EFFECT OF HARVESTING AGE ON THE CHEMICAL AND MORPHOLOGICAL PROPERTIES OF DHAINCHA (*SESBANIA ACULEATA*) AND ITS PULPABILITY AND BLEACHABILITY

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Dhaincha (*Sesbania aculeata*) is one of the annual plants in Bangladesh that has potential as a fiber source. It is a crop generally cultivated for its nutritive value to soil. This paper describes the effect of harvesting age of dhaincha on chemical, morphological, pulp, and papermaking properties and its bleachability. Dhaincha is a short length fiber that can be used as a substitute to hardwood. Fiber length was found to increase slightly with increasing age of the plant. The alpha-cellulose content in dhaincha increased and pentosan decreased with increasing age. Dhaincha pulp was prepared from 2, 3, 4, 5, and 6 month old plants under identical cooking conditions. Pulp yield (43-45 %) and kappa number (26-30) did not follow direct correlation with plant age. But physical properties of unbleached pulp increased linearly with the increase of age. Bleachability of dhaincha pulp was quite poor. The bleachability of pulp was improved when the age of dhaincha was increased.

Keywords: Sesbania aculeate; Harvesting age; Chemical properties; Pulping; Bleaching

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INTRODUCTION

A large amount of the paper produced today in developing countries is derived from various annual non-wood fiber sources, where adequate forest resources are lacking. Despite this, there are still some potentially important annual plants that have not been utilized on a commercial scale by the paper industry.

Forests are declining at the alarming rate of 13.0 million hectares per year in developing countries (Anon. 1997). Population growth has increased dramatically since 1960, adding 1 billion people per 15 years (Hartmann 1998), and the world population could double to 12 billion by 2075 (FAO 1999). Rising population, better literacy, improving communication, and industrialization in developing countries are expected to increase the demand for paper and paperboards by 4.3 % per annum as compared to 1.2% in developed countries (Anon. 1997). New legislative regulations endorsed in response to the demand of environmentalists, environmental groups, and nongovernmental organizations (NGOs) in various countries are restricting the logging of trees, which is expected to affect the supply and price of wood to the international pulp and paper industry (Anon. 1996). To meet these changes, a variety of fiber resources will be required to meet projected demand, including fast growth wood plantations, increased recycling, and non-wood plant fibers from crop residues, as well as fiber crops.

In this paper we will consider *Sesbania aculeate* as a pulping raw material, which is not commercially utilized yet. *Sesbania aculeate* is commonly known as Dhaincha in Bengali. It is a shrub and belongs to the family *Leguminoceae* that helps in fixing of atmospheric nitrogen with its root nodules. About 18 MT of *Sesbania aculeate* ploughed in 1 ha yield about 77 kg of nitrogen (Anon. 1950). In our previous investigation it was observed that dhaincha is a potential raw material for kraft linear paper pulp (Jahan et al. 2007).

It is generally recognized that harvesting age affects the fiber properties, pulp yield, and quality (Jahan and Mun 2004). The effect of tree age on wood properties varies with wood species (Orme et al. 1996). Growth site is also known to affect wood properties.

The objectives of this study were to quantify the effects of plant age (a) on the yield obtained throughout 6 months, (b) on the chemical composition changes of the raw material during that period, (c) on the physico-chemical characteristics of the pulp and paper obtained, and (d) on the bleachability of the produced pulp. The kraft process showed better results in dhaincha pulping than the soda-AQ process (Jahan et al. 2007), so pulping was carried out by the kraft process in this study.

MATERIALS AND METHODS

Materials

Dhaincha were collected from the BCSIR agricultural research field, Dhaka after one month intervals. Dhaincha was cut to 2-3 cm in length, and ground (40/60 mesh) in a Wiley mill for chemical analysis.

For the measurements of fiber length, the sample was collected from the middle part of the plant and macerated in a solution containing 1:1 HNO_3 and $KClO_3$. A drop of macerated sample was taken on a slide, and fiber length was measured under a profile projector (Nikon V-12, Japan). Fiber width was determined from the cross section photograph taken in an image analyzer using software.

