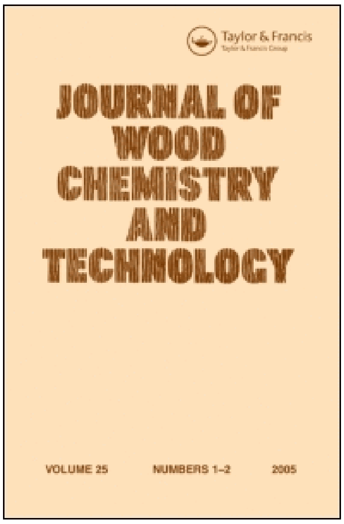

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CHEMICAL AND SODA PULPING PROPERTIES OF KENAF AS A FUNCTION OF GROWTH

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ABSTRACT

Kenaf (*Hibiscus canabinus*) is a promising non-wood source of fibre for pulping and papermaking. Because the chemical and physical composition of the plant changes as the plant develops, research is needed to determine its pulping properties at various stages of growth in order to establish the optimum harvesting time. In the present work, the chemical composition and pulping properties of kenaf as a function of growth have been studied. Kenaf plants were harvested at the end of 90, 120, 150, and 200 days (maturity). Extractive-free ground samples of the stem were cooked at three different temperatures, 140, 155, and 170°C, using soda cooking liquor of 32 g/L NaOH, with an active alkali charge of 15% as Na_2O, and a liquor-to-wood ratio of 6:1. The differences in the holocellulose and lignin content for 90, 120, and 150-day old kenaf were not significant, while 200-day-old kenaf was significantly different from others. Pulping of kenaf at various stages of growth indicated that soda pulping properties were not significant. In comparing the yields of kenaf pulps, it is observed that; over the whole range of cooking times and temperatures studied, the average yield for 150-day-old kenaf is highest at 60.4%. Kenaf can be harvested at the end of 150-day growth period, based on the results of chemical analyses to achieve higher yield, with lower lignin content.

INTRODUCTION

Kenaf (*Hibiscus cannabinus*), a herbaceous plant of the family Malvacae, is an example of a non-wood pulp source that is already used in parts of the world.[1] The plant grows to a height of 3.7–5.5 m with a diameter of 25–51 mm. Kenaf has been grown successfully in North America and could become a pulp alternative to tree species in the southern U.S. Non-wood pulps such as those from kenaf bark have long fibers (2.5–5.0 mm), similar to softwood fibers (2.7–4.6 mm), kenaf core short fibres (0.5–0.7 mm), much like temperate zone hardwoods (0.7–1.6 mm).

It is known that different parts of a plant have different chemical and physical properties. That is, the chemical composition and fiber properties of plant tissue taken from the roots, stem, trunk, and leaves are different. What is not so well known is that the chemical composition and fiber properties of plant tissue are also different at different stages of the growing season. There may be an advantage in harvesting fiber for paper at some time earlier than from a mature plant.[2] For example, fiber from an immature plant may be low in lignin which could be used for paper there would be little pulping chemical required to remove the lignin. There may be an advantage in chemical and energy use to harvest early. In the case of annual plants, it may be possible to harvest two crops in one season to give the same yield of fiber but with much less lignin.[2] The objective of this study was to investigate the kenaf fibres in terms of their chemical, physical, soda pulping properties at various stages of growth in order to establish the optimum harvesting time.

MATERIALS AND METHODS

Raw Material

Kenaf plants were grown in the greenhouse of the Faculty of Forestry, University of Toronto, using Pro-mix ‘‘GSX’’ greenhouse select growing medium. Pots of 4 L size were completely filled with pro-mix, taking care not to compact and making sure that all containers had adequate drainage. Seeds of kenaf were sown 0.5 in deep, 4 to 6 in apart in a row and 15 in

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between the rows in pots. Then these pots were placed on benches in the greenhouse. The soil was kept wet for about 2–3 weeks after the seeding.

