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JUTE AND KENAF

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1. Introduction	466
2. Formation of Fiber and Extraction	467
3. Fiber Structure	468
4. Chemical Composition	470
5. Acetyl Content	471
6. Changes in Chemical and Fiber Properties During the Growing Season	472
7. Fine Structure	474
8. Physical Properties	476
9. Grading and Classification	477
10. Fiber and Yarn Quality	478
11. Chemical Modification for Property Improvement	479
11.1 Acetylation	481
11.2 Cyanoethylation	483
12. Photochemical and Thermal Degradation	484
13. Moisture Effects	486
14. Fastness to Light	486
14.1 Undyed jute	486
14.2 Dyed jute	487
15. Woolenization	488

*Retired

16. Applications and Markets	490
16.1 Composites	490
16.2 Fiber-thermoplastic blends	495
16.3 Fiber matrix thermoplasticization	499
16.4 Fiber-thermoplastic alloys	500
17. Future Trends	502
References	502

1. INTRODUCTION

Jute is the common name given to the fiber extracted from the stems of plants belonging to the genus *Corchorus*, family Tiliaceae. Kenaf is the name given to the rather similar fiber obtained from the stems of plants belonging to the genus *Hibiscus*, family Malvaceae, especially the species *H. cannabinus* L. Only two species of *Corchorus*, namely, *C. capsularis* L. and *C. olitorius* L., are grown commercially, although around 40 wild species are known, but other species of *Hibiscus*, particularly *H. sabdariffa* L. are sometimes also marketed as kenaf.

These plants are examples of a number of woody-stemmed herbaceous dicotyledons grown in the tropics and subtropics, from the bast of whose stems fibers can be extracted. Most of the plants cultivated for fiber, including jute and kenaf, are grown from seed annually, but a few are grown as perennials. Jute is the most important fiber of this type, and it is probable that, in the industrial and engineering uses of textiles, more jute fiber is used than any other single fiber. Kenaf is used in many countries where it is grown, but its international market is much smaller than that for jute, and estimates of world kenaf production are liable to be in error. In many marketing statistics, figures given for the production or utilization of “jute and allied fibers” include all the fibers in this group. “Allied fibers” are suitable for processing on jute spinning systems.

Favorable conditions for jute cultivation are found in the deltas of the great rivers of the tropics and subtropics—the Ganges, the Irrawaddy, the Amazon, and the Yangtze, for example—where irrigation, often by extensive flooding, and alluvial soils combine with long day lengths to provide opportunity for considerable vegetative growth before flowering.

Kenaf requires less water to grow than jute does and is now grown in several countries in Europe, South America, Mexico, the United States, Japan, and China.

Both jute and kenaf reach 2.5-3.5 m in height at maturity; but kenaf, although still requiring a long day length for vegetative growth, flourishes in drier conditions than jute can, and can adapt to a wider variety of soils and climates. As a result, it has been preferred to jute as a fiber crop by many countries in Africa and Latin America, although usually only for internal use. Bangladesh remains the world’s principal exporter of this type of fiber, with exports of jute fiber currently running at around 500,000 tons/year. This compares with an FAO forecast for world consumption of manufactured jute goods of 4 million tons in 1985.

The commercial use of the base fibers goes back more than 150 years, and although during that time there has been little change in the nature of the technical fiber, considerable developments have taken place in the techniques of conversion to yarn and

fabric, and in the end-uses for these products. Scientific studies began around 60 years ago; and although they never received publicity on the scale given to studies of cotton and wool, the broad features of the internal structure and physical characteristics of these fibers were elucidated sufficiently long ago for a great deal of common knowledge to have been built up. The literature is now extensive and contained throughout a variety of journals. A number of books have become standard reading, and critical reviews of the literature have appeared from time to time [1-5]. In the description that follows of the structure and properties of jute and kenaf, this common knowledge is presented without critical annotation of references; a list of the principle books and papers that are considered relevant is appended instead.

2. FORMATION OF FIBER AND EXTRACTION

Jute and kenaf fibers develop in the phloem, or bast, region of the stem of the plants; in transverse sections of the stem, they appear as wedge-shaped bundles of cells intermingled with parenchyma cells and other soft tissue (Fig. 7.1). In the growing part of the stem, a circumferential layer of primary fibers develop from the protophloem, but, as vertical growth ceases in the lower parts, secondary phloem fibers develop as a result of cambial activity. In mature plants, which reach a height of 2.5-3.5 m and

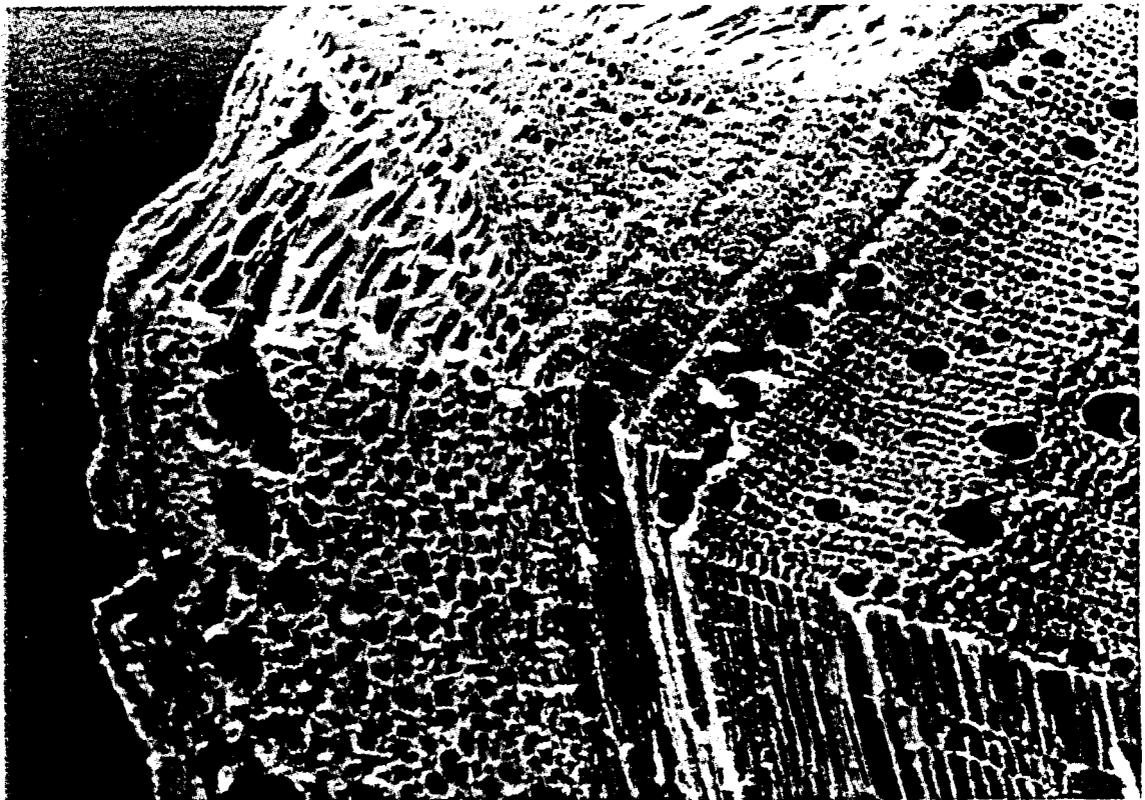


Figure 7.1 Jute stem (combined transverse section and longitudinal section). Magnification x 70. (Courtesy of Dr. C. G. Jarman, Tropical Development and Research Institute, London, U. K.)

a basal diameter of about 25 mm, the secondary fiber accounts for about 90% of the total fiber bundles.

The plants pass from vegetative to reproductive phase when the day length falls below 12.5 hr. Vertical growth then ceases, and cambial activity declines. The production of cell bundles is much reduced, but, at the same time, the secondary fiber cells begin to mature rapidly. Their walls, which have remained thin during the vegetative period, become thicker, and they increase in weight and strength.

Harvesting the plants at the correct time is most important and requires long experience. For kenaf, the optimum time for harvesting is when about 10 of the flowers are in bloom and the older flowers have already set their seed. With jute, the correct time is judged to be when the plants are in the small-pod stage. Harvesting before flowering generally results in lower yields and weaker fiber; and if the seeds are allowed to mature, the fiber becomes harsh and coarse and difficult to extract from the plant.

The plants are harvested by hand with a sickle and cut close to the ground. The cut stems are then tied into bundles, the leaves removed as much as possible, and the bundles submerged in water for retting. This is the process by which the bundles of cells in the outer layers of the stem are separated from the woody core and from nonfibrous matter by the removal of pectins and other gummy substances. The action involves water, microorganisms, and enzymes, and takes between 5 and 30 days to completion, depending on the temperature of the water. Constant supervision is required, and the time of removal is critical, because if the degree of retting is insufficient, the fiber cannot easily be stripped from the woody core and may be contaminated with cortical cells; and if retting proceeds too far, the fiber cells themselves may be attacked and weakened by microorganisms. Stripping the fiber from the stem is done by hand, after which the fibers are washed and dried.

A difficulty in the retting procedure is that the thicker parts of the stem take longer to ret than the thinner parts do; consequently, if the butt ends of the stem are fully retted, the top ends are overretted and damaged. This can be avoided by stacking the bundles of stems upright with the butt ends in water for a few days before immersing the whole stem; but with fiber intended for export, it is usual to cut off the partly retted butt ends and sell these separately as "cuttings."

Correct retting is an essential first step in the production of good quality fiber. A comprehensive account of the techniques used, and their effects on fiber quality has been given by Jarman [6a]. Controlling the quality of water along with improving microorganisms used in the process are the keys to improved fiber quality.

3. FIBER STRUCTURE

In each plant, the rings of fiber cell bundles form a tubular mesh that encases the entire stem from top to bottom. Two layers can usually be distinguished, connected together by lateral fiber bundles, so that the whole sheath is really a lattice in three dimensions [6b]. The cell bundles form the links of the mesh, but each link extends only for a few centimeters before it divides or joins up with another link. After extraction from the plant, the fiber sheath forms a flat ribbon in three dimensions.

The jute or kenaf fiber or commerce refers to the sheath extracted from the plant stems, whereas a single fiber is a cell bundle forming one of the links of the mesh.

Staple length, as applied to cotton and wool fibers, has no counterpart in the base fibers, and, as a preliminary to spinning, it is necessary to break up the sheaths by a carding process. The fragments so produced are the equivalent of the staple fibers of the cotton and wool industries.

When a transverse section of a single jute fiber is examined under the microscope, the cell structure is seen clearly. Each cell is roughly polygonal in shape, with a central hole, or lumen, comprising about 10% of the cell area of cross section, as shown in Fig. 7.2. In longitudinal view, the fiber appears as in Fig. 7.3, which shows the overlapping of the cells along the length of the fiber. The cells are firmly attached to one another laterally, and the regions at the interface of two cells is termed the middle lamella. Separation of cells can be effected by chemical means, and they are then seen to be threadlike bodies ranging from 0.75 to 5 mm in length, with an average of about 2.3 mm. The cells are some 200 times longer than they are broad, and in common terminology are referred to as *ultimate cells*. A single fiber thus comprises a bundle of ultimates.

Transverse sections of single fibers show that the number of ultimate cells in a bundle ranges from a minimum of 8 or 9 to a maximum of 20-25. Bundles containing up to 50 ultimate cells are sometimes reported, but in such cases it is questionable whether the fiber is truly single in the botanical sense or is comprised of two fibers adhering together. A minimum number of cells in the cross section is evidently necessary to provide a coherent and continuous overlapping structure.

