Chemical Composition of Components Comprising Bast Tissue in Flax

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Components from the bast region of flax (*Linum usitatissium*) were removed by hand from seed flax and fiber flax and analyzed by gas chromatography and mass spectrometry. Stems soaked in water were separated by hand into an outer layer, which consisted of epidermis with cuticle and parenchyma cells, and fiber bundles. Each of the two fractions were evaluated for chemical markers that could be used to predict the degree of retting and possibly fiber quality. The outer layer was subsequently treated with a mixed enzyme preparation to remove the carbohydrate portion, thus providing another fraction consisting primarily of cuticle. Four main constituent groups were investigated: dihydroxy fatty acids, long-chain fatty acids and alcohols, sterols, and aromatics. Long-chain fatty acids and alcohols located in the outer layer accounted for 80−92% of the total found in both fractions. Aromatics and sterols in the outer layer accounted for 29−72 and 27−67%, respectively, of the total and do not appear to be a reliable marker for the degree of retting. The best markers for retting were the dihydroxy fatty acids, of which 98−99% were accounted for in the outer layer. The main dihydroxy fatty acids were a mixture of 8,16- and 9,16-dihydroxyhexadecanoic acids and represented 87−89% of the total dihydroxy fatty acids measured. As a constituent of cuticle, this compound may serve as an excellent marker for indicating the degree of retting as well as a possible marker for fiber quality because this compound is almost always exclusively associated with the outer layer and not the fiber.

Keywords: *Flax; retting; waxes; dihydroxy fatty acids; cuticle*

INTRODUCTION

Flax (*Linum usitatissimum*), the source for linen, is an important commercial crop in Europe and is gaining attention in the United States. Considerable interest exists in the United States and Canada for production of flax for use in textiles and a variety of high-value products. Seed flax straw is an option for production of a lower grade fiber for certain composites (*1*), whereas fiber flax could supply the textile market with short fiber that can be blended with cotton. Currently, the residual stem portion of flax grown for seed is burned with a small percentage used in the production of specialty paper and pulp (*2*). If properly retted and processed, the residual fiber can be used for composites and low-quality short staple fiber products. Retting is the microbial activity that separates the flax fibers from the core or shive and "outer layer" (OL) that consists of epidermis with cuticle and parenchyma cells (Figure 1a). Earlier work has shown that possible important markers for the effectiveness of retting and/or the quality are cuticular dihydroxy fatty acids and straight-chain fatty acids and alcohols (*3*, *4*). Separation of the bast fibers from the core and OL is accomplished by water, dew, or enzyme retting.

The cuticle, which is composed of cutin, a lipid polymer, is a network of interesterified hydroxy fatty acids, embedded in a layer of waxy material that serves as an outer barrier for the plant and plays a crucial role in the interaction of the plant with the environment (*5*). For flax it is the primary barrier protecting the plant from indigenous microorganisms important in retting. Akin et al. (*6*) have shown that disruption of the OL by crimping greatly increases the uptake of an enzyme solution. Effective retting requires penetration of the cuticle by some means to allow the action of pectinases to make contact with tissue and effectively release the fiber bundles from the surrounding tissues. Incomplete retting results in small particles of the OL remaining attached to the fibers.

Currently, expert graders and buyers use subjective eye and hand evaluations to determine flax quality. Pieces of the OL and shive that remain attached to the fibers have been associated with lower quality fibers and yarn (*3*, *4*). The present study was undertaken to identify the chemical composition of the OL and to determine which components would serve as a marker-(s) for retting efficiency and possibly as one of many indicators of fiber quality.

MATERIALS AND METHODS

Flax Samples. Flax cultivars Omega (seed flax) and Laura (fiber flax) were grown under identical conditions in Florence, SC, as a winter crop (1999−2000). A third cultivar, Ariane (fiber flax), was grown as a fiber crop (early, harvested May 5, 1999) and also for fiber and seed (late, harvested May 27, 1999), as a winter crop the previous year. Ten centimeter sections were taken from the center of the plant and placed in water for ∼3 h. After soaking, the stems were removed from the water, the OL was removed by hand, and fiber bundles were then removed from the OL-free sections. OL and fiber samples were dried at 35 °C in a vacuum oven. The samples were ground to a fine powder in a Spex 5100 mixer/mill. The

