromatic skeleton ring vibration in lignin is also reduced after treatment as shown in the curve for residue 3 (curve B). This effect is more noticeable in the spectrum of the ultimate fibers (curve C), which consist mostly of cellulose, as it was further confirmed by TGA (next section). However, as it was already discussed, a small concentration of the other components is still present (as indicated by the content of Klason lignin), which is considered the main reason for the presence of a small peak in the carbonyl region.

Comparison of the spectra shows that the treatment with NaOH solution produces a change in the 1248 cm^{-1} band, which was assigned to the C—O bond of the aryl eters in lignin. After treatment with NaOH, the band is substantially reduced. This effect has been reported previously in the literature and discussed as characteristic of the alkaline treatment of lignocellulosic materials.[21–23] The low intensity of this absorption peak in spectrum C is again a confirmation of the low contribution of the original components of the fibers other than cellulose (hemicellulose and lignin) present in the ultimate fibers.

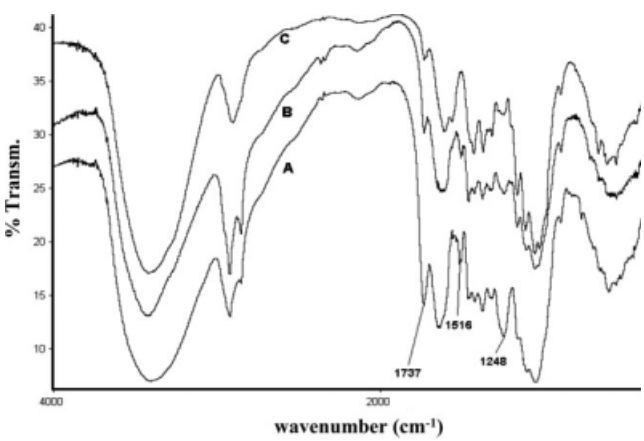

Figure 5 FTIR spectra of Caranday Palm: original leaves (A), Residue 3 (B), elementary fibers (C).

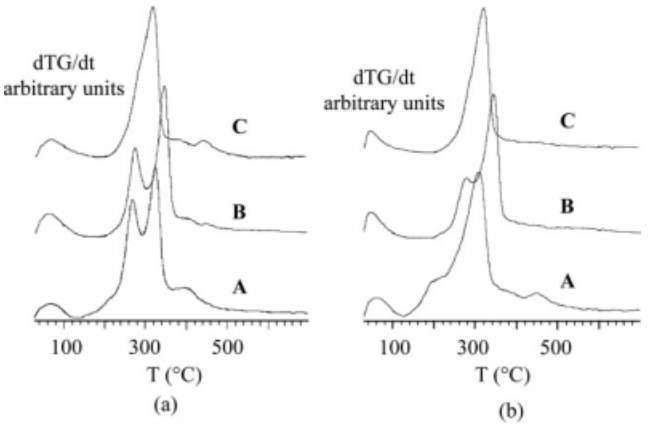

Figure 6 Derivative signal of the thermogravimetric curves for Caranday Palm (a) and Phormium (b): Original leaves (A), Residue 2 (B), and Residue 3 (C).

Similar results were found in phormium fibers, as a result of the different treatments (not shown).

Thermogravimetric analysis

The changes suffered by the vegetable materials after each extraction process were also monitored by TGA. The derivatives of the TG curves (% mass loss per unit time) are shown in Figure 6. They correspond to the materials that were not treated and to the solids named as Residue 2 and Residue 3 (Method 2), illustrating that thermal degradation occurs as a multistep process for all the materials analyzed. The temperatures for each maximum in the degradation rate and the weight losses occurring in each of the steps are summarized in Table III, for all the residues produced by Method 2.

In Figure 6(A) four thermal decomposition peaks can be distinguished. The first one with a maximum at 62°C is related to humidity loss. Above 150°C the carbohydrates (pectins, hemicelluloses, and cellulose) and the lignin are decomposed. The cellulose is, in the group of the carbohydrates, the most thermally resistant, decomposing at a temperature near the 350°C.[24,25] It has been found that although amorphous polysaccharides decompose between 300 and 370°C, hemicellulose's degradation has been reported at such low temperature as 230°C.[26]

Regarding the lignin, its thermal degradation occurs between 180 and 400°C. There is a fourth step that appears at a temperature above 400°C and may be attributed to the decomposition of tars, produced by the pyrolysis of the carbohydrates. The shoulder at 120–220°C, which appears in the thermogram of the original leaves was associated to fats and pectins.[26]

After the first extraction (fats and waxes), TGA of the Residue 1 reveals that the material becomes more hygroscopic, varying the water content from 4–5 wt % to 8–9 wt % in both plants (Table III). This result was expected due to the fact that the fats and waxes act as a barrier against humidity. Furthermore, there is evidence proving that the removal of the fats produces an augmentation in the size of the micropores present in the internal structure of the fibers, allowing the absorption of higher amounts of water.[27]