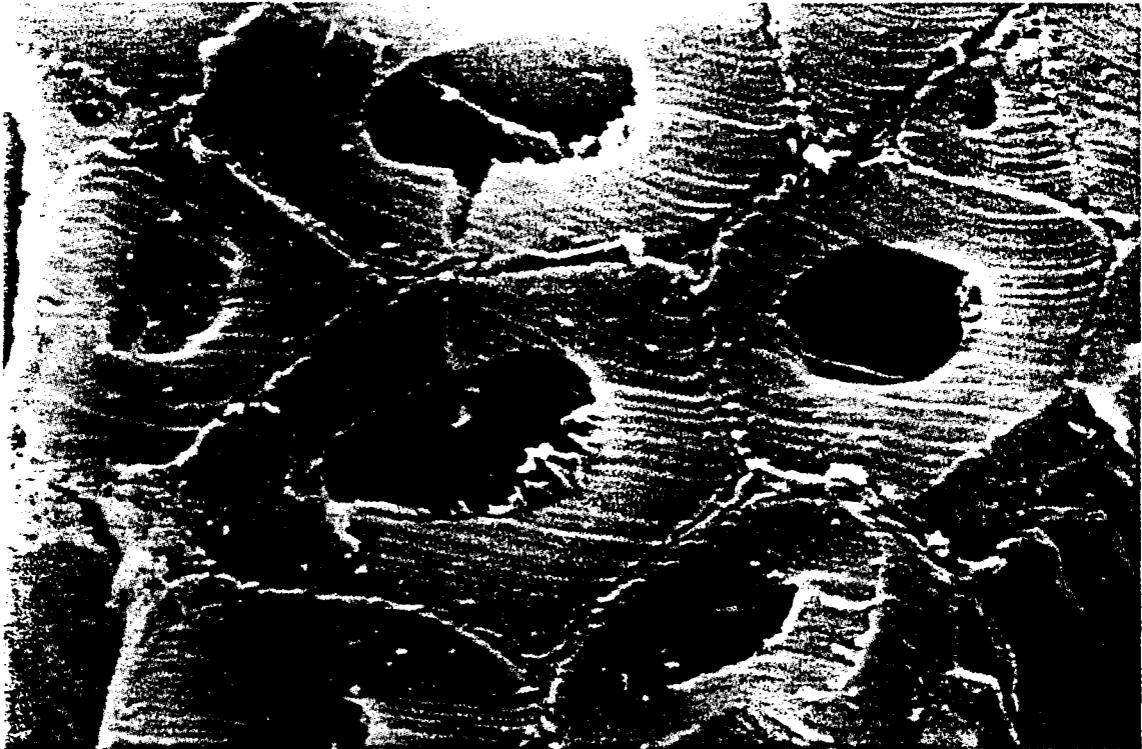


Figure 7.2 Part of a fiber bundle of jute as seen in transverse view under the scanning electron microscope. The cementing material between the ultimate fibers can clearly be seen. Magnification $\times 7600$. (Scanning electron micrography by Mr. A. J. Canning. Courtesy of Tropical Development and Research Institute, London, U. K.)

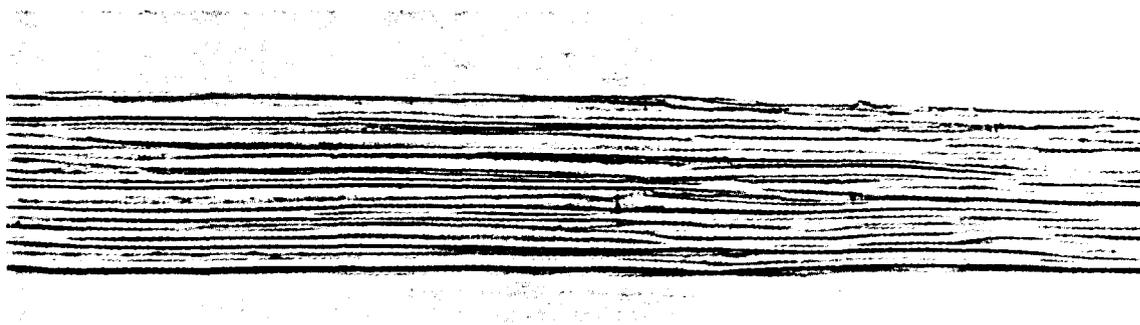


Figure 7.3 Longitudinal view of a single fiber strand of jute showing ultimate fibers. The tips of the ultimate can be seen slightly to the right of center. Stained in safranin. Magnification $\times 500$. (Photomicrograph by Mr. A. J. Canning. Courtesy of Tropical Development and Research Institute, London, U. K.)

The ultimate cell dimensions of kenaf and many other fiber-bearing dicotyledons are similar to those of jute. A distinction must be made between jutelike fibers and flax, however, for although all are described as base fibers, the ultimate cells in flax are much longer, averaging 20-25 μm . They are also greater in cross-sectional area, and, because of the longer length, a coherent fiber structure can be built up from only two or three overlapping ultimates. The single fibers of flax are thus much finer than those of jute.

4. CHEMICAL COMPOSITION

Retted fibers such as jute and kenaf have three principal chemical constituents, namely α -cellulose, hemicelluloses, and lignin. The lignin can be almost completely removed by chlorination methods in which a soluble chloro-lignin complex is formed, and the hemicelluloses are then dissolved out of the remaining holocellulose by treatment with dilute alkali. The final insoluble residue is the α -cellulose constituent, which invariably contains traces of sugar residues other than glucose.

The hemicelluloses consist of polysaccharides of comparatively low molecular weight built up from hexoses, pentoses, and uronic acid residues. In jute, *capsularis* and *olitorius* have similar analyses, although small differences occur among different fiber samples. For fiber extracted from jute plants grown in Bangladesh, the range of composition has been given as lignin, 12-14%; α -cellulose, 58-63%; and hemicellulose, 21-24% [7].

In addition, analysis of the hemicellulose isolated from α -cellulose and lignin gives xylan, 8-12.5%; galactan, 2-4%; glucuronic acid, 3-4%; together with traces of araban and rhamnosan. The insoluble residue of α -cellulose has the composition glucosan, 55-59%; xylan, 1.8-3.0%; glucuronic acid, 0.8-1.2%; together with traces of galactan, araban, mannan, and rhamnosan. All percentages refer to the weight of dry fiber.

As well as the three principal constituents, jute and kenaf contain minor constituents such as fats and waxes, 0.4-0.8 %; inorganic matter, 0.6- 1.2 %; nitrogenous matter, 0.8- 1.5 %; and traces of pigments. In total, these amount to about 2%.

The detailed molecular structure of the hemicellulose component is not known with certainty, although in the isolated material the major part is stated to consist of a straight chain of D-xylose residues, with two side-branches of D-xylose residues, whose position and length are uncertain [3]. In addition, there are other side-branches formed from single residues of 4-*O*-methyl glucuronic acid, to the extent of one for every seven xylose units.

The third major constituent, lignin, is a long-chain substance of high molecular weight which, like the hemicelluloses, varies in composition from one type of vegetable material to another. The molecular chains are built up from comparatively simple organic units which may differ from different sources as well as in the way in which they are combined.

Most of the studies in lignin have been concerned with wood; the bast fibers have been rather neglected. It seems unlikely, however, any major differences will exist between jute and wood lignin, but in any case many details of the molecular structure still remain unresolved.

5. ACETYL CONTENT

Jute and kenaf, like most vegetable fibers, contain a proportion of acetyl groups that are readily hydrolyzed by dilute alkali to acetic acid. Estimation of the quantity of acetic acid produced per unit weight of fiber then provides an index of the acetyl content.

The acetyl content of any particular type of fiber shows some variation according to where it is grown and under what conditions, but often these intrafiber variations are small compared with the variations among fiber types. This is the case with hibiscus and corchorus fibers, for example; Soutar and Brydon [8] have reported acetyl contents averaging 110 for *Hibiscus*, 89 for *C. capsularis*, and 76 for *C. olitorius*, all expressed in milliequivalents of acetic acid per 100 g of dry fiber. The higher acetyl content in *capsularis* over than in *olitorius* has since been confirmed by Khuda et al. [9].

Soutar and Brydon's results show no significant difference between *H. cannabinus* and *H. sabdariffa*, which is, perhaps, surprising in view of the difference between the two jute varieties; but the acetyl content does appear to offer a means of differentiating between jute and kenaf. For such a comparison to be valid, of course, there must have been no prior treatment of the fibers with alkali, such as occasionally happens in chemical retting experiments.

An interesting feature of this study is the measurement of acetyl content for other fibers covering a wide range of hemicellulose and lignin contents. The study concludes that acetyl content shows a steady increase with increases of hemicellulose content as the latter ranges from about 4 to 26%, but the correlation with varying lignin content is not marked.

An alternative method of distinguishing between jute and kenaf is by means of the crystals in the ash of the fiber after incineration. These crystals are present in the parenchyma and retain their original form during asking. In kenaf, cluster crystals are commonly found in the ash, whereas they are relatively uncommon in the case of jute. Jarman and Kirby [10], however, have shown that jute can be distinguished by the fact that the ash contains solitary crystals occurring in chains. Solitary crystals may occur in kenaf but not in chains.

6. CHANGES IN CHEMICAL AND FIBER PROPERTIES DURING THE GROWING SEASON

It is well known that different parts of a plant have different chemical and physical properties. That is, the chemical compositions and fiber properties of plant tissues taken from the roots, stem, trunk, and leaves are different from each other. What is not so well known is that the chemical composition and fiber properties of plant tissue is also different at different stages of the growing season.

The University of Manchester, the Shirley Institute, and the British Textile Technology Group in the United Kingdom have spent years working on jute. While some of the research has been published, the results relating to the changes in properties of jute fiber as a function of the growing season were done for the International Jute Organization in Bangladesh and never published [11]. The research records are stored in Bangladesh, and attempts to gain access have failed. Personal communications concerning these results indicate that juvenile jute fiber looks and feels like silk, but this has never been documented in print.

Working at the Technological Jute Research Laboratories in Caicua, India, Chatterjee first reported the changes in chemical composition at different stages of jute plain growth [12]. Table 7.1 shows a summary of his results. These results show that there is little difference in cellulose, holocellulose, and lignin contents, but that xylan, ash, and iron contents decrease as the plant matures. The aggregate fiber length increases as the growing season progresses. Without defining what is meant by "best," Chatterjee reports that the best fiber is obtained at the bud stage.

Later, Mukherjee et al., working at the Indian Jute Industries Research Association in Calcutta, studied the surface characteristics of jute fiber at different stages of growth [13]. At the early stages of growth, they found that there was an incomplete formation of the middle lamella in the cell wall and that the parallel bundles of fibrils were oriented as an angle with respect to the fiber axis that gradually decreased with growth.

Table 7.1 Changes in Chemical Composition of Jute at Different Stages of Plant Growth*

Component	Stage of plant growth				
	Pre-bud	Bud	Flower	Small pod	Large pod
α -cellulose	58.3	57.6	59.4	58.7	59.1
Holocellulose	86.8	87.8	87.3	87.1	86.8
Xylan	15.5	14.8	14.4	13.7	13.9
Lignin	12.1	12.7	12.4	12.0	12.0
Ash	0.57	0.53	0.47	0.67	0.47
Iron	0.020	0.018	0.009	0.011	0.008
Reed length (ft)	6.6	9.1	9.3	9.6	10.7

*Reported on 100 g of dry material.

Source: Ref. 12.

Table 7.2 Changes in Chemical Composition of Kenaf at Different Stages of Plain Growth* (percent by weight)

Component	Stage of plant growth (by days after planting)					
	90	120	138	147	158	244
Hot-water extractives	25.0	22.7	21.6	17.6	16.6	9.7
Lignin	7.0	9.0	9.9	10.7	11.4	13.4
α -Cellulose	23.5	25.5	28.0	31.2	31.9	35.3
Pentosan	8.9	13.2	14.9	16.7	14.6	23.4
Protein	14.8	12.1	9.3	7.6	9.2	6.0

*Data from analysis of bottom part of the plant.

Source: Ref. 14.

After about 35 days of growth, the fibrils run parallel to the fiber axis. In the mature plant, a few helically oriented fibrils in the Z-direction were observed just below the primary cell wall layer.

Clark and Wolff carried out the first studies on the changes in chemical composition of kenaf as a function of the growing season [14]. They also studied the chemical differences along the stem and between leaves and stem. The values shown in Table 7.2 were taken from the bottom (all but the top 0.66 meter of the plant) and show that the pentosans, lignin, and cellulose content increase with age, while the protein and hot water-extractives content decrease with age. Data taken from the top part of the plant (Table 7.3) show similar trends, but the top part has less cellulose, pentosans, and lignin but higher hot-water extractives and protein than the bottom part of the plant.

Clark et al. also studied the changes in kenaf fiber properties during the growing season [15]. Table 7.4 shows that the bast single fibers are longer than the pith fibers and that both decrease in length with age. Pith single fibers are twice as wide and have

Table 7.3 Changes in Chemical Composition of Kenaf at Different Stages of Plant Growth* (percent by weight)

Component	Stage of plant growth (by days after planting)					
	90	120	138	147	158	244
Hot-water extractives	37.4	39.0	35.2	31.6	30.6	12.8
Lignin	4.5	3.9	6.4	7.2	7.4	11.4
α -cellulose	10.6	14.5	18.5	18.1	20.6	29.8
Pentosan	5.0	12.5	16.1	17.0	16.7	20.1
Protein	25.0	17.9	16.1	13.3	14.9	11.1

*Data from analysis of top 0.66 m of the plant.

Source: Ref. 14.

Table 7.4 Changes in Fiber Properties of Kenaf at Different Stages of Plant Growth

Component	Stage of plant growth (days since planting)			
	90	120	150	180
Bast fiber				
Length (mm)	3.34	2.28	2.16	2.42
Width (microns)	18.3	14.5	13.6	15.1
Lumen width (microns)	11.1	5.4	6.8	7.7
Cell wall				
Thickness (microns)	3.6	4.6	3.4	3.7
Pith fiber				
Length (mm)	0.55	0.54	0.45	0.36
Width (microns)	36.9	31.2	32.0	31.6
Lumen width (microns)	22.7	14.8	18.6	18.7
Cell wall				
Thickness (microns)	7.1	8.2	6.7	6.4

Source: Ref. 15.

twice the cell wall thickness as bast single fibers; both dimensions decrease with age. Finally, the lumen width is greater in pith fibers as compared to that of bast single fibers, and both decrease with age.