Initial plant development occured without immediate fertilization. To ensure plants received adequate nutrients, all purpose fertilizer (20-20-20) was used. The plants were fertilized every two weeks, following the instructions given by the producer. Two plants from each pot were harvested at the end of 90, 120, 150, and 200-day growing period. The plants flowered at about 200 days. This was considered as the maturity.

The kenaf plants were cut at the base and the leaves were discarded. The kenaf plant stems were chipped into specified sizes (about 2 in) for grinding and air dried for some time (about 3–4 weeks depending on the conditions). The samples for chemical analysis and pulping were prepared following Tappi standard (T 257 cm – 85). After grinding the chips in a Wiley laboratory mill (Model 4), the finer material was separated by sifting on a 40-mesh (0.40 mm) power-driven screen.

Chemical Characterization

The ground samples of kenaf at various stages of growth were extracted following Tappi standard (T 264 om – 82) with ethanol : benzene (1:2 by volume) for 6 h and with ethanol for four hours. Hollocellulose and alpha-cellulose were determined following the procedure originally pioneered by Zobel *et al*.[3] The carbohydrate (holocellulose) fraction of fiber was isolated by removing the lignin from extractive-free fiber using the acid chlorite method. NaOH (17.5%) was applied to holocellulose to dissolve hemicelluloses for determination of alpha-cellulose in kenaf fibers. Klason lignin content was determined by hydrolysing the carbohydrates with 72% sulfuric acid as per Tappi standard T222 om – 83; the acid-soluble lignin was also measured on the filtrate specimen as per Tappi standard um 250 using UV absorption at 205 nm. To determine the ash content the procedure outlined in TAPPI Standard T 211 om – 85 was followed.

Soda Micro-Pulping Procedure

Soda cooking liquor of 32 g/L NaOH with active alkali charge of 15% as Na_2O was used. The extractive-free ground fibers samples were placed in a stainless steel reactor (25 mL) with screw caps, and soda cooking liquor was added to maintain a liquor to raw material ratio of 6:1. A presoaking period of 30 min. at room temperature was provided before immersing the reactors in a silicone oil bath, thermostatically controlled at the desired temperature. The temperatures studied were 140, 155 and 170°C. The time to reach the reaction temperature is the time required to regain the temperature after the reactors are immersed in the oil bath and it varied with temperature, (12 min. at 170°C, 10 min. at 155°C, and 8 min. at 140°C). The reaction time reported is the time at temperature, and it does not take into account the time to reach the temperature. The reaction was stopped by quenching the reactor in a bath of running cold water. The resulting pulp with liquor was transferred to a Buchner funnel and washed with enough distilled water to transfer the pulp with liquor from the reactors. The pulp was washed with water and the yield and residual lignin content of the pulp were determined. The reported residual lignin content is the sum of the Klason lignin and the acid soluble lignin.

RESULTS AND DISCUSSION

Yield per Acre

The weight of 90-day-old greenhouse-grown kenaf plant was (average of 4 air dried plant stems) 17.1 g per plant at the end of growing period. Using the 15 in between the row and 4 to 6 in apart within a row, (which is 60 to 90 in^2 per plant) the yield is 1.2 to 1.8 tons/acre/year. The extractives content was 20.2% and holocellulose content 81.4%. Taking the extractives content into consideration the holocellulose content is 61.2% and the yield for 90-day-old kenaf is 0.7 to 1.0 tons/acre/year. The weight of 120, 150 and 200-day-old kenaf (average of 4 a.d. plant stems) 22.4, 43.9 and 75.8 g per plant respectively at the end of growing period. Using 61.2, 66.2, and 66.3% for the holocellulose contents when the extractives contents taken into consideration, the yield for 120, 150 and 200-day-old kenaf is 0.7 to 1.0, 2.0 to 3.0, and 3.5 to 5.2 tons/acre/year respectively.

Chemical Composition of Kenaf as a Function of Growth

Holocellulose Content

The results of chemical analysis of kenaf at various stages of growth are given in Table 1, and shown in Figure 1 and Figure 2. The Duncan's Test for the means of the holocellulose contents showed that the variations among 90, 120, 150-day-old kenaf are not significant, except for 200-day-old kenaf, at 95% confidence level. Han *et al.*[4] found that the juvenile kenaf samples had low holocellulose values which gradually increased as the plant aged.

