

Nonwood Fibers

Tear and tensile properties of soda pulps from kenaf bast fibers

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ABSTRACT

Kenaf (*Hibiscus cannabinus*) is an annual nonwood fiber source for pulping and papermaking. In this paper, an attempt is made to understand the tear and tensile properties of kenaf pulp fibers in terms of morphological and chemical factors. The discussion is limited to the bast fibers only and excludes the core fibers. The tear–tensile plot for kenaf bast fibers resembles that for the softwoods. Multiple regression models are discussed for tear, tensile, and strength index (tear $\times$ tensile) for bast fibers. It is concluded that the morphological properties explain most of the variations in breaking length, tear factor, and strength index. Elasticity, or the relative rate of change, is also calculated and used to explain the percentage change in paper properties as a result of a change in morphological or chemical property. For example, in kenaf bast fibers, a 1% change in fiber coarseness results in a 0.35% change in breaking length.

INTRODUCTION

The kenaf plant (*Hibiscus cannabinus*) is considered one of the most promising alternatives to virgin softwoods and hardwoods for paper production. A herbaceous annual plant of the Malvaceae family related to cotton and okra, kenaf is a member of the mallow family indigenous to West Africa. The United States Department of Agriculture (USDA) began researching kenaf in the 1940s, when World War II put a stop to jute imports from Asia. The work started with a scheme for evaluating potential plants as new sources of pulp and paper. The evaluation procedure involved consideration of botanical characteristics, chemical composition, yield, and dimensional measurements of fibrous constituents, and a qualitative and visual appraisal of the plants (1–4). A total of 58 plant species representing 11 families were reported, and laboratory-scale pulping studies were carried out on them (3). This work was extended by analysis and evaluation of 208 additional species (32 families) (4). Characteristics of pulpwood and other accepted pulping materials served as a guideline in determining which properties of the nonwoody species should be measured. Among the species that were subjected to the entire screening evaluation, kenaf and hemp were the most promising (5).

Kenaf is an annual nonwood fiber plant having low density (0.256 g/cm³). It is grown in rows using standard farm equipment. It reaches heights of 3–5 meters and yields 4–8 metric tons of dry fiber per acre, depending on silvicultural conditions. Kenaf can be cultivated under a wide

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range of conditions and requires relatively little care. It has straight slender stems and is largely unbranched in dense strands. (6, 7). Kenaf plant consists of two morphologically and chemically distinctive regions in its stem. The outer portion of the kenaf stem is known as bark, and it contains long bast or cordage fibers, while the inner or woody portion is termed as core, which contains short fibers. The whole stalk of kenaf constitutes about 60–65% of core fibers, and 35–40% of bast fibers (8). At present, kenaf is being used on a limited scale as a substitute for wood in the production of pulp and paper in Thailand and the Peoples Republic of China (9). In 1998, a newsprint mill with an annual production capacity of 70,000–80,000 tons from 100% whole-stalk kenaf fiber is planned in LaSara, TX.

Softwood pulp fibers are long and approximately cylindrical. They vary in length, diameter, and cell-wall thickness. Hardwood fibers are shorter than softwood fibers with tapering ends, and are accompanied by parenchyma cells and vessel elements. Similarly, nonwood fibers vary in length and diameter, depending on whether they are derived from the outer bark or the inner core. They generally contain a lot of parenchyma cells, epidermal cells, and vessel elements, which account for their higher percentage of fines. The nonwoods generally have lower lignin, comparable cellulose and hemicelluloses, higher ash, higher extractives, and higher silica than the woods. Thus any relationship between fiber dimensions, chemical composition, and paper properties that holds good for softwoods or hardwoods may not hold for nonwoods. The literature includes various attempts to determine which fiber characteristics influence various paper properties. The usual experimental approach in early studies was to hold all fiber characteristics constant except one, which was allowed to vary in order to determine its influence on sheet quality (10). A more recent approach allows several fiber characteristics to vary simultaneously, and multiple regression analysis is then used to quantify the contribution of each characteristic to paper properties (11). A comprehensive review of literature on the relationship between fiber morphology and paper properties for wood fibers is reported by Dinwoodie (12). More recently, Paavilainen (13) has studied the effect of morphological, fine structural, and chemical properties of wood fibers on pulp fiber properties and on fiber network properties. Mathematical models for paper strength properties like tear and tensile have been developed by various authors (11, 14–16).

