

Physical characteristics of pulps obtained from mesta (*Hibiscus sabdariffa*) plants of different ages

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Paper made from pulp of older mesta plants yields paper of higher strength.

Kenaf (*Hibiscus cannabinus*) and mesta (*Hibiscus sabdariffa*) are excellent substitutes for traditional softwood pulp for the manufacture of newsprint (1). Pilot plant studies showed that it would be possible to produce high-strength mechanical pulp suitable for newsprint from whole mesta plant (1). Of the two species of mesta (2-4), *Hibiscus cannabinus* is used as textile fiber, while fiber from *Hibiscus sabdariffa* is coarse, with more attention paid to this variety for making paper.

Some of the properties of the pulp have been studied by Kulkarni *et al.* (1). Paper made of pulp from 150-day-old mesta plant was stronger by about 12% than that from 120-day-old mesta plant (5). We undertook our study to see the effect of age of mesta plant on the properties of the pulp and to find out the reason for the changes of the properties of paper.

Age and fiber structure

Figures 1-4 show the scanning electron micrographs of pulp fibers from 120-day-old mesta plants and 150-day-old mesta plants. These micrographs show that there are two types of fibers—long and fine fibers are from the bast portion, while the short ones are from the woody stalk. The fibers are longer in the pulp of 120-day-old mesta plant. At higher magnification, the details of fiber structure are seen. Transverse cracks are in the fiber from 120-day-old plant, and fibrillation is better in the fiber from 150-day-old plant. Thus, some of the properties favorable for the development of better strength in the paper are found in the pulp from 150-day-old mesta plant.

Table I shows that the lengths of both short and long fibers from 150-day-old plant are smaller from those from 120-day-old plant, whereas the cell wall thickness of fibers of 150-day-old plant is greater than that from 120-day-old plant. Greater cell wall thickness appears to play an important role toward increasing the strength of paper from pulp of 150-day-old plants.

The decrease in the length of fibers after 120 days of growth is in agreement with the result obtained with jute fibers (6). The lignin network and turgor pressure incite

I. Average length of ultimate fibers, average cell wall thickness and crystallinity index for the two pulps

	Pulp from plants	
	120 days	150 days
Average length of ultimate fibers (mm) with standard error of measurement in parentheses		
Long fiber	2.47 (0.06)	2.25 (0.07)
Short fiber	0.61 (0.01)	0.54 (0.01)
Average cell wall thickness (μm) with standard error of measurement in parentheses		
Long fiber	3.56 (0.023)	4.17 (0.027)
Short fiber	4.58 (0.031)	4.72 (0.041)
X-ray crystallinity index (x)	39	46
I. R. crystallinity	1.67	1.88

cell extension during growth. At later stages, the constancy of lignification and loss of natural water content may be responsible for the shortening of the fibers, in accordance with the hypothesis proposed by Stockmann (7, 8).

Table I shows that both X-ray and infrared crystallinities for pulp from 150-day-old plants are higher than those for pulp from 120-day-old plants. The higher crystallinity value is also responsible for the development of higher strength of the paper obtained from 150-day-old plants.

Figure 5 shows the infrared spectra of the two pulps. The peak at 1740 cm^{-1} is due to free carboxyl acid or ester stretching vibration. This peak is present in the spectrum of 150-day-old plants but does not exist in that of 120-day-old plants. The peak at 1600 cm^{-1} is present in both. The peak at 1500 cm^{-1} is smaller for 120-day-old plants. These peaks are characteristics of aromatic stretching bands.

The peak at 1460 cm^{-1} is present in both pulps. For the pulp made from 150-day-old plants, the height of the peak at 1430 cm^{-1} is greater than that in the pulp from 120-day-old plants. The case with the peak at 1370 cm^{-1} and 1330 cm^{-1} is similar. At 1250 cm^{-1} , there is a prominent band for the pulp from 150-day-old plants.

The bands from 1460 cm^{-1} to 1250 cm^{-1} are caused by lignin. These peaks are either small or insignificant in the spectra of pulp from 120-day-old plants, indicating that there is more lignin in the pulp from 150-day-old plants. From the peak at 1740 cm^{-1} , it appears that the hemicellulose content of 150-day-old plants is also greater, which favors the development of the strength of the paper from pulp obtained from 150-day-old plants. Because the lignin

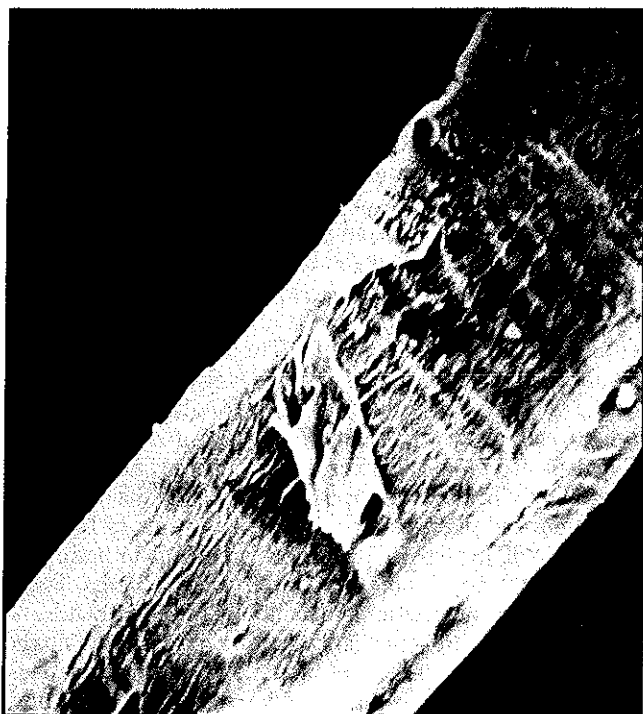
1. SEM of fibers from 120-day-old mesta plant (31X)



2. 1300X magnification of fibers from 120-day-old mesta plant



3. SEM of fibers from 150-day-old mesta plant (29X)



4. 2600X magnification of fibers from 150-day-old mesta plant



content is higher with older plants of higher ages, the extraction of noncellulosic encrustants by the same pulping process is also less difficult with the pulp from older plants.