In a recent study, Han and Rowell reported changes in kenaf as a function of the growing season [16]. They found that the average length of a bast and core (stick) fiber increased as the plant aged. Protein content went from 1.48% at 48 days to 1.84% at 70 days. Lignin content went from 5.4% at 48 days to 10.5 at 70 days. Solvent extractive content increased from 2 % to 13% in the same length of time. Arabinose, rhamnase, galactose, and manose content decreased from day 48 to day 70 while glucose and xylose content increased over this same period.

7. FINE STRUCTURE

The location of the three main chemical components of the fibers are reasonably well established. α -Cellulose forms the bulk of the ultimate cell walls, with the molecular chains lying broadly parallel to the direction of the fiber axis. The hemicellulose and lignin, however, are located mainly in the area between neighboring cells, where they form the cementing material of the middle lamella, providing strong lateral adhesion between the ultimates. The precise nature of the linkages that exist between the three components and the role played by the middle lamella in determining the fiber properties are incompletely understood. Some years ago, in the interesting literature survey on the middle lamella of base fiber, Lewin [17] brought together a great deal of relevant information that illuminated many of the problems; but a thorough understanding of the intercell structure is still awaited.

X-ray diffraction patterns show the basic cellulose crystal structure. But in jute and kenaf, although the crystallite orientation is high, the degree of lateral order is relatively low in comparison with, for example, that of flax. There is also considerable background x-ray scattering arising from the noncellulosic content of the fiber.

The cellulose molecular chains in the secondary walls of ultimate cells lie in a spiral around the fiber axis. The effect of this is to produce double spots in the x-ray diffraction patterns, the centers of the spots being separated by an angular distance of twice the Bragg angle. For large angles such as occur in coir fiber and some leaf fibers such as mauritius hemp, the two spots are visibly separated; but for the small angles found in jute and kenaf, the spots overlap. In this case, the distribution of intensity across the width of the spots, instead of reaching a peak at the center of each, is spread out into a single, flatter, peak. The [002] equatorial reflection shows these effects particularly well, and analysis of the intensity distribution allows calculation of the Hermans RMS spiral angle. A wide range of base and leaf fibers have been examined in this way [18], with results showing the Hermans angle to range from about 80 for jute and kenaf to 23° for sisal. Coir fiber, *Cocos nuciferos*, is exceptional in having a Hermans angle of about 45°.

The leaf fibers in this study are particularly interesting, because as well as covering a good range of spiral angles, they also cover a wide range of ultimate cell dimensions. The results indicate that, among this group of fibers, the spiral structure averages a constant number of turns per unit length of cell, about 10/mm; with this arrangement, the spiral angle then depends solely on the breadth of the cell. Whether this constancy of turns applies to individual cells, or whether the longer cells tend to have steeper spirals (as in wood), was not, however, investigated.

For the secondary bast fibers, the cell dimensions show little variation among plant species, but the number of spiral turns per unit length of cell averages only about four per millimeter, appreciably fewer than for the leaf fibers.

The importance of the spiral angle measurements lies in the control that the spiral structure exercises on the extension that the fiber can withstand before breaking. Regarding the structure as a helical spring, the extension necessary to straighten a spring of initial angle q to the axis is $(\sec q - 1) \times 100\%$. A 10° spring will thus extend by 1.54%. a 20° spring by 6.4%, and a 30° spring by 15.5%.

The coconut fiber, coir, is a particularly interesting case, with a spiral of about 45°, and a helical spring extension of 41.4%. Such a large extension is easily measured and has been shown to be reasonably correct. Moreover, it is possible to carry out the extension in stages and to measure the angle while the fiber is stretched and under tension. X-ray measurements showed the angle to decrease with the extension, as predicted by the spring structure, and it was concluded that the extensibility of coir fiber is due almost entirely to the spiral structure of the ultimate cells [19]. This has been confirmed by other studies in which the spirality of the cell wall was investigated microscopically using replica and ultrathin sectioning techniques [20].

It is interesting to note that when coir fiber relaxes after stretching, it shortens in length, and the spiral angle increases according to the spring theory. There is usually a semipermanent set left in the fiber after relaxation, but this can be removed by steaming, and the fiber can be restored virtually to its original unstretched length.

To carry out similar measurements of the extension/spiral angle relationship for low-angle fibers such as jute and kenaf is difficult. because of the changes in angle are small, the overlapping of the spots in the x-ray diffraction pattern could introduce significant errors. With coir, the angles over most of the extension range are measurable to a higher degree of accuracy,

Assuming, therefore, that the coir results are of general applicability to fiber cells, it appears that the helical spring theory could be used to calculate the order of magnitude of the extensibility of the fiber and to rank fibers accordingly.

8. PHYSICAL PROPERTIES

Jute and kenaf are strong fibers but exhibiting brittle fracture and have only a small extension at break. They have a high initial modulus, but show very little recoverable elasticity. Tenacity measurements recorded in the literature vary widely, and although some of this variation is due to differences in the methods of measurement, a major part is due to variations in linear density of the fibers themselves. All linear densities will be given in Tex units of grams per kilometer.

Taking account of all the available evidence, a tenacity of 70 g/Tex is a reasonable middle value for a wide range of jute fibers, based on single fiber test lengths of 10 mm or less. and a time to break of 10 sec. This value of tenacity is appropriate to fibers of linear density 1.8 Tex; it is important to state the linear density, for an increase of 0.1 Tex reduces the tenacity by about 1.5 g/Tex. This inverse dependence of tenacity on linear density is common to most fibers and also to fine metal wires.

The elongation at which a fiber breaks is a more invariant and fundamental property than the load at which it breaks. It is not affected significantly by changes in linear density, or by changes in the method of loading. Length of test specimens does have an effect, however, as irregularities in diameter prevent all sections of a long fiber from being elongated equally. For test lengths of 10 mm, the elongation is generally between 1% and 2% of the initial length, but is difficult to measure accurately such short lengths. In one case, 500 fibers from a bulk of medium-quality jute had a mean elongation of 1.60% (of the 10-mm test length) with a coefficient of variation (CV) of 25%. The breaking load of the fibers, however, had the much higher CV of 40% [21]. It may be noted that 1.6% elongation corresponds to a spiral angle of $10^{\circ}12'$, which, although slightly greater than the Hermans angle reported, is still within the uncertainty of the comparison.

The initial Young's modulus of the fibers, calculated from the slope of the load-elongation curve, has a mean value of about 4×10^3 g/Tex at 100% extension. The value for any particular group of fibers, of course, will be dependent on the linear density, to some extent owing to the dependence of tenacity values on this factor.

The bending of jute fibers has been studied by Kabir and Saha, who calculated the Young's modulus from measurements of the force required to deflect the free ends of a fringe of fibers arranged cantilever-fashion [22]. For this calculation, it is necessary to know the fiber diameter instead of the linear density, and this causes a difficulty because the cross section of the fiber is irregular in outline and often far from circular. The authors assumed an elliptical configuration, and measured the minimum and maximum diameters of a number of cross sections microscopically for insertion in the appropriate formula. Their calculations showed that, over a wide range of commercial fiber qualities, Young's modulus decreased from about 2.0×10^{11} dynes/cm² at 46 mm average diameter, to 0.8×10^{11} dynes/cm² at 68 mm. These values correspond to 3050 and 815 g/Tex at 100% extension, respectively, and again demonstrate the marked effect of variations in fiber dimensions. Extrapolations of Kabir and Saha's data to smaller diameters shows that the tensile value for the modulus of 4000 g/Tex at 100% extension would be reached at a mean diameter of about 40 mm.

Young's modulus may also be calculated from the fundamental frequency of transverse vibration of a single fiber fixed at one end as a cantilever. For a variety of bast fiber types, including jute and kenaf, the modulus lies between 3 and 8×10^{10} dynes/cm², and for jute fibers appears almost independent of diameter, unlike the tensile values [23].

In making these dynamic measurements, it was found that each fiber had two resonant frequencies corresponding to vibration along the major and minor axes of the cross section, respectively. The extent of the difference between the two frequencies gives an indication of the departure from a circular outline.

Kabir and Saha also examined the effect of delignification on the bending modulus of jute, using the fringe technique, and showed that successive extractions of lignin from the same fibers resulted in an increasing flexibility and a decreasing Young's modulus [24]. The delignification method was treatment with sodium chlorite solution followed by extraction with sodium bisulfate. Removal of 10% of lignin reduced the modulus from 1.10 to 0.79×10^{10} dynes/cm². At the same time, however, the diameter of the fibers was reduced significantly, and this may have affected the flexibility.

9. GRADING AND CLASSIFICATION

The grading and classification of bast fibers such as jute and kenaf for commercial purposes has a long history but is still done subjectively by hand and eye. Official standards have been formulated, but these are purely descriptive, and no quantitative values are assigned to the stated criteria. Nevertheless, there is a surprising degree of consistency, particularly for export purposes, and experience buyers and sellers do not find it too difficult to agree on whether or note the grade assigned to a particular consignment of fiber is correct.

For jute fiber exported from Bangladesh, for example, the current grading system first separates *C. capsularis* and *C. olitorius* into white and tossa categories, respectively, and then further classifies each into five grades denoted by the letters A to E. The highest prices are paid for Grade A, although a special grade may be introduced for which a higher price can be demanded.

The principal criteria are color, luster, strength, cleanliness, and freedom from retting defects. From a spinning point of view, color is irrelevant but certain end-users traditionally prefer fibers in particular colors for the sake of appearance. Luster is commonly an indication of strength: if, for example, the fiber has been overretted so that the cellulose, or middle lamella, have been attacked and weakened, the surface appears dull. A lack of luster thus downgrades the fiber, although occasionally this same effect may result from inadequate washing, without any loss of strength. Strength of fiber is also assessed by snapping a few strands by hand, a procedure that gives a useful indication of quality to an experienced operator.

Cleanliness and freedom from nonfibrous matter is an important feature. In this respect, the physical imperfections that may result from improper retting can have a profound effect on the allotted grade. Adhering bark in any form results in downgrading, irrespective of the intrinsic value of the fiber; and in the case of plants grown on flooded land, which stand in water, the bark becomes so difficult to remove that, for export, the root ends are cut off and sold separately as "cuttings," to be used in heavy yarns of low quality.

The linear density of the individual fibers making up the network is given little consideration in grading, despite the importance of this characteristic in staple fibers, where it is a major factor controlling the levelness of the spun yarn. Adhering bark increases the linear density of the fiber and makes subjective assessment difficult.

Khuda et al. [9] have studied the variation in chemical constituents of jute fiber taken from different grades of both white and tossa and concluded that certain correlations exist between the analytical results and the commercial grade. Thus it is claimed that, as the grades go from higher to lower, the lignin content increases. The ash content and copper number show similar negative correlations with level of fiber grade.

Although it might be expected that variations in chemical composition would result in variations in physical characteristics, a correlation with fiber grade is surprising. The chemical composition is that of the fiber itself, and can scarcely take account of the physical imperfections resulting from inadequate retting, which are so important in commercial grading.

The essential feature of any system of grading is that it be self-consistent in the sense that buyers and sellers can agree on the attributes that place a fiber in a particular grade. But it does not follow that a subjective system based on appearance and feel will classify fiber in the same way that an objective system based on measurement will. Both systems may be valid but in different ways, and there is no need to seek a close correlation between them except, perhaps, for classification of the top and bottom grades.

Commercial buying and selling takes place according to a subjective system. A buyer selects a range of fiber grades from which blends will be made up appropriate to the different yarn qualities required. If these fiber grades can now be measured for quality according to an objective system, more precision in blending will be possible.

Any system of objective grading based on measurable characteristics, in fact, must be concerned with the fiber as it is, including nonfibrous matter, and not merely with the single fibers themselves. With the condition in view, Mather [25], in work extending over a decade at the British Jute Trade Research Association laboratories in Scotland, studied the classification of a bulk of jute for its spinning quality.

10. FIBER AND YARN QUALITY

The principal uses for jute yarns are for industrial purposes in which adequate strength is essential. Appearance and color are of little significance. For jute yarns, "quality" relates specifically to tensile properties. An objective classification of fiber in bulk thus requires the identification of those attributes of the raw fiber which affect yarn strength. Each grade of fiber bought commercially must then have these attributes measured and the grades assessed for corresponding yarn quality. By blending together fibers having different values of these attributes, the average value will serve to predict the strength of the yarn spun from the blend.

From an extensive series of correlations between fiber properties and yarn properties, Mather [25] concludes that the tensile properties of a yarn can be predicted from only two measurements on the raw fiber: the linear density, and the ballistic work of rupture of uncarded strands of fiber.

The linear density was measured by an air-flow method using a modification of apparatus designed for cotton and wool. A sample of fiber weighing 27 g was used, and care was taken to include in the sample a similar amount of nonfibrous matter as was contained in the bulk. The nonfibrous component effectively increases the linear

density, and a less regular yarn results when spinning to a fixed count.

