Introduction of a New Vegetable Fiber for Textile Application

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ABSTRACT: Finding textile applications for a new or underused fiber is always very attractive, especially when the fiber is natural and indigenous. Leafiran fiber is derived form the leaves of a plant called *Typha australis,* which belongs to the family Typhaceae. The fiber was obtained by chemical retting. Some properties of this fiber, such as its tensile strength, chemical composition, thermal properties, moisture absorption, and IR spectra, were determined. Leafiran is a lignocellulosic fiber having a cellulose content of 54%, a moisture regain of 8–10%, and a tenacity of 29 cN/tex. The results show that Leafiran could be an ideal replacement for some widely used natural textile fibers.
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Key words: Leafiran; chemical retting; Typha australis; tensile strength; fibers

INTRODUCTION

Numerous textile applications, from apparel to industrial textiles, require fibers with various properties obtained through a variety of processes.[1] Some plant fibers, particularly bast fibers, have not been adequately characterized to evaluate their potential as industrial raw materials. Natural fibers from plant sources are of particular interest because they possess a wide range of physicochemical properties, some of which are dependent on the nature of the yielding plants or on the specific parts from which the fibers are derived.[2] There exist a myriad of nonconventional fiber yielding plants with potential for use in various applications that remain to be explored. One is the wildly grown *Typha australis,* which belongs to the family Typhaceae.[3] It is commonly known as *T. australis Schem* with various other names, such as *T. angustifolia Smith, T. domingensis var. australis Geze,* and *T. domiatica Ehrenb.*[4] *T. australis* is an erect, perennial, freshwater aquatic herb that grows to a height of 3 m or more. The linear cattail leaves are thick, with a ribbonlike structure that has a spongy cross section exhibiting air channels. The subterranean stem arises from thick creeping rhizomes.

Typha can be found in wetlands or along slow moving streams, river banks, and lake shores and in areas of widely fluctuating water levels, such as roadside ditches, reservoirs, or other disturbed wet soil areas.[5]

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Because its leaf fiber is very similar to that of hemp or jute, it can be used in the textile industry for the same purposes as they are. Typha leaves are currently wasted because of the lack of knowledge about their textile potential.

For *T. australis* to be put to industrial use, it must first undergo a decortication process to free the leaf fibers. It was the objective of this study to investigate the chemical retting of the fiber in decortications methods. We also dealt with the extraction of the fiber from the *T. australis* leaf, called Leafiran. The major properties of the fiber, such as the fiber strength, linear density, and moisture absorption, and its chemical composition have already been studied for suitability for commercial use.

EXPERIMENTAL

Materials

T. australis was collected from locally grown plants in Isfahan, washed, and dried. The leaf of the Typha plant was then cut into 10- cm lengths. Sodium hydroxide (98%, Shiraz Petrochemical, Shiraz, Iran), sodium tripolyphosphate (Shanghai, China), and ethylene diamine tetraacetic acid (EDTA; Merck, Germany) were used.

Methods

Fiber extraction

For conventional chemical retting, a sample was treated with a 2% caustic soda solution at 80°C for 4 h.[6] In this study, *T. australis* leaves were treated in polymath [AHIBA AG B-100 (in a solution

containing 0.5–6% w/v sodium hydroxide, 0.1–0.3 w/v EDTA, anthraquinone 0.01% w/v, and 1–3% w/v sodium tripolyphosphate)] for 2 and 4 h at 80°C with $L : R = 100 : 1$ (liquor ratio). The treated fiber was thoroughly washed in warm tap water to remove dissolved substances. The fibers were then neutralized with dilute acetic acid 0.2% w/v, rinsed in water, and dried under ambient conditions.

