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Evaluation of Pulp and Paper making characteristics of certain fast growing plants

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Summary Four species of fast growing annual plant viz. *Hibiscus sabdariffa*, *Crotalaria juncea*, *Tephrosia candida* and *Hibiscus cannabinus* and a variety of reed, Neyraudia reynaudiana were evaluated in the laboratory for their pulp and paper making properties. Data on proximate chemical analysis of raw materials, unbleached and bleached pulp properties, morphological properties of fibres and physical strength properties of paper sheets were evaluated. The unbleached pulp yields were 44.90–53.20% with all the five plant materials, while bleached pulp yields varied from 39.80–50.60%. The average fibre lengths and diameters of the unbleached pulp obtained from the five plant species were within the range of 0.75 mm–2.15 mm and 20–22 μm respectively. Paper hand sheets, made with bleached pulps (45° SR) obtained from the five plant species, gave burst indices 2.75–3.92 kPa m^2/g, tear indices 3.73–9.61 m N m^2/g and tensile indices 37.27–47.81 N mg^{-1}, indicating adequate strength properties for writing and printing type of paper.

Introduction

A global shortage of conventional pulpable raw material is visualised by the turn of this century (Anon, 1989). Search for new fibre crops has been underway during last three decades or so, to find and develop new fibre supplies for paper and cellulose industries. The most promising amongst the fibre bearing plants, some of which have already proven to be ideal for pulp and paper making, is the group of fast growing plant species. Many fast growing annual and perennial plants, have been identified, cultivated and studied for their suitability as alternative source of raw material for paper industry (Nelson et al. 1966; Cunningham et al. 1970; Watson et al. 1976; Cunningham et al. 1978; Zarges et al. 1980; Mohanrao et al. 1982). Development of scientific cultivation technology for raising some of these fast growing annual and perennial plants, to give wood production, has helped to a great extent to fill the gap between demand and supply of pulpable

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raw materials. Paper industry in India, is also facing a grave raw material crisis today. Depletion of forest areas in the country has badly hit the supply of fibrous raw material to the industry. Great importance has, therefore, been given to raise fast growing plant species. The potentiality of some of the fast growing plants for use as raw material for paper and cellulose industries has also been studied (Dutta, Fotedar 1979; Mohanrao et al. 1982; Kulkarni et al. 1985; Sarma et al. 1990; Saikia et al. 1991).

The annual plants like *Hibiscus sabdariffa*, *Crotalaria juncea*, *Tephrosia candida* and *Hibiscus cannabinus* can be grown in the temperate and tropical climates and these have long been cultivated in many parts of India. Under agro-climatic conditions of Assam, India, all these annual plants attain luxuriant growth. These annual plants have been cultivated in the experimental farm of Regional Research Laboratory (RRL) and experiments were conducted to determine various parameters of growth and biomass yields to optimise cultivation technology and silvicultural methods to get the best output under local conditions (Sarma 1990; R&D Report 1991). *Neyraudia reynaudiana* is one of the many varieties of reeds that grows in abundance in the forests of Northern Eastern part of India. It belongs to the family Poaceae and is a fast growing perennial grass, with thick, terete, smooth and shinning culms (Bor 1984).

The present work was undertaken to evaluate the suitability of the above plant materials as pulp and paper making raw materials.

Experimental

Raw materials

Fully matured annual plants were obtained from the experimental farm of RRL, Jorhat, Assam, India. The reed was collected from a forest near Jorhat. The plant materials were first cleaned and the sample along with bark was cut into 2.5 × 1.5 cm chips. The chips were air dried (moisture 12%) before use.

Evaluation of raw materials, pulp and paper sheets

Proximate chemical analyses

The proximate chemical analyses of the raw material were carried out as per TAPPI standard methods (Tappi, 1980). For this purpose, air dried chips were made into powder and screened through a 40 BS sieve. Powder (−40 and +80) was taken for analysis. The results are recorded in Table 1.

Pulping and bleaching

Kraft pulping was carried out in a 10 L electrically heated rotary stainless steel reactor, with temperature controlled system. The oven dry (O.D.) chips (1000 g) were charged into the digester along with required amount of chemical solution at a material to liquor ratio 1 : 4. The cooking was done with 16.5% active alkali ($NaOH + Na_2S$) on OD raw materials, keeping sulphidity at 20%. The chips were cooked at a controlled temperature of 163 ± 2 °C for 3 h (including 45 min to raise the temperature to maximum). After the cooking, the pulp was washed thoroughly with deionised water and the unbleached pulp thus obtained was analysed for characteristic properties (Table 1).

