# **FLAX FIBER**

# 1. Introduction

Flax (*Linum usitatissimum* L.) is a versatile crop that is grown throughout the world and in a variety of climates. The translation of its scientific name, "linen most useful" (1,2), aptly describes its versatility. Linen, which is used for apparel and interior textiles, comes from the long, strong bast fibers that form in the outer portions of the flax stem (3). Flax fibers also are used in industrial applica-

tions, eg, composites, geo-textiles, insulation, and specialty papers (4,5). Flax seeds are the source of linseed oil, which has been widely used in paints, varnishes, cosmetics, and linoleum (6). More recently, flax seeds are being recognized as a health food, with nutritional benefits from lignans and omega-3 fatty acids (7,8). Even the woody core tissue (shive), which is removed during cleaning of fiber, is used for particleboards and animal bedding (9). Linen, which is valued for comfort and its distinctive appearance, remains a favorite in the textile industry. With the burgeoning interest in natural fibers for a variety of industrial uses (4,10), flax fibers provide the potential to supply these applications from diverse, nontraditional linen sources.

# 2. History and Status of Flax and Linen

There can be no doubt that the history of flax and linen is long and storied. Hamilton (3) states that the use of the long, strong fibers from flax stems for making linen is one of the earliest successes in textiles. Evidence throughout the world attests to the widespread knowledge and use of flax and linen. Linen samples have been reported in the remains of Swiss lake dwellings dating back some 10,000 years (3). Flax was reportedly known as far back as 8000-9000 years to inhabitants in the ancient seacoast regions of modern day Denmark and Turkey (11). Franck (12) speculates that how ancient peoples discovered the process of extracting flax fibers from the stems, possibly an accidental observation of weathering and mechanical handling of stems, will likely never be known. Flax as a major textile in ancient Egypt, however, is well documented and frequently referenced (13). While flax is considered to have been first cultivated in Egypt, there is speculation that the origins of the plant might have been in other regions (eg, between the Baltic and Caspian Sea), subsequently coming to Egypt via China or India (14). The high value products were important to Egyptian society as shown in artistic depictions of the cultivation and processing of this crop. Linen shrouds used to wrap mummies have been reported to remain for  $\sim$ 7000 years. Notably, the high quality linen from Tutankhamun's tomb has survived  $\sim 3500$  years (3). Linen continued to be produced and used in the ancient Middle East and surrounding countries. References to linen are prevalent in the Old and New Testaments of the Bible, with Hebrews regarding the material as symbolic of purity and cleanliness (15). Linen production and use expanded beyond the Mediterranean countries to central and northern Europe (12). Linen-making was likely introduced to Great Britain  $\sim 2000$  years ago from the Middle East by Phoenician traders (3). Linen along with wool were the primary fibers for Europe throughout the Middle Ages and the Renaissance, with flax fibers used extensively for clothing and a variety of other applications.

Flax fiber has been particularly important to Russia and its economy through various stages of its political history (16). In 1653, English merchants reported Russian hand-spun yarn and linen to be of very high quality. Early on in Russia, flax was graded for quality based on retting type and spinning characteristics. The old czarist "tax flax" was so called because taxes could be paid with it. Flax became the greatest export item and the basis of economic life in Russia in the late 1800s and into the twentieth century. At one time, Russia produced  $\sim$ 80% of the world's fiber flax crop and before 1936 was the greatest exporter of flax. Commerce and processing assessments depended on a judging and classification system, which was based originally on the ability to spin the flax fibers.

Fiber flax was introduced to North America by European colonists, with reports of the crop grown in Connecticut as early as 1640 (Jenkins in 17). Early colonial law required every family to grow  $\sim 0.05-0.1$  hectares of flax or hemp, and in the early 1800s two Connecticut countries led all of New England in flax production. With the widespread settlement by English colonists, flax and linen were used throughout the eastern coast of the United States, as indicated, eg, by historical markers along the Blue Ridge Parkway in North Carolina reporting the use of flax for a wide variety of farm and household needs. While flax was grown in several regions of the United States, particular states had wellorganized efforts. Robinson and Hutcheson (18) reported that fiber flax was grown on a commercial scale for several years in eastern Michigan and the Willamette Valley in Oregon. Flax has been grown in Oregon since 1844, with the seed carried across the plains during early settlement (15,19). Flax fiber produced in Oregon and exhibited at the Philadelphia Exposition in 1876 won the Bronze Medal and Certificate of Merit for its outstanding quality (15). Afterward, little progress occurred in developing a flax industry in Oregon until 1915, when the Oregon state legislature appropriated funds to establish a flax processing plant in the state penitentiary. With labor available noncompetitively in this environment, supporters of the project hoped to foster a flax fiber industry. In 1932, the U.S. Department of Agriculture and the Oregon Agricultural Experiment Station began agronomic work on flax production (19). Earlier work begun in Michigan was moved to Oregon, and efforts continued on agronomic, engineering, and marketing aspects of flax fiber. Accounts of the Oregon experience, including production yields, processing mills, and advancements in many areas, are well documented (15,16,19). As in Europe, specially designed equipment to pull, turn, deseed, and scutch flax was developed to increase agricultural efficiency. In 1953, F.E. Price stated in the foreword of the university bulletin (19) that "Oregon is the only state in the Union that is growing fiber flax and the only state with the people...for flax processing". Flax work in Oregon, however, ended in the 1950s due to introduction of synthetic fibers and loss of government subsidies (17).

The advent of synthetic fibers, such as nylon and polyester for apparel, also caused a decline in the linen industry in Europe (3,12). Before the arrival of synthetics, however, cotton preempted flax as the natural fiber of choice for textiles. Cotton production on plantations in the southern United States effectively overtook the high position of linen and industrial flax fibers, which had existed for millennia. Large scale, economic production of cotton, brought about by the invention of the cotton gin, and its exportation to Europe and the northern United States coincided with the start of the Industrial Revolution (12). Inexpensive cotton, available in large amounts, and improved mechanical processing allowed cotton to quickly and globally overcome flax as the main plant fiber. By and large, flax has been preempted by cotton since this time, with only short perturbations such as blockades during the American Civil War (1861–1865) and disruptions caused by World War II (1939–1945). For example, in World War II

a 100% increase in price and production of Oregon flax straw occurred with the increased fiber demand to supply military and civilian needs (19). These increases, which were tied to the war effort, were short lived and soon the lower production levels returned.

World production of flax fiber decreased from 803,387 metric tons in 1965 to 636,067 metric tons in 2001 (20). Particular countries, however, continue to dominate in producing flax fiber (Table 1) and in hectares cultivated (21; Table 2). Sizeable collections of germplasm for fiber, linseed, and intermediate (ie, both) uses exist in several countries (22). Russia, which maintains a large collection of flax germplasm, and former Soviet Union countries continuously rank high in production and cultivation. Other countries, such as Spain and Great Britain, that had virtually no production for some years produced large amounts of flax in the 1990s; current production in these countries, however, is very low. Government subsidies in Europe since the 1990s have influenced production levels and regions of production, and evolving payment structures continue to affect the flax fiber industry. Production of flax by mainland China, which has varied over the last two decades, has occupied a prominent position in the last several years. France and Belgium lead western Europe, and because of the favorable conditions for retting, flax from these countries along with the Netherlands is historically prized for high quality fiber for textiles (3).

Promotional programs by linen industries in Northern Ireland and western European countries in the 1960s led to a strategic organization to promote linen

	Metric tons			
Country	Avg (1997–2000)	2001		
China	160,000	220,500		
Argentina	1,950	1,900		
Belarus	29,975	31,500		
Belgium-Luxembourg	14,458	17,000		
Chile	2,025	2,200		
Croatia	10	10		
Czech Republic	12,441	15,100		
Egypt	13,575	62,533		
Estonia	54	105		
France	69,750	75,000		
Italy	150	150		
Latvia	1,383	840		
Lithuania	5,575	4,000		
Netherlands	28,344	24,712		
Poland	4,300	5,000		
Romania	900	300		
Russian Federation	33,000	58,000		
Slovakia	2,000	2,000		
Spain	66,511	75,000		
Turkey	13	17		
Ukraine	8,000	12,000		
United Kingdom	26,750	28,000		
World	481,426	636,067		

#### Table 1. Annual Flax Fiber Production<sup>a</sup>

<sup>a</sup> Source—FAO Statistics (20).