Tensile testing

The tensile tests of the fibers were performed under ambient conditions with a Zwick universal testing machine 1446 60 (Germany) according to ASTM D 3822-01. The gauge length was 20 mm, and the crosshead speed was 2 mm/min. The values are reported as averages of 30 test replicates.[7]

Linear density

We determined the linear density with ASTM D 1577-96 by weighing known lengths of the fibers.[8]

Determination of the density

The density of fibers was calculated from the following equation:

$$\rho = \frac{A}{A-B}(\rho_0 - \rho_L) + \rho_L \quad (1)$$

where ρ is the density of the sample, A is the weight of the sample in air, B is the weight of the sample in the auxiliary liquid, and ρ_0 and ρ_L are the densities of the auxiliary liquid and air (0.0012 g/cm^3), respectively. In this test, distilled water ($\rho = 0.99707$ at 25°C) was used as the auxiliary liquid.[9]

Moisture absorption

We determined the fiber moisture regain with ASTM D 2654-89a by oven drying the fibers at 105°C for 4 h. The samples were then allowed to absorb water under standard testing conditions (21°C and 65% relative humidity) for 24 h. The moisture regain was calculated as the ratio of the amount of water absorbed to the dry weight of the sample.[10]

Fiber composition

The fiber composition was determined with Goeering and Van Soest[11] methods, which provided the percentages of substances present in the fiber obtained through extraction.

IR study

The IR spectra of Leafiran were recorded in a Bomem Hartmann & Braun B-100 spectrophotometer. A pellet was made from dry KBr (potassium bromide) (Merck, Germany), and the powdered sample was placed in the sample holder of the instrument. The spectra were recorded over the range 400–4000 cm^{-1}, with a resolution of 4 cm^{-1}, and averaged over 20 scans.

Thermal analysis

Extracted fiber samples weighing between 5 and 12 mg were analyzed with Linsels thermal analyzer model L70/200171 operated in the dynamic mode, with heating from ambient temperature to 500°C at 10 C/min in air purged at 150 mL/min with an empty pan used as a reference. Differential thermal analysis (DTA) curves and thermogravimetric analysis (TGA) curves were obtained.

RESULTS AND DISCUSSION

Fiber extraction

Natural cellulosic fibers (except for cotton and kapok) are multicellular fibers used in industrial applications as groups of individual cells, known as *fiber bundles*. Lignin, hemicellulose, and other binding substances bind the individual cells into a fiber bundle suitable for textile and other industrial applications. Fiber bundles are obtained from larger vascular bundles in plants by the partial removal of lignin and other constituents, such as hemicelluloses, pectin, and wax, in a process called *retting*, which may employ bacteria and fungi in the atmosphere, chemicals, and enzymes.[12] The challenge in obtaining long fiber bundles from Typha leaf is to prevent the Typha leaf from breaking down into individual

TABLE I
Leafiran Fiber Composition

	Fiber composition											
	ADF (%)	CV (%)	NDF (%)	CV (%)	NCWM (%)	CV (%)	Hemi (%)	CV (%)	Cellulose (%)	CV (%)	Lignin (%)	CV (%)
Typha leaf	53.28	1.62	74.85	2.86	25.15	1.68	21.42	4.3	35.83	3.45	17.60	5.7
Leafiran fiber	82.63	1.44	92.37	1.61	7.62	5.46	9.87	7.6	53.47	8.5	28.54	5.9

Where ADF is Acid Detergent Fiber; NDF is Natural Detergent Fiber; NCWM is Non Cell Wall Material; Hemi is Hemicellulose.

TABLE II
Tensile Properties of the Fibers Extracted at 80°C for 2 h

Chemical bath (w/v)	Linear density (tex)	CV (%)	Modulus (cN/tex)	CV (%)	Tenacity (cN/tex)	CV (%)	Elongation (%)	CV (%)	Work of rupture (cN/tex)	CV (%)
1.5% NaOH + 0.1% EDTA	5.38	7.3	1427.22	17.92	23.08 ± 6.31	27.33	1.19	30.01	0.37	39.9
3% NaOH + 0.1% EDTA	4.60	9.20	1707.64	34.47	28.30 ± 8.97	31.69	1.49	37.6	0.54	39.76
4.5% NaOH + 3.0% EDTA	5.40	12.3	1495.89	21.51	22.66 ± 7.62	28.6	1.35	26.4	0.48	30.4
6.0% NaOH + 3.0% EDTA	5.25	7.40	1544.85	15.18	28.49 ± 6.38	22.40	1.44	22.90	0.54	27.0
8.0% NaOH + 3.0% EDTA	4.31	8.30	1595.83	38.25	24.48 ± 5.76	23.54	1.32	34.93	0.53	31.30

cells. The controlled alkali treatment of Typha leaf developed in this study avoids this breakdown by the partial removal of encrusting substances, such as hemicellulose, lignin, and pectin; this results in fiber bundles suitable for textiles and other applications.[1]