The unbleached pulp was bleached by using hypochlorite alkali extraction – hypochlorite (HEH) sequence, with approximately 5.50–6% total chlorine consumption, to yield pulp of 78–81% brightness.

Fibre morphology

The length (L), diameter (D), wall thickness (W) and lumen diameter (d) of well digested pulp fibre were determined under a Dokuval photomicroscope (JEOL, Japan) at different magnifications.

Brightness

The brightness of the bleached pulp sheets was determined by using an EIL Reflectance Spectrophotometer and the results were expressed on the basis of MgO = 100.

Infra-red (IR) spectra

The IR spectra of the bleached pulp fibres were collected in a Perkin Elmer 297 spectrometer from 4000 to 200 cm^{-1} with resolution of 2 cm^{-1} with a five times scan per sample. All spectra were measured with air as background and were background subtracted to make them directly comparable.

Scanning electron microscopy

Fibres were separated in the pulp samples and were mounted on specimen holders with the help of electroconductive tape. The samples were coated with gold in an Ion sputter coater (JFC 100, JEOL, Japan) in low vacuum with a layer of 150–200 nm thick. The observation was made in a JEOL, JSM-35M-35CF electron microscope at an accelerating potential of 15 KV. Micrographs were taken at this potential.

Papersheet formation

The unbleached and bleached pulps were beaten in a laboratory valley beater to 45° SR (Schopper Reigler) freeness at 1.57% consistency and standard sheets of 60 ± 1 gm^{-2} were made from the pulps in a British Standard laboratory hand sheet forming machine, followed by pressing and drying.

Table 1. Proximate chemical analysis of plant raw materials and evaluation of unbleached pulp properties (values in brackets)

Parameters (%)	Plant raw materials				
	H. cannabinus	*H. sabdariffa*	*T. candida*	*C. juncea*	*N. reynaudiana*
Solubility in					
Cold water	3.55	3.97	3.10	4.70	5.75
	(1.85)	(0.99)	(1.97)	(1.80)	(1.08)
Hot water	6.55	7.25	5.25	6.30	7.35
	(1.02)	(1.12)	(0.66)	(0.59)	(2.06)
1% Alkali	34.20	30.40	22.70	28.58	32.58
	(9.16)	(9.95)	(4.00)	(4.36)	(6.88)
Alcohol	7.85	4.80	5.01	3.20	2.64
benzene	(5.71)	(3.75)	(2.37)	(2.22)	(2.23)
Lignin	17.33	17.92	21.05	20.50	22.15
	(4.02)	(3.01)	(4.76)	(3.68)	(3.70)
Pentosan	18.10	18.35	19.57	17.80	20.65
	(14.52)	(17.44)	(14.45)	(14.89)	(15.90)
Cross & Bevan	59.50	58.40	52.22	56.70	62.45
Cellulose	(85.14)	(84.49)	(85.82)	(87.16)	(81.44)
Ash	2.25	1.15	1.30	1.85	2.35

Testing of paper sheets

The dried paper sheets were conditioned at 65% relative humidity at 27 °C for 2 h and then tested for various physical properties.

Results and discussion

Table 1 gives the results obtained in the characterization of the plant raw materials. The lignin and pentosan contents are found to be more in the reed plant in comparison to the annual plant species. So also, the cellulose contents. The cellulose contents of all the five species of plants were between 52.22–62.45%. The chemical compositions indicate their suitability as pulpable cellulosic materials.

The optimum kraft pulping conditions were fixed up after carrying out digestions at different conditions of chemical concentrations, temperature and time periods. The unbleached pulp yields from all the species were between 44.90 and 52.50% (Table 2). The results obtained in the evaluation of unbleached pulp characters are shown in brackets in Table 1. The lignin and pentosan contents and the solubility figures of the unbleached pulps are proportionately reduced in comparison to wood raw material. The cellulose contents of the pulps from all the plant raw materials vary from 81.44–87.16%, exhibiting suitability of the cellulose for paper making.