Table 2. Annual Cultiva	Hectares cultivated			
Country	Avg (1997–2000)	2001		
Austria	497	130		
Belarus	75,086	$NA^b$		
Belgium	12,110	16,990		
Bulgaria	135	210		
China	$80,809^{c}$	$\mathrm{NA}^b$		
Czech Republic	4,242	7,095		
Denmark	34	19		
Egypt	8,418	$NA^b$		
Estonia	176	27		
Finland	855	405		
France	48,542	67,970		
Germany	664	297		
Latvia	1,920	$NA^b$		
Lithuania	7,680	9,600		
Netherlands	3,710	4,415		
Poland	2,549	4,520		
Portugal	3,101	0		
Russia	107,303	127,361		
Spain	79,044	342		
Śweden	608	32		
Ukraine	26,857	28,280		
United Kingdom	15,120	4,430		

Table 2. Annual Cultivated Areas of Flax for Fiber<sup>a</sup>

<sup>*a*</sup> Source—Euroflax Newsletter (21).

<sup>b</sup> Data not available.

<sup>c</sup> Average of 1996–1999.

(12). Further programs emphasizing other fibers likely facilitated a sociological shift over the last several years to a greater demand for natural fibers in textiles. In the 1980s, the FAO (Food and Agriculture Organization of the United Nations) sponsored workshops on flax, and in 1993 the "FAO Flax Group" became the "European Cooperative Research Network on Flax", with coordination through the Institute of Natural Fibres, Poznan, Poland (23). This program, which was broadened in 1996 to include other bast crops, compiles data on crop production, facilitates interaction of several working groups, and sponsors numerous workshops thus promoting global interests in flax fiber (21).

Efforts to establish a flax fiber industry in the United States have persisted over the years, with several records that document the work (likely many efforts were not recorded). In 1989, Clemson University in South Carolina initiated work on flax fiber, supported mostly by the Ecusta Division of P.H. Glatfelter Co, for specialty paper (24). For several years, field trials were conducted on varieties and agronomic conditions that optimized fiber and seed yields. Despite earlier conclusions (18) that quality flax production was unlikely in the southeastern United States, winter production (October to May) in South Carolina resulted in good fiber yields. Soils in the coastal plains region with higher organic matter tended to be more productive than the more sandy ones. Other work confirmed the positive prospects of winter-grown flax for fiber in this region (24,25). Support by Ecusta for large scale flax production ended in the 1990s, and only experimental work has continued. At this writing, collaborative studies are being carried out between the U.S. Department of Agriculture and Clemson University on winter-grown flax and more efficient processing methods. A commercial venture, Eastern Flax, is attempting to establish a cottonized flax fiber industry in South Carolina.

In 1992, variety trials were initiated at the Connecticut Agricultural Experiment Station, New Haven, Connecticut, in an effort to reintroduce flax fiber in the region (26). Stephens (17,26) published guidelines for production and a series of reports on agronomic data and seed and fiber yields from numerous varieties and from various origins. Lack of a sustainable, commercial industry in the region and retirements of key personnel led to a discontinuation of major efforts. U.S. Flax and Linen operated for a short time in the northeastern part of the United States, but this operation no longer exists. Presently, however, there are renewed plans for commercial operations to grow and process flax fiber in this region.

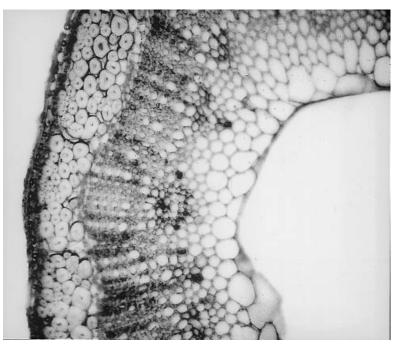
Linseed straw offers a potentially large resource for flax fibers, although the quality would be for lower grade fibers rather than for fine linen textiles. Institutions in Germany, which is a major producer of linseed in Europe (21), have conducted considerable research on flax fibers in composites and other industrial products (27,28). Canada, which is the largest global producer of linseed (21), currently has a strong interest in developing a flax fiber industry. About 15-20% of the straw from the linseed industry currently satisfies the specialty paper (mostly cigarette) industry. The remainder of this straw by-product (>1 million tons from western Canada), which is now burned or chopped to spread on fields, offers an opportunity to provide a value-added product and improve farm economy (29). Biolin Research, Inc. in Saskatchewan, Canada, conducts a modest research effort on fiber yield from varieties and diverse environments (30, Ulrich, personal communication). Canada also maintains a large collection of flax germplasm on both fiber and seed varieties and carries out research to describe plant diversity (31,32). Durafibre Inc., a part of Cargill Ltd, for several years supplied various grades of flax fiber derived from linseed straw, but this operation in western Canada has ceased production. North Dakota in the United States grows linseed and continues to increase in the area cultivated, which in 2002 was  $\sim$ 316,000 hectares and  $\sim$ 95% of the U.S. crop (J.F. Carter, personal communication). While mostly emphasizing linseed, researchers in North Dakota have shown greater interest recently in flax fiber (7). Other than for specialty paper and other low value uses, there are currently no major commercial industries based on linseed straw fibers in North America.

Linen is  $\sim 2-3\%$  of the fiber used in the textile industry (3,33). Despite the reduction in production and usage from previous times, linen imparts characteristics of comfort, drape, and a distinctive appearance that have maintained a share of the market, particularly the luxury market for textiles (12). Blending with cotton and other fibers offers a potential to use nontraditional linen fibers for value-added properties in textiles. For example, adding blended weft yarns made of flax–cotton fibers to cotton warp yarns resulted in fabrics with improved air permeability and wicking rates for moisture; improvement in each property occurred when flax levels in the weft blend were further increased from 25 to 75\%.

The use of flax fibers in other industrial applications such as composites and nonwoven materials, however, may provide the greatest potential for expanded use of flax fibers in the future (3,28,34–38). In particular, composites using flax fibers for the automotive industry are gaining widespread interest due to improved structural properties, processing benefits, and design flexibility and ease (39). Over 136 million kilograms of flax and other natural fibers were reportedly used in automobiles in 1998, mostly in nonvisible areas for reinforcement or sound dampening (40). Composites of flax-sisal-polypropylene for car interiors in Germany has increased over three-fold from 1996 to 1999 (28). Daimler-Chrysler in Germany reported that since 1992 tests have been conducted using flax and natural fibers with plastic for exterior car parts (41). Governmentmandated environmental guidelines for biodegradable products bode well for flax and other natural fibers that can be mixed with polypropylene or other resins for composites. Flax fiber provides a low cost alternative for glass in reinforced composites. The replacement of glass fibers with flax for this application is a considerable challenge but with important advantages. For example, use of flax in automobile parts results in weight reduction, improved sound absorbancy, deep draw potential, and better impact shatter characteristics. Compared to glass, flax fibers are lower in cost, lower in density, biodegradable, and similar in elongation at break; tensile strength is lower for flax (42). When density and cost are considered, flax fibers become more competitive with glass for strength (37). Woven flax fibers as insets with resins particularly provide good strength and rigidity in composites. Substantial savings in energy costs are possible with natural fiber mats, which reportedly require  $\sim 80\%$  less energy than those made with glass (41). Consistency in supply and in fiber characteristics must be addressed when flax fiber is sought for large-scale industrial usage. The degree of processing for fiber cleanliness will depend upon the end product desired, but at least for some products the levels of cleanliness and processing costs are considerably less than for linen fabrics. Treatment of flax to improve surface bonding or to control moisture absorption, such as through acetylation or other methods to alter surface properties, is an area of current research in flax composites (37, 43, 44).

# 3. Structure and Chemistry of Flax

**3.1. Anatomy.** Bast fiber plants, which in addition to flax include other industrially important crops such as hemp, kenaf, and ramie, have been studied over a long period of time. The anatomical structure is well described in numerous publications and reviews (3,33,45,46). Bast fibers are produced in the outer regions of the stem between the outermost cuticle–epidermis layer and the innermost, woody tissues. The structure of the stem is important in retting, which is the process of separating fiber and nonfiber fractions and described in detail later, and for the quality of the fibers derived for industrial applications. Tissues in the stem cross section (Fig. 1) are identified as follows: outermost cuticle layer covering the epidermis, thin-walled parenchyma cells inside the epidermis and woody core cells. The fibers exist in bundles of ultimate (ie, individual) fibers in a ring encircling the core tissues. About 20–50 bundles form in cross-sections of flax stems, with 10–40 spindle-shaped ultimate fibers of 2–3 cm long and 15–



**Fig. 1.** Light microscopy of cross section of flax stem showing the organization of tissues: epidermis with protective cuticle to the outside; parenchyma underneath the epidermis and between fiber bundles; ultimate fibers in bundles; cambium, and woody core cells.