Fiber density

The density of a fiber is the ratio of the mass of the material to the mass of an equal volume of water at 4°C. This is an important characteristic of any fiber, as it affects the way a fabric will drape.[13] The density of the Leafiran obtained from approximately 400 samples was 1.257 ± 0.0612 g/cm^3. This fiber showed a lower density relative to other natural fibers, such as cotton, flax, jute, ramie, and hemp, among others, which typically have densities on the order of 1.4–1.5 g/cm^3.

Fiber composition

Fiber composition affects appearance, structure, properties, and processability.[1] Chemically, Typha leaf fiber is multicellular and lignocellulosic in nature and is composed mainly of polysaccharides, lignin, and other compounds, such as fat, wax, pectin, and hemicellulose. The fibers are cemented together by a gummy-matter-like lignin, which contributes to the strength of the fiber. Natural cellulosic fibers contain anywhere between 60 and 95% cellulose. Hemicellulose, lignin, pectin, waxes, and proteins are the remaining constituents, with their proportions depending on growth conditions, fiber source, and method of fiber extraction.[12] Table I gives the percentages of some of the substances present in the fibers as obtained by extraction. It is clear that the cellulose content of the fibers accounted for about 54% of their total mass, which is much lower than that of ramie and flax, in which the cellulose contents are approximately 73 and 75%, respectively. Most of the hemicellulose is removed during fiber extraction, whereas the remaining hemicellulose, lignin, and pectin hold the individual cells in the form of fiber bundles.[14] Higher amounts of binding materials in the fiber bundle will not only make it coarser but will also decrease the tensile strength of the fiber bundle.[1] It is clear from Table I that lignin, which is a large-molecule compound with a three-dimensional net structure, was relatively stable and was reduced to a smaller molecule under the alkali conditions of the extraction process. In contrast, the noncellulosic wall materials (NCWMs) were removed more significantly during these treatments, from 25.15 to 7.62%.[12] Alkalis, particularly sodium hydroxide, readily react with hemicellulose but have little effect on lignin at low concentrations.[15]

Moisture sorption properties

The moisture absorptivity of a fiber is a major factor affecting the comfort of the final product.[2] Natural fibers are hygroscopic in nature and absorb released moisture, depending on environmental conditions. The higher moisture regain of the Typha fibers (8.5–10.5%) in comparison to cotton was due to the presence of noncellulosic substances, especially hemicellulose and pectin, which are hydrophilic. The high moisture regain of the Typha fibers suggests that apparel made from Typha fibers would be comfortable to wear.[12]

TABLE III
Tensile Properties of the Fibers Extracted at 80°C for 4 h

Chemical bath (w/v)	Linear density (tex)	CV (%)	Modulus (cN/tex)	CV (%)	Tenacity (cN/tex)	CV (%)	Elongation (%)	CV (%)	Work of rupture (cN/tex)	CV (%)
1.5% NaOH + 0.1% EDTA	5.31	15.0	1336.3	24.25	26.3 ± 4.63	12.83	2.46	23.94	1.2	40.96
3% NaOH + 0.1% EDTA	6.32	9.7	818.35	44.03	20.93 ± 10.77	42.3	3.75	32.35	1.28	41.05
4.5% NaOH + 3.0% EDTA	5.03	22.1	1225.95	29.44	18.09 ± 4.23	32.37	1.23	34.56	0.47	18.89
6.0% NaOH + 3.0% EDTA	5.60	21.1	1044.32	42.31	22.78 ± 7.63	33.51	1.76	36.35	0.48	36.6
8.0% NaOH + 3.0% EDTA	6.47	14.2	1247.08	35.10	21.44 ± 8.05	37.54	1.35	28.63	0.39	33.08