The characteristic morphological properties of the bleached pulp fibres are given in Table 3. *Hibiscus cannabinus* and *Hibiscus sabdariffa* have long fibres in comparison to other three plant fibres. The average fibre lengths (L), varied from 0.75 mm (*T. candida*) to 2.15 mm (*H. cannabinus*); the average fibre width (D) from 20.00 µm (*T. cannabinus*) to the 22.00 µm (*H. sabdariffa*); the average lumen diameter (d) from 12.70 µm (N. reynaudiana) to 17.10 µm (*H. cannabinus*) and the cell wall thickness (W) from 3.90 µm (*H. cannabinus*) to 4.75 µm (*T. candida*). Thus, the Runkel ratio (2 W/d) and the shape factor (D^2-d^2/D^2+d^2) varied from 0.47 (*N. reynaudiana*) to 0.63 (*T. candida*) and 0.23 (*H. cannabinus*) to 0.47 (*N. reynaudiana*) respectively. The fibre characteristics are collectively represented by Runkel ratio and shape factor. The paper formation and its strength properties are largely dependent on these properties. The results show that all the five species of plants are suitable for paper making. The classification of unbleached pulp fibres showed well-balanced fibre sizes as given in Table 4.

Table 2. Analytical data on unbleached and bleached pulps

Parameters	Pulp from plant raw materials				
	H. cannabinus	*H. sabdariffa*	*T. candida*	*C. juncea*	*N. reynaudiana*
Yield of unbleached pulp (%)	52.50	51.70	45.80	53.20	44.90
Kappa Number (%)	25.00	23.50	19.90	16.60	22.10
Yield of bleached pulp (%)	50.60	47.00	40.80	46.10	40.35
Brightness of bleached pulp (%)	81.70	80.50	78.90	80.10	80.00
CED Viscosity at 27° (Cp)	45.00	40.00	38.50	48.00	38.22
Degree of polymerization (DP) (average)	387	328	325	475	322

Table 3. Morphological properties of the plant fibres

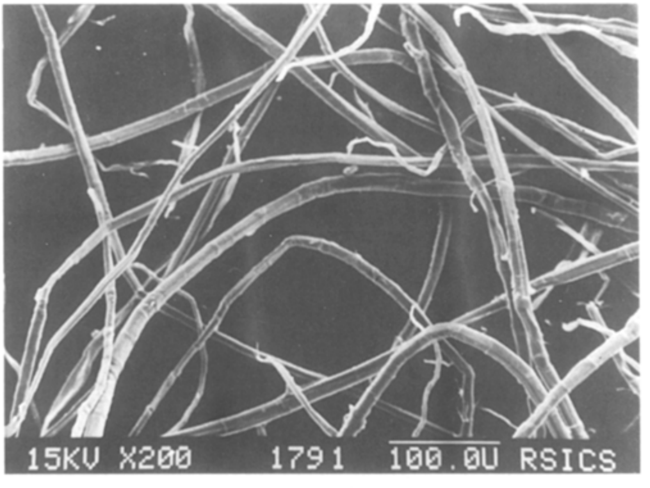
Properties	Plant raw materials									
	H. cannabinus		*H. sabdariffa*		*T. candida*		*C. juncea*		*N. reynaudiana*	
	I	II	I	II	I	II	I	II	I	II
Fibre length, L (mm)	3.80–0.50	2.15	3.70–0.45	2.00	1.50–0.60	0.75	3.30–0.60	1.45	1.70–0.45	1.00
Fibre width, D (μm)	25.20–18.00	21.60	25.00–12.50	22.00	23.00–12.00	20.00	23.00–15.00	21.00	22.60–10.50	21.10
Cell wall thickness W (μm)	3.90–6.60	3.90	3.50–5.00	4.20	3.20–5.12	4.75	3.12–5.25	4.00	2.82–4.48	4.15
Lumen diameter d (μm)	7.50–19.00	17.10	8.50–17.10	15.50	6.86–15.80	15.20	8.00–14.80	14.65	4.80–13.80	12.70
Slenderness ratio, L/D	–	99.54	–	90.91	–	37.50	–	69.05	–	47.40
Runkel ratio, 2 W/d	–	0.46	–	0.54	–	0.63	–	0.54	–	0.62
Flexibility Co-efficient, (d/D × 100)	–	79.17	–	70.45	–	76.00	–	69.76	–	60.19
Ratio of twice cell wall thickness to fibre diameter	–	0.36	–	0.38	–	0.47	–	0.38	–	0.39
Ratio of twice cell wall thickness to fibre width	–	0.23	–	0.27	–	0.31	–	0.27	–	0.33
Shape factor, $(D^2-d^2)/(D^2+d^2)$	–	0.23	–	0.33	–	0.27	–	0.35	–	0.47