Table 1. Chemical Composition of 90, 120, 150 and 200-day-old Kenaf (Mature Kenaf) Grown Under Greenhouse Conditions, Kenaf Grown in Mississippi,[5] Trembling Aspen, and Black Spruce[6]

Characteristics	Holocellulose* (%)	Alpha-cellulose* (%)	Hemicellulose* (%)	Lignin* (%)	Ethanol-benzene extract (%)	Ash* (%)
90-day-old kenaf	81.4	51.0	30.4	18.6	20.2	2.9
120-day-old kenaf	81.3	51.6	29.7	18.7	15.0	2.6
150-day-old kenaf	81.1	51.4	29.7	18.9	14.9	2.5
200-day-old kenaf	80.5	50.6	29.9	19.5	14.2	2.1
Kenaf (Mississippi)	76.5	44.1	32.4	16.2	3.2	2.1
Trembling aspen (Populus tremuloides Michx.)	80.3	49.4	30.9	18.1	3.8	0.4
Black spruce (Picea mariana (Mill.) B.S.P.)	71.7	51.1	20.6	27.3	2.6	0.2

*Measurements made on extractive-free samples.

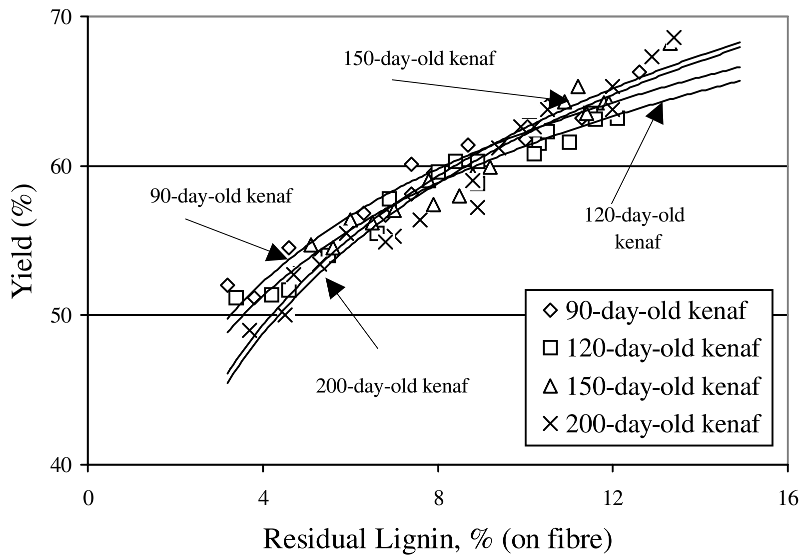

Figure 1. Delignification selectivity curves for 90, 120, 150 and 200-day-old (mature) kenaf.

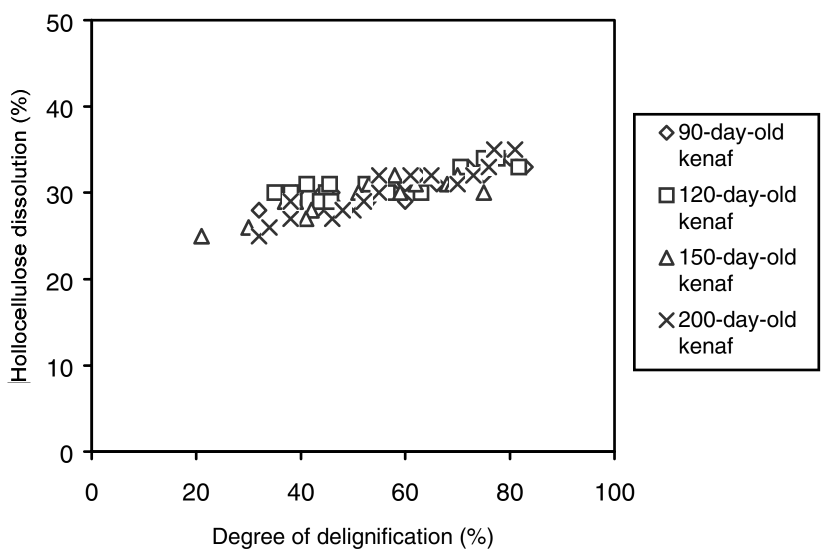

Figure 2. Selectivity of soda pulping (holocellulose dissolution vs. degree of delignification) of kenaf at various stages of growth.