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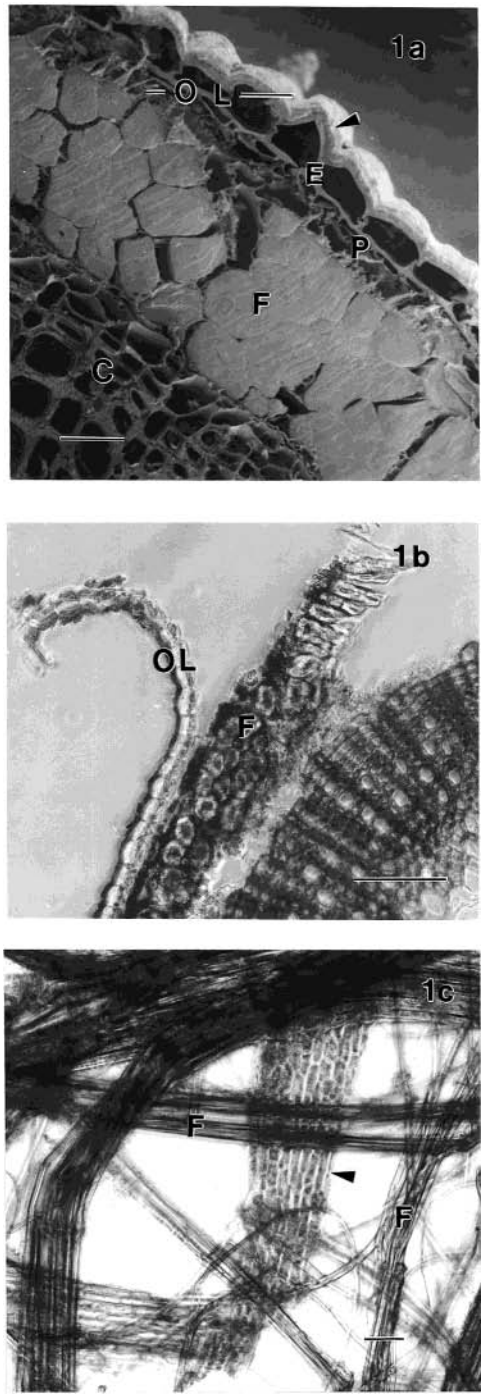


Figure 1. Microscopy of flax stem: (a) scanning electron micrograph of stem cross section showing tissue arrangement [core (C), fiber (F) cell in bundles, and components of the outer layer (OL) including parenchyma (P), epidermis (E) covered with cuticle (arrow); bar = 20 μm]; (b) light micrograph of stem cross section partially retted with a separated OL from the fibers (F) (bar = 100 μm); (c) fiber sample that has been enzyme-retted and cleaned through a Shirley Analyzer showing the presence of cuticle sheets (arrow) with fibers bundles (F) (bar = 100 μm).

percentages of OL and fiber were determined in duplicate by removal of the OL and underlying fiber bundles by hand, drying, and weighing each fraction. The percentage of each fraction was based on the combined dry weights of each fraction.

Enzyme Treatment. Light microscopy of cross sections indicated that the OL consisted of cuticle closely attached to the epidermis, along with attached parenchyma cells. To remove cell wall carbohydrates and obtain a pure cuticle, 100 mg of OL was incubated overnight with a proprietary enzyme mixture (2% in acetate buffer) having high cellulase activity at pH 5 at 50 °C. Another aliquot of enzyme was added, and the sample was incubated for another 24 h. The enzyme solution was decanted, the remaining cuticular material washed with 3 × 10 mL of H_2O, and the sample dried. The yield of pure cuticle was ~10 mg.

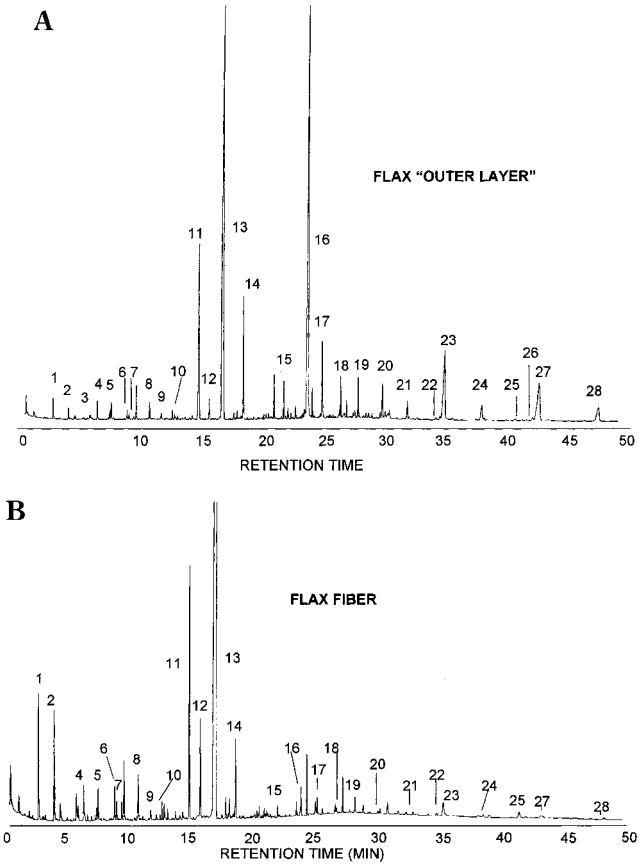


Figure 2. Gas chromatogram of silylated products: (A) OL and (B) fiber after digestion with 4 M NaOH at 170 °C for 2 h, acidification, and extraction with ether.