The ballistic work of rupture was measured by stretching strands of fiber of known linear density transversely across the path of a falling pendulum and recording the energy lost by the pendulum in breaking the strands. The energy lost per Tex is then a measure of the specific work of rupture and is related to the product of tensile strength and extensibility. The particular feature of the work of rupture is that it appears to control the average length of fiber after carding. Staple length has no meaning in the bulk fiber, and it is only after the mesh has been fragmented by carding that average length becomes meaningful.

The yarns were spun on a standard system of carding, drawing, and spinning frames, programmed to produce yarn of linear density 275 Tex. Different spinning systems and different linear densities will also affect yarn strength, and these must be taken into account. It is inappropriate to discuss the technology of jute spinning in this article; a detailed account of the experimental work on which Mather's conclusions are based has been compiled by Stout [26].

Quantitatively, Mather concludes that, for jutes exported from Bangladesh (or the erstwhile East Pakistan), the range of linear density is about 1.3–2.4 Tex, and work of rupture ranges from 4.0 to 8.3 g/cm/Tex. For kenaf, although work of rupture is little different, the linear density is often higher than that for jute; and a survey of *H. sabdariffa* grown in Thailand showed a range of 1.9–3.0 Tex.

Moreover, in the overlapping region of linear density of 1.9–2.4 g/cm/Tex, it was noticeable that the kenaf fibers were intrinsically coarse but free from nonfibrous matter, whereas the jute fibers were intrinsically much finer but carried a significant amount of adhering bark.

It was also concluded from the statistical correlations, that the change in tenacity of a jute yarn resulting from a certain percentage change in fiber linear density is about three times greater than that resulting from a similar percentage change in ballistic work of rupture. Moreover, no correlation was found between linear density and work of rupture, so these two parameters must exercise their effects quite separately.

The fiber linear density is a measure of the average number of fibers in the cross section of a given yarn, and this controls the yarn irregularity. The more fibers in the cross section, the more uniform will be the yarn thickness from point to point. As yarns break at their thinnest points, the greater will be the breaking load, irrespective of the intrinsic fiber strength.

The high modulus of jute has made jute materials a partial substitute for glass fiber as a reinforcement for polyester or epoxy resins in resin transfer technologies. It has not, however, found general acceptance in this reinforcement field, partly because it provides lower impact strength than glass does and partly because the economic advantages are not sufficiently attractive. Jute and kenaf have found success as reinforcement fillers in thermoplastic composites, which will be covered in another section in this chapter.

11. CHEMICAL MODIFICATION FOR PROPERTY IMPROVEMENT

The performance of any lignocellulosic fiber composite is restricted by the properties of the fiber itself. Agro-based composites will change dimensions with changes in moisture content, will burn, and will be degraded by organisms and ultraviolet radi-

tion. If these negative properties of the natural fiber can be improved, all types of agro-based composites could have greatly improved performance. To understand how agro-based fiber can be used in property-enhanced applications, it is important to understand the properties of the components of the cell wall and their contributions to fiber properties.

All agro-based fibers are three-dimensional, polymeric composites made up primarily of cellulose, hemicelluloses, lignin, and small amounts of extractives and ash. The cell wall polymers and their matrix make up the cell wall and in general are responsible for the physical and chemical properties of the jute and kenaf fiber. Properties such as dimensional instability, flammability, biodegradability, and degradation caused by acids, bases, and ultraviolet radiation are a result of the environment trying to convert the natural composites back into their basic building blocks (carbon dioxide and water).

Agro-based fibers change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding. The hemicelluloses are mainly responsible for moisture sorption, but the accessible cellulose, noncrystalline cellulose, lignin, and surface of crystalline cellulose also play major roles. Moisture swells the cell wall, and the fiber expands until the cell wall is saturated with water. Beyond this saturation point, moisture exists as free water in the void structure and does not contribute to further expansion. This process is reversible, and the fiber shrinks as it loses moisture.

Agro-based fibers are degraded biologically because organisms recognize the carbohydrate polymers (mainly by hemicelluloses) in the cell wall and have very specific enzyme systems capable of hydrolyzing these polymers into digestible units. Biodegradation of the high-molecular-weight cellulose weakens the fiber cell wall because crystalline cellulose is primarily responsible for the strength of the cell wall. Strength is lost as the cellulose polymer undergoes degradation through oxidation, hydrolysis, and dehydration reactions. The same types of reactions take place in the presence of acids and bases.

Agro-based fibers exposed outdoors to ultraviolet light undergo photochemical degradation. This degradation takes place primarily in the lignin component, which is responsible for the characteristic color changes. The lignin acts as an adhesive in the cell walls, holding the cellulose fibers together. The surface becomes richer in cellulose content as the lignin degrades. In comparison to lignin, cellulose is much less susceptible to ultraviolet light degradation. After the lignin has been degraded, the poorly bonded carbohydrate-rich fibers erode easily from the surface, which exposes new lignin to further degradative reactions. In time, this "weathering" process causes the surface of the composite to become rough and can account for a significant loss in surface fibers.

Agro-based fibers burn because the cell wall polymers undergo pyrolysis reactions with increasing temperature to give off volatile, flammable gases. The hemicellulose and cellulose polymers are degraded by heat much before the lignin is. The lignin component contributes to char formation, and the charred layer helps insulate the composite from further thermal degradation.

Because the properties of the agro-based fiber result from the chemistry of the cell wall components, the basic properties of a fiber can be changed by modifying the basic chemistry of the cell wall polymers.

Dimensional stability can be greatly improved by bulking the fiber cell wall either with simple bonded chemicals or by impregnation with water-soluble polymers. For example, acetylation of the cell wall polymers using acetic anhydride produces a fiber composite with greatly improved dimensional stability and biological resistance. The same level of stabilization can also be achieved by using water-soluble phenol formaldehyde polymers followed by curing.

Biological resistance of fiber-based materials can be improved by several methods. Bonding chemicals to the cell wall polymers increases resistance due to the lowering of the equilibrium moisture content below the point needed for microorganism attack and by changing the conformation and configuration requirements of the enzyme-substrate reactions. Toxic chemicals can also be added to the composite to stop biological attack. This is the basis for the wood-preservation industry.

Resistance to ultraviolet radiation can be improved by bonding chemicals to the cell wall polymers, which reduces lignin degradation, or by adding polymers to the cell matrix to help hold the degraded fiber structure together so that water-leaching of the undegraded carbohydrate polymers cannot occur. Fire retardants can be bonded to the fiber cell wall to greatly improve the fire performance. Soluble inorganic salts or polymers containing nitrogen and phosphorus can also be used. These chemicals are the basis of the fire-retardant wood-treating industry.

The strength properties of fiber-based composites can be greatly improved in several ways. The finished composites can be impregnated with a monomer and polymerized in situ or impregnated with a preformed polymer. In most cases, the polymer does not enter the cell wall and is located in the cell lumen. By using this technology, mechanical properties can be greatly enhanced. For example, composites impregnated with acrylates, methacrylate, epoxy, or melamine monomers, and polymerized to weight-gain levels of 60% to 100%, show increases (compared to untreated controls) in density of 60% to 150%, compression strength of 60% to 250%, and tangential hardness of 120% to 400%. Static bending tests show increases in modulus of elasticity of 25%, modulus of rupture of 80%, fiber stress at proportional limit of 80%, work to proportional limit of 150%, and work to maximum load of 80%, and at the same time a decrease in permeability of 200% to 1200%.

Many chemical-reaction systems have been published for the modification of agrofibers. These chemicals include ketene, phthalic, succinic, malic, propionic and butyric anhydride, acid chlorides, carboxylic acids, many different types of isocyanates, formaldehyde, acetaldehyde, difunctional aldehydes, chloral, phthalaldehydic acid, dimethyl sulfate, alkyl chlorides, β -propiolactone, acrylonitrile, ethylene, propylene, butylene oxide, and difunctional epoxides [27, 28].

11.1 Acetylation

By far, the most research has been done on the reaction of acetic anhydride with cell wall polymer hydroxyl groups to give an acetylated fiber. Both jute and kenaf have been reacted with acetic anhydride [29-33]. Without a strong catalyst, acetylation using acetic anhydride alone levels off at approximately 20 weight percent gain (WPG). The equilibrium moisture content (EMC) and thickness swelling (TS) at three relative humidities for fiberboards made from these fibers is shown in Table 7.5.

Table 7.5 Equilibrium Moisture Content (EMC) and Thickness Swelling (TS) of Fiberboards Made from Control and Acetylated Fiber

Fiber	Weight percent gain	EMC and TS at 27°C					
		30% RH		65% RH		90% RH	
		TS	EMC	TS	EMC	TS	EMC
Kenaf	0	3.0	4.8	9.6	10.5	33.0	26.7
	18.4	0.8	2.6	2.4	5.8	10.0	11.3
Jute	0	3.7	5.8	8.6	9.3	17.4	18.3
	16.2	0.6	2.0	1.7	4.1	7.3	7.8

The rate and extent of thickness swelling in liquid water of fiberboards made from control and acetylated fiber is shown in Table 7.6. Both the rate and extent of swelling are greatly reduced as a result of acetylation. At the end of 5 days of water-soaking, control boards swelled 45%, whereas boards made from acetylated fiber swelled 10%. Drying all boards after the water-soaking test shows the amount of irreversible swelling that has resulted from water-swelling. Compared with boards made from acetylated fiber, control boards show a greater degree of irreversible swelling.

Chemically modified jute and kenaf cloth have been tested against brown-, white-, and soft-rot fungi and tunneling bacteria in a fungal cellar (Table 7.7). Control cloth was destroyed in less than 6 months, while cloth made from acetylated fiber above 16 WPG showed no attack after 1 year. [34].

The modulus of rupture (MOR), modulus of elasticity (MOE) in bending and tensile strength (TS) parallel to the board surface are shown in Table 7.8 for fiberboards

Table 7.6 Rate and Extent of TS in Liquid Water of Kenaf Fiberboards Made from Control and Acetylated Fiber and a Phenolic Resin*

Fiber	←—minutes—→					%	←—hours—→			
	15	30	45	1	2		3	4	5	
Control	15.5	17.1	21.1	22.6	24.7	26.8	31.1	32.6		
18.4 WPG	6.7	6.8	6.8	7.0	7.0	7.0	8.0	8.1		
Fiber	←—Days—→					%	Oven drying	Weight loss after test		
	1	2	3	4	5					
Control	37.7	41.5	42.6	43.5	44.5	19.0	2.0			
18.4 WPG	8.5	8.5	8.7	8.8	9.0	0.7	2.8			

*Resin content of boards: 8%.

Table 7.7 Fungal Cellar Tests of Jute Cloth Made from Control and Acetylated Fiber*

Weight percent gain	Rating at intervals (months) [†]							
	2	3	4	5	6	12	24	36
0	2	3	3	3	4	—	—	—
7.4	0	1	1	2	3	4	—	—
11.5	0	0	0	1	2	3	4	—
13.3	0	0	0	0	0	1	2	3
16.8	0	0	0	0	0	0	0	0
18.3	0	0	0	0	0	0	0	0

*Nonsterile soil containing brown-, white-, and soft-rot fungi and tunneling bacteria.

[†]Rating system: 0 = no attack; 1 = slight attack; 2 = moderate attack; 3 = heavy attack; 4 = destroyed.

Source: Ref. 34.

made from control and acetylated kenaf fiber. Acetylation results in a small decrease in MOR but about equal values in MOE and TS. All strength values given in Table 7,8 are above the minimum standard as given by the American Hardboard Association [35]. The small decrease in some strength properties resulting from acetylation may be attributed to the hydrophobic nature of the acetylated fiber, which may not allow the water soluble phenolic or isocyanate resins to penetrate into the flake. The adhesives used in these tests have also been developed for unmodified lignocellulosics. Different types of adhesives may be needed in chemically modified boards [36].

11.2 Cyanoethylation

Jute can be made to react with acrylonitrile in the presence of alkali under conditions that do not reduce the tensile strength of the fibers to any important extent. The properties of cyanoethylated cotton have been known for some time [371, and this particular

Table 7.8 Modulus of Rupture (MOR), Modulus of Elasticity (MOE), and Tensile Strength (TS) Parallel to the Board Surface of Fiberboards Made from Control or Acetylated Kenaf Fiber and 8% Phenolic Resin

Board	MOR <-MPa->	MOE <-GPa->	TS <-MPa->
Kenaf			
Control	47.1	4.6	31.0
18.4 WPG	38.6	5.1	27.1
ANSI Standard	31.0	—	10.3

chemical modification is claimed to provide increased stability against degradation by acids and heat. Cotton containing more than 3% nitrogen is also said to show high resistance to microbiological deterioration [38].

Experiments with jute yarn at the British Jute Trade Research Association, have shown that, whereas untreated yarn subjected to hydrolysis with 0.2 N sulphuric acid at 100° C for 60 min retained only 20% of its initial strength, a yarn cyanoethylated to 4.6% nitrogen content retained 80% of its strength under similar conditions.

Jute yarns cyanoethylated to different extents showed increasing resistance to degradation by heating; whereas untreated yarn heated at 150° C for 24 hr retained only 55% of its initial strength, similar yarn with a nitrogen content of 4.9% retained 90%.