Chemical Analysis

The extractives (T204 om88), Klason lignin (T211 om 83), and pentosan (T223 cm-84) were determined in accordance with TAPPI Test Methods. Holocellulose was determined by treating extractive free wood meal with $NaClO_2$ solution. The pH of the solution was maintained at 4 by adding CH_3COOH-CH_3COONa buffer. Alfa cellulose content was then determined by treating holocellulose by 17.5% NaOH.

Alkaline nitrobenzene oxidation of dhaincha meal was carried out according to Mun's modified method (Mun and Wi 1991). GC analysis was conducted using a Shimatzu GC 17A gas chromatograph equipped with a CBP1 capillary column (25 m x 0.25 mm). Conditions used were as follows: column temperature was programmed to increase from 150 to 270 ^oC at the rate of 5 ^oC/min; injection and detection temperature were 220 and 270 ^oC, respectively; column flow was rate 1ml/min; and split ratio was 20.

Pulping

Kraft pulping was done in a 5 L capacity thermostatically controlled electrically heated rotary digester under constant cooking conditions. The active alkali and sulphidity were constant at 18% and 25 %, respectively as Na_2O on oven dried (o.d.) sample in the liquor ratio of 1:6. The cooking was continued for 120 min at the maximum temperature (170 ^{o}C). At the end of pulping, pressure was relieved to atmospheric level, and pulp was taken out from the digester, disintegrated, and washed by continuous flow of water. Pulp was screened in a Yasuda flat vibratory screener yield and reject determined gravimetrically. Pulp yield was determined as dry matter obtained on the basis of o. d. raw material. Kappa number was determined in accordance with T 236 cm-85. Three replicates were carried out for each experiment.

All pulps were beaten in a PFI mill to 3000 revolution, and handsheets of about 60 g/m^2 were made in a Rapid Kothen Sheet Making Machine according to German Standard Methods DIN 106. The physical properties of handsheets were determined by the method T 220 sp-96.

D_oEpD_1 Bleaching

All dhaincha pulps were bleached by $D_oE_pD_1$ bleaching sequences (where D represents chlorine dioxide and E_p represents peroxide reinforced alkaline extraction). The kappa factor was 0.22 in the first stage of $DoEpD_1$ bleaching sequences. The temperature was 70 ^{o}C in Do stage for 60 min. Pulp consistency was 5%. The pH was adjusted to 2.5 by adding dilute H_2SO_4. In the alkaline extraction stage, temperature was 70^{o}C for 60 min in a water solution of 2% NaOH and 0.5% H_2O_2 (on oven-dry pulp). Pulp consistency was 10 %. In the final D_1 stage, pH was adjusted to 4 by adding dilute H_2SO_4. The ClO_2 charge in the D_1 stage was half of first stage. Pulp consistency in D_1 was also 5%. After each of the stages pulp was washed with deionized water. Pulp brightness was determined according to the TAPPI standard method T272. The physical properties of the bleached pulps were determined after 3000 revolutions in a PFI mill.

RESULTS AND DISCUSSION

Biomass and Morphology

Figure 1 shows the air-dried biomass production (Stem) of dhaincha harvesting at 2, 3, 4, 5, and 6 months. The increase in biomass of dhaincha was negligible after 5 months. The biomass was increased to 0.59 kg/m^2 at 5 months age from 0.51 kg/m^2 at 2 months age. The biomass produced by dhaincha on an annual basis is higher than wood but lower than hemp (Kamat et al. 2002). Although classified as an annual plant, dhaincha at its mature stage has a structure similar to that of conventional hardwoods, with a woody stem, which is covered with bark. The bark consists of a thin layer of bast fiber (Fig. 2).