Microscopy. For scanning electron microscopy, 2-mm sections of unretted flax stems were prepared as described (*11*). For light microscopy of retted flax, free-hand sections of flax stems were enzyme-retted (using an experimental endo-polygalacturonase plus 50 mM EDTA) under conditions similar to those described in Akin et al. (*6*) and examined under phase contrast. Other sections of manually separated OL were cryosectioned at 16 μm thick cross sections and positioned to show the various layers observed using phase contrast light microscopy. Fibers that were cleaned by processing through a Shirley Analyzer were spread onto a microscope slide and observed by light microscopy as above.

Chemical Analysis. Ground samples of fiber, OL, and cuticle from the enzyme treatment of the OL were treated with 4 M NaOH for 2 h at 170 °C, adjusted to pH 2.5 with 2 N HCL, and extracted with ethyl ether. Heating was carried out by placing the sample and base in a screw-capped Teflon vial. The solution was purged with nitrogen, and the vial was capped and placed in a steal reaction vessel containing 7 mL of water. The vessel was sealed and placed in an oven at 170 °C for 2 h. After acidification and extraction with diethyl ether, the ether layer was dried and the residue treated with *N*,*O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA). Analyses of aromatics, fatty acids, alcohols, and sterols were previously described (*3*) on a Finnigan 9001 gas chromatograph using a 0.25 mm i.d. × 30 m DB-5 capillary column (0.25 μm film thickness). Compound identification was by comparison of their mass spectra with published spectra of dihydroxy fatty acids (*7*) or authentic mass spectra (fatty acids, alcohols, and

Table 1. Chemical Composition (Milligrams per Gram) of Flax Outer Layer (OL)[a] and Fiber