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Klason Lignin (Acid-Insoluble Lignin)

As with the holocellulose content, the differences in lignin content of kenaf at various stages of growth are significant only for mature kenaf. Clark and Wolff [7] studied the changes in chemical composition of kenaf as a function of the growing season. They found that lignin content increased with age for the values taken from the bottom (all but the top 0.66 m of the plant). Data taken from the top part of the plant shows similar trends but the top part has less lignin but higher protein than the bottom part of the plant.

Han *et al.*[4] determined the weight ratios of core to bast as a function of growth. They found that weight ratios between kenaf bast and core (core/bast) increased as the growing days advanced. The maximum of 1.8 was reached at 175 days after planting.

Extractives Content

The analyses indicate that the differences in the mean values for extractive contents of kenaf as a function of the growing season are significant among 90, 120, 150, and 200-day old (mature) kenaf. And the extractive content of 90-day old kenaf is significantly higher than the others. These results also show that the extractives content of kenaf decreases as a function of growth and this is in good agreement with the results reported in literature. Han *et al.*[4] reported that solvent extractives content varied as a function of growth. In general, they were high at the beginning, decreased during the first part of the growing time, and then increased again.

In this study, one of the reasons why extractives content values are higher than field-grown kenaf may be due to the fact that the top of the plant stems had not been removed prior to testing. This part of the plant is reported to have a large amount of protein and extractives as reported by Clark and Wollf.[7] It is indicated that in most cases for a green chop, removal of the leafy top portion is desirable. Clark *et al.*[8] also reported that the upper 3 feet part of the plant had to be removed to reduce the amount of foliage before chopping the stalks to 1-in lengths. Foliage does not contribute to a pulp of good quality, but only the leaves were removed, and the upper 3 ft of the plant stem were not removed prior to this study. The other reason for field-grown kenaf to have lower extractives content than greenhouse-grown kenaf may be due to the fact that field-grown kenaf may be retted before processing.

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Ash Content

The differences in the mean values of ash content of kenaf at various stages of growth are significant as can be seen in Table 1. Ash content of 90-day old kenaf is the highest at 2.9% and there is a decrease to 2.6% then to 2.5% for 120-day and 150-day old kenaf. A decrease in ash content of kenaf as a function of growth is in good agreement with the reported trend in literature. The ash content of mature kenaf grown in the greenhouse at 2.1% is also in good agreement with the ash content of mature kenaf grown in Mississippi.[5] It is reported that ash content of kenaf decreased as the plant reached maturity.[4,7,8]

Soda Micro-Pulping Characteristics of Kenaf as a Function of Growth

Pulp Yield

The results of this study indicate that the differences in the mean values of percentage yields of kenaf at various stages of growth are not significant. In comparing the yields of pulps from kenaf at various stages of growth, it is observed that 150-day-old kenaf gives about 2% higher yield (60.4%) than 90-day-old (58.8%), 120-day-old (58.7%) and 200-day-old (mature) kenaf (58.8%), for all cooking times and temperatures studied. For example, cooks at 170°C for 180 min gave pulp yields of 52.0, 51.2, 54.7, and 49.0% respectively for 90, 120, 150 and 200-day-old (mature) kenaf. Mature (200-day-old) kenaf pulp yield is lower than the others in the group compared. For pulping conditions similar to the industrial scale, cooking at 170°C for 120 min., gave pulp yields of 54.5, 51.7, 55.6, and 52.7% respectively for 90, 120, 150 and 200-day-old (mature) kenaf.