20 µm in diameter per bundle (3,14). Fibers vary in length with position on the stem. Oval-shaped bundles indicate high quality fiber, while irregularly shaped bundles indicate poor quality (14). A polygonal cross-sectional shape (3–7 sides) and thick cell walls provide better quality fibers. Separated fibers and fiber bundles appear stiff and brittle in longitudinal views under the microscope (Fig. 2). Nodes, recently termed "fibernodes" (47), are dislocations that appear as horizontal bands in the fibers and bundles and are easily recognized (48-50). These nodes, whose origin is not fully understood, are regions where moisture, dyes, and enzymes can penetrate and influence fiber properties. They also represent weak points in the fibers and result in a blunt, distinct appearance at fiber fractures after breaking tests (Fig. 3). The occurrence of kink bands, which appear similar to nodes, arises from processing methods and has been implicated in failures of compression tests (51). A thin cambium layer separates fibers and core tissues. These core tissues are comprised of lignified woody cells, which constitute the "shive" fraction produced during fiber cleaning. Flax variety, climate, and production practices influence the stem and fiber anatomies.

**3.2.** Chemistry. The stem cuticle of flax contains waxes, cutin, and aromatics (45,52,53). This structure serves as a barrier to protect plants from invading organisms and water loss (54). The cuticle closely covers the epidermis, and this relationship constitutes a rigid and formidable structure that influences the ease of retting. The cuticularized epidermis in flax must be breached for microbes and enzymes to reach the inner tissues and loosen fibers from nonfiber tissues.



**Fig. 2.** Scanning electron microscopy of ultimate fibers in flax showing a stiff nature and "disruptions" that have been termed nodes or fibernodes. These disruptions in the fiber allow access to the fiber for moisture, dyes, and enzymes and represent mechanically weak regions.

During retting, microorganisms enter the stems through cracks and disruptions in the cuticle, partially degrade tissues, and thereby separate the cuticle/epidermal barrier from the fibers. Incomplete degradation, ie, poor retting, leaves this protective barrier and fibers still attached and contributes to reduced fiber and yarn quality. Amounts of cuticular fragments are inversely related to quality of



**Fig. 3.** Fibers and fiber bundles in flax that have been broken by Stelometer showing the distinct, blunt appearance at the break points arising from nodes.

Treatment	Uronic acids	Noncellulosic carbohydrates	Glucose	Total phenolics
unretted fiber	0.21	104.2	434.0	7.2
dew-retted fiber	0.08	94.1	649.5	Trace
enzyme-retted (1% Flaxzyme)	0.09	99.8	623.5	$\mathrm{NP}^b$

Table 3. Chemical Composition of Commercially Grown (Holland) Ariane Fiber Flax  $mg/g^{\alpha}$ 

<sup>a</sup> Adapted from Akin and co-workers (45,56).

<sup>b</sup> Not present = NP.

yarn and fiber (52). The cuticle is particularly problematic in retting mature or seed flax stems (55).

Thin-walled parenchyma cells, which occur between the epidermis and fibers and between the fiber bundles, are rich in pectin and other matrix polysaccharides. The cambium, a specialized tissue for secondary cell growth, exists between fibers and woody cells and is also rich in pectin. The separation of fibers from the woody core at the cambium can easily occur, especially when stems have been stored in dry climates for an extended time.

Flax fibers are primarily comprised of cellulose, but pectins, hemicellulose, and phenolic compounds also are present (45,56, Table 3). Compared with cotton fibers, which typically contain  $\sim$ 95% cellulose (57), flax has a lower percentage of cellulose and more pectin and hemicellulose (49). For example, in retted "Ariane" flax glucose was the predominant sugar (650 mg/g dry wt.) followed by mannose (39.2 mg/g) and galactose (35.0 mg/g); rhamnose, xylose, arabinose, and uronic acids were also present (45). The increase in mannose and galactose along with glucose after retting suggests an intimate involvement of noncellulosic sugars in the secondary fiber walls of flax. Further, hemicellulosic constituents such as galacto-gluco-mannans and xylose are often reported as substantial components in flax fibers (58-60). The presence of these noncellulosic carbohydrates is thought to impart distinguishing characteristics, such as high moisture regain, to flax. Further complicating the structure of flax fiber is the association of proteins and proteoglycans with secondary walls, which possibly provides a structural dimension to flax fibers (61). Cellulose in flax shows a "notable region of order" by X-ray diffractometry, with a higher crystallinity index than other natural fibers (62). In contrast to cotton, flax fibers stained with Oil Red, which indicates the presence of wax (63, Akin, unpublished data) gave no positive reaction, indicating little or no waxes present. Analytical studies of fibers manually separated and cleaned of all other tissues confirmed the presence of only low levels of waxes, cutins, and sterols, with amounts of  ${\sim}0.2\%$  of fiber dry weight and 1/20th or less of levels in cuticularized epidermis (53). Therefore, while flax fiber is considered primarily a cellulosic fiber, its characteristics differ from cotton and many other natural fibers and allow application in a range of industries.

Woody core tissues are the most highly lignified cells in flax stems based on wet chemical, histochemical and spectroscopic comparisons with fiber and other tissues in the stem (45). Lignin imparts rigidity and strength to plants tissues generally. Electron microscopy showed thick core cell walls with distinctive secondary, primary, and middle lamella layers. Guaiacyl and syringyl lignins both occurred in core cells, with the guaiacyl type more prevalent in cultivars examined. These lignified core cell walls were little affected by the microorganisms during dew-retting, indicating the recalcitrance of this tissue to fungal attack.

Pectin, which is strategically located in plants, is particularly important to maintain the structure of flax stems; its degradation is of fundamental importance for retting and, therefore, the quality of flax fibers (14). Parenchyma, cambium, and the middle lamella binding fibers in the bundles are rich in this component. Pectin is a heteropolysaccharide consisting mainly of 1,4-linked α-D-galacturonic acid, with various degrees of methylesterification at the carboxyl position and with various attached side chains (64). Reports indicate in some cases that pectin in primary walls generally may have a high proportion of oligosaccharide chains on the backbone and longer chains than the pectin in the middle lamellae (64). A rhamno-galacturonan structure of type I pectin, which is a prominent form in plants, likely forms the backbone of the high molecular weight polysaccharides in flax fiber as shown by nuclear magnetic resonance (nmr) (65). Regarding pectin and retting of flax, Meijer and co-workers (66) indicated that pectin degradation was faster in flax harvested during flowering compared with mature flax stems. They also found that a residual pectin level of 7-10 g/kg remained after retting, suggesting the presence of a more resistant pectin. Morvan and co-workers (67) reported that methoxylated pectins were synthesized during the elongation stage of flax growth; amounts of both the highly and less methoxylated pectins remained the same during maturation, thus preventing a determination of the type of pectin preferentially synthesized. Nonmethoxylated carboxyl groups on galacturonic acid are often cross-linked by  $Ca^{2+}$  or other cations that form stable bridges across pectin molecules (64). Microscopic evaluation of specific tissues in flax hypocotyls indicated that acidic polygalacturonans and calcium were more prominent in the epidermal than cortical cells (68,69). Results from these microscopic studies are substantiated by inductive coupling plasma (ICP) emission spectrometry showing in one example a 5.6-fold higher amount of calcium in cuticularized epidermis compared with fiber cells (Table 4). The enzyme endopolygalacturonase, which degrades pectin and disorganizes flax hypocotyls, was reportedly inhibited by steric hindrance through calcium linkages in pectin (69,70). Mapping with mid-infrared (ir) microspectroscopy of different varieties of mature flax fiber confirmed that pectin types

	Tria	1
$Sample^{b}$	1	2
whole bast epidermis/cuticle fibers	$2,219 \\ 11,244 \\ 2,463$	$3,234 \\ 12,750 \\ 1,890$

Table 4. Calcium Levels (ppm) in Flax Tissues<sup>a</sup>

<sup>a</sup> Calcium determined by ICP.