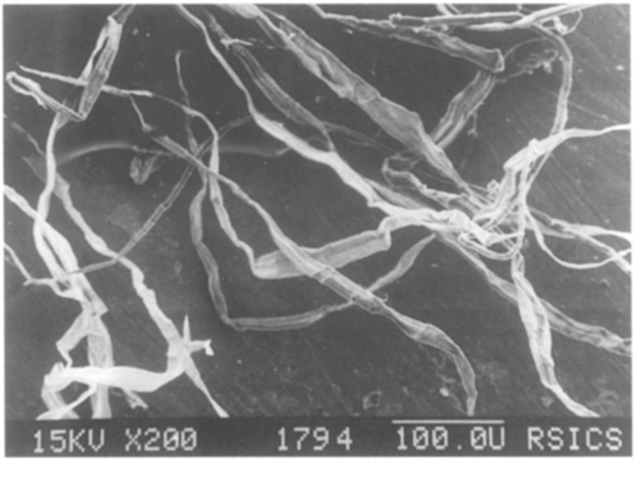
I = Range
II = Average value

Table 4. Bauer McNett Fibre classification

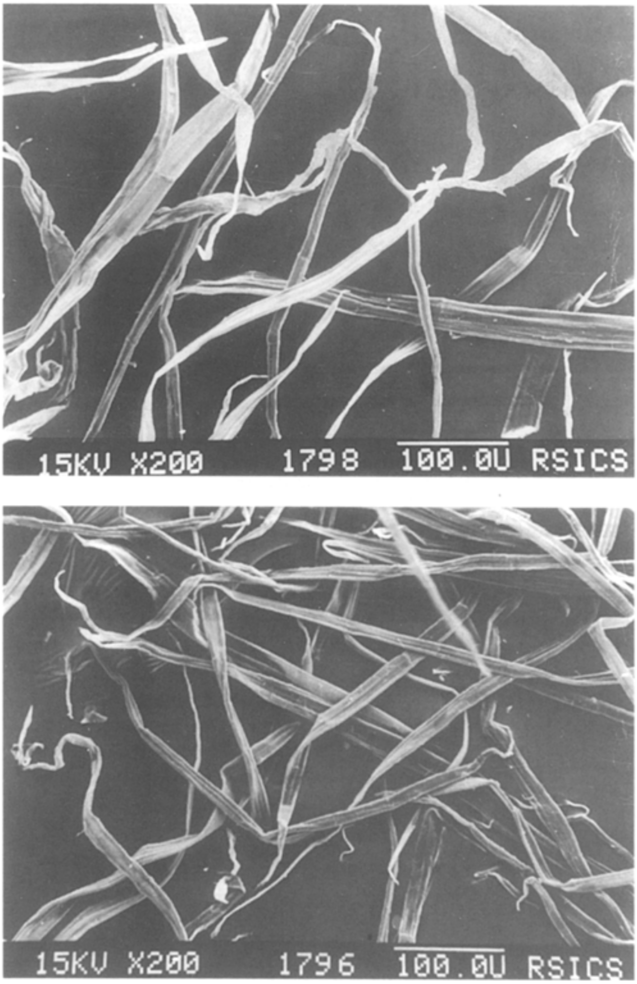
Mesh	Retention (%) Pulp from plant raw materials				
	H. cannabinus	*H. sabdariffa*	*C. juncea*	*T. candida*	*N. reynaudiana*
+14	44.50	46.00	35.50	8.60	6.20
−14,+30	20.75	18.70	22.60	7.80	8.10
−30,+50	11.80	10.20	7.80	47.90	45.60
−50,+100	3.85	5.10	3.50	26.30	22.70
−100	19.10	20.00	29.50	15.20	17.40

Figures 1–5 show the scanning electron micrographs of pulp fibres of H. *cannabinus* (1), *H. sabdariffa* (2), C. juncea (3), *T. candida* (4) and *N. reynaudiana* (5). Some of the fibres of H. cannabinus are long cylindrical with distinct nodes, whereas, some are though long and cylindrical, but smaller in diameter. The fibres





Figs. 1 and 2. SEM of pulp fibres of *H. sabdariffa* respectively. (×200)



Figs. 3 and 4. SEM of pulp fibres of *C. juncea* and *T. candida* respectively. (×200)