Conclusions

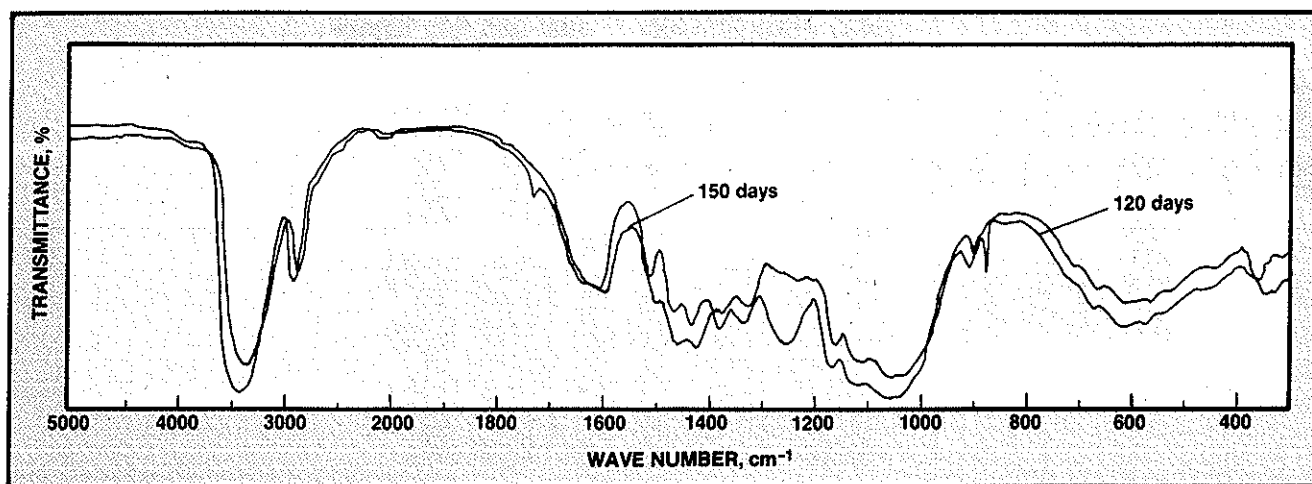
The physical properties of the pulp vary with the age of the plant. The pulp fibers from older mesta plants are shorter, but the cell wall thickness is higher. The crystallinity of the pulp from older plants is more pronounced. The

properties of the pulp are reflected in the properties of the paper made of the pulp from mesta plants of different ages. Paper made of the pulp from mesta plant of higher age yields paper of higher strength.

Materials

The experimental plants were grown around the Agricultural Research Station, Amadalavalasa, A.P. The plants were harvested 120 days and 150 days after sowing. After

5. Infrared spectra of pulp obtained from mesta plant



harvesting, the plants were left in the field for about 7 days to shred leaves. The air-dried plant was cut in chips of 1-2 in.

A 1-kg portion of chips was digested with 7 L of water at 140°C for 5 min. The steam was blown up rapidly, and the chips were passed through a Sprout Waldran disk refiner in three stages under three spacings. The crude pulp was bleached with calcium hypochlorite to brightness of 60. The pulp was then subjected to a short beating to attain a freeness of 40 mL. The handsheet made from the 150-day-old mesta whole plant was much stronger than that made from the 120-day-old mesta whole plant.

Experimental procedures

Optical

The pulp fibers were separated after soaking the pulp in water, dried in air, and examined in a projection microscope. Two types of fibers were seen, long and fine fibers from the bast portion and short and coarse fibers from the woody stalk. About 100 fibers from the randomly selected pulp portion were examined for each sample, and the values shown are the average of these readings.

Infrared examination

The pulp samples were powdered in a vibratory ball mill and mixed with powdered KBr of spectroscopic purity in suitable proportions. Pellets were made under vacuum and high pressure. The infrared spectra were taken in a Shimadzu 440 spectrophotometer. From each pulp, three samples were selected and three spectra from each pulp were taken. Crystallinity was determined following the methods reported earlier (9, 10) by taking the infrared absorption bands at 1420-30 cm⁻¹ and 900 cm⁻¹ as crystalline and amorphous bands, respectively.

X-ray examination

A fixed weight (5 mg) of pulp sample was used to prepare a pellet. The pellet of specified dimensions was made by pressing the fibers in a cylindrical hole in a brass plate. The pellet was finally mounted in a sample holder, and X-ray photographs were taken in a flat camera in a Philips X-ray tube, following the method described earlier (11).

The crystallinity index was measured by using the empirical method of Segal *et al.* (12). The Method of

calculation was based on the following relation:

$$\text{Index} = (I_{002} - I_{\text{am}} / I_{002}) \times 100 \quad (1)$$

where

Index = crystallinity index

I_{002} = maximum intensity (in arbitrary units) of the (002) lattice diffraction at $2\theta \times 22.6^\circ$

I_{am} = intensity of diffraction in the same unit at $2\theta \times 18^\circ$.

Scanning electron microscopy

Fibers were separated in the pulp samples and were mounted on specimen holders with the help of conducting copper tape. The samples were coated in a vacuum coating unit with a silver layer about 20 nm thick and were examined in a Hitachi S-430 electron microscope at an accelerating potential of 5 kV and 15 kV. Micrographs were taken at each potential.

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