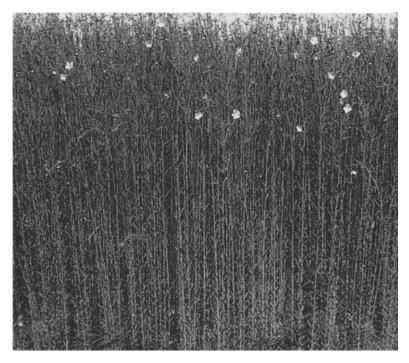
 $^{b}$  Tissues of Ariane fiber flax were manually separated into fractions.

could vary among samples and regions (71), thereby influencing pectinase activity and retting efficiency. Immunocytochemical staining methods, using goldlabeled antibodies against specific pectin structures, have further indicated sites of specific pectin types within areas and layers of flax fibers (72,73). These results support the hypothesis that different pectins in regions of the bast, and their susceptibility to different pectinases, eg, pectate or pectin lyases, polygalacturonases (64), could significantly influence the efficiency of retting by enzymes. The presence of calcium-stabilized pectin molecules in high amounts in the cuticularized epidermis further shows the formidable barrier that must be overcome for effective retting by microorganisms. These data suggest potential strategies, which are discussed later, that could be employed to improve fiber extraction in flax.

Lignin, consisting of recalcitrant compounds with a complex polyphenylpropanoid structure, is a major limitation generally to microbial degradation of plant carbohydrates (74,75). Often lignin is mentioned as a detriment to the quality of flax fiber (49). Studies to localize sites of lignin and aromatic compounds within cells, using histochemical stains (45,76) and ultraviolet (uv) absorption microspectrophotometry (45), showed that these compounds occurred nonuniformly in middle lamellae between fibers, with the greatest levels in cell corners. Lignin, however, does not appear to impede fiber separation from the core cells (55), particularly with subsequent processing to clean fiber. Heavily localized areas of aromatics that remain on retted fiber, however, could influence properties (76) or reduce processing efficiency. Chemically extracted aromatics from bast tissues of flax inhibited the activities of pectinase and other enzymes (77), suggesting a possible role for such compounds in limiting retting. Use of a water rinse to remove contaminants, including colored materials, was a widespread practice before water-retting flax bundles (78). Using this practice as a basis for removing potential inhibitors to retting, laboratory tests were conducted using a water presoak before retting with cell free-enzymes; only moderate and inconsistent effects, however, occurred in these studies (79). Most of the lignin occurs in the woody core cells (see section above on Chemistry) and is removed as this shive fraction is separated from the fiber during cleaning. Rapid analysis of lignin and aromatics could provide a useful method for assessing shive content as a contaminant of fiber for grading fiber quality.

# 4. Production

Flax can be grown for fiber or linseed. For fiber, seeds from high fiber varieties are densely sown to give a final plant density of ~2000 plants/m<sup>2</sup> (80). Planting in this way gives thin-stemmed, straight, and tall plants for high fiber yield and quality (Fig. 4). For optimal quality of fiber, flax plants are harvested before full seed maturity. Fiber yields as well as quality vary due to variety, environment, and agronomic practices, but total fiber yields of 25–30% of straw dry weight are possible (26). Linseed varieties, in contrast, are sown in low densities (~750 plants/m<sup>2</sup>) to maximize branching for greater seed production. Plants grown in this manner to full seed maturity have thick stems and produce fiber of low quality.



**Fig. 4.** Flax plants growing in the field for fiber and near ready for harvest. Thin, tall stems are produced from a densely packed (suggested at 2000 plants /  $m^2$ ) cultivation.

Flax is a temperate weather crop, generally cultivated in areas where the daily temperature remains  $<30^{\circ}$ C (80). Production of flax is environmentally friendly in that few chemicals are required for crop production. Herbicides and pesticides are not required or only in small amounts. Often, herbicides are used only initially to establish plants. Control of pathogens is by crop rotation and use of disease-resistant varieties. Nitrogen requirements are low at  $\sim$ 70–80 kg/ hectare (80) and should be kept low because high levels tend to induce lodging of plants. Typically, in the better regions for flax production in western Europe, fiber flax is planted in March–April for harvest in mid-July–August (3). In Egypt, flax is sown in November for a May harvest (13). Similarly, winter flax cultivation (October–April/May) in the southeastern United States produced high yields of fiber and seed (24,25).

In traditional production of linen such as that practiced in Europe, flax plants are pulled from the soil, manually in early times and now with specialized equipment (Fig. 5). Plants can be harvested by mowing (Fig. 6) when short flax fiber, rather than long line for linen, is the objective. While costs are likely to be less with mowing, fiber remaining in the stubble reduces yield. With linseed, the fiber for paper or low-value composites may be in tangled straw that results from the combine used to harvest seeds. Other methods of harvest, eg, a stripper header to collect seeds (Fig. 7), may be used that leave the linseed straw residue in a more suitable state for collection and processing of fiber. The end product sought and economics determine the best method for harvest.



**Fig. 5.** Pulling of flax using specialized equipment produced in Europe. Stems with roots attached are pulled to maintain a high yield of long, strong fiber bundles for traditional linen production.



**Fig. 6.** Drum mower successfully used to harvest flax fibers for nontraditional applications. With mowing, fiber alignment is not maintained and fiber yield is less due to the remaining stubble. Costs may be less due to use of general-purpose farm equipment.



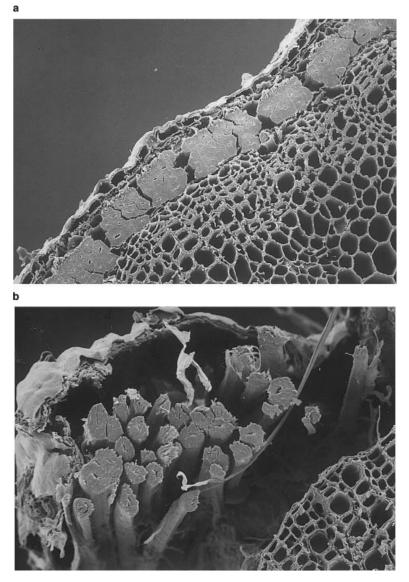
**Fig. 7.** A stripper-header used to harvest linseed, leaving the stems upright for subsequent harvesting for fiber.

Research has been carried out to optimize flax harvesting using typical farm equipment rather than specialized equipment, ie, pullers and turners, used for traditional linen. In South Carolina, drum mowers, rakes, and round balers have been used to collect and store flax. In some trials, seeds were collected by a stripper-header leaving stems for subsequent mowing, baling, and storage for fiber (24,35).

### 5. Processing

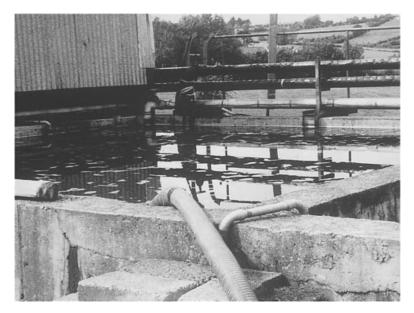
**5.1. Retting.** Retting, which is the separation or loosening of fiber bundles from nonfibrous tissues, is a major problem in processing flax. In retting, fiber bundles are separated from the cuticularized epidermis and the woody core cells and subdivided to smaller bundles and ultimate fibers (Fig. 8). The quality of retting determines both yield and quality of the fiber, and plant development and seasonal variation in turn influence retting (78). Under-retted flax results in coarser fibers heavily contaminated with shive and cuticular fragments, while overretting can reduce fiber strength due to excessive thinning of bundles or microbial attack on fiber cellulose. Two primary methods for retting, namely water- and dew-retting, have been used traditionally over millennia to separate fibers for textile and other commercial applications.

Water-retting depends on fermentation by anaerobic bacteria, such as *Clostridium felsinium*, to degrade pectins and other matrix substances (14). In early times, bundled flax stalks were submerged in natural bodies of running



**Fig. 8.** Scanning electron microscopy of unretted and retted flax stems. (a) Unretted stem showing the cuticle on the epidermis, fibers, and woody core cells. (b) Similar but dew-retted stem showing the separation of fibers from the cuticularized epidermis and woody core. Fiber bundles have also been subdivided into smaller bundles and ultimate fibers.

or still water (eg, lakes, rivers, dams) for 5-7 days and then dried in the field for 1-2 weeks. Particular reference is often made to the river Lys (for Courtrai flax) in Belgium, where the suitability for cold water-retting and excellent quality fiber led to an active linen industry (14,16). Retting pits or tanks were constructed (Fig. 9) where temperature and other conditions could be controlled.