Delignification Selectivity

The selectivity of the pulping chemicals toward delignification determines the yield of the pulping process and to some extent the pulp properties. Delignification selectivity curves, residual lignin, (% of extractive-free fibre) versus pulp yield (% of extractive-free fiber) for 90, 120, 150, and 200-day-old (mature) kenaf are shown in Figure 1. The average yield for 150-day-old kenaf over the whole range of cooking times and temperatures studied is the highest at 60.4% among the group compared.

Degree of delignification versus holocellulose dissolution is shown in Figure 2. It shows that the bulk phase of delignification started at approximately 20% delignification, and up to this point, in the initial phase of delignification, most of the carbohydrates dissolved (25 to 30%). In the bulk delignification phase of pulping, holocellulose dissolution is slow (0.06% per minute) and stays almost constant at percentage increases of (28 to 33%), (30 to 33%), (25 to 32%), and (25 to 35%) for 90, 120, 150, and 200-day-old (mature) kenaf respectively for all the cooking times and temperatures studied. But the degree of delignification increases from about 21 to 83% during the whole cooking times and temperatures. As stated before, Figure 2 also does not show any trend that any of the age groups of kenaf studied is more selective than the other.

The percentage residual lignin content based on raw material as a function of H-factor for 90, 120, 150 and 200-day-old kenaf is shown in Figure 3. Delignification studies of kenaf at various stages of growth indicated that all of the age groups studied showed similar trends in lignin removal and holocellulose dissolution and these trends are not significantly different from each other. It is also shown that pulping of kenaf can be stopped at the end of 120 min. of cooking to prevent further degradation

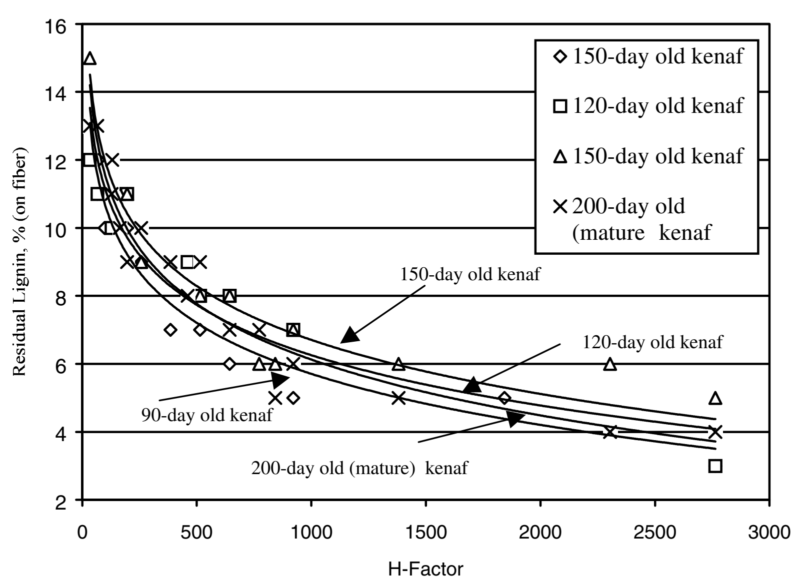

Figure 3. Lignin removal in soda pulping of 90, 120, 150 and 200-day old (mature) kenaf as a function of H-Factor.

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of polysaccharides and to obtain maximum pulp yield with low lignin content.

CONCLUSIONS

The chemical analyses indicate that the differences in holocellulose, and lignin content for 90, 120, 150-day-old kenaf are not significant, except for mature kenaf. Mature kenaf had a higher lignin and lower holocellulose content. The higher lignin content indicates the need for high consumption of chemicals for pulping and bleaching. Lower holocellulose content, on the other hand, gives lower pulp yield and strength properties. The Duncan's test indicated that the differences in the mean values of soda pulping properties of kenaf at various stages of growth are not significant. The average yield for 150-day-old kenaf over the whole range of cooking times and temperatures studied is the highest (60.4%) amongst the group studied. Based on this study, kenaf can be harvested at 150 day to achieve optimum pulp yield with lower lignin content.

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