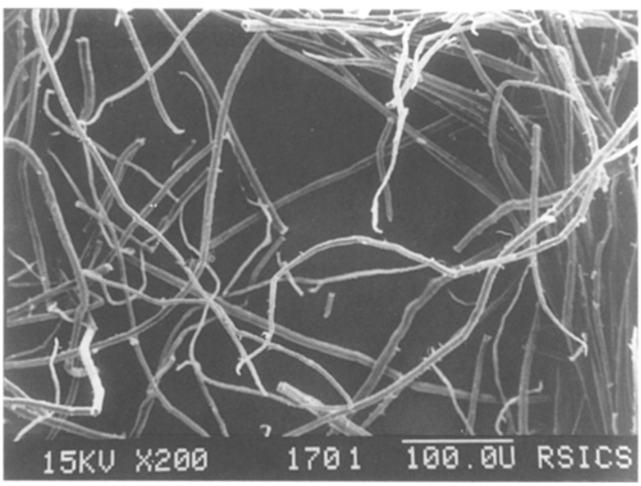


Fig. 5. SEM of pulp fibres of *N. reynaudiana* (×200)

of *H. sabdariffa* are flat, rosette like and wavy, comparatively larger in diameter than that of *H. cannabinus* fibres. *C. juncea* has two types of fibres - some are flat and large in size and others are small and cylindrical. The fibres of T. candida are though flat but uniform in size with no distinct nodes, whereas, the fibres of *N. reynaudiana* are narrow cylindrical and are mostly of uniform size.

The Infra-red spectra of bleached pulps from all the five species of plant and that of bleached bamboo pulp were found to be identical. The region from 4000 to 1900 cm^{-1} would be largely responsible for the O—H and aliphetic C—H stretching frequencies (Liang, Marchessault 1959a; Higgins et al. 1961; Sao et al. 1987; Stewart, Morrision 1992). The strong band at 1640 cm^{-1} was due to vibration of adsorbed water molecules in the non-crystalline region of the cellulose (Liang, Marchessault 1959a; Williams, Fleming 1976). The region between 1530 and 670 cm^{-1} represented carbon-hydrogen vibrational modes of the glucose unit of the cellulose chain. The bands at 1365 cm^{-1} and 1315 cm^{-1} were due to CH deformation and CH_2 wagging frequencies and attributed to the xylan fractions in cellulose (Liang, Marchessault 1959b; Pray 1972).

The physical strength properties of paper sheets, made out of unbleached and bleached pulps that were beaten to 45°SR are shown in Table 5. The data obtained for bulk densities (1.42–1.58 c/g), burst indices (3.82–5.19 kPa m^2/g^{-1}), tear indices (4.41–10.79 m N m^2/g^{-1}), tensile indices (51.68–63.26 N mg^{-1}) and double folds (210–420) of the unbleached pulp sheets made from the pulp obtained from all the five species of plant, are found to be more or less comparable, although pulp sheets made from *Crotalaria juncea* and *Hibiscus cannabinus* show higher values of physical strength properties. So also, the physical strength properties of pulp sheets made from bleached pulp of all the five plant species exhibit that paper with adequate physical strength properties can be produced.

It can, therefore, be concluded that the plant species under investigation would be ideal sources of raw material for pulp & paper industry.

Table 5. Physical strength properties of paper sheets made from pulp obtained from different plant raw materials

Plant raw materials	Bulk density (cc/g)	Burst index (kPa m^2/g^{-1})	Tear index (m N m^2/g)	Tensile index (N mg^{-1})	Folding endurance (double folds)
Hibiscus cannabinus					
Ubps	1.58	5.19	10.79	60.31	350
Bps	1.42	3.43	5.88	37.27	110
Hibiscus sabdariffa					
Ubps	1.52	4.90	11.77	62.03	320
Bps	1.38	3.73	6.37	42.17	120
Crotalaria juncea					
Ubps	1.47	5.09	10.79	63.26	420
Bps	1.39	3.92	9.61	47.81	255
Tephrosia candida					
Ubps	1.44	4.71	5.69	57.62	300
Bps	1.35	3.43	4.32	40.45	150
Neyraudia reynaudiana					
Ubps	1.42	3.82	4.41	51.68	210
Bps	1.38	2.75	3.73	38.15	120

Ubps = Unbleached pulp sheet
Bps = Bleached pulp sheet

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