**Fig. 9.** Pits, or tanks, for water-retting of flax. Flax stems are bundled and submerged. Conditions can be altered in the pits, such as using warm water for more uniform retting, aeration to change the microbial metabolism, inoculation of specific retting microorganisms, and addition of supplements to promote retting.

Van Sumere (14) reported that such pits have been used since the 1920s for water-retting. Similarly, retting pits were also constructed in Oregon early in the 1900s (16). These pits could be flushed with an initial rinse water to remove contaminants, heated to controlled temperatures, and even inoculated with specific microorganisms. Aeration of the tanks has been attempted to modify the microbial types and subsequently the anaerobic metabolism (ie, reduce acidity and toxins to retting microorganisms). Different microbial consortia and more complete oxidation of organic materials result from aerated conditions. Water-retted stems were then sun-bleached and dried naturally (Fig. 10), reportedly giving the finest and highest quality fibers (78). Van Sumere (14) has given a historical perspective of retting, and Sharma and co-workers (81) reviewed details of the microbiology in retting.

Despite the fact that the highest flax fiber quality is produced by waterretting, this practice has been largely discontinued in western Europe due to the high costs and the stench and pollution arising from fermentation of the plant material (78). Fermentation products absorbed by the fibers during water-retting impose an unpleasant odor (14). Dew-retting is now the most common practice for separating flax fibers, even though some water-retted fiber is still marketed (82).

Dew-retting is reportedly the oldest method of retting, being used by Egyptians for millenia (14). Even though the flax fiber is of lower quality than that from water-retting, lower labor costs and higher fiber yields make dew-retting attractive. Stalks are pulled (Fig. 5) or mowed, spread in uniform and



Fig. 10. Drying of retted flax stems in "wigwams".

thin, nonoverlapping swaths (Fig. 11), and left in the field where the moisture and temperature encourage colonization and partial degradation of flax stems by consortia of fungi, yeasts, and aerobic bacteria. Flax plants are turned over on a regular basis to produce more uniform retting. Primarily, indigenous fungi effect dew-retting, and successions of various species and groups occur during the process (78). Typical saprophytic, soil fungi are the major components of these consortia, including species of Aspergillus, Cladosporium, Fusarium, *Rhizopus*, and *Trichoderma* (14,83,84). Various structures in flax stems that are undergoing attack by dew-retting fungi are shown in Figure 12. Sharma and Van Sumere (78) noted that secondary colonists produce the most cellulase, which can weaken the fiber and reduce quality. For example, *Epicoccus nigrum*, which is often isolated in the consortia (81,83-85), degrades fiber cellulose, resulting in loss of fiber strength and poor quality. Van Sumere (14) noted particular fungi with retting periods, eg, Cladosporium herbarum for summer retting, Mucor stolonifer for autumn retting, and M. hiemalis for snow retting. Reports of the mycological consortia are often from the United Kingdom and western Europe, where flax has been grown over long periods of time. Possibly, other microorganisms dominate in different regions and affect various fiber parameters. Flax bales from different regions vary in color, suggesting among other factors the possible variation in dominant retting microorganisms. To this point, Henriksson and co-workers (84) isolated fungi from winter-grown flax that was dew-retted in South Carolina. The most prevalent species was *Rhizopus oryzae*,

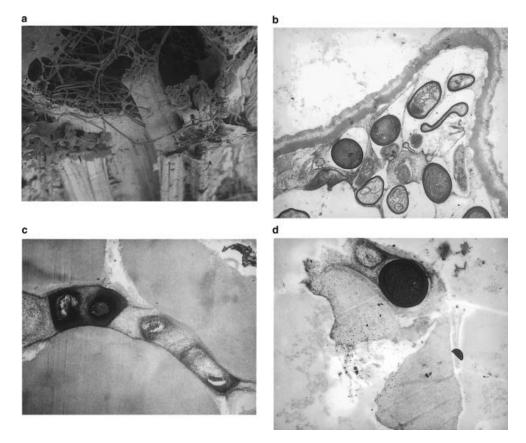


**Fig. 11.** Swaths of flax pulled and laid in uniform rows by specialized pulling equipment for dew-retting. With proper moisture and temperature, indigenous microorganisms from the soil and plant colonize and partially degrade the flax stems. When fibers have been loosened from the nonfiber cells and before fibers begin to be degraded, flax is harvested in round or square bales for further processing.

originally identified as *Rhizomucor pusillus*, that in laboratory studies effectively retted flax without loss in fiber strength noted for some other fungi (86,87).

Stand-retting, which is another method of retting in the field with indigenous fungi for the most part, was attempted in the 1960s-1970s to overcome limitations for dew-retting in northern Ireland (85). In these trials, glyphosate (*N*-phosphonomethyl glycine) was used successfully as a preharvest desiccant to facilitate retting. Stand-retted fiber pretreated with glyphosate retained more strength than dew-retted flax, although fungal colonization and retting were slower (88). Dry weather during production and harvest, however, proved problematic for use of glyphosate as an aid to retting (3,88). Recent research reports indicate a continuing interest, however, in glyphosate treatment and stand-retting (89,90).

Dew-retting suffers from several disadvantages. Sharma and Faughey (91) reported that the quality of flax fiber has declined in the years since dewretting replaced water-retting. In addition to poor and inconsistent fiber quality, dew-retting requires occupation of agricultural fields for several weeks and restriction to geographical regions that have the appropriate moisture and temperature for effective microbial growth (14,85). Unsuitable weather for dewretting in particular regions, such as the United Kingdom, that previously were large fiber producers now prevents long fiber production for linen. In western



**Fig. 12.** Electron microscopy of flax stems colonized and partially degraded by fungi as occurs in dew-retting. (**a**) Scanning electron microscopy of fungal attack on cut stems from laboratory studies showing growth of the thread-like fungal mycelium over the plant cells and separation of fibers from nonfiber cells. (**b**). Transmission electron microscopy of fungal colonization showing attack and degradation of plant cells underneath the recalcitrant cuticle. Fungal hyphae are prevalent in regions now devoid of plant cell walls. (**c**) Transmission electron microscopy of fungal degradation of middle lamella between fiber cells showing a hypha completely filling the region between fibers. Selective degradation of middle lamellar material, comprised of pectin in part, results in finer fibers. (**d**) Transmission electron microscopy showing progressive fungal growth and attack on the fiber secondary wall, resulting in destruction and weakening of fiber. In part from Akin and co-workers (86).

Europe, which reportedly produces the highest quality linen due to a favorable climate for dew-retting (1,3), crop losses of  $\sim 33\%$  often occur. The prolific fungal colonization and contact with the soil during dew-retting result in a heavily contaminated product, which creates another problem for United States textile mills that import flax fiber for blending with cotton and spinning on high efficiency, short staple systems. Because of these considerable problems, numerous studies have been proposed or carried out to improve dew-retting, including inoculating with favorable microorganisms or chemical additives such as urea, sucrose, and ethylenediaminetetraacetic acid (EDTA) (81,83,92,93). Experiments have been conducted with enzymes to improve or tailor properties of dew-retted fibers or

flax roving from dew-retted fibers (94,95). From considerable experience, Sharma and co-workers (81) stated that the weather dictates success in dew-retting, although improvements through chemical treatments could occur but with additional costs. Despite these problems, dew-retting still remains the method of choice for extracting flax and linen fibers for most of the world.

Considerable research has been undertaken to find a replacement for dewretting. Chemical-retting has been evaluated using a variety of methods, including EDTA or other chemical chelators at high pH, detergents, strong alkali, and steam explosion (96–99). Sharma (98) patented a chemical-retting method using chelating agents. The Reutlingen Steam Explosion Treatment (27) uses impregnation and steam to remove pectins and hemicelluloses from decorticated flax; a fast decompression separates bundles to smaller bundles and ultimate fibers. Steam explosion provides fiber of a constant quality that can be designed for different applications. Successful laboratory results have been reported with chemical-retting methods, but at times fiber properties are less satisfactory than those from other methods. Chemical treatment increases cost, but cost efficiencies are usually not reported. To date, no chemical-retting methods are used commercially.