For kenaf fibers, Touzinsky *et al.* (17) have applied statistical techniques to develop a general quadratic model relating the strength properties to active alkali charged and the cooking time. Another study on kenaf fibers by Clark *et al.* (18) uses analysis of variance (ANOVA) to relate the effects of type of pulping chemicals and the level of applied chemicals on sheet strength properties. However, there is no comprehensive study on kenaf fibers relating the strength properties of paper to the morphological characteristics and the chemical pulp properties.

In this paper, an attempt is made to relate the various chemical and morphological characteristics to the paper strength properties like tear and tensile for kenaf fibers. Multiple regression techniques were used to develop the relationships, and the concept of elasticity was used to explain the relative rate of change in the paper property as a result of a change in the chemical or morphological property. The paper mainly discusses the relationships for kenaf bast fibers.

MATERIALS AND METHODS

PULP

The raw materials used in this study—kenaf bast and core fibers—were procured from Mississippi State University. Pulping was carried out in M.K. Systems computerized laboratory digester. The rate of heating was held constant at 150°C/h, and the concentration of the soda cooking liquor was 15% (as Na₂O). The ratio of raw material to liquor was maintained at 1:6 for all experiments, while the variables were cooking temperature (155°C and 170°C) and time (90, 120, 150, 180, and 210 min). The cooked pulp was washed and screened, and from the ten sets of cooks, five sets with a range of residual lignin content were selected for further processing in a PFI mill.

CHEMICAL ANALYSIS

The chemical analysis carried out included Klason lignin content, acid soluble lignin, holocellulose, and alpha cellulose. The reported hemicellulose content is obtained from the holocellulose and alpha-cellulose content. Extractives, ash, and trace element analysis was done for the raw material samples only. The reported residual lignin content is the sum of the Klason lignin and the acid-soluble lignin content, determined as per TAPPI T 222 om-83 and TAPPI UM 250. Holocellulose and alpha-cellulose were determined using the procedure described by Zobel *et al.* (19). Ash content was determined using TAPPI T 211 om-85.

BEATING AND MORPHOLOGICAL CHARACTERISTICS

Freeness was measured using the Canadian Standard Freeness (CSF) tester. A Fiber Quality Analyzer (FQA) was used to measure the fiber length, coarseness, fines, and fiber curl. The samples were prepared to achieve a fiber frequency of 5–20 events per second during the test. To achieve this range of fiber frequency, 3.5 mg/L of bast fibers and 1.25 mg/L of core fibers had to be taken.

FIBER WIDTH AND CELL-WALL THICKNESS

Fibers from samples were first layered one on top of the other by tweezers. Agar solution (2%) was then applied to the fiber layers. Fibers in the gelatinlike layers were dehydrated by soaking them in 50% ethanol for 15 min, 70% ethanol for 15 min, 90% ethanol for 20 min, and twice in 100% ethanol for 20 min. This was followed by the introduction of spurr resin. Approximately 23 g of resin was freshly prepared from 5.0 g of vinylcyclohexane dioxide (ERL), 3.0 g of diglycidyl ether of polypropylene glycol (DER), 13.0 g of nonenyl succinnic anhydride (NSA), and 0.2 g of dimethylaminoethanol (S-1). The dehydrated fibers were soaked in the spurr resin in 1:3 ratio with 100% ethanol for one hour. The amount of spurr resin was increased to obtain ratios of 1:1 and 3:1, each with soaking times of 1.5 h. Infiltration with resin was achieved by soaking the samples overnight in 100% resin. To avoid exceeding the three-day shelf life of the resin, a fresh mixture was prepared for the remainder of the procedure: 5.0 g ERL, 3.0 g DER, 13.0 g NSA, 0.6 g S-1. Embedding of the samples with spurr resin was then done, after which they were cured at 60°C for 16–24 h. The resin blocks were cut and mounted for transverse sections. Facing was achieved with the use of an ultramicrotome equipped with either a glass

knife or a diamond knife. The cross sections were measured for cell-wall thickness and fiber width using a light microscope. Fiber width is a measurement that is rarely used on its own. It is usually used in ratios with other anatomical variables such as cell-wall thickness, fiber length, and lumen diameter (11). All these ratios express the potential of fiber to collapse. Collapsed fibers provide more bonding area, and subsequently stronger papers are produced. One possibility that has been mostly overlooked in the literature is to consider fiber width as a variable on its own. In this research, fiber width is taken as an explanatory variable.