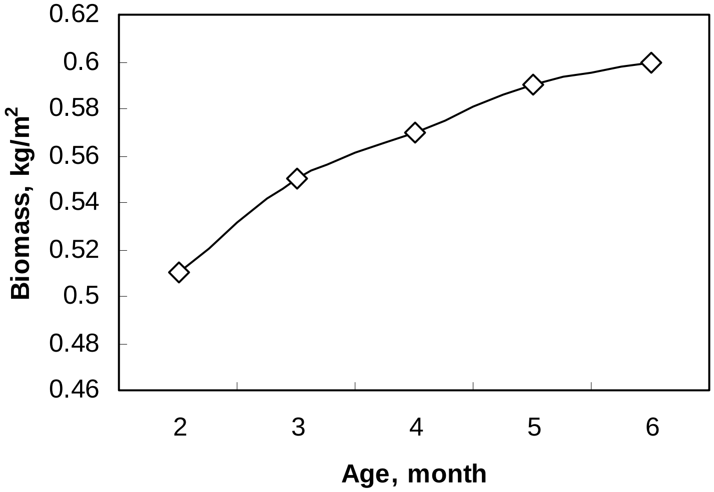

Fig. 1. Effect of plant age on biomass production

Table 1. Fiber Dimensions of Dhaincha at Different Ages

Age of plant	2 months	3 months	4 months	5 months	6 months
Fiber length, mm	0.98	1.15	1.19	1.34	1.34
Fiber width, µm	18.6	18.9	18.5	19.1	18.3
Slender ratio	52.7	60.8	64.3	70.1	73.2

Table 1 shows the fiber dimensions of dhaincha harvested at different ages. Fiber length is one of the most important parameters from the papermaking point of view. Fiber length is increased with the increase of plant harvesting age. It is increased from 0.98 to 1.34 mm with the increase of harvesting age from 2 months to 6 months. It is also positively correlated with harvesting age (R = 0.9181). Dhaincha has a relatively low slenderness ratio, in comparison to wood, and it is increased with plant age, which in turn reduces tearing resistance dramatically. Short and thick fibers do not produce good surface contact and fiber-to-fiber bonding (Ogbonnaya 1997).

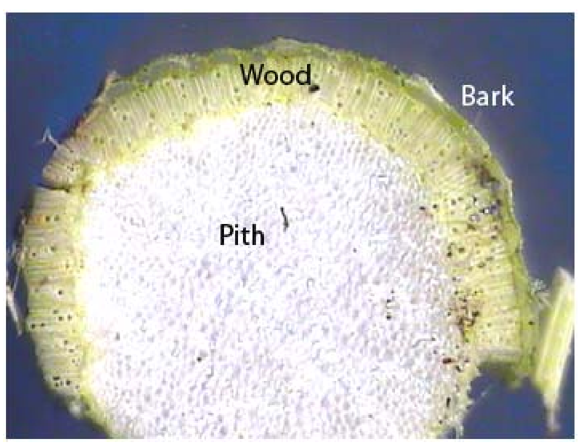

Fig. 2. Cross section of dhaincha plant

Chemical Properties

Chemical analysis and evaluation of fiber properties of the dhaincha were used to assess the dhaincha's potential for substitution of woody raw materials used in pulp and paper production. Table 2 summarizes the extractives content of dhaincha at different ages. Extractives dissolved in 1% alkali declined significantly from the 2nd month to the 6th month. Acetone extract also decreased from month 2 to 5. This reduction in extractive content with the age of the plants was expected some extent. The extractives content in the hemp also declined significantly from 30-day plants to 120-day old plants (Kamat et al. 2002). Reduction in the extractive content with age has also been reported for bamboo and kenaf (Rowell et al. 1997). Lopez et al (2008) also showed that extractives of Leucaena species in hot water and 1% alkali decreased from year 1 to year 2. The very high amount of extractives observed in 2-month plants could possibly be attributed to the large amounts of proteins and chlorophyll that were also extracted during the extraction process. Extractives obtained in this experiment were higher than what was observed by Dutta et al. (2004) for *Sesbania sesban*, which might affect pulp yield.