Enzymes have been considered for some time as a potential replacement for dew-retting flax. Early work with water- and dew-retting microorganisms showed conclusively that pectinases were required for effective retting (14). Other matrix-degrading enzymes, eg, various hemicellulases, reportedly contribute to effective retting. Recent work indicated that endopolygalacturonase, a hydrolytic pectinase that attacks nonmethoxylated pectin, alone could effectively separate fibers from nonfiber cells (100–102). Successful enzyme-retting could provide considerable advantages including: high and consistent quality flax fiber, tailored properties for specific applications, and broadened geographic regions for production of flax and linen. Such potential for enzyme-retting has prompted in depth research projects to develop effective processes.

A major research effort took place in Europe in the 1980s to develop enzyme-retting as a replacement for dew-retting (14). The strategy was to submerge flax in an enzyme solution, thereby simulating water-retting by replacing bacteria with enzymes. Flazzyme, a commercial enzyme mixture from Aspergillus species (Novo Nordisk, Denmark) was evaluated at the State University of Ghent in Belgium, at the Lamberg Industrial Research Association in Northern Ireland, and in France (78). Several patents on the use of enzymes for flax retting came from this work (14,103,104). Flaxzyme, which is a mixture of cell-wall degrading enzymes including pectinase, hemicellulase, and cellulase, produced fiber with good properties, having yield, strength, and fineness equal to waterretted fiber (105). A pilot plant scale study (106) using 80 kg of flax stems submerged in SP 249 (Novo Industries) at 0.3% v/v (11:1 liquid/fiber ratio, 45°C, 24 h) carried out in Europe in the 1980s produced fibers of equal yield and quality to that from water- and chemical-retting in the same tests. The little known company Lyven (Caen, France) markets Lyvelin, which is reportedly a pectinase from Aspergillus niger for flax retting. Despite apparent success in the use of enzymes, a commercial process for enzyme-retting was not established.

Research was initiated on enzyme-retting in the 1990s by the Agricultural Research Service, U.S. Department of Agriculture. The goal of this work was to

develop a method for consistent quality cottonized flax fiber, rather than traditional linen, from the entire stem and from diverse sources of flax. Viscozyme L (Novozymes North America, Inc.), which proved to be an effective, commercially available enzyme mixture, was used in several studies. Flaxzyme, as least by that name, is no longer available according to information provided by the company. SP 249 (Novozymes) is equally effective and likely is similar to Novozym 249 used by Sharma (98). Other pectinase mixtures also were effective for retting in laboratory studies. Chelators, which had been previously shown to ret flax, were included with enzymes to remove Ca<sup>2+</sup> from pectin cross links and destabilize cell wall structures. The inclusion of chelators significantly reduced enzyme levels required for effective retting as shown by the in vitro Fried Test (107). This test, as described in Van Sumere (14), is a relatively simple and quick method to evaluate flax stems for the ability of enzymes to separate fibers from core. The Fried Test has been used extensively with small batches of stems to determine the most effective levels of enzymes and chelators. Recent work (96,108), in which chelators representing diverse chemical types (eg, polyphosphates, phosphonic acids, and aminopolycarboxylic acids) and costs were included with enzymes, showed that EDTA was most effective at binding Ca<sup>2+</sup>, particularly at pHs (eg, 5–6) required for enzyme activity.

Through laboratory and small pilot scale (10 kg) tests, an enzyme-retting method was developed using Viscozyme L and EDTA (109,110). After this research was reported, the commercial EDTA product, Mayoquest 200 (Callaway Chemical, Smyrna, Georgia), was used and found to be effective for retting with Viscozmye L. The features of this work that vary from other reports are physical crimping of flax stems to disrupt the cuticle barrier, inclusion of chelators with enzymes at pH 5 or 6 (depending on enzyme employed) in specific formulations from 0.05 to 0.3% of product as supplied, and spraying of formulations rather than immersing to reduce the liquid/fiber ratio from 11:1 to  $\sim$ 2:1. Tests of fibers produced in pilot plant evaluations (Table 5) and laboratory yarn blends made with flax and cotton fibers (Table 6) indicated that fibers from both fiber and

Sample	Retting treatment	Fineness (air flow)	Strength (g/tex)	Elongation (%)	Length (UQL)	Fine fiber yield (%)
seed flax	enzyme (0.05)-EDTA	6.0	19.6	1.7	1.2	23.6
fiber flax	enzyme (0.05)-EDTA	5.7	20.9	2.0	1.4	37.9
fiber flax	enzyme (0.3)-EDTA	4.6	15.8	1.8	1.2	58.7
fiber flax	dew-retted	5.3	36.2	2.3	1.3	43.0

Table 5. Effect of Source and Retting Treatment on Fiber Flax Properties<sup>a</sup>

<sup>a</sup> Seed flax and Ariane fiber flax experimentally spray-enzyme-retted (Akin and co-workers, 2000) with Viscozyme L (Novozymes North America, Inc., Franklinton, North Carolina) and EDTA. Retted fiber cleaned through commercial equipment including the Unified Line and La Roche cottonizing system at Ceskomoravsky len, Czech Republic. Properties analyzed at the Cotton Quality Research Station, ARS-USDA, Clemson, South Carolina, using standard or modified cotton methods as follows: fineness by modified airflow method, strength and elongation by Stelometer, length by array method. Fine fiber yield is the amount of fiber obtained by passing cleaned, cottonized fiber through the Shirley Analyzer (SDL America, Inc., Charlotte, North Carolina). Adapted from Akin and co-workers (110).

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		Properties			
Sample	Retting treatment	Single end strength (g/tex)	Mass evenness (cv)	Nep imperfections	
seed flax	enzyme (0.05)-EDTA	13.0	27.1	647	
fiber flax	enzyme (0.05)-EDTA	13.9	25.2	597	
fiber flax	enzyme (0.3)-EDTA	13.9	26.1	571	
fiber flax	dew-retted	13.7	24.6	555	
100% upland	cotton	17.4	19.4	461	

Table 6. Effect of Source and Retting Treatment on Properties of Flax–Cotton Blended Yarn<sup>*a*, *b*</sup>

 $^{a}$  Adapted from Akin and co-workers (110).

<sup>b</sup> Seed flax and Ariane fiber flax experimentally spray-enzyme-retted (Akin and co-workers, 2000) with Viscozyme L (Novozymes North America, Inc., Franklinton, North Carolina) and EDTA. Retted fiber cleaned through commercial equipment including the Unified Line and La Roche cottonizing system at Ceskomoravsky len, Czech Republic. Fine fiber obtained through the Shirley Analyzer (SDL America, Inc., Charlotte, North Carolina, USA) was used to make and test flax:cotton (1:9) blended yarn at the Cotton Quality Research Station, ARS-USDA, Clemson, South Carolina).

seed flax could be successfully retted and processed by this method (110). Laboratory tests indicated that high enzyme levels or longer incubation times could reduce fiber strength, while higher chelator levels tended to increase fiber fineness (111). Retting with polygalacturonase without cellulases, compared with pectinase in mixtures with cellulase, produced fibers with significantly greater strength (102). Microscopy showed that cellulases preferentially attacked nodes, thereby weakening fibers. Before a commercial method can be developed, additional research is required to optimize enzyme-chelator levels in formulations, based on fiber properties and cost, and to integrate retting and cleaning processes.

**5.2.** Mechanical Cleaning. In traditional linen production, mechanical cleaning follows retting to remove shive and cuticularized epidermis from the fiber. Figure 13 shows a large round bale of flax entering a bale opener and beginning the cleaning process. The first phase of cleaning breaks the stems by passage through fluted rollers and then the scutching process beats and strokes the fiber to remove shive (112). The quality of retting determines the quantity and quality of the fiber remaining after scutching. From the primitive manual tools such as hammers and boards used to scutch flax, modern equipment, although automated, scutches flax more or less by the same methods. Scutching mills clean long fiber by gripping the broken stems and beating first the top portion and then the lower portion with paddles or blades. As the long line flax is beaten, a short fiber fraction, called tow, is removed along with contaminants and cleaned separately. Prior to breaking the stems, modern mills may align and carry out other processes to improve the efficiency of scutching. Maintenance of the integrity of the long fibers, which are to be spun into linen yarn, is maintained during the mechanical cleaning processes (Fig. 14).