The thermograms corresponding to the Residue 2 in both species [curves (B) in Figures 6(a,b)] reveal that above the 150°C, the decomposition peaks of the Caranday Palm are shifted towards higher temperature [Fig. 6(a)]. In the case of the Phormium, the material without treatment presented a wide decomposition peak [curve A in Fig. 6(b)]. After the pectin and fat extraction, this peak was resolved in two peaks [curve B in Fig. 6(b)], which maxima coincide with those reported for the Residue 2 of Caranday Palm (Table III). It was also observed, that the shoulder at 120–220°C present in the original material, disappears in Residue 2. The successive extractions with toluene–ethanol, water, ammonium oxalate, and EDTA eliminate fats, waxes, salts, and soluble carbohydrates, pectins, and the majority of the proteins. Therefore, Residue 2, in both cases, is principally made up of cellulose, hemicellulose, and lignin. This purification is clearly reflected in the thermal decomposition curves, which present flatter base lines and better resolved peaks. The components degrade at well-defined temperatures, independently from the plant species, from which they were derived. In particular, the 343–344°C peak is assigned to the thermal degradation of the cellulose.

The ratio of the content of cellulose to the material extracted with NaOH (Ext.Mat2) can be calculated from the results summarized in Table I. This ratio was compared with the ratio of the mass lost during the step of degradation centered at 343–344°C (assigned to cellulose degradation) to the mass lost in the step centered at 273–277°C (Table III).

Caranday Palm: % cellulose/% Ext.Mat.2
$= 42.36/23.12 = 1.83$
mass loss (peak centered at 344°C)/mass loss (peak centered at 273°C)
$= 33.82/19.23 = 1.76$
Phormium % cellulose/% Ext.Mat.2
$= 37.63/15.96 = 2.36$
mass loss (peak centered at 344°C)/mass loss (peak centered at 273°C)
$= 42.90/17.86 = 2.40$

The agreement between the ratios measured and calculated by the two methods is quite good and corroborates the assignment of the 273–277°C peak to the thermal decomposition of hemicellulose majorly.

On the other hand, the lignin decomposition cannot be distinguished in the thermograms, probably

TABLE III
Summary of the Thermogravimetric Analysis Results

	T_{max} (°C)	Weight Loss (%)
Caranday Palm		
untreated	62	4.27
	268	24.76
	323	26.43
Residue 1	70	8.36
	264	22.31
	327	28.32
Residue 2	62	8.36
	273	19.23
	344	33.82
Residue 3	67	8.95
	315	48.29
Elementary Fibers*	41	5.46
	345	72.56
Phormium		
untreated	66	4.78
	306	50.63
Residue 1	53	9.01
	310	51.40
Residue 2	49	7.71
	277	17.86
	343	42.90
Residue 3	43	7.60
	318	52.97
Elementary Fibers*	43	7.46
	341	70.89
Filter Paper	61	5.77
	348	75.04

Temperatures at the maxima of the weight loss rate and weight lost during each degradation step.
* From reflux extraction (Method 3).

because it is present in a low percentage and also, because its degradation is a process that covers a wide temperature range (as it was discussed previously) and overlaps other degradation processes.[26]

Residue 3 in both vegetables presents only one wide peak of decomposition [curves C in Fig. 6(a,b)]. This residue is the solid remaining after the alkaline extraction, in which most of the hemicellulose was eliminated, as corroborated by the disappearance of the 273–277°C peak. However, a small shoulder around this temperature suggests that the hemicellulose removal was not complete. Additionally, although Residue 3 is composed mainly of cellulose, the decomposition temperature (maximum) suffers a shift towards lower temperature as compared to the cellulose-decomposition temperature (maximum) observed in the thermogram of Residue 2. This shift can be associated to the fact that the fibers undergo swelling in the alkaline medium that, without modifying the crystalline phase, can make the amorphous regions more accessible to chemical modifications and to degradation. In particular, it has been observed that NaOH solutions that are not highly concentrated (<8% in weight) reduce the thermal resistance of hemp, sisal, jute, and kapok fibers without altering their crystalline structure.[28] It has also been reported that the cellulose contaminated with sodium salts decomposes at temperature lower than 350°C.[29]

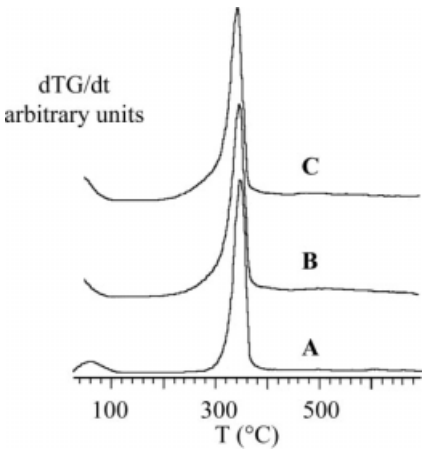

Figure 7 Derivative TG curves for the elementary fibers of filter paper (A), Caranday Palm (B), and Phormium (C).