Resistance to rotting was also examined by incubation of yarns under degrading conditions which caused complete breakdown of strength after 2 weeks. Cyanoethylation up to 1.5% nitrogen resulted in little improvement; but for 2.8% nitrogen and over, even 16 weeks of incubation reduced strength by only 10%. By comparison, yarns undergoing copper naphthenate treatment with 1.2% copper retained only 30% of strength under similar conditions of exposure. Thus, provided the nitrogen content approaches about 3%, cyanoethylation gives most effective protection against rotting, just as is said to be the case for cotton [38].

12. PHOTOCHEMICAL AND THERMAL DEGRADATION

All cellulose-containing fibers lose strength on prolonged exposure to sunlight. This effect is attributable mainly to the ultraviolet component of the radiation, and its scale is such that in cotton, for example, about 900 hr exposure will reduce strength to 50% of the initial value. In jute, however, a similar strength-reduction occurs after only about 350 hr exposure, and so, although these exposure times are not precise, it is clear that jute loses strength at more than twice the rate for cotton.

There will be a loss in strength in both fibers due to primary bond breakages in the cellulose constituent; but in seeking an explanation for the difference in behavior the important question is whether it arises entirely from a greater rate of bond breakage in jute than in cotton, or whether the cohesion between the ultimate cells in jute is also reduced as a result of changes in the middle lamella.

The rate of breakage of cellulose bonds in cotton is readily found from the changes in the degree of polymerization (DP) as exposure continues, using the cuprammonium fluidity as a measure of DP. In jute, however, this method is not always satisfactory, because it is difficult to achieve complete dissolution of the cellulose component in cuprammonium hydroxide due to interference from the lignin in the fiber. Moreover, preliminary removal of lignin is not advisable, as whatever process is used is always liable to cause some degradation of the cellulose.

Nitration techniques that do not degrade the cellulose component have been used successfully to determine the DP of wood cellulose [39]; similar methods are equally satisfactory for jute or other lignified materials [40 and 41]. In one study, the nitrated lignin and hemicellulose components were first removed by solvent extraction and fractional precipitation, and the DP of the residual cellulose nitrate was then determined from viscosity measurements in acetone solution [42]. The viscosities have to be referred to a standard rate of shear, and the whole procedure is rather lengthy; but the results showed that, after the same exposure conditions, jute and cotton had similar DPs

within experimental limits of error. Moreover, a plot of $1/DP$ against time of exposure in standard sun hours was linear, suggesting that the kinetic equation for random breakdown of a polymer chain $2/(DP)_t - 2/(DP)_0 = kt$ applies in this case. $(DP)_0$ and $(DP)_t$ are the DPs measured before exposure and after exposure for time t ; k is a constant representing the rate of bond breakage.

Exposure to sunlight for periods up to 600 hr gave values of k equal to 15.4×10^{-7} and 13.3×10^{-7} for jute and cotton, respectively, in units of reciprocal DP per hour exposure. Exposure to artificial sources of UV light, such as a mercury arc lamp or a xenon arc lamp, gave lower values of k than those for sunlight exposure, but, again, the effects on jute and cotton were similar. With the mercury lamp, the k values were 8.0×10^{-7} and 8.9×10^{-7} units for jute and cotton, whereas the xenon lamp gave 5.9 and 6.4×10^{-7} units, respectively, for jute and cotton.

The rate of photochemical breakdown of cellulose thus appears largely independent of whether or not lignin is present; contrary to views that have been expressed in the past, lignin is not acting as a photosensitizer for the breakdown. The greater loss in strength of jute compared with cotton must therefore be related to photochemical changes taking place in the middle lamella, which reduce the cohesion between ultimate cells.

Cellulose-containing fibers also lose strength on prolonged exposure to elevated temperatures, and cotton and jute show only minor differences in strength losses under similar heating conditions. At 140°C , both fibers have lost 50% of strength after 80-85 h.r exposure; at 160°C , only about 10 hr exposure is required for the same fall in strength. Thus, although the temperature is a major factor determining the rate of loss in strength, cotton and jute behave similarly, and there is no suggestion that the cohesion of the middle lamella is changed by exposure to heat.

Measurement of the change in DP in heating presents difficulties, as the cellulose nitrate now becomes insoluble in acetone or other solvents. This may be due to cross-linking between reactive groups produced in the cellulose molecules by the thermal exposure. In any case, however, it appears that the chemical changes taking place in thermal degradation are different from those occurring in light-induced degradation.

The nitration techniques used in the measurement of the DP of α -cellulose merit further discussion, particularly with respect to the effect of time of nitration on the cellulosic constituents of the fiber. Nitration of lignin results in products that are soluble either in the nitrating acids or in methanol. By a suitable extraction procedure, the lignin component of the fiber can be completely removed.

After removal of lignin, the nitrated cellulosic products can be separated into three fractions, which will be designated A, B, and C, and of which fraction A is insoluble in acetone; fraction B is soluble in acetone but insoluble in water; and fraction C is soluble in both acetone and water. Analysis shows the acetone-soluble fractions B and C to consist of nitrated α -cellulose of DP about 4450, and nitrated hemicellulose, respectively. Both these products are also to be found in the acetone-insoluble fraction A.

The amount of α -cellulose that is released as the acetone-soluble fraction B increases as the time of nitration is increased. Although small at first, it finally reaches the analytical value of about 60% of the whole fiber. The time required for the analytical value to be reached is temperature-dependent; and although Lewin and Epstein [41]

report that at 3°C over 24 hr nitration is required, they point out that Timell [40] obtained a similar result in only 1 hr at 17° C.

As the acetone-soluble fraction B increases, so the acetone-insoluble fraction A decreases. Interpolation in Lewin and Epstein's results suggests that the two fractions become equal after 11 or 12 hr nitration, and that at this point of equality their value is about 50% of the maximum value achieved by fraction B, namely the analytical value.

Lewin and Epstein consider this pattern of behavior to indicate the presence in jute of chemical linkages between α -cellulose and hemicellulose that hold these components together in fraction A and render the complex insoluble in acetone. The release of increasing amounts of acetone-soluble nitrated α -cellulose in fraction B then arises from breakage of the links by the nitrating acids, with more breakages occurring as the time of nitration is increased.

13. MOISTURE EFFECTS

The equilibrium moisture held by jute when exposed to atmospheres of different relative humidity (RH) shows appreciable hysteresis according to whether absorption from low humidities or resorption from high humidities is concerned. Thus, at 65% RH and 20°C, the equilibrium moisture regain is about 12.5% for absorption by dry fiber and 14.6% for resorption of wet fiber; whereas exposure to 100% RH gives an equilibrium regain of 34-35%. These are average values, and different samples of fiber may show minor differences. It will be noted that, at 65% RH, the equilibrium regain of jute is about 6% higher than that of cotton.

Jute swells in water to the extent of about 22%, a value similar to that of cotton, despite the greater proportion of noncrystalline material in jute. Delignification has a pronounced effect, and it is reported that when the lignin content has been reduced to 0.78% the swelling may reach almost 40% [43].

Apart from swelling, delignification also affects the equilibrium regain of jute fiber. Kabir et al. have shown that when jute fiber is delignified by 10%, using a chlorite treatment followed by sodium bisulfite solution extraction, the absorption and resorption regains at 65% RH are both increased by about 1% [44].

14. FASTNESS TO LIGHT

14.1 Undyed Jute

A major practical difficulty affecting the performance of dyed or bleached jute materials is the change in color that occurs when jute fiber is exposed to sunlight. In the UV region of the spectrum, exposure to light of wavelengths between about 3000 and 3600 Å results in yellowing of the fiber, whereas exposure to wavelengths between about 3800 and 4000 Å, on the fringe of the visible spectrum, has a bleaching effect. The final color is the result of the two processes; in general, the initial color change is an obvious yellowing, or darkening, of the fiber, but, on longer exposure, this color slowly gets lighter and less intense.

Bleaching before exposure generally accentuates the discoloration of the fiber, compared with the color of unbleached jute, although part of this is due to the heightened

contrast between the nearly white bleached fiber and the exposed fiber. The onset of yellowing varies considerably with the use of different bleaches. Alkaline or neutral hypochlorite, a cheap bleaching medium, gives a product with a rather rapid yellowing tendency, whereas alkaline hydrogen peroxide gives a good white color and a less marked yellowing. Sodium chlorite, applied under acid conditions, results in the least yellowing tendency, but care must be taken that, in obtaining the best conditions to prevent yellowing, no drastic loss of strength takes place.

A bleaching process developed in the United States and patented for jute by Fabric Research Laboratories involves treatment with hydrogen peroxide and acid permanganate and gives a rather better resistance to yellowing than does chlorite bleaching. Treatment with acid permanganate alone leaves the natural color of the jute almost unchanged but also provides a higher resistance to yellowing. This improvement probably represents a true reduction in yellowing, although that may be due in part to the smaller contrast between the original bleached color and the exposed color than that which results from the use of whiter bleaches.

Improvements in the stability of jute to light exposure result from acetylation or methylation. Treatment with acetic anhydride in xylene solution, for example, combined with a reduction process using sodium borohydride, may confer virtually complete stability, whereas methylation with diazomethane confers a marked improvement without preventing yellowing entirely.

Color changes in jute are associated with the lignin content of the fiber, the isolated α -cellulose and hemicellulose fractions being unaffected by exposure to UV light of the correct wavelength. The importance of lignin has also been demonstrated by irradiating cellulosic fibers of different lignin contents. For a series of fibers covering lignin content ranges of 0% (cotton) to 13% (*Phormium tenax*), it was evident that the intensity of yellowing became more pronounced as the lignin content increased [45].

The formation of colored products from irradiated lignin involves complex reaction chains that are difficult to elucidate fully. It is probable that *ortho*-quinone groups are responsible for the yellow color, formed from *ortho*-phenol groups as intermediates. Acetylation blocks both phenolic and aliphatic hydroxyl groups and prevents the objectionable reactions from taking place. The less effective methylation, however, blocks only the phenolic hydroxyl groups.

14.2 Dyed Jute

Jute can be dyed with a wide range of dyestuffs. All those generally used for cellulosic fibers, such as direct, vat, and reactive dyes, can be used successfully on jute, but, in addition, jute has a strong affinity for both acid dyes and basic dyes which normally have little or no dyeing capacity for cotton or rayon, although they are used extensively for wool.

Many acid and basic dyes give strong, bright colors to jute, but performance is disappointing with regard to color fastness on exposure to sunlight. Although some of the dyes used have intrinsically poor light fastness, it has long been apparent that many acid dyes that give excellent light fastness on wool become fugitive when applied to jute. Yellowing of the jute background causes an apparent change in color of dyed jute; and although poor fastness to light usually means fading of color, any change in color is in fact regarded as a lack of fastness.

Although systematic studies of dyestuffs on jute have not been frequent, the comprehensive studies carried out at the British Jute Trade Research Association merit discussion [45]. In these studies, a number of dyes were taken from each of several different classes and used to dye standard jute fabric, both natural and after bleaching. The fastness to light of these dyed samples was then assessed by exposure to xenon light, alongside a series of light-fastness standards, and the results compared with the known fastness value of the dyestuff on cotton. There are eight standards in all, no. 1 being the most fugitive and no. 8 the most resistant; the experimental conditions for assessment are well standardized [46].

The results indicated that, with vat dyes, accelerated fading of the dyestuff on jute compared with cotton was largely absent, and that the yellowing was the main factor on which the apparent light-fastness depended. With acid and basic dyes, however, accelerated fading appeared to be the predominant effect, although the balance with yellowing varied with both color and chemical structure of the dye. A number of acid dyestuffs known to give fastness ratings of 6 or more on wool were rated only 3-4 on jute, with loss of dye color the main cause, whereas a group of basic dyes giving ratings of 6 or over on acrylic fibers were reduced to ratings of 2-3 on jute. Again, although yellowing was evident, accelerated fading was the principal cause.

A selection of 200 direct dyes, representative of the range of chemical types in this class and all having fastness ratings of 4 or over on cotton, were used for test-dyeings on jute samples which were natural, chlorite-bleached, and peroxide-bleached. On cotton, 66% of dyes had a fastness grade of 6 or over, but on natural jute only 17% retained this grade, the number falling further to 12% on chlorite-bleached jute and 5% on peroxide-bleached. The average grading was 5.7 on cotton and 4.8 on natural jute, 4.7 on chlorite-bleached, and 4.6 on peroxide-bleached. On average, the grade on jute was about 1.0 lower than on cotton.

The drop in grade, however, was far from regular for different dyes, and the balance between yellowing and accelerated fading did not follow a predictable pattern. Dye color played an important role: whereas yellow dyes dropped only about 0.5 grade on jute against cotton, for blue colors the average drop was 1.4 grades.

In the case of reactive dyes, test dyeings were done on natural and chlorite-bleached jute. Of the dyes used, 55% were graded 5-6 and over 6; but on jute, only 2% were retained in this category. The mean grade for cotton was 5.3, compared with 4.2 on natural jute and 4.1 on chlorite-bleached.