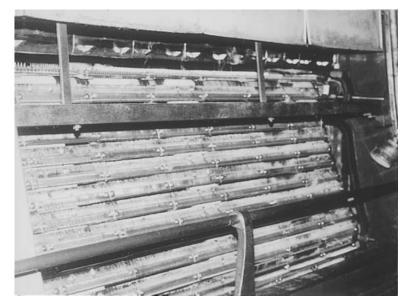
Scutched flax is then cleaned using a combing action called hackling, which removes smaller contaminants, disentangles and aligns the long fibers, and separates the bundles without destroying length (113). A short fiber fraction,



**Fig. 13.** Round bales of dew-retted flax entering a bale opener, which is the beginning of mechanical processing where stems will be broken and fibers cleaned of contaminants.



**Fig. 14.** Flax stems that have been retted and scutched for traditional linen production are long, aligned, and clean fiber bundles. These fibers, which will be further cleaned by hackling, are eventually wet spun for high value, 100% linen materials.



**Fig. 15.** Hackling machines comb through scutched flax, thereby aligning and subdividing fiber bundles. This machine has a series of pins (upper left) that are progressively closer and closer together to clean long fiber.

called hackling tow, is produced as a by-product of the long-line fiber. Automated hackling systems with progressively finer and finer pinned rollers (Fig. 15) comb through scutched, long-line fibers to produce the long hackled fibers for traditional linen textiles. As in the scutching process, the integrity of the long line fibers is maintained. Fibers are then processed into sliver (a continuous strand of loosely assembled fibers) and then roving (sliver with reduced diameter and a slight twist to hold fibers together). From this material, yarns are made using a wet, ring spinning system that is relatively slow and expensive in comparison to cotton spinning. The tow fibers are cleaned and refined for cottonized flax, blended with cotton or other fibers, and spun on efficient dry ring or rotor spinning systems. Tow also is used in various nontextile industrial applications, such as composites and nonwoven materials.

"Total fiber" scutching can be carried out to process only one type of fiber from the flax stems rather than long-line and tow for traditional linen. This process is simpler than that for traditional linen in that alignment of stems is not as critical for processing, and nontraditional sources of fiber (eg, linseed straw) may be used. Equipment is often quite expensive for refining and shortening clean fiber for blending with cotton and processing on short staple equipment. Generally, for total fiber the retted stems are broken and then cleaned of shive and contaminants through a beating or carding action, where fibers of nonuniform length result (39). Fibers are chopped to uniform length or reduced in size in some way and further refined and cleaned for cottonized flax (Fig. 16). These fibers, now similar in length to cotton, can be blended with cotton or other staple length fibers and spun on high- efficiency, short staple spinning systems as indicated earlier. Short flax fibers, such as tow, originating from cleaned long-line or



**Fig. 16.** Bales of cottonized flax from total fiber processing systems. Flax cleaned in this way can be non-uniform in length or further processed or cut for a more uniform length. Cottonized flax is cleaned and refined for blending with cotton and other fibers for short staple spinning systems.

from total fiber processing do not have the properties or generally bring the high price of long-line fiber used in traditional linen mills (82). Government subsidies (114), environmental concerns (29), and perhaps other situations, however, have at times caused a significant effort to be made to use linseed straw or lower quality stems as a fiber source. Research has been carried out to develop equipment for more efficient decortication (eg, removal of shive from fibers) of linseed straw for fibers of lower technical grade where traditional linen is not the object (114). Depending on the application, total fiber of various levels of cleanliness (ie, amount of shive remaining with fibers) can be produced in these systems for use in geotextiles, composites, and other nontextile uses.

### 6. Flax Fiber Properties and Grading

**6.1. Measurement of Fiber Properties.** While many natural fibers such as cotton and wool exist as individual units or cells, the occurrence of flax as ultimate fibers connected in bundles of various sizes, as well as the inherent chemistry of the fiber, influences properties and methods of analysis. Retting and cleaning processes, such as scutching, hackling, etc, tend to further divide bundles resulting in smaller bundles and even ultimate (ie, individual) fibers. This division of fibers, which can continue to occur during processing or physical disturbance, also affects strength and length of the fibers.

For traditional long-line flax used in textiles, a number of factors are subjectively judged by experienced graders and include weight in hand, strength,

Retting process	L*	a*	b*
dew retted $(N=3)$ water retted $(N=2)$ enzyme retted $(N=6)$	$\begin{array}{c} 59.4 \pm 1.4 a \\ 67.5 \pm 1.1 b \\ 72.0 \pm 3.3 b \end{array}$	$\begin{array}{c} 2.87 \pm 0.85 a \\ 2.60 \pm 0.10 a \\ 3.45 \pm 0.75 a \end{array}$	$\begin{array}{c} 11.08 \pm 1.66a \\ 14.54 \pm 0.49b \\ 16.30 \pm 1.57b \end{array}$

Table 7. Comparative CIELAB Color Values of Flax Fibers<sup>*a*, *b*</sup>

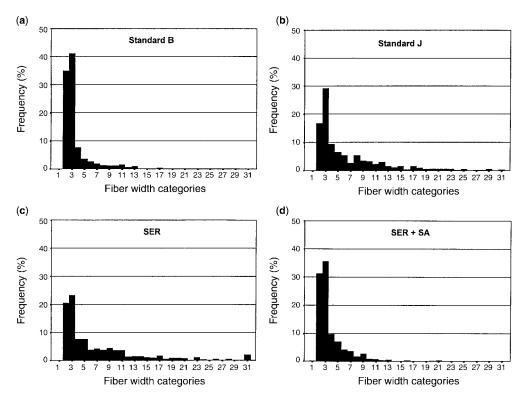
 ${}^{a}L^{*}$  is lightness with a more positive number indicating a lighter sample; a\* is red/green color with a higher number indicating a more red sample; b\* is the yellow/blue color with a higher number indicating a more yellow sample.  ${}^{b}$  Source: Akin and co-workers (116).

fineness, handle (softness, smoothness, pliability), luster, cleanliness, parallelism of fiber bundles, freedom from knots and entanglements, length and shape (115). High fiber strength reduces breaks during spinning, which improves efficiency, and has been considered the "best single measure of yarn quality" (115). Color also is important. With the advent of dew-retting over water-retting, the color of fibers tended to be darker. Weathering and fungal colonization impart various degrees of gray, black, or brown to the fibers. These colors have been used also to indicate the degree of retting within certain constraints. Fiber lots having different colors may be blended to provide a final product with a desired color. With various retting systems, however, the range in fiber colors can be greatly expanded (116,117, Table 7). For blending of tow or total fiber with cotton, fiber length and length distribution affect spinning efficiency and should be uniform in various lots. In the high efficiency cotton spinning systems used for flax-cotton blends, cleanliness is especially important as it influences breaks during spinning and final yarn grades. Textile mills using efficient, short staple systems would be more accepting of flax if properties were more uniform in various lots and the product less contaminated.

As mentioned previously, the nature of fibers and fiber bundles in flax and their propensity to fracture to smaller sizes throughout processing presents difficulties in assessment. Nevertheless, methods to derive objective values for various parameters, such as fiber strength, length, and fineness, are available (115,118) and routinely used by research and industrial organizations for inhouse testing of flax samples. The Stelometer, eg, provides data for fiber strength, with elongation at break measured at the same time (110,118,119). The method is time consuming and requires skill by operators to obtain consistent results for test and reference materials.

Flax fiber fineness can be calculated gravimetrically as the weight per unit length, as is done with textiles generally. Resistance to airflow for a known fiber mass in a known volume has been used as a quick method to indicate fiber fineness. With constant mass and volume, fine fibers have more specific surface area than coarse fibers and, therefore, greater resistance to airflow. The International Standard (ISO) 2370 (120) entitled "Textiles—Determination of Fineness of Flax Fibres—Permeametric Methods to Determine Fineness" was developed using airflow. This method, using reference samples based on the tex system, "permits compensation for the fact that the fineness of flax fibers cannot be defined in an absolute manner" (120). Both parallel (reference method) and random fibers are analyzed. The ASTM Standard D1448 (121) using airflow for cotton fineness, measured in micronaire (units of  $\mu$ g/in.), was modified with flax fibers cut to 2.54 cm and loaded at 5 g and evaluated against a series of IFS grades of flax samples (119). A high relationship ( $\mathbb{R}^2 = 0.99$ ) occurred between the IFS and the modified micronaire method. Fibers are routinely evaluated using airflow (110,122), but this method provides only a relative scale for comparison, since the number has not been related to quality or processing efficiency as in cotton. Image analysis provides a method to assess fineness, with determination of the fiber and fiber bundle diameters directly (28,110). This method is not a rapid method, although automation has improved the efficiency of these systems. Image analysis has the advantage of providing fiber width distributions that occur with flax due to variation in fiber bundle sizes caused by myriad factors. Fiber width distribution and the relationship to the value obtained with the modified micronaire system for a series of flax samples is shown (Fig. 17**a**-**d**; Table 8).