Figure 7 shows the thermal decomposition curves of the Caranday Palm and Phormium elementary fibers. The curve corresponding to the thermal degradation of filter paper (essentially pure cellulose) is also included for comparison. It can be noticed that the ultimate fibers of both species are mainly constituted by cellulose, and differ from the paper in that their decomposition peaks present a small tail at low temperature. This tail can be attributed to rests of hemicellulose present in these materials (as it was indicated previously in the discussion of the chemical characterization and the FTIR analysis of the fibers).

TABLE IV
Tensile Mechanical Properties of the Technical Fibers

	Young modulus (GPa)	Tensile strength (MPa)	Ultimate strain (%)	Refs.
Caranday palm	17.85 ± 0.98	196 ± 15	1.25 ± 0.40	this work
Phormium	17.10 ± 1.76	280 ± 29	2.17 ± 0.80	this work
Jute	10–30	400–800	1.8	4,13
Sisal	9.4–38	600–700	2–3	4,13
Flax	27.6	345–1035	2.7–3.2	4
Cotton	5.5–12.6	287–597	7–8	4

TABLE V
Mechanical Properties of Polypropylene-Caranday Palm Composite

	Fiber wt %	Density (Kg/m^3)	Young modulus (GPa)	Tensile strength (MPa)	Ref.
PP	0	905	1.12 ± 0.26	31.6 ± 0.1	this work
PP- Caranday Palm	50	1005	4.61 ± 1.11	50.5 ± 0.5	this work
PP-glass fibers	30	1330	5.18 ± 0.17	92 ± 2	9

The properties of the unfilled polymer and a glass fiber composite are included for comparison

Mechanical properties of the technical fibers

Technical fibers longer than 2 cm were manually selected for the mechanical tests. The diameter of the tested technical fibers was determined by optical microscopy and calculated as the average of measurements done in different points of the fibers and assuming that the fibers were cylindrical. The calculated average diameters were 240 ± 87 μm and 254 ± 53 μm for the Caranday Palm and Phormium technical fibers, respectively.

The tensile mechanical properties of the technical fibers were determined and are reported in Table IV. Both fibers show tensile properties comparable to those of other commercial vegetable fibers, with exception of a lower strength, and being closer to those of the jute. Moreover, the Young modulus of Caranday Palm and Phormium fibers are much higher than the corresponding one for thermoplastic polymers, such as polyolefins (0.5–3 GPa), so that they could be an effective reinforcement for this kind of matrices.[2]

Use of caranday palm fibers as reinforcement of polypropylene

To evaluate the performance of the vegetable fibers as potential reinforcement for polyolefins, a composite was prepared using polypropylene as the polymeric matrix and Caranday Palm technical fibers as reinforcement (50 wt %), as described in the Methods section. The fibers used in the preparation of the composite were also characterized by microscopy, resulting in average lengths of 2.46 ± 1.48 mm and diameters of 240 ± 87 μm, the aspect ratio being 10.3 ± 0.2. The composite was tested in extension and the tensile properties are reported in Table V. The results obtained for the unfilled polymer as well as a glass fiber–polypropylene composite are also included in the Table for comparison.

The modulus and tensile strength of the Caranday Palm-composite are both higher than those corresponding to the neat polypropylene (more than three times and 60% higher than the neat polymer, respectively).

Although the properties of the composite prepared from the natural fiber are lower than those measured for the glass fiber composite, they can be acceptable for many applications. The moduli are comparable and the low density of the Caranday Palm composite can be an interesting advantage. The neat polymer showed a density of 0.905 g/cm^3, while the 50 wt % composite had a measured density of 1.005 g/cm^3, not too far from the calculated value of 1.070 g/cm^3 (rule of mixtures considering the density of the fiber as ~ 1.3 g/cm^3). The density of this natural fiber composite is quite low if compared with the density of a glass fiber composite of the same concentration (~ 1.34 g/cm^3, by the rule of mixtures).

From the earlier results, the performance of these natural fibers as polyolefin reinforcements is promising and thus, the properties of their derived composites will be the subject of further studies.

CONCLUSIONS

Two South-American plants, Caranday Palm and Phormium, were characterized to investigate their suitability as reinforcements for polymer composites.