In general, therefore, few dyestuffs will retain the same light-fastness on jute, natural or bleached, as they will on cotton. Reduction of the underlying yellowing will be helpful in many cases, but there will remain examples of accelerated fading on jute. Acetylation and methylation can improve the fastness considerably by preventing the background yellowing, and possibly this prevention may also affect the accelerated fading. But these treatments are expensive and not simple to use, and alternative methods of obtaining light-fastness are needed if the standard of jute dyeing is to be raised.

15. WOOLENIZATION

When jute fiber is treated with strong alkali, profound changes occur in its physical structure. Lateral swelling occurs, together with considerable shrinkage in length, as a result of which the fiber is softened to the touch and develops a high degree of crimp

or waviness. The crimp gives a woollike appearance to the fiber, and much attention has been given to assessing the commercial possibilities for this chemical modification.

On stretching the fibers to break, the crimp is straightened and thereby the extensibility of the fiber is increased. The effect is small at alkali concentrations of up to about 10%, but the extensibility increases rapidly at concentrations of 15% and upward and may reach 8 or 9%. At the same time, however, the tensile strength of the fiber decreases with an increased alkali concentration, but it is interesting to note that the product of extensibility and tensile strength, the breaking energy appears to pass through a maximum at 15-20% concentration [47]. This has a beneficial effect on spinning, because the carded fiber has a longer average length than normal, which results in a more uniform yarn.

The rapid change in extensibility in the vicinity of 15% concentration is similar to the effect of slack mercerization on cotton. The nature of the chemical changes occurring in jute on mercerization have been discussed by Lewin [17], especially with regard to the role played by lignin in the fiber structure. The sheathing of the ultimate cells by a lignified membrane affects the free swelling of the cells and produces tension, and the irregular shape of fibers in cross section will lead to folding under tension once the middle lamella material is weakened by the treatment.

The crimp statistics have been studied in detail at the Institute for Fibers and Forest Products Research in Jerusalem, and much information has been brought together by, among others, Lewin et al. [48]. Two parameters are measured to define the crimp, namely the RMS value of the width (D) and the number of crimps per unit length of the stretched fiber (n). As the crimp is three-dimensional, the fiber is rotated during the measurements. Typical values for jute fibers immersed in 12.5% NaOH for 1 hr at a temperature of 2°C are reported to be about 1.6 mm for D , with a standard deviation of 0.55 mm, and about 0.098 mm⁻¹ for n , with a standard deviation of 0.035 mm⁻¹. The extension of the fibers at break are reported to be 15%, relative to the initial length of the crimped fiber under a load of 10 mg; the crimp disappears for loads of about 2000 mg. The energy required to uncrimp the fiber is equivalent to about 3.9 g per 1% of extension.

The above figures refer specifically to an alkali concentration of 12.5%. At concentrations below 6%, no crimp is formed, whereas at 9% alkali, D reaches a maximum value of about 1.9 mm. At concentrations of 15% and upward, D takes a reasonably constant value of about 1.35 mm. The value of n , however, is scarcely affected by changes in alkali concentration.

It is reported that the optimum temperature for crimp formation is about 2°C, and that, at higher temperature, the crimp parameters are reduced, becoming zero at 40°C. An immersion time of at least 0.5 hr is necessary for the crimp to be formed.

Banbaji [49] has examined the tensile properties of jute fibers before and after alkali treatment and has shown that the tenacity decreases with increasing the alkali concentration, an initial value of 3.6 g/den falling to 2.5 g/den at 9% alkali and to 1.5 g/den at 24% alkali (at 2°C and 1 hr immersion). The extension at break, referring to the fiber length before immersion, increases from 1.2% without alkali treatment to 3.6% at 9% alkali, and then falls slightly to 2.4% at 24% alkali.

The tenacity changes are no doubt linked with the losses in weight that occur with alkali treatment, but there may be more profound changes taking place internally within the ultimate cells. Such changes are at present imperfectly understood, but if useful

commercial developments are to be made, further investigation of structural changes appears essential. Moreover, the crimp is a “once-only” effect; to be really useful, some small degree of elasticity must be introduced into the fiber.

The stability of the crimp is poor. Once the fiber has been straightened under tension, there is no tendency to revert to the crimped state when the tension is removed. That is to say, the woolenizing treatment does not confer elasticity on the fiber.

Under mercerizing conditions, the fibers lose considerable weight, 15% or more, and give the appearance of being opened up. It is commonly said that there is a considerable reduction in diameter, which implies a lower linear density and, hence, more regular yarns. But just as in natural jute, there appears to be a limit below which the diameter does not fall.

The physical effects of the mercerizing process are different when the jute material is kept under tension, instead of being slack. Experiments reported from the Bangladesh Jute Research Institute with treated jute yarns [50] show that the shrinkage is greatly reduced by tension, falling from 11–12% when slack to 1.5–2.5% under 3 kg tension. The loss in weight of 12–13% when slack was reduced by a few percent under 3 kg tension. The effect of temperature change from 30°C to 60°C was small in all cases.

The appearance and handle of jute fabrics is much improved by the woolenizing process, and bleached and dyed fabrics appear to have commercial possibilities. The problem is the cost of treatment, but to achieve similar effects more cheaply may require a deeper knowledge of the internal changes that take place in the fiber.

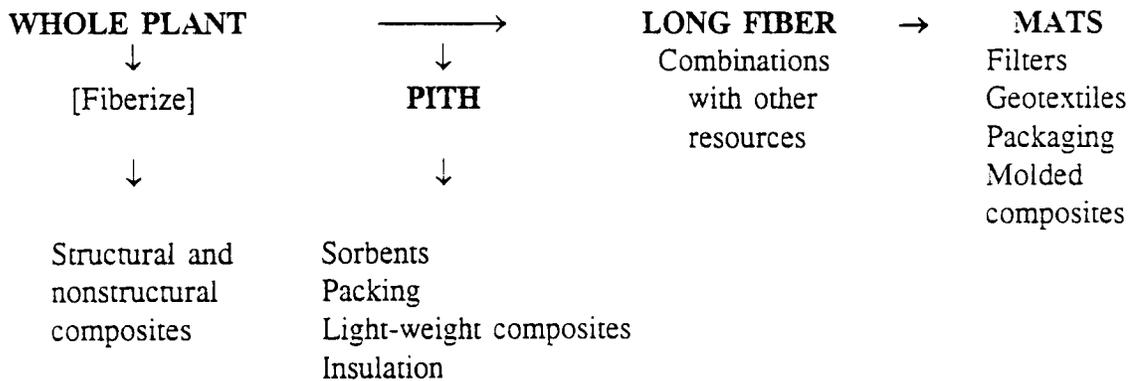
16. APPLICATIONS AND MARKETS

The large historic markets for jute in sacking, carpet backing, cordage, and textiles have decreased over the years as jute has been replaced by synthetics. Fiber from jute and kenaf can be used in the handicraft industries, to make textiles, to make paper products, or to produce a wide variety of composites. A great deal of research is presently going on in each of these fields; however, the largest potential markets are in composite products. These composites range from value-added speciality products to very large-volume commercial materials. These markets are potentially larger than the past markets for jute and kenaf and could lead to new dynamic uses for this and other natural fibers.

16.1 Composites

A composite is any combination of two or more resources held together by some type of mastic or matrix. The mastic or matrix can be as simple as physical entanglement of fibers to as complex as systems based on thermosetting or thermoplastic-polymer production. The scheme shown below gives possible processing pathways that lead to the composite products that can come from each fraction of the plant. The entire plant (leaves, stock, pith, and roots) can be used directly to produce structural and non-structural composites. By using the entire plant, processes such as retting, fiber separation, fraction purification, etc. can be eliminated, which increases the total yield of plant material and reduces the costs associated with fraction isolation. This also gives farmers a different option for their crop utilization—that is, taking the entire plant to

Another option is to separate the higher-value long fiber from the other types of shorter fiber and use it in combination with other materials to make value-added structural composites. When the long fiber is separated, the byproduct is a large amount of short fiber and pith material that can be used for such products as sorbents, packing, light-weight composites, and insulation. By utilizing the byproduct of the long-fiber isolation process, the overall cost of long-fiber utilization is reduced.



The isolated long fiber can then be used to make mats which have value-added applications in filters, geotextiles, packaging, molded composites, and structural and non-structural composites.

Composites can be classified in many ways: by their densities, by their uses, by their manufacturing methods, or other systems. For this report, they will be classified by their uses. Eight different use classes will be covered: geotextiles, filters, sorbents, structural composites, non-structural composites, molded products, packaging, and combinations with other materials. There is some overlap between these areas. For example, once a fiber web has been made, it can be directly applied as a geotextile, filter, or sorbent, or can be further processed into a structural or non-structural composite, molded product, used in packaging, or combined with other resources. Within each composite made there are opportunities to improve the performance of that composite by improving the performance of the fiber going into the composite.

Geotextiles

The long bast or leaf fibers can be formed into flexible fiber mats, which can be made by physical entanglement, nonwoven needling, or thermoplastic fiber melt matrix technologies. The two most common types are carded and needle-punched mats. In carding, the fibers are combed, mixed, and physically entangled into a felted mat. These mats are usually of high density but can be made at almost any density. A needle-punched mat is produced in a machine which passes a randomly formed machine-made web through a needle board that produces a mat in which the fibers are mechanically entangled. The density of this type of mat can be controlled by controlling the amount of fiber going through the needle board or by overlapping needled mats to give the desired density.

Numerous articles and technical papers have been written, and several patents have been issued, on both the manufacture and the uses of nonwoven fiber mats containing combinations of textile and lignocellulosic fibers or lignocellulosic fibers alone. This technology is particularly well known in the consumer products industry. Medium- to

high-density fiber mats have several uses. One is as a geotextile. Geotextiles derive their name from *geo* and *textile* and, therefore, mean fabrics in associated with the earth.

Geotextiles have a large variety of uses. They can be used for mulch around newly planted seedlings. The mats provide the benefits of natural mulch; in addition, controlled-release fertilizers, repellents, insecticides, and herbicides can be added to the mats as needed. The addition of such chemicals could be based on silvicultural prescriptions to ensure seedling survival and early development on planting sites where severe nutritional deficiencies, animal damage, insect attack, and weed problems are anticipated. Research results on the combination of mulch and pesticides in agronomic crops have been promising.

Medium-density fiber mats can also be used to replace dirt or sod for grass seeding around new homesites or along highly embankments. Grass or other type of seed can be incorporated in the fiber mat. Fiber mats have good moisture retention and promote seed germination. Low- and medium-density fiber mats can be used for soil stabilization around new or existing construction sites. Steep slopes without roots to hold the soil erode and top soil is lost.

Medium- and high-density fiber mats can also be used below ground in road- and other types of construction as natural separators between different materials in the layering of back fill. It is important to restrain slippage and mixing of the different layers by placing separators between the various layers. Jute and kenaf geotextiles have been shown to work very well in these applications, but the potential exists for any of the long agro-based fibers.

Filters

Medium- and high-density fiber mats can be used for air filters. The density of the mats can be varied, depending on the size and quantity of material that needs to be filtered and the volume of air required to pass through the filter per unit of time. Air filters can be made to remove particulate and/or can be impregnated or reacted with various chemicals as air fresheners or cleansers.

Medium- to high-density mats can also be used as filtering aids to take particulates out of waste water and drinking water or solvents.

Sorbents

In several cities in the United States, tests are presently under way to use agro-based sorbents to remove heavy metals, pesticides, and oil from rain water runoff. Medium- and high-density mats can also be used for oil-spill clean-up pillows. It has been shown that agro-based core material from kenaf, when saturated with water, will preferentially sorb oil out of sea water. There are many other potential sorbent applications of agro-fiber core resources, such as the removal of dyes, trace chemicals in solvents and in the purification of solvents,

It is also possible to use core materials as sorbents in cleaning aids such as floor sweep. While this is not a composite as such, it does represent another way agro-based resources can be used as sorbents.

Structural Composites

A structural composite is defined as one that is required to carry a load in use. In the housing industry, for example, structural composites are used in load-bearing walls,

roof systems, subflooring, stairs, framing components, furniture, etc. In most if not all cases, performance requirements for these composites are spelled out in codes and/or in specifications set forth by local or national agencies.

Structural composites can range widely in performance from the high-performance materials used in the aerospace industry down to wood-based composites which have lower performance requirements. Among the wood-based composites, performance varies, from that of multilayered plywood and laminated lumber to low-cost particle-board. Structural wood-based composites, intended for indoor use, are usually made with a low-cost adhesive which is not stable to moisture, while exterior-grade composites are made with a thermosetting resin that is higher in cost but stable to moisture.

Performance can be improved in wood-based as well as agro-based composites by using chemical modification techniques, fire-retardant and/or decay-control chemicals, etc.

Nonstructural Composites

As the name implies, nonstructural composites are not intended to carry a load in use. These can be made from a variety of materials such as thermoplastics, textiles, and wood particles, and are used for such products as doors, windows, furniture, gaskets, ceiling tiles, automotive interior parts, molding, etc. These are generally lower in cost than structural composites and are subject to codes and specifications associated with them.