FIBER DENSITY

Fiber density in this study is determined based on coarseness and fiber width measurements, and as given in the literature (11) can be expressed as:

$$\text{FiberDensity} = \frac{\text{Mass}}{\text{Volume}} = \frac{\text{Coarseness}}{(\text{Fiber width})^2} \quad (1)$$

PAPER PROPERTIES

Breaking length was measured by an Instron No. 1130 following CPPA standard D.6H. Four-ply tear strength was measured using an Elmendorf tear tester following CPPA standard D.9. Zero-span breaking length, which is considered as a morphological property in the statistical analysis, was measured with a Pulmac tester following TAPPI T 231 cm-96.

STATISTICAL ANALYSIS

A correlation coefficient matrix for all the variables was made, and those variables having a correlation coefficient $r \geq 0.4$ at a significance level of 5% were selected for further analysis. A regression of all the explanatory variables screened above against the selected paper strength property as a dependent variable was run using SPSS statistical software. After various trials, the best possible regression that gave the best F -value, t -values, and adjusted r^2 was selected and reported. Multicollinearity affecting the explanatory variables was removed using different transformations of explanatory variables. Elasticity as used in this study is defined as the relative rate of change in dependent variable (Y) as a result of a change in the explanatory variable (X). If $Y = f(X)$, the elasticity (ε) of Y with respect to X is:

$$\varepsilon = \left(\frac{dY}{dX} \right) \left(\frac{X}{Y} \right) \quad (2)$$

A concept that is used extensively in econometrics (20) is utilized in this research to quantify the relative change in paper properties resulting from a change in the morphological property or chemical property. For example, elasticity of breaking length with respect to fiber length can tell us how much change can be expected in breaking length if fiber length is changed by 1%.

RESULTS AND DISCUSSION

BAST VS. CORE FIBERS

In their previous work (21), the authors have clearly demonstrated that there is a significant difference in the delignification behavior of the different fractions of kenaf stem. The activation energies for the two fractions, bast and core, are significantly different. The bast fraction is easier to delignify and has a lower activation energy (68 kJ/mole) than the core fibers (91 kJ/mole). The chemical analysis of kenaf fibers with 95% confidence limits is given in **Table I** (21).

Average composition	Bast	Core	Whole
Lignin, %	11.8 ± 0.45	18.3 ± 0.25	16.2 ± 0.66
Hollocellulose, %	81.1 ± 1.09	71.6 ± 2.15	76.5 ± 0.78
Alpha cellulose, %	51.0 ± 0.58	34.9 ± 0.47	44.1 ± 0.74
Extractives, %	2.8 ± 0.09	4.8 ± 0.34	3.2 ± 0.28

Table I. Chemical composition of different fractions of kenaf stem

TEAR-TENSILE PLOT

Tensile and tear strengths are two important mechanical properties of paper. Tear strength is the ability of the paper sheet to resist the propagation of tears initiated by flaws such as cracks. Tensile strength determines the ability of a sheet to withstand in-plane stress. For papermaking, both high tensile and tear are desired. Tear-tensile plots are thus very useful tools for comparing different pulps. They are also useful for predicting the degree of delignification at which optimum tear and tensile would be obtained (22). The tear-tensile plot for the kenaf bast fiber used in this study is given in **Fig. 1**. It resembles the tear-tensile plot for softwoods in the literature (23). Tensile strength is given as breaking length (measured in kilometers) and tear strength is expressed as tear factor.