		Laura		Omega		Ariane Early		Ariane Late	
peak	compound[b]	OL	fiber	OL	fiber	OL	fiber	OL	fiber
1	BHT (from ether)								
2	(A) vanillin	1.36 ± 1.3	0.06 ± 0.01	1.54 ± 1.4	0.29 ± 0.11	0.41 ± 0.2	0.15 ± 0.03	0.49 ± 0.2	0.27 ± 0.07
3	(A) acetovanillone	0.44 ± 0.4	0.02 ± 0.01	0.37 ± 0.16	0.10 ± 0.03	0.11 ± 0.03	0.04 ± 0.02	0.09 ± 0.03	0.06 ± 0.02
4	(W) C-12 fatty acid	1.7 ± 0.4	0.09 ± 0.02	0.51 ± 0.13	0.17 ± 0.02	1.54 ± 0.6	0.10 ± 0	0.68 ± 0.03	0.11 ± 0.06
5	(A) syringaldehyde	0.45 ± 0.3	0.02 ± 0.01	0.18 ± 0		0.1 ± 0.02	0.03 ± 0.01	0.58 ± 0.06	0.08 ± 0.01
6	(A) vanillic acid	0.44 ± 0.3	0.03 ± 0.01	0.47 ± 0.3	0.1 ± 0.03	0.08 ± 0.04	0.08 ± 0.01	0.3 ± 0.01	0.11 ± 0.01
7	(A) acetosyringone	0.72 ± 0.6	0.03 ± 0.02	0.83 ± 0.6	0.26 ± 0.2	0.37 ± 0.25	0.21 ± 0.25	0.18 ± 0.01	0.11 ± 0.01
8	(W) C-14 fatty acid	0.95 ± 0.2	0.14 ± 0.01	0.81 ± 0.1	0.32 ± 0.03	0.85 ± 0.18	0.13 ± 0.02	0.76 ± 0.03	0.11 ± 0.06
9	(A) syringic acid	0.19 ± 0.1		0.10 ± 0.03		0.05 ± 0.2	0.03 ± 0.0	0.20 ± 0.03	0.03 ± 0.01
10	(A) *p*-coumaric acid	0.35 ± 0.1	0.05 ± 0	0.47 ± 0.23	0.13 ± 0.1	0.42 ± 0.06	0.08 ± 0.01	0.34 ± 0.03	0.06 ± 0.01
11	(W) C-16 fatty acid	16.9 ± 2.8	0.65 ± 0.08	8.77 ± 0.7	1.44 ± 0.14	14.5 ± 3.9	0.81 ± 0.10	9.66 ± 0.5	0.74 ± 0.03
12	(A) ferulic acid	1.89 ± 1.1	0.29 ± 0.16	3.6 ± 2.2	1.17 ± 0.7	0.65 ± 0.31	0.21 ± 0.06	0.81 ± 0.01	0.35 ± 0.03
13	C-17 fatty acid (std)								
14	(W) C-18 fatty acid	10.8 ± 0.3	0.22 ± 0.06	6.09 ± 0.36	0.54 ± 0.13	6.7 ± 0.47	0.24 ± 0.01	5.3 ± 0.27	0.22 ± 0.03
15	(W) C-20 fatty acid	1.30 ± 0.2	0.04 ± 0.01	0.80 ± 0.02	0.07 ± 0.01	1.46 ± 0.12	0.11 ± 0.05	1.42 ± 0.13	0.04 ± 0
16	(C) 8(9),16-dihydroxy C-16 fatty acid (mixture)	73.7 ± 15	0.07 ± 0.01	31.0 ± 2.4	0.01 ± 0	71.9 ± 2.9	0.02 ± 0.02	38.4 ± 1.8	0.11 ± 0
17	(C) 8(9),17-dihydroxy C-17 fatty acid (mixture)	6.1 ± 0.6	0.03 ± 0	2.29 ± 0.1	0.06 ± 0.02	5.4 ± 0.41	0.03 ± 0	3.91 ± 0.09	0.1 ± 0.4
18	(C) 9(10),18-dihydroxy C-18 fatty acid (mixture)	4.3 ± 0.6	0.01 ± 0	1.8 ± 0.14	0.01 ± 0	3.48 ± 0.31	0.01 ± 0.01	1.43 ± 0.05	0.05 ± 0.02
19	(W) C-24 fatty acid	1.93 ± 0.2	0.03 ± 0	0.79 ± 0.09	0.1 ± 0.05	0.97 ± 0.26	0.08 ± 0.04	1.67 ± 0.03	0.06 ± 0.01
20	(W) C-26 alcohol	3.03 ± 0.4	0.02 ± 0.01	2.09 ± 0.1	0.04 ± 0.01	1.78 ± 1.2	0.01 ± 0.01	1.71 ± 0.1	0.03 ± 0.01
21	(W) C-26 fatty acid	0.81 ± 0.1	0.01 ± 0	0.56 ± 0.01	0.02 ± 0	0.6 ± 0.12	0.01 ± 0	0.96 ± 0.09	0.01 ± 0
22	cholesterol	0.47 ± 0.1	0.05 ± 0.02	0.35 ± 0.07	0.11 ± 0.03	0.14 ± 0.15	0.04 ± 0.01	0.14 ± 0.03	0.02 ± 0
23	(W) C28 alcohol	18.9 ± 2.4	0.04 ± 0.04	12.6 ± 0.45	0.15 ± 0.02	6.03 ± 2.6	0.05 ± 0.02	7.92 ± 1.02	0.13 ± 0.06
24	(W) C-28 fatty acid	1.61 ± 0.2	0.01 ± 0	1.9 ± 0.01	0.02 ± 0	0.71 ± 0.12		1.27 ± 0.2	0.04 ± 0.02
25	sitosterol	0.87 ± 0.12	0.08 ± 0.06	0.36 ± 0.09	0.26 ± 0.02	0.06 ± 0.06	0.06 ± 0.02	0.16+0.02	0.04 ± 0.02
26	β-amyrin	0.36± 0.09	0.06 ± 0.02	0.21 ± 0.06	0.07 ± 0.02	0.06 ± 0.03	0.01 ± 0	.010 ± 0.02	0.01 ± 0
27	(W) C-30 alcohol	4.85 ± 1.3		1.0 ± 0.18		1.08 ± 0.31		5.93 ± 1.1	0.04 ± 0.04
28	(W) C-30 fatty acid	0.83 ± 0.1		0.51 ± 0		1.46 ± 0.12		2.1 ± 0.41	0.04 ± 0
	percentage of component	18.3	81.7	24.1	75.9	13.9	86.1	21.0	79.0
	total aromatics	5.84	0.50	7.56	2.05	2.19	0.83	2.99	1.07
	total wax	63.6	1.25	36.4	2.78	37.7	1.54	39.4	1.57
	total cutin	84.1	0.11	35.1	0.08	80.8	0.06	43.7	0.26
	total sterols	1.7	0.19	0.92	0.44	0.26	0.11	0.31	0.07

[a] OL = epidermis + cuticle + parenchyma tissues. [b] (A), those compounds counted as aromatics; (W), those compounds counted as wax; (C), those compounds counted as cutin.

sterols). Cell wall sugars present in the OL were analyzed as their alditol acetates by gas chromatography according to the method of Hobler et al. (*8*) with the initial digestion time increased from 30 to 90 min at 40 °C. The column used was a 0.25 mm i.d. × 30 m DB-225 capillary column (0.15 μm film thickness).