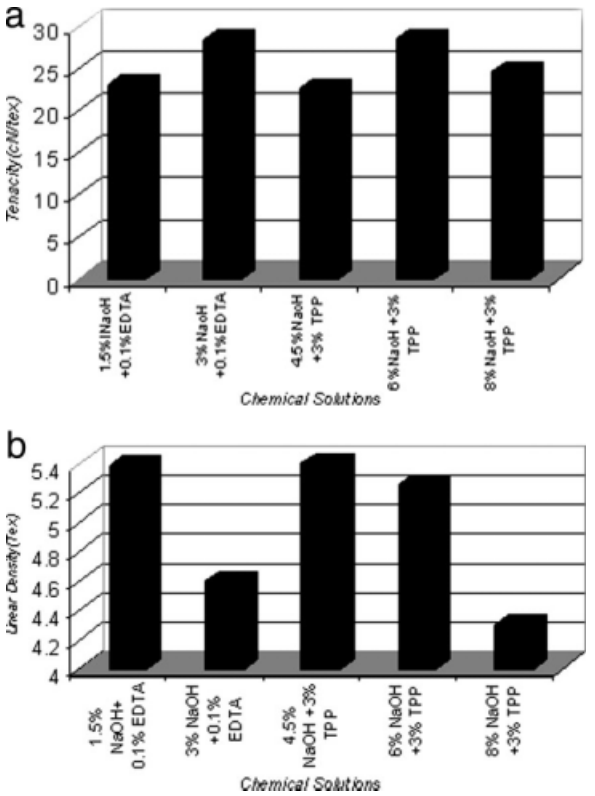

Figure 1 Linear density of the fiber extracted at 80 °C for 4 h.

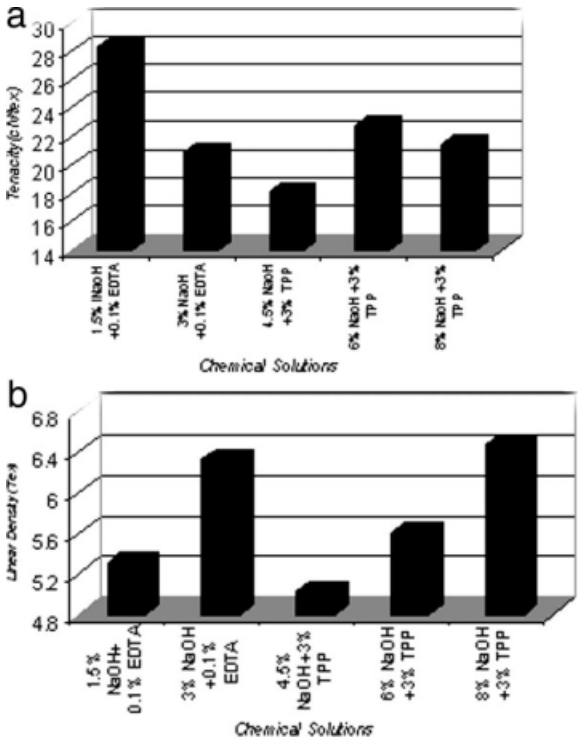

Figure 2 (a) Tenacity and (b) linear density of the fiber extracted at 80°C for 2 h.

Tensile properties

Tensile tests measure the behavior of fibers when a force of deformation is applied along the fiber axis in terms of its tenacity, percentage elongation, initial modulus, and work of rupture. Generally, natural fibers have a characteristic higher tenacity and a lower elongation.[12] *Tenacity* is defined as the specific stress corresponding to the maximum force on a force–extension curve and indicates the load a fiber can bear before it breaks.

Leafiran was subjected to tensile tests, the results of which are reported in Tables II and III and Figures 1 and 2.