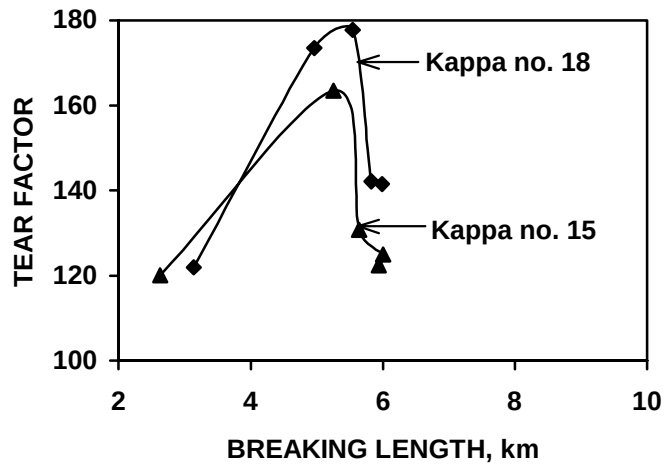


Fig. 1. Tear–tensile plot of kenaf bast fibers

From the tear–tensile plot in Fig. 1, it is clear that when the degree of bonding between fibers in a paper sheet is increased, both tensile strength and tear strength increase initially. Then, after passing through a maximum, the tear factor appears to decrease sharply with increased bonding. In this work, bonding was increased by beating the pulp in a PFI mill.

BREAKING LENGTH OF KENAF BAST FIBERS

The breaking length or the tensile strength of a sheet depends on the fiber strength and the bond strength. When fibers are stronger than the bonds, the bonds tend to break. When the bonds are stronger than the fibers, the fibers break. The strongest fibers have the thickest walls and will probably be least bonded. The importance of the relationship has been quantified in the form of the equation by Page (15). In kenaf fibers, the breaking length for the bast and the core fibers are comparable, unlike the tear values, which are quite different. A multiple regression equation for the breaking length (km) was developed using the following 13 explanatory variables:

- Pulp freeness, mL CSF
- Lignin, %
- Hemicellulose, %
- Cellulose, %
- Fiber width, μm
- Cell-wall thickness, μm
- Length-weighted fiber length, mm
- Fiber coarseness, mg/m
- Fines, %
- Length-weighted curl index
- Fiber density, kg/m^3
- Sheet bulk, m^3/kg
- Zero-span breaking length, km

A total of 25 pulp samples from five different cooks and five beating points were analyzed for all these properties. The regression equation developed for breaking length of bast fibers is given in Eq. 3.

$$BL = 7.5273 - 10.4248 FC + 2.7535 \times 10^{-4} (FD \times HC) - 1.7005 \times 10^{-4} (FW \times CSF) \quad (3)$$

(2.18) (5.85) (3.48)

where

FC = fiber coarseness

FD = fiber density

HC = hemicellulose

FW = fiber width

CSF = pulp freeness

Numbers in parentheses give the *t*-values of the coefficients of the explanatory variables. Equation 3 has an adjusted r^2 value of 0.87 at 5% significance level, which means that 87% of the variation in the breaking length is explained by the above equation. The *F*-value of 52.06 (significance level = 0.00) indicates the statistical significance of the equation as a whole. All the coefficients of the explanatory variables in the equation are statistically different from zero at 5% significance level. The elasticity, or the relative rate of change of breaking length with respect to the explanatory variables in Eq. 3, is given in **Table II**. Thus a 1% change in fiber coarseness will result in a 0.35% change in breaking length, while a 1% change in either fiber density or hemicellulose content will result in 0.3% change in breaking length. Similarly, a 1% change in either fiber width or CSF will translate to a 0.23% change in breaking length.

	Explanatory variable	Elasticity of breaking length
	Fiber coarseness	– 0.35
	Fiber density	0.30
	Hemicellulose	0.30
	Fiber width	– 0.23
	CSF	– 0.23

Table II. Elasticity of breaking length with respect to the explanatory variables

Breaking length is positively related to fiber density and hemicelluloses content, while it is negatively related to fiber width, CSF, and coarseness. A stepwise regression of the variables in Eq. 3 shows that the fiber density and hemicellulose content explain 63% of the variation. A regression of breaking length and hemicellulose alone points out that the hemicellulose term explains about 24% of the variations in the breaking length. In other words, the major part of the variations in breaking length of kenaf bast fibers is explained by the morphological factors.

TEAR FACTOR OF KENAF BAST FIBERS

The tear test measures the energy consumed or the work done when a piece of paper is torn a certain distance. In tearing, there are two possible situations:

1. The fiber is torn out without being ruptured, and breakage takes place in the areas where the fiber is bonded to other fibers. This will be the situation in loosely bonded sheets. As the bonding increases, the work done by tearing also increases.
2. When the bonds have reached a certain number, the fiber will be so strongly held by all the bonds together that the fiber itself will break, and such a rupture of fiber will only consume a limited amount of energy. Although the force required might be high, the energy requirement in this case is far less than when the fiber had to be pulled out intact. The degree of bonding at which breakage switches over from multibond rupture to fiber rupture depends on the strength of the fiber and the bonds.