Table 2. Chemical Properties of Dhaincha at Different Ages

Age of plant	2 months	3 months	4 months	5 months	6 months
Extractives, %					
Cold water	8.3	10.1	8.4	7.5	7.5
Hot water	12.8	9.4	12.2	11.5	11.0
Acetone	4.8	4.2	1.3	0.70	1.6
1 % alkali	43.6	30.2	36.9	36.7	35.2
Lignin %	23.0	23.0	22.9	22.2	21.9
Hollocellulose, %	70.0	70.4	70.3	72.3	72.8
α-cellulose, %	34.3	35.0	34.9	35.7	37.9
Pentosan, %	19.5	19.5	19.6	19.4	19.7
Ash, %	3.4	3.7	3.5	3.6	3.8

The percentage of holocellulose, α-cellulose, lignin, and pentosan on extractive free dhaincha are also shown in Table 2. Holocellulose and α-cellulose content were increased with the increase of plant age. The α-cellulose in dhaincha was 34-37%, which is lower than hardwood (Jahan and Mun 2003; Jahan and Mun 2004) and almost similar to straw (Han 1998). Fang and Yang (2003) observed an increasing trend of cellulose content in poplar clone with cambial age. Lignin exhibited slight or no variation as a function of growth. In the first 3 months, there was no change of lignin, but over the 6 months of growth it decreased by 1 %. Miranda and Pereira (2002) observed that the lignin content in *Eucalyptus globulus* decreased with tree age in both irrigated and fertilized trees. Lignin content in dhaincha in this investigation is almost similar to that observed by Jackson et al (1997) in *Sesbania bispinosa*. Pentosan content did not vary significantly with plant age; it was about 19.5 %, which is similar to *Sesbania sesban*, as observed by Datta (2004). The ash content was 3.4-3.8 %, which is much higher than usual for tropical species (1-3 %) (Kristova et al. 1997).

Alkaline Nitrobenzene Oxidation

Table 3 shows the yield of alkaline nitrobenzene oxidation products obtained from different ages plant meals. In Table 3 the predominant product is identified as syringaldehyde (S), which comprised 19-21% of lignin. It resulted from the degradation of noncondensed syringyl units. Vanillin (V) appeared as the second major degradation product, resulting from the noncondensed guaiacyl unit. The relative ratio of S to V was 2. The results appeared to be in general agreement with the range of S to V ratios obtained from hardwood lignin (Creighton et al 1940). The yield of *p*-hydroxy-benzaldehyde in dhaincha lignin was negligible. The yield of total oxidation products increased with plant age, suggesting that the noncondensed structural units are increased with the maturity of plant. Morrision et al. (1999) showed that the total amounts of phenolics were slightly lower in the top portion of each cultivar.

Table 3. Alkaline Nitrobenzene Oxidation Products of Dhaincha at Different Ages

Age, month	Aldehyde, %				
	P	V	S	Total	S/V (molar ratio)
2	0.5	9.8	19.3	29.1	2.0
3	0.5	10.1	20.2	30.8	2.0
4	0.3	10.4	20.8	31.5	2.0
5	0.4	10.5	20.9	31.8	2.0
6	0.1	10.8	21.4	32.2	2.0

P= *p*-hydroxy benzaldehyde; V= vanillin; S= syringyldehyde

Pulping

The pulp yield and kappa number of 2 to 6 month old dhaincha were 42-45 % and 26-30, respectively in 18% active alkali and 2 h cooking at 170 ^{o}C (Table 4). The 5 month old dhaincha gave the highest and 6 month old the lowest pulp yield. The 6 month old dhaincha plant had the highest reject, while other samples had almost no reject. Dutt et al. (2004) observed 49.8 % pulp yield with kappa number 30.4 in kraft pulping of *Sesbania aculeate*, while *Sesbania sesban* showed 47.0 pulp yield with kappa number 31.0 (Dutt et al. 2004a). The lower pulp yield of dhaincha in this investigation possibly comes from the low α-cellulose level (Table 2). There was no trend observed in pulp yield with plant age. Duke et al. (2003) observed that mimosa plant gave 0.8 % higher pulp yield and lower kappa number in 2 year plants than 1 year plants under similar cooking conditions.