Figure 1 shows that the tenacity of the fiber increased with increasing caustic soda concentration because of the enhanced removal of impurities and axis alignment of the fibers. The addition of NaOH improved fiber separation, possibly by neutralizing

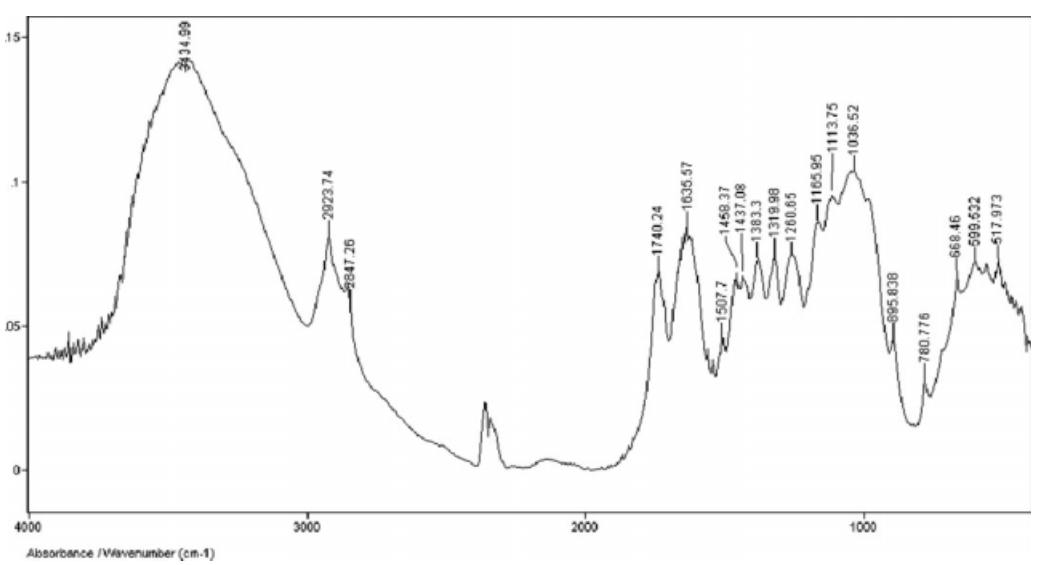

Figure 3 Fourier transform infrared spectra of the Leafiran fiber.

TABLE IV
IR Spectra of the Leafiran Fiber

Bond type	Leafiran
—OH stretching	3434.99
C—H vibration	2923.74
C=O stretching	1740.24
C=C stretching	1635.57
C—H bending	1383.3
C—C stretching	1000–1162
C—H stretching	895.84
—OH	668.9

the acid produced and removing more of the impurities.[16]

The best chemical extractions were obtained at the 3% w/v caustic soda plus 0.1% w/v EDTA concentration and 1.5% w/v caustic soda plus 3% w/v EDTA concentration for 4 h. Caustic soda at 30 g/L plus 1 g/L EDTA at 2 h resulted in greater strengths than the other concentration levels tested. The tensile strength was 29.30 cN/tex, and the linear density was 4.6 tex, whereas at 4 h with caustic soda at 1.5% (w/v) plus 0.1% (w/v) EDTA, the tensile strength was 36 cN/tex, and the linear density was 5.31 tex. The results show that lower caustic soda concentrations were needed when the treatment time was increased.