Molded Products

The present wood-based-composite industry mainly produces two-dimensional (flat) sheet products. In some cases, these flat sheets are cut into pieces and glued/fastened together to make shaped products such as drawers, boxes, and packaging. Flat sheet wood-fiber composite products are made by making a gravity-formed mat of fibers with an adhesive and then pressing. If the final shape can be produced during the pressing step, then the secondary manufacturing profits can be realized by the primary board producer. Instead of making low-cost flat-sheet-type composites, it is possible to make complexly shaped composites directly using the long bast fiber.

In this technology, fiber mats are similar to the ones described for use as geotextiles except, during mat formation, an adhesive is added by dipping or spraying the fiber before mat formation, or it is added as a powder during mat formation. The mat is then shaped and densified by a thermoforming step. Within certain limits, any size, shape, thickness, and density is possible.

These molded composites can be used for structural or non-structural applications as well as for packaging, and can be combined with other materials to form new classes of composites. This technology will be described later.

Packaging

“Gunny” bags made from jute have been used as sacking for products such as coffee, cocoa, nuts, cereals, dried fruits, and vegetables for many years. While there are still many applications for long fiber for sacking, most of the commodity goods are now shipped in containers that are not made of agro-fibers. But there is no reason why they cannot be. Medium- and high-density agro-based fiber composites can be used for small containers, for example, in the tea industry and for large sea-going containers for

commodity goods. These composites can be shaped to suit the product by using the molding technology described previously, or made into low-cost, flat sheets to be made into containers.

Agro-based fiber composites can also be used in returnable, reusable containers. These containers can range from simple crease-fold types to more solid, even nestable, types. Long agro-fiber fabric and mats can be overlaid with thermoplastic films such as polyethylene or polypropylene to be used to package such products as concrete, foods, chemicals, and fertilizer. Corrosive chemicals require the plastic film to make them more water-resistant and to reduce degradation of the agro-based fiber. There are also many applications for agro-based fiber as paper sheet products for packaging. These vary from simple paper wrappers to corrugated, multifolded, multilayered packaging.

Combinations with Other Resources

It is possible to make completely new types of composites by combining different resources. It is possible to combine, blend, or alloy leaf, bast and/or stick fiber with other materials such as glass, metals, plastics, and synthetics to produce new classes of materials. The objective is to combine two or more materials in such a way that a synergism between the components results in a new material that is much better than its individual components.

Composites of agro-based fiber and glass fiber can be made by using the glass as a surface material or combined, as a fiber, with lignocellulosic fiber. Composites of this type can have a very high stiffness-to-weight ratio. The long bast fibers can also be used in place of glass fiber in resin injection molding (RIM) or used to replace, or in combination with, glass fiber in resin-transfer-molding (RTM) technologies. Problems of dimensional stability and compatibility with the resin must be addressed, but such composites could also lead to new markets for property-enhanced agro-based materials.

Metal films can be overlaid onto smooth, dimensionally stabilized fiber composite surfaces or applied through cold-plasma technology to produce durable coatings. Such products could be used in exterior construction to replace all-aluminum or vinyl siding, markets where agro-based resources have lost market share.

Metal fibers can also be combined with stabilized fiber in a matrix configuration in the same way metal fibers are added to rubber to produce wear-resistant aircraft tires. A metal matrix offers excellent temperature-resistance and improved strength properties, and the ductility of the metal lends toughness to the resulting composite. Application for metal matrix composites could be in the cooler parts of the skin of ultrahigh-speed aircraft. Technology also exists for making molded products: perforated metal plates are embedded in a phenolic-coated fiber mat, which is then pressed into various shaped sections.

Bast or leaf fiber can also be combined in an inorganic matrix. Such composites are dimensionally and thermally stable, and they can be used as substitutes for asbestos composites. Inorganic bonded bast-fiber composites can also be made with variable densities that can be used for structural applications.

One of the biggest new areas of research in the value-added area is in combining natural fibers with thermoplastics. Prices for plastics have risen sharply over the past few years, but adding a natural powder or fiber to plastics reduces cost (and in some

cases increases performance as well). To the agro-based industry, this represents an increased value for the agro-based component.

16.2 Fiber-Thermoplastic Blends

Before 1980, the concepts of blends and alloys were essentially unknown in the plastics industry. Today, there are more than 1000 patents relating to plastic blends and alloys, and it is estimated that one out of every five kilograms of plastic sold in the United States is a blend or alloy [52]. Blends and alloys have revolutionized the plastics industry, as they offer new materials with properties never before available and materials that can be tailored for specific end uses. The jute and kenaf industries have the same opportunity to follow this trend and greatly expand markets for new materials based on blends and alloys with other resources.

Newer materials and composites that have both economic and environmental benefits are being considered for applications in the automotive, building, furniture, and packaging industries. Mineral fillers and fibers are used frequently in the plastics industry to achieve desired properties or to reduce the cost of the finished article. For example, glass fiber is used to improve the stiffness and strength of plastics, although there are several disadvantages associated with its use. Glass-fiber production requires a great deal of energy—processing temperatures can exceed 1200°C. They tend to abrade processing equipment and increase the density of the plastic system. The use of lignocellulosic fibers in thermoplastics has received a lot of interest due to their low densities, low cost, and non-abrasive nature. The inherent polar and hydrophilic nature of the jute and kenaf fibers and the nonpolar characteristics of the polyolefins lead to difficulties in compounding and result in inefficient composites. Proper selection of additives is necessary to improve the interaction and adhesion between the fiber and matrix phases.

Recent research on the use of jute and kenaf fiber suggests that these fibers have the potential for being used as reinforcing fillers in thermoplastics; a brief preliminary account of this potential was published earlier [53]. The use of annual-growth agricultural crop fibers such as kenaf has resulted in significant property advantages as compared to those of typical wood-based fillers/fibers such as wood flour, wood fibers, and recycled newspaper [54-58]. Results indicate that kenaf-fiber-polypropylene (PP) composites have significant advantages over conventional inorganic filled/reinforced PP systems for certain applications. The low cost and densities and the nonabrasive nature of the fibers allow high filling levels and thereby result in significant cost savings. The primary advantages of using these fibers as additives in plastics are: low densities, low cost, nonabrasion, high filling levels, low energy consumption, high specific properties, renewability, wide distribution, biodegradability, and improvement of the rural/agricultural-based economy.

The two main disadvantages of using jute and kenaf fibers in thermoplastics are the high moisture absorption of the fibers and composites [53] and the low processing temperatures permissible. The moisture absorbed by the composite and the corresponding dimensional changes can be reduced dramatically if the fibers are thoroughly encapsulated in the plastic and there is good adhesion between the fiber and the matrix. If necessary, moisture absorption of the fibers can be significantly reduced by acetylation of the hydroxyl groups present in the fiber [59], although at some increase in

cost. The disadvantage of the high moisture absorption of the composite can be minimized by selecting applications where high moisture absorption is not a major drawback. For example, polyamide and its composites absorb large amounts of water, but its applications are such that this deficiency is not of prime importance. The processing temperature of the lignocelulosic fibers in thermoplastics is limited due to potential fiber degradation at higher temperatures. Thus the plastics that can be used with it are limited to those with low melting temperatures. In general, no deterioration of properties due to fiber degradation occurs when processing temperatures are maintained below about 200°C for short periods.

Kenaf bast fibers with a filament length of more than 1 m are common. These filaments consist of discrete individual fibers, generally 2 mm to 6 mm long, which are themselves composites of predominantly cellulose, lignin, and hemicelluloses. Filament and individual fiber properties can vary depending on the source, age, and history of the fiber and the separating techniques that were used. The properties of the fibers are difficult to measure, so we have made no attempt to measure the properties of kenaf.

Kenaf filaments, about 15-20 cm long, a maleic anhydride grafted polypropylene (MAPP used as a coupling agent to improve the compatibility and adhesion between the fibers and matrix), and polypropylene were compounded in a high-intensity kinetic mixer, with the only source of heat being that which was generated through the kinetic energy of rotating blades. The blending was accomplished at 4600 rpm, which resulted in a blade-tip speed of about 30 m/s, and then automatically discharged at 190°C. The mixed blends were then granulated and dried at 105°C for 4 hr. Test specimens were injection-molded at 190°C. Tensile tests were conducted according to ASTM 638-90, Izod impact strength tests according to ASTM D 256-90, and flexural testing using the ASTM 790-90 standard. The cross-head speed during the tension and flexural testing was 5 mm/min. Although all the experiments were designed around the weight percent of kenaf in the composites, fiber volume fractions can be estimated from composite density measurements and the weights of dry kenaf fibers and matrix in the composite. The density of the kenaf in the composite was estimated to be 1.4 g/cc. The results are shown in Table 7.9.

To develop sufficient stress-transfer properties between the matrix and the fiber, two factors need to be considered. First, the MAPP present near the fiber surface should be strongly interacting with the fiber surface through covalent bonding and/or acid-base interactions. This means that sufficient MA groups should be present in the MAPP so that interactions can occur with the -OH groups on the fiber surface. Second, the polymer chains of the MAPP should be long enough to permit entanglements with the PP in the interphase. Polar polymers that can develop hydrogen bonding between chains tend to reach mechanical integrity at lower molecular weights.

A small amount of the MAPP (0.5% by weight) improves the flexural and tensile strength, tensile energy absorption, failure strain and unnotched Izod impact strength. The anhydride groups present in the MAPP can covalently bond to the hydroxyl groups of the fiber surface. Any MA that has been converted to the acid form can interact with the fiber surface through acid-base interactions. The improved interaction and adhesion between the fibers and the matrix leads to better matrix-to-fiber stress transfer. There is little difference in the properties obtained between the 2% and 3% (by weight) MAPP systems. The drop in tensile modulus with the addition of the MAPP is probably due to the molecular morphology of the polymer near the fiber surface or in the

Table 7.9 Comparison of Filled PP Composites

Filler/reinforcement in PP	ASTM standard	None	Kenaf	Jute	Talc	CaCO ₃	Glass	Mica
% filler by weight		0	50	50	40	40	40	40
% filler by volume (estimated)		0	39	39	18	18	19	18
Tensile modulus, GPa	D638	1.7	8.3	7.8	4	3.5	9	7.6
Specific tensile modulus, GPa		1.9	7.8	7.2	3.1	2.8	7.3	6.0
Tensile strength, MPa	D638	33	68	72	35	25	110	39
Specific tensile strength, MPa		37	58	67	28	20	89	31
Elongation at break, %	D638	>>10	2.2	2.3	—	—	2.5	2.3
Flexural strength, MPa	D790	41	91	99	63	48	131	62
Specific flexural strength, MPa		46	85	92	50	38	107	49
Flexural modulus, GPa	D790	1.4	7.8	7.7	4.3	3.1	6.2	6.9
Specific flexural modulus, GPa		1.6	7.3	7.1	3.4	2.5	5.0	5.5
Notched Izod impact- J/m	D256A	24	32	31	32	32	107	27
Specific gravity		0.9	1.07	1.08	1.27	1.25	1.23	1.26
Water absorption %- 24 hr	D570	0.02	1.05	—	0.02	0.02	0.06	0.03
Mold (linear) shrinkage cm/cm		0.028	0.003	—	0.01	0.01	0.004	—

Source: Refs. 61 and 62.

bulk of the plastic phase. Transcrystallization and changes in the apparent modulus of the bulk matrix can result in changes in the contribution of the matrix to the composite modulus and will be discussed later.

There is little change in the notched impact strength with the addition of the MAPP, while the improvement in unnotched impact strength is significant. In the notched test, the predominant mechanism of energy absorption is through crack propagation, as the notch is already present in the sample. Addition of the coupling agent has little effect on the amount of energy absorbed during crack propagation. In the unnotched test, on the other hand, energy absorption is through a combination of crack initiation and propagation. Cracks are initiated at places of high stress concentrations such as the fiber ends, at defects, or at the interface region where the adhesion between the two phases is very poor. The addition of MAPP increases the energy needed to initiate cracks in the system and thereby results in improved unnotched impact-strength values. Entanglement between the PP and MAPP molecules results in improved interphase properties and the strain-to-failure of the composite. There is a plateau after which further addition of coupling agent results in no further increase in ultimate failure strain.

There is little difference in the tensile strength of uncoupled composites compared with that of the unfilled PP, irrespective of the amount of fiber present. This suggests that here is little stress transfer from the matrix to the fibers due to incompatibilities between the different surface properties of the polar fibers and the nonpolar PP. The tensile strengths of the coupled systems increase with the amount of fiber present; strengths of up to 74 MPa were achieved with the higher fiber loading of 60% by weight or about 49% by volume.

As in the case with tensile strength, the flexural strength of the uncoupled composites are approximately equal for all fiber loading levels, although there is a small improvement as compared to the unfilled PP. The high shear mixing using the thermokinetic mixer causes a great deal of fiber attrition. Preliminary measurements of the length of fibers present in the composite after injection molding show that few fibers are longer than 0.2 mm. The strengths obtained in our composites was thus limited by the short fiber lengths. Higher strengths are likely if alternative processing techniques are developed which could reduce the amount of fiber attrition and at the same time achieve good fiber dispersion.