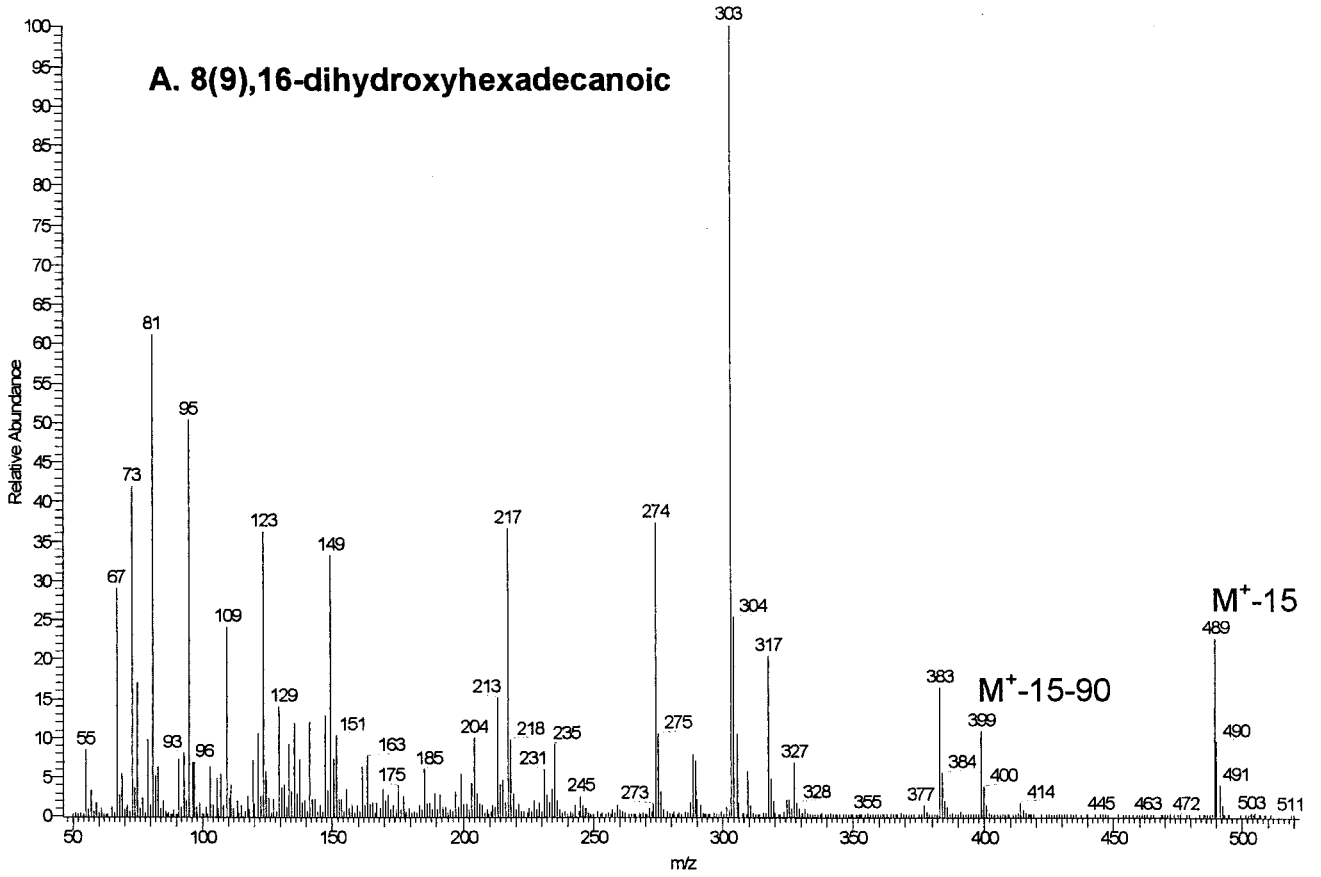
RESULTS AND DISCUSSION

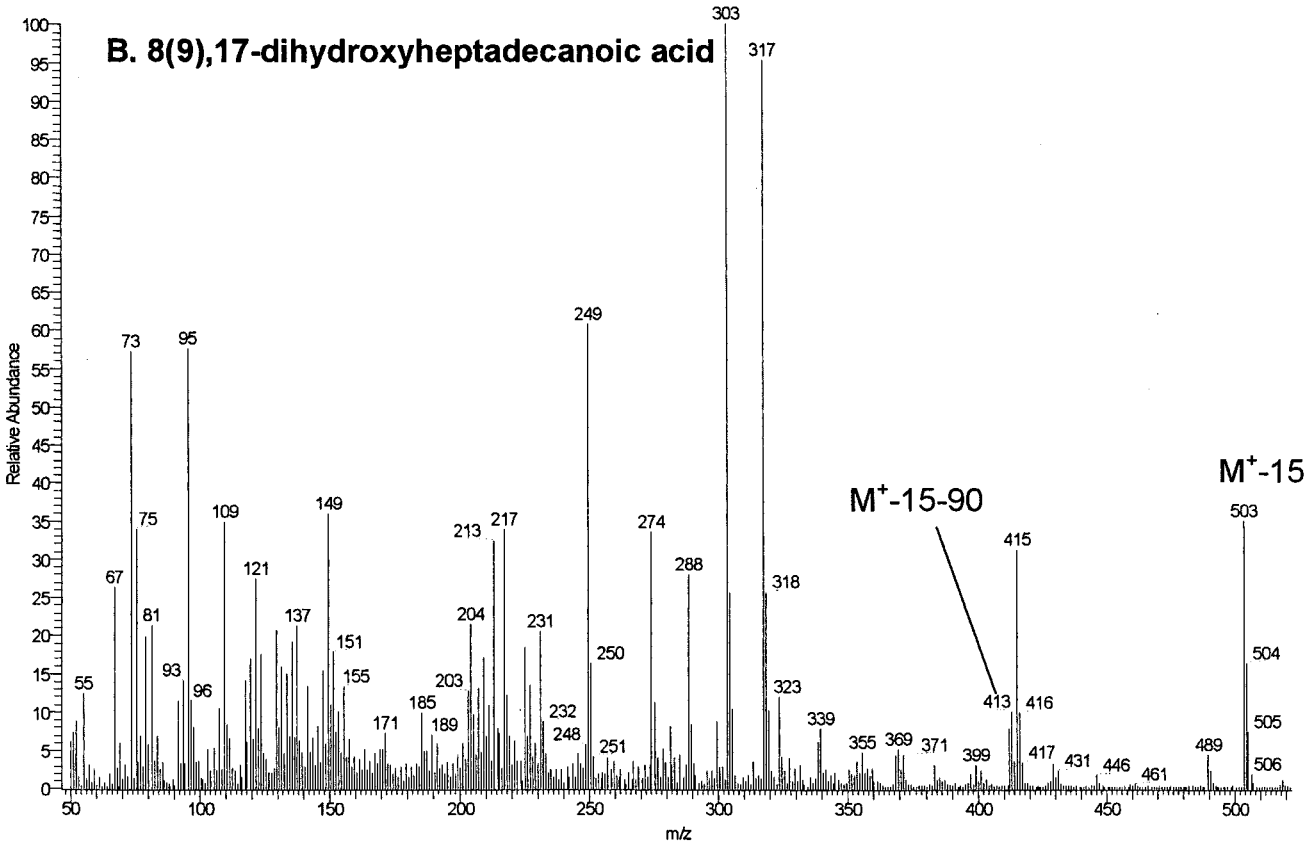
Figure 1a shows a scanning electron micrograph of a cross section of a flax stem indicating the tissue arrangement. The OL consists of the closely attached cuticle and overlaying epidermal cells, followed by a layer of parenchyma cells (Figure 1b). Scanning electron microscopy shows the arrangement of tissues in intact, unretted flax stems (Figure 1a). The OL is the material to the outer side of and excludes the bast fiber bundles. The OL consists of the cuticle tightly adhered to and on the outer side of the epidermal cells, all of which overlays an inner tissue of parenchyma cells, which appear collapsed in the micrograph. The parenchyma cells are adjacent to the fiber bundles in intact stems. Retting degrades pectins and matrix polysaccharides in the parenchyma cells, thus permitting separation of the epidermis/cuticle barrier of the OL from the fibers (Figure 1b). Furthermore, retting results in degradation of the region between the fiber and core (Figure 1, F and C), further separating fiber bundles from other nonfiber tissues. Incomplete retting leaves a portion of the OL attached to the flax fibers (Figure 1b). This residual OL is often difficult to remove even after subsequent processing to clean fibers as shown by cuticle sheets associated with fibers (Figure 1c).