The models developed for tear have very little application due to the hook-shaped nature of the curve during beating. A multiple regression equation was developed for tear factor using the same 13 explanatory variables that were used for the breaking length regression model discussed earlier. The regression equation for the tear factor is given in Eq. 4.

$$\begin{aligned} \text{TF} = & -258.6517 + 0.2770 \text{ CSF} + 236.1734 \text{ FC} + 12.3136 \text{ ZBL} + 20.6095 \text{ Fines} - 8.8428 \text{ FW} + \\ & 644.0641 \text{ Curl} \end{aligned} \quad \begin{matrix} (3.94) & (2.78) & (3.21) & (3.90) & (3.28) & (2.69) \end{matrix} \quad (4)$$

where

CSF = pulp freeness

FC = fiber coarseness

ZBL = zero-span breaking length

FW = fiber width

Equation 4 explains about 57% variations in the tear factor (adjusted $r^2 = 0.57$ at 5% significance level), and the equation is statistically significant (F -value = 6.13 at significance level = 0.00). All the explanatory variables in the equation are statistically different from zero at 1% significance level (t -values in parentheses). The elasticity of tear factor with respect to the explanatory variables in Eq. 4 is given in **Table III**.

Explanatory variable	Elasticity of tear factor
Zero-span	1.35
Fiber width	− 1.23
Fines	0.96
CSF	0.86
Curl	0.62
Fiber coarseness	0.34

Table III. Elasticity of tear factor with respect to the explanatory variables

It is clear from Table III that tear factor is very sensitive to small changes in morphological properties. For example, a 1% change in zero-span results in 1.35% change in tear factor.

STRENGTH INDEX OF KENAF BAST FIBERS

From the papermaker's point of view, both high tensile and tear are desired. An empirical strength index can be used to characterize pulps (23–25). Strength index is defined in Eq. 5.

$$\text{Strength index} = \text{Breaking length} \times \text{Tear factor} \quad (5)$$

We used this definition of strength index to account for variations in the tear and tensile properties of kenaf bast fibers. The regression equation for the strength index was developed using the same 13 explanatory variables that were used to develop the models for breaking length and tear factor. The regression equation is given in Eq. 6.

$$SI = -80.6275 + 1.9233 \text{ Hemicellulose} + 38.4419 \ln (\text{ZBL} \times \text{Fines}) - 1.9980 \text{ FW} \quad (6)$$

Equation 6 explains about 77% of the variation in strength index at a 5% significance level, and the equation is statistically significant (F -value = 26.76, significance level = 0.00). All the explanatory variables in the equation are statistically different from zero at 1% significance level (t -values in parentheses). The elasticity values for strength index with respect to the explanatory variables in Eq. 6 are given in **Table IV**. The table shows that a 1% change in either zero-span, fines content, or fiber width results in a 0.5% change in strength index, and a 1% change in hemicellulose content results in a 0.3% change in strength index. A stepwise regression of the variables in Eq. 6 shows that the $\ln$ (zero-span $\times$ fines) term alone explains about 44% of the variation in the strength index.

	Explanatory variable	Elasticity of strength index
	Zero-span	0.50
	Fines	0.50
	Fiber width	– 0.49
	Hemicellulose	0.28

Table IV. Elasticity of strength index with respect to the explanatory variables

CONCLUSIONS

The behavior of tear–tensile plots leads to the conclusion that the papermaking potential of bast fibers in kenaf is similar to that of softwoods. The results of this study provide an initial exploratory tool for a better understanding of the development of tear and tensile properties of kenaf fibers. The breaking length of the bast fibers is a function of fiber coarseness, fiber density, hemicellulose content, fiber width, and freeness, while the tear factor is a function of zero-span, fiber width, fines, freeness, fiber curl, and fiber coarseness. A combined strength index has been calculated and analyzed in this study, and for the bast fibers, it is a function of zero-span, fines, fiber width, and hemicellulose content. The elasticity values suggest that a 1% change in the morphological parameters that are significant for the development of strength index results in about a 0.5% change in strength index, while a 1% change in chemical factors results in about a 0.3% change in strength index. For the bast fibers, it can be concluded that the morphological factors contribute more than the chemical factors toward the development of tear and tensile properties. Results for the bast core fibers will be discussed in another paper.

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