Fourier transform infrared analyses of the alkaline-treated fibers

The absorbance peaks of interest in this study are identified and shown in Figure 3 and presented in Table IV. In various articles, the strong broad band observed at 3350 cm^{-1} in the spectra is due to hydrogen-bonded O—H stretching vibrations.[3] The hydroxyl groups are also involved in hydrogen bonding with the carboxyl groups, perhaps of fatty acids, available on the surface of natural fibers. This is indicated by the reduction in the peaks between 3200 and 3600 cm^{-1}.[15] The medium-intensity band at 2847–2923 cm^{-1} is attributed to the C—H stretching frequency in methyl and methylene groups.[3] The medium-intensity absorption band at 1730 cm^{-1} is assigned to the C=O stretching of carboxyl and acetyl groups in the hemicellulose content of the fiber, which appeared as a strong peak at 1740 cm^{-1} in the case of Typha fibers and which indicated a higher hemicellulose content. The absorption band at 1635 cm^{-1}, due to vibrations of adsorbed water molecules in the noncrystalline region of cellulose, appeared as a shoulder in the spectra. The sharp peak at 1507 cm^{-1} was caused by aromatic C=C skeletal in-plane vibrations and were diagnostic of the aromatic structure of the lignin present in the fiber and indicated their lignocellulosic characteristics. The band at 1383 cm^{-1} was assigned to C—H deformation (symmetric) arising from lignin and α-cellulose. The peak at 1160 cm^{-1} was assigned to the asymmetric bridge C—O—C stretching in cellulose and hemicellulose, to C—O stretching or O—H bonding of the C—OH group, and also to the aromatic C—H in-plane deformation in lignin. The sharp weak band at 895 cm^{-1} was characteristic of a β-glycosidic linkage contributed by both cellulose and hemicellulose in the fiber.[3,15] The analysis of the IR spectra of the Typha fiber showed characteristic features of lignin and hemicellulose components, which indicated that the fiber was lignocellulosic in nature.

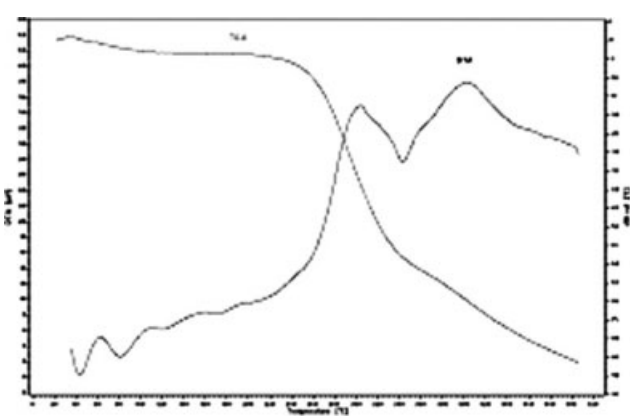

Figure 4 Thermal analysis curves of the Leafiran Fiber.

TABLE V
DTA Peak Temperatures of the Leafiran Fibers

	Temperature (°C)		
	Peak 1	Peak 2	Peak 3
Leafiran fiber	64.25	304.0	402.3

Thermal analysis

The peaks shown in Figure 4 are peaks in the DTA curves that corresponded to points of maximum slope in the original TGA curves. For each of these peaks, the mass loss associated with it was calculated (between two DTA minima), and the corresponding peak temperatures are reported in Tables V and VI. The DTA curves were found to have three exothermic peaks coinciding with regions of weight loss observed through TGA, as shown in Figure 4 for the extracted

TABLE VI
DTA Mass Losses of the Leafiran Fibers

	Mass loss (%)			
	m1	m2	m3	Ash
Leafiran fiber	5.3	57.4	28.6	8.7

Where m1 is weight loss of fiber corresponding to the peak 3 of DTA peak temperatures; m2 is weight loss of fiber corresponding to the peak 2 of DTA peak temperatures; m3 is weight loss of fiber corresponding to the peak 1 of DTA peak temperatures.

fiber. The initial temperature of decomposition was obtained from the first exothermic peak, where the fibers began to decompose. The first peak (64.25°C) below 100°C was due to the evaporation of moisture. The second peak at 304°C and the third one at 402°Cwere due to the decomposition of the hemicellulose and cellulose, respectively.

CONCLUSIONS

Leafiran is a natural vegetable fiber that is derived from the leaves of *T. australis,* which belongs to the family Typhaceae. This cellulosic fiber is similar in structure to common natural cellulosic fibers. Studies of stress–strain relationships have indicated that the fiber is sufficiently strong. The fiber has an initial modulus of 140–200 N/tex, a tensile strength of 25–35 cN/tex, and an elongation at break of 1.2–1.6%. It is lignocellulosic in nature with a 54% cellulose and 28% lignin content. Its low density (1.26 g/cm^3), relatively higher moisture regain (8.5–10%), and good thermal stability give it unique properties.

The results of our ongoing studies of the properties of Leafiran, including better extraction methods, its bleaching and dyeing behavior, and modifications to improve the fiber properties, will be reported in the future.

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