Our goal was to determine if the cutin and wax markers observed in earlier work that were associated with fiber quality (*3*) are specific to the OL or fiber. For this study, the OL and underlying fibers were removed by hand from stems of fiber and seed flax as well as fiber flax that had been harvested early and late. The aromatic, cutin, sterol, and wax components of the OL and underlying fiber are shown in Figure 2, and the identification of the compounds is shown in Table 1.

The principal dihydroxy fatty acids associated with cutin were mixtures of 8,16- and 9,16-dihydroxyhexadacanoic (M^+, *m*/*z* 504), 8,17- and 9,17-dihydroxyheptadecanoic acids (M^+, *m*/*z* 518), and 9,18- and 10,18-dihydroxyoctadecanoic acids (M^+, 532) (peaks 16, 17, and 18, respectively, Figure 2). The mass spectra of their trimethylsilyl (TMS) ethers are shown in Figure 3. The major component of these dihydroxy fatty acids was a mixture of 8,16- and 9,16-dihydroxyhexadecanoic acids. The expected α-cleavage of these compounds produced fragments at *m*/*z* 303 and 317 for the 8,16- and 9,16-dihydroxy isomers (*7*) (Figure 3A). In equal amounts were mixtures of 8,17- and 9,17-dihydroxyheptadecanoic acids producing analogous α-cleavage produced at *m*/*z* 303 and 317 (Figure 3B), whereas α-cleavage produced fragments at 9,18- and 10,18-dihydroxyoctadecanoic acids with products at *m*/*z* 317 and 331 (Figure 3C) (*7*). All exhibited weak parent ions and *m*/*z* of $M^+ - 15$ and $M^+ - 15 - 90$ at the expected *m*/*z*.