The cellulose content in the plant leaves was high enough to be considered for the intended use, although the Caranday Palm fibers showed a higher percentage of cellulose than Phormium. FTIR and TGA techniques allowed for a good characterization of the residues obtained at each step of the extraction route selected to investigate the nature of the plant leaves. It was shown that extraction performed with acetic acid and hydrogen peroxide lead directly to obtaining elementary fibers consisting mainly of cellulose, with small amounts of hemicellulose and lignin, as detected from TGA and chemical analysis (Kappa number). The mechanical properties of the technical fibers were of the same order of magnitude that those of other vegetable fibers. Additionally, the preliminary results obtained from using Caranday Palm fibers in the production of polypropylene composites showed that these fibers can be effectively used as reinforcements in polyolefin composites.

References

1. Wambua, P.; Ivens, J.; Verpoest, I. Compos Sci Technol 2003, 63, 1259.

2. Reboredo, M. M.; Aranguren, M. I.; Marcovich, N. E. In Polyolefin Composites; Nwabunma, D., Kyu, T., Eds.; John Wiley and Sons., Inc.: Hoboken, New Jersey, 2008; Chapter 6, p 150.
3. Zarate, C. N.; Aranguren, M. I.; Reboredo, M. M. J Appl Polym Sci 2000, 77, 1832.
4. Bledzki, A. K.; Gassan, J. Prog Polym Sci 1999, 24, 221.
5. Zarate, C. N.; Aranguren, M. I.; Reboredo, M. M. J Appl Polym Sci 2003, 89, 2714.
6. Available at. http://www.pacsoa.org.au/palms/Trithrinax/campestrisB.html (accessed June 3, 2009)
7. de Guth, E.B. Revista de Investigaciones Agropecuarias, INTA, Biología y Producción Vegetal, No. 8, 1963, 105.
8. Available at http://www.britannica.com/EBchecked/topic/457357/phormium (accessed June 3, 2009)
9. Krause-Sammartino, L. E.; Lucas, J. C.; Reboredo, M. M.; Aranguren, M. I. Plast Rubber Compos 2006, 35, 117.
10. Goering, H. K.; Van Soest, P. J. USDA - ARS Agricultural Handbook 379; US Government Printing Office: Washington DC, 1970.
11. Morvan, C.; Abdul-Hafez, A.; Morvan, O.; Jauneau, A.; Demarty, A. Plant Physiol Biochem 1989, 27, 451.
12. Catherine, G.-J. Thèse de Doctorat, Université Joseph Fourier: Grenoble, France, 1995.
13. Gritsch, C. S.; Murphy, R. J. Ann Bot 2005, 95, 619; Available at http://aob.oxfordjournals.org/cgi/content/full/95/4/619
14. Van Soest, P. J. In Nutritional Ecology of the Rumiant, 2nd ed.; Comstock Publishing Associates, Cornell University Press: Ithaca and London, 1994.
15. McDougall, G. J.; Morrison, I. M.; Stewart, D.; Weyers, J. D. B.; Hillman, J. R. J Sci Food Agric 1993, 62, 1.
16. McGovern, J. N. Encyclopedia of Chemical Technology, 3rd ed.; Kirk, R. E., Othmer, D. F., Eds.; Wiley: New York, 1984; Vol 10, pp 182–197.
17. de Guth, E. B.; Martire, M. B. IDIA - Suplemento Forestal, No. 7, 1972, 57.
18. de Guth, E. B. Revista Investigaciones Agrícolas XV, 1961, 4, 619.
19. Método TAPPI 236-cm-85 (SCAN-C1:77).
20. Marcovich, N. E.; Reboredo, M. M.; Aranguren, M. I. Compos Interface 1996, 4, 119.
21. Roy, A. K.; Sen, S. K.; Bag, S. C.; Pandey, S. N. J Appl Polym Sci 1991, 42, 2943.
22. Reddy, S. S.; Bhaduri, S. K.; Sen, S. K. J Appl Polym Sci 1990, 41, 329.
23. Aranguren, M. I.; Reboredo, M. M.; Demma, G.; Kenny, J. Holz als Roh und Werkstoff 1999, 57, 325.
24. Beall, F. C. In Encyclopedia of Materials Science and Engineering, 1st ed.; Bever, M. B., Ed.; Pergamon Press: Oxford, 1986; Vol. 7, pp 4933–4935.
25. Marcovich, N. E.; Aranguren, M. I.; Reboredo, M. M. Thermochim Acta 2001, 372, 45.
26. Van de Velde, K.; Baetens, E. Macromol Mater Eng 2001, 286, 342.
27. Mannan, K. M.; Talukder, M. A. I. Polymer 1997, 38, 2493.
28. Mwaikambo, L. Y.; Ansell, M. P. Die Angew Makromol Chem 1999, 272, 108.
29. Huang, M. R.; Li, X. G. J Appl Polym Sci 1998, 68, 293.