The specific tensile and flexural moduli of the 50%-by-weight kenaf coupled composites were about equivalent to or higher than typical reported values of 40% by weight coupled glass-PP injection-molded composites (see Table 7.9) [60]. The specific flexural moduli of the kenaf composites with fiber contents greater than 40% were extremely high and even stiffer than a 40% mica-PP composite. Table 7.9 shows some typical data of commercially available injection-molded PP composites and the comparison with typical kenaf-PP composites. Data on the talc, mica, calcium carbonate, and glass composites were compiled from the Resins and Compounds, Modern Plastics Encyclopedia [61] and Thermoplastic Molding Compounds, Material Design [62]. The properties of kenaf-based fiber composites have properties superior to those of typical wood-fiber (newsprint)-PP composites. The specific tensile and flexural moduli of a 50% by weight of kenaf-PP composite compares favorably with that of the stiffest of the systems shown, glass-PP and mica-PP.

The failure strain decreases with the addition of the fibers. Addition of a rigid filler/fiber restricts the mobility of the polymer molecules to flow freely past one another

and thereby causes premature failure. With the addition of MAPP, there was a similar trend to that of the uncoupled system, although the drop in failure strain with increasing fiber amounts was not as severe. There is a decrease in the failure strain with increasing amounts of kenaf for a coupled system. The stress-strain curve is not linear, which is due to the plastic deformation of the matrix. The distribution of the fiber lengths in the composite can influence the shape of the stress-strain curve because the load taken up by the fibers decreases as the strain increases; detailed explanations are available elsewhere [63]. The tensile energy absorption, the integrated area under the stress-strain curve up to failure, behaves in roughly the same manner as does the tensile failure strain. The difference between the coupled and uncoupled composites increases with the amount of fibers present, although the drop in energy absorbed for the coupled composites levels off after the addition of about 35 vol% of fiber.

The impact strength of the composite depends on the amount of fiber and the type of testing, i.e., whether the samples were notched or unnotched. In the case of notched samples, the impact strength increases with the amount of fibers added, until a plateau is reached at about 45% fiber weight, irrespective of whether MAPP is used or not. The fibers bridge cracks and increase the resistance of the propagation of the crack. Contribution from fiber pullout is limited, since the aspect ratio of the fibers in the system are well below the estimated critical aspect ratio of about 0.4 mm [64]. In the case of the unnotched impact values of the uncoupled composites, the presence of the fibers decrease the energy absorbed by the specimens. Addition of the fibers creates regions of stress concentrations that require less energy to initiate a crack. Improving the fiber-matrix adhesion through the use of IMAPP increases the resistance-to-crack initiation at the fiber-matrix interface, and the fall in impact strength with the addition of fibers is not as dramatic.

The two main disadvantages of kenaf-PP as compared to glass-PP are the lower impact strength and higher water absorption. The lower notched impact strength can be improved by using impact-modified PP copolymers and flexible maleated copolymers, albeit with some loss in tensile strength and modulus, which will be discussed in a later paper.

Care needs to be taken when using these fibers in applications where water absorption and the dimensional stability of the composites are of critical importance. Judicious use of these fibers will make it possible for agro-based fibers to define their own niche in the plastics industry for the manufacture of low-cost, high-volume composites using commodity plastics.

An interesting point to note is the higher fiber volume fractions of the agro-based composites when compared to those of the inorganic filled systems. This can result in significant material cost savings as the fibers are cheaper than the pure PP resin, and far less expensive than glass fibers. Environmental and energy savings by using an agriculturally grown fiber instead of the high energy-utilizing glass fibers or mined inorganic fillers are benefits that cannot be ignored, although a thorough study needs to be conducted to evaluate these benefits.

16.3 Fiber Matrix Thermoplasticization

There has been much research over the years studying ways to thermoform lignocellulosics. Most of the efforts have concentrated on film formation and thermoplas-

tic composites. The approach most often taken involves the chemical modification of cellulose, lignin, and the hemicelluloses to recrystallize/modify the cellulose and to thermoplasticize the lignin and hemicellulose matrix in order to mold the entire lignocellulosic resource into films or thermoplastic composites [65-70].

Jute and kenaf fibers are composites made up of a rigid polymer (cellulose) in a thermoplastic matrix (lignin and the hemicelluloses). If a nondecrystallizing reaction condition is used, it is possible to modify the lignin and hemicellulose chemically, but not the cellulose. This selective reactivity has been shown to occur if uncatalyzed anhydrides are reacted with wood fiber [71]. The goal is to modify only the matrix of agro-based fibers, allowing thermoplastic flow but keeping the cellulose backbone as a reinforcing filler. This type of composite should have less of the heat-induced deformation (creep) that restricts thermoplastic-based composites from structural uses.

Modification of the kenaf bast fibers using succinic anhydrides (SA) has been carried out performed using either solution reactions with xylene or solid-state reactions using SA in a melt state [72]. Since xylene does not swell the fiber, it is there only as a carrier for the reagent. The rate of reaction is fastest at higher concentration of SA in xylene and at temperatures above 140°C. The rate of reaction in the melt state has not been determined. It has not yet been determined what level of modification is needed to give the desired thermoplasticity, so it is not known what optimum reaction time is needed.

Thermal analysis (DSC) has shown that the first glass transition temperature decreases from 170° C to about 133°C [71]. Samples of reacted fiber were pressed into pellets by a powder-pressing die consisting of a heavy walled steel cylinder with a separate bottom and a ram diameter 10.4 mm). Fiber was placed in the preheated cylinder and then compressed to a pellet thickness of 8.7 mm (target density 1.5 g/cm³, target volume 0.736 cm³) for 10 mimes at 190° C. Scanning electron micrographs (SEM) were then taken of the pressed control and SA-reacted fiber specimens [72]. Figure 7.4 shows the SEM of the hot pressed control and esterified kenaf fiber. The SA fiber is derived from a reaction carried out according to the solid-state reaction method and pressed at 190° C for 10 min. The weight percent gain due to esterification is 50. The control fiber (a) shows little tendency to thermal flow under the pressure of the hot press, whereas the esterified fiber (b) shows thermal flow at this temperature. Views (a), (b), and (d) are taken from the top of the compressed pellet, and (c) is taken from the side of the pellet. The side view, (c), shows a definite layering of the fiber has occurred and view (d) shows that fiber orientation is still evident.

The research done so far in this area shows that kenaf fiber can be reacted with succinic anhydride to give high weight gains of esterification of the cell wall polymers either by solution or solid state chemistry. The esterified fiber shows a reduced transition temperature from about 170°C down to about 135°C regardless of the weight gain obtained. Electron microscopy of hot pressed fiber indicates matrix thermoplasticity with a rigid fiber structure still in existence.

16.4 Fiber-Thermoplastic Alloys

Research to develop jute and kenaf fiber-thermoplastic alloys is based on first thermoplasticizing the fiber matrix as described above and then the grafting of the modified fiber with a reactive thermoplastic. In this type of composite, the thermoplastic is bonded onto the lignocellulosic in such a way that there is only one continuous phase

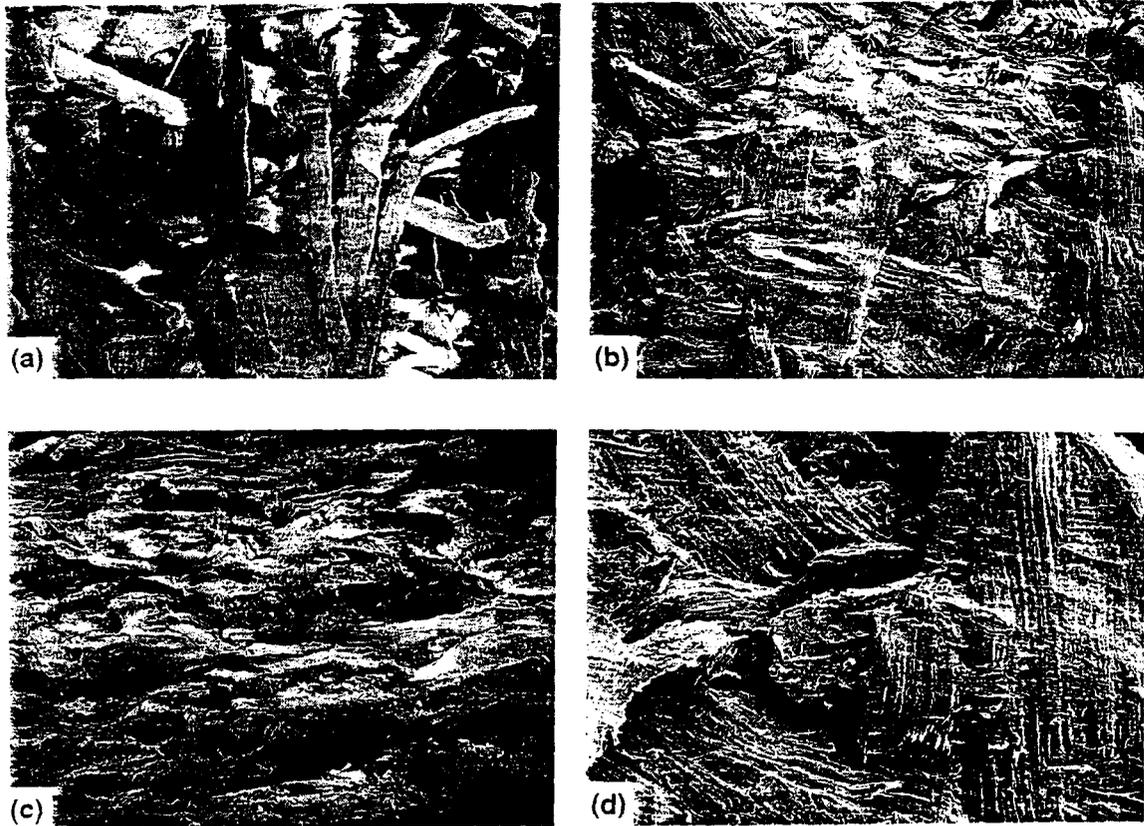


Figure 7.4 SEM of pressed kenaf fiber: (a) control (30 \times); (b) SA-reacted (50 WPG, 30 \times); (c) SA-reacted (50 WPG, 50 \times); (d) SA-reacted (50 WPG, 100 \times). (From Ref. 72.)

in the molecule. This is done by one of two methods. In one case, the matrix is reacted with maleic anhydride, which results in a double bond in the grafted reacted molecule. This can then be used in vinyl-type additions or in free radical polymerization to either build a thermoplastic polymer or graft one onto the lignocellulosic backbone. In the second method, the matrix is reacted first with a bonded chemical and then with a low-molecular-weight thermoplastic that has been grafted with side-chain anhydride groups.

The anhydride functionality in the compatibilization research described above may react with the lignocellulosic, but there is no evidence to support that possibility at this time. A higher level of grafted anhydride on the polypropylene would be required for the alloy reactions, and it would be expected that the reaction between grafted thermoplastic and lignocellulosic would take place both on the matrix polymers (lignin and hemicelluloses) and in the cellulose backbone. Some recrystallization of the cellulose may be desired to give more thermoplastic character to the entire composite.

Preliminary results indicate that maleic anhydride reacts with the lignocellulosic matrix in both liquid and solid-state reactions to similar weight gains as given for succinic anhydride. Research in this area is continuing.

Combining jute and kenaf fibers with thermoplastics provides a strategy for producing advanced composites that take advantage of the enhanced properties of both types of resource. It allows the scientist to design materials based on end-use requirements within a framework of cost, availability, recyclability, energy use, and environmen-

tal considerations. These new composites make it possible to explore new applications and new markets in such areas as packaging, furniture, housing, and automotive uses.

17. FUTURE TRENDS

The main commercial developments in the jute industry have been concerned with spinning and weaving technology, and considerable improvements in productivity have indeed taken place. But it is now timely to consider what new innovations would assist the spread of jute materials into textile uses outside the traditional fields of packaging and carpets.

Agricultural developments to breed *Corchorus* or *Hibiscus* plants containing fibers of significantly lower linear density would allow yarns of lower count than is now feasible, to be spun and so enable the production of light-weight fabrics. Such fabrics could have increased potential for decorative and furnishing uses, especially if the constraint of color-instability could first be removed and a process devised to produce additional elasticity that is more permanent basis than that of woolenized jute.

Kenaf is now being grown in several states in the United States, where the bast fiber is used for geotextiles and the pith is used in sorbents for oil-spill clean-up and animal litter. Whole-plant utilization of both jute and kenaf are being considered for structural and nonstructural composites. Interior automobile door panels are now being produced in Germany and the United States out of jute and kenaf bast fiber.

Research is being conducted in the areas of paper and composite production in several countries, and it is expected that whole new markets will be developed for both jute and kenaf as well as for many other agro-based fibers.

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