Of the two types of groups of tissue removed, the OL (consisting of cuticle, epidermis, and parenchyma) and the fiber, the percentage of OL ranged from 13.9 to 24.1% (dry weight) (Table 1). These figures were used


A. 8(9),16-dihydroxyhexadecanoic
Relative Abundance
m/z
M+-15
M+-15-90


B. 8(9),17-dihydroxyheptadecanoic acid
Relative Abundance
m/z
M+-15
M+-15-90

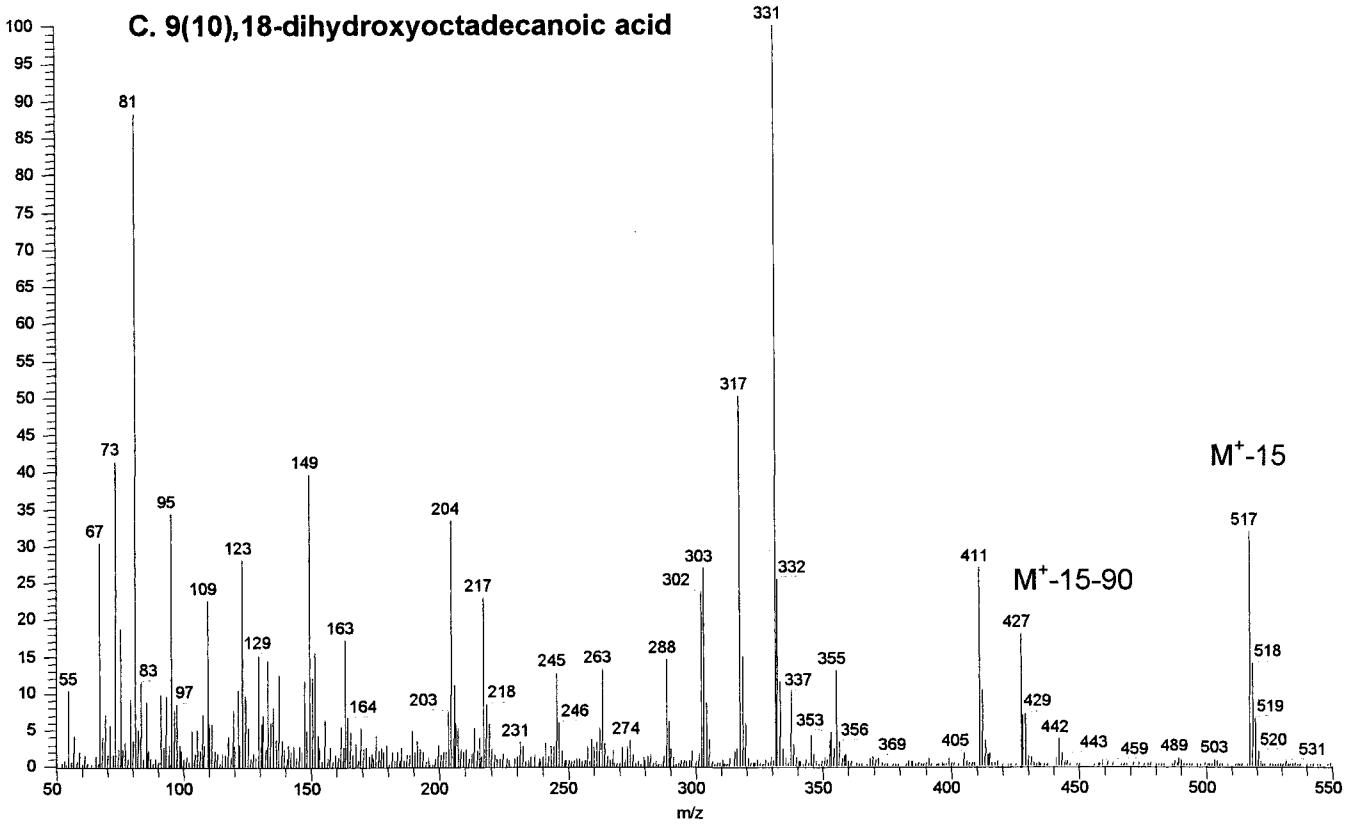


Figure 3. TMS ethers: (A) peak 16, 8(9),16-dihydroxyhexadecanoic acid; (B) peak 17, 8(9),17-dihydroxyheptadecanoic acid; (C) peak 18, 9(10),18-dihydroxyoctadecanoic acid.

to determine the contribution of aromatics, waxes, cutin, and sterols to the OL and fiber fractions by multiplying the dry weight percentage of OL and fiber times the weight of each component in that fraction. These results represent actual amounts of each component in the OL and fiber on a milligram per gram basis. Of the total dihydroxy fatty acids, 97.8−99.6% were located in the OL fraction and the remainder in the fiber fraction. This proportion suggests that any dihydroxy fatty acids remaining on the fiber after processing are a result of incomplete removal of the OL.