Wet ring spinning systems use long-line, fine flax fiber for high value, traditional 100% linen textiles. For composites, long and strong fibers with low elongation are sought to provide reinforcement of resins, with coarseness and levels



**Fig. 17.** Image analysis to determine flax fiber widths and width distribution. The frequency % is shown for selected width categories (multiply  $\times$  10 for width in µm) to compare various samples. Most of the fibers are in the 20–30 µm range regardless of treatment or source. The finer samples (see Table 8) have a higher frequency % in the 10–30 µm range and a lower % in the larger width categories. (**a**) Finest fiber (IFS 21) from Institut Textile de France, Lille. (**b**) Coarsest fiber (IFS 72). (**c**) Experimental sample of enzyme-retted flax. (**d**) Similar to c above but processed through the Shirley Analyzer.

	Width frequency $\mu m$ (image analysis)				Fineness
Sample	10-30	40-100	110 - 200	210-300	$(air-flow)^a$
IFS Grade B <sup>b</sup> IFS Grade J <sup>b</sup> SER <sup>c</sup> SER-SA <sup>d</sup>	$76.3 \\ 46.1 \\ 43.9 \\ 67.1$	$19.6 \\ 36.3 \\ 35.2 \\ 30.1$	$\begin{array}{c} 4.1 \\ 13.7 \\ 14.6 \\ 2.2 \end{array}$	0 3.9 6.3 0.6	3.7 7.4 7.8 4.8

Table 8. Fineness Measurements of Flax Fibers

<sup>a</sup> Fineness test based on the modified cotton micronaire method.

<sup>b</sup> From set of standard fineness grades, Institut Textile de France, Lille. B represents the finest fibers (IFS score 21.7), and J represents the coarsest fibers (IFS score 72.1). <sup>c</sup> Spray enzyme retted, experimental sample.

<sup>d</sup> Spray enzyme retted and further processed through the Shirley Analyzer.

<sup>e</sup> Adapted from Akin and co-workers (110,122).

of nonfiber components perhaps allowable based on application. Cottonized fibers for blending with cotton on short staple spinning systems require uniform lengths for efficient processing. Often, total fiber is processed sufficiently for length or cut to specific lengths for various applications.

Attempts have been made to employ instruments that rapidly and objectively analyze cotton to assess flax fiber as well. Hardware and software modifications allowed some success over time, but optimum performance of the equipment for flax analyses has not been achieved as of yet. In order to measure flax fibers successfully with cotton equipment, a major redesign in the mechanics and software of instruments likely will be required. The amount of development needed, a predicted small market size, and lack of standards have prevented progress in this area.

Recent attempts have been made to use rapid, spectroscopic methods to assess flax fiber quality in place of time-consuming older methods. Models using near infrared (nir) reflectance spectroscopy (123) were used to monitor the degree of enzyme-retting. Faughey and Sharma (124) used particular wavelength ranges from nir spectroscopy for models to assess flax fiber fineness (using calibration data from derivative thermogravimetric analysis and airflow methods) and fiber strength. Components in the flax stem (eg, carbohydrates, aromatics, waxes) as well as changes in fiber cellulose have been detected using Fourier transform (FT) Raman spectroscopy (71,125). The use of ir imaging for flax (126,127) has potential to identify the site of specific components that relate to retting efficiency, utilization, and quality. These near and mid-ir spectroscopic methods must be related to calibration sets from some other assessment method, eg, wet chemical, strength, fineness. With advances in rapid, nondestructive chemometric methods, success has been made in assessing flax fiber yield and properties. These methods, furthermore, could facilitate the development of standards for an objective classification system for flax and linen.

**6.2. Standards.** Even though flax is considered the oldest textile fiber known, objective standards recognized for the industry, such as for cotton (128), for the most part do not exist at this time for flax (4). The need for such standards and a classification system for judging quality, commerce, and processing efficiency is widely recognized (21,129-131). The only publicized standard available is ISO 2370 (120) for fineness. Instead, flax is traditionally bought

and sold by the subjective judgment of experienced graders who appraise by look and feel, called organoleptic tests. Various classification schemes that include the source (eg, Belgium, France, Russia, or China), processing history (eg, water- or dew-retted), or application (eg, warp or weft yarn) have been used within an industry segment. Grading systems for traditional linen attempt to assess fineness, length and shape of fibers, strength, density, luster, color, handle, parallelism, cleanliness, and freedom from neps and knots (113). Within particular countries, measurement of flax fibers is done by more or less consistent means and, therefore, a limited classification system may exist. For example, in past years Russia used an elaborate judging and grading system for commerce and processing of flax (16). Various grades of flax fibers are identified for marketing within a company.

The European Union project COST 847 (Textile Quality and Biotechnology) group reported "the situation regarding the characterization of flax and other bast fibres is certainly not satisfactory" (21). The development of standards for judging flax fiber quality has been held back by difficulty in assessing flax due to its complex physical structure, inconsistent measurement practices, lack of industry support, and a rather small, confined market for traditional long-line flax and tow. The early efforts by ISO, which resulted in ISO 2370 (120) and working documents for other properties, have been discontinued. Current interest, however, in expanding the use of flax fiber in composites for diverse applications and blending refined flax with cotton for high speed, efficient short staple spinning systems requires the establishment of standards, much like those that have helped the cotton industry (128). The need for standards, therefore, is certainly recognized. Cost Action 847 has as a stated objective acquiring knowledge "to set up quality standards for assessing flax fibre" (21). In the United States, subcommittee D 13.17, entitled Flax and Linen, of the Textile Committee in ASTM International was established in 1999 with the goal of establishing a set of standards for flax fiber. As of this writing, a terminology standard has been approved (ASTM D 6798-02) and other documents on fineness, color, and trash are in various stages of preparation and discussion.

# 7. Future Outlook

Linen has about a 2-3% share of the consumer textile market, compared with cotton at ~65%. Comfort, drape, and distinctive appearance, however, continue to command market share for linen (12). Emphases in the fashion industry will likely continue to dictate a periodicity in use and value of linen and flax fibers for textiles. Cotton and flax blends, both as intimate blends and with flax used as weft yarns, continue to be popular in the United States, which is a major importer. The greatest total value of flax fiber in the future likely will be as cottonized fibers for distinctive textiles and as industrial fibers for nonwoven materials and composites. In textiles, the cottonized flax fibers are blended with cotton or other fibers and spun into yarns on advanced and efficient, short staple spinning equipment. Cottonization of flax demands different methods of processing from traditional long-line flax, and any new retting procedure should take advantage of this opportunity. Flax and other natural fibers are in demand for reinforced

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composites, and the replacement of glass fiber with flax allows large savings in energy costs, provides advantages in the environment through biodegradability, and offers opportunities for new, value-added crops in agriculture. Commercially viable chemical- or enzyme-retting methods to improve the quality and consistency of flax fiber are needed to expand applications for flax fiber. Such methods, if made cost efficient, could expand production beyond regions where weather now limits dew-retting. Important as well is the opportunity to employ enzymes to tailor properties for specific applications. Improved retting could take advantage of the vast amounts of seed flax straw as a by-product of the linseed industry available in Europe and North America. Linseed straw, which is becoming an increasing environmental problem for disposal, likely would not suffice for traditional long-line flax but could provide a value-added resource at the farm level for use in a wide range of composites and nonwoven materials.

In addition to the need for improved retting practices, methods to measure and assess fiber quality in quick and accurate ways are lacking. The use of spectroscopy and high speed computers for chemometrics may offer a strategy to overcome current difficulties in this area. The development of a set of standards for objective assessment of fiber properties and value, as well as the basis for a classification system, is needed to expand the use of flax fibers that potentially could be grown and produced from climates and sources worldwide.

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