The predominant long-chain fatty acids found in both tissues were palmitic and stearic acids, with 70−80% being found in the OL. The remaining fatty acids ranged from C_{20} to C_{30}. The long-chain alcohols identified ranged from C_{24} to C_{30}, with octacosanol and hexacosanol predominating. The relative percentages of the fatty acids and alcohols found in flax were in good agreement with the results of Tulloch and Hoffman (*9*). Collectively, the OL contained 80−96% of these alcohols and acids but they were not exclusively associated with either fraction to any extent, as were the dihydroxy fatty acids. To prevent problems with dyeing, the wax content in flax yarn should be <0.3% (*10*). These data suggest that some long-chain fatty acid alcohols and acids remain on the fiber after processing and may be associated with the fiber and independent of the composition of the OL. The relative percentage of palmitic and stearic acids in the fiber is higher than the remaining acids and alcohols, which suggests a preferential deposition of these acids on the fibers.

Earlier work has shown that within the fibers, primarily at cell wall junctures and middle lamellae (*11*), there is a substance that gave an intense reaction with acid phloroglucinol (a stain for lignin). Sharma et al. (*12*) and McDougall et al. (*13*) report lignin in flax is ∼1.5−2%. The differences in the levels of aromatics between fiber and cuticle were not as large as the levels of the cutin and wax markers. Earlier work has shown that the composition of lignin with regard to syringyl/guaiacyl lignin can be determined by caustic treatment at elevated temperatures, followed by analysis of the aromatic degradation products (*14*). Using this procedure, 29−72% of the aromatics can be accounted for in the OL (Table 1). Because the main source of aromatics in the flax stem is associated with the core, most of the aromatics associated with the fiber result from core material remaining attached to the fiber after processing. Treatment of the OL with a cellulase-rich preparation followed by analysis of the residual cuticle for aromatics gave lower levels of the phenolics associated with just the OL. Ferulic and *p*-coumaric acids occur in plants as part of the aromatic constituents; often these compounds are ester-linked with cell wall arabinoxylans (*15*−*18*). In an earlier study, the amount of arabinose, xylose, and glucose in samples of dew-retted fiber averaged 6.8, 10.7, and 661 mg/g, respectively (*19*). The OL contained 52, 28, and 105 mg/g arabinose, xylose, and glucose, respectively. These data indicate that hemicellulose is the primary polysaccharide in the outer layer. Phenolic acids in the fiber fraction could be from incomplete removal of the parenchyma tissue and residual core material, the main source of lignin in the flax stem, during hand separation as well as from aromatics in the bast fiber middle lamella.

The sterols identified were cholesterol, sitosterol, and *β*-amyrin and are associated with cutin and suberin waxes (*20*). Sterols in the OL accounted for 27−67% of the total from both fractions (Table 1). Therefore, it does not appear that these compounds would serve as reliable

markers for retting efficiency because they were not associated exclusively with either fraction.

In our earlier work (*3*, *4*) we suggested that dihydroxy fatty acids and long-chain fatty acids and alcohols could serve as markers for the quality of flax fiber and yarn. Microscopic examination of graded fiber samples revealed more OL attached to samples that were graded as being of low quality. Fiber samples that had undergone a scouring step (i.e., a caustic treatment of the fiber) in the production of yarn revealed bits of OL remaining on the fibers that would contribute waxes and cuticle as contaminants. Fiber graded as being of high quality contained 14.1 mg/g dihydroxy fatty acids, whereas low-quality fiber contained 15.3 mg/g (*3*). Processed yarns still contained measurable amounts of these acids, with high-quality yarn containing 3.7 mg/g and low-quality, 9.9 mg/g. These values are considerably higher than those found in the fiber in this study that has had the OL removed by hand and reflect the difficulty in completely removing these tissues during processing flax fiber to yarn. "Wax" markers for high- and low-quality fibers were 6.9 and 11.5 mg/g, respectively, as compared to an average of ~1.7 mg/g found on the hand-separated fibers in this study. High-quality yarn contained 0.6 mg/g compared to 4.5 mg/g for low-quality yarn, again reflecting the difficulty of removing these compounds during processing. Although our initial values for long-chain alcohols and acids did show an inverse relationship with quality (*3*), it appears that 8(9),16-dihydroxyhexadecanoic acid, as a constituent of the cuticle, may serve as an better single marker indicating the degree of retting efficiency. Furthermore, this may also serve as one of many indicators of quality, as it is almost exclusively associated with the OL.

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