Table 4. Pulp Yield and Kappa Number of Dhaincha at Different Ages

Plant Age	Screened pulp yield, %	Reject	Kappa number	Brightness
2	43.1±0.8	0	30.0	15.3
3	43.0±0.7	0	27.8	16.0
4	43.4±0.7	0.2	25.8	21.4
5	44.7±0.9	0	26.9	21.4
6	42.1±0.6	0.6	27.0	19.6

The unbleached pulps were beaten in a PFI mill with 3000 revolutions, and handsheets were prepared for physical properties. The drainage resistance (^{o}SR) was about 45. All physical properties increased linearly with plant age (Figs. 3-6). The correlation coefficient (R) was about 90. Best physical properties were obtained with 5 months old dhaincha, where tensile, tear, burst index, and fold number were 89 N.mg^{-1}, 13.7 mN·m^{2}g^{-1}, 7.6 kPa·m^{2}g^{-1}, and 4648, respectively. These data indicate that dhaincha gives finer and more comfortable fibers. Mimosa plant pulp also showed better physical properties in mature plants (Duke et al. 2003).

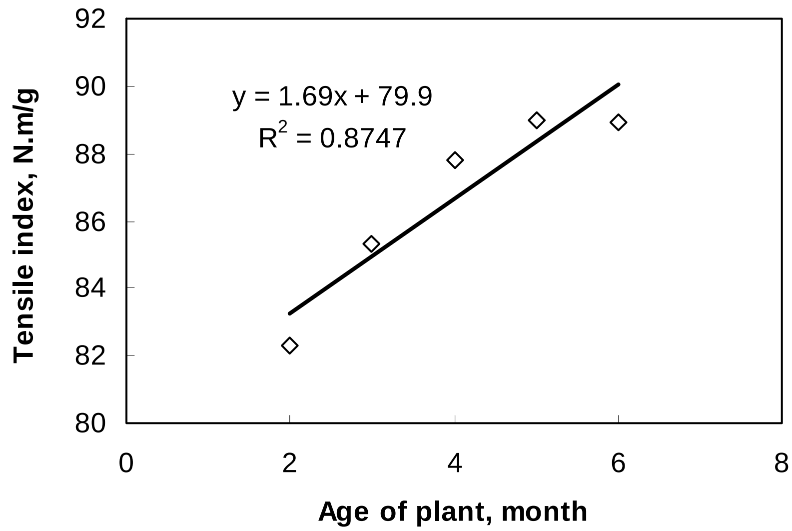

Fig. 3. Effect of harvesting age on tensile index of dhaincha pulp

Fig. 4. Effect of harvesting age on tear index of dhaincha pulp

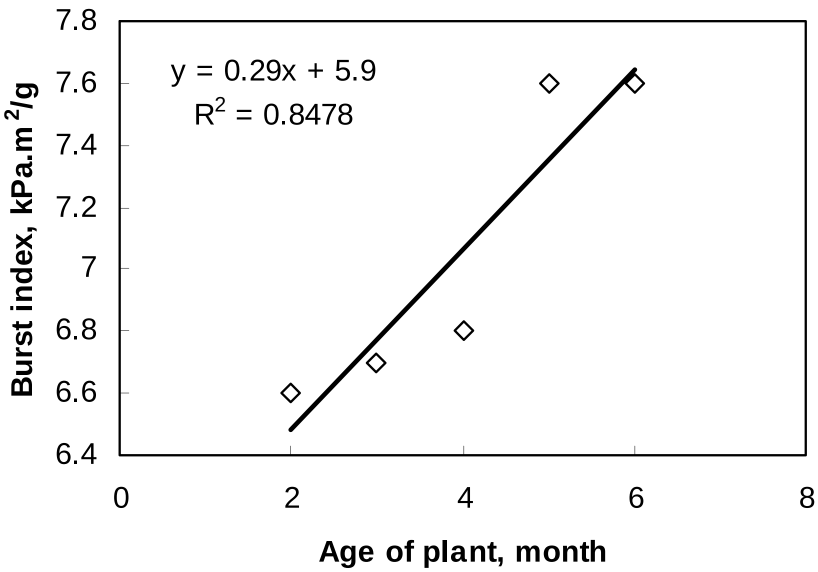

Fig. 5. Effect of harvesting age on burst index of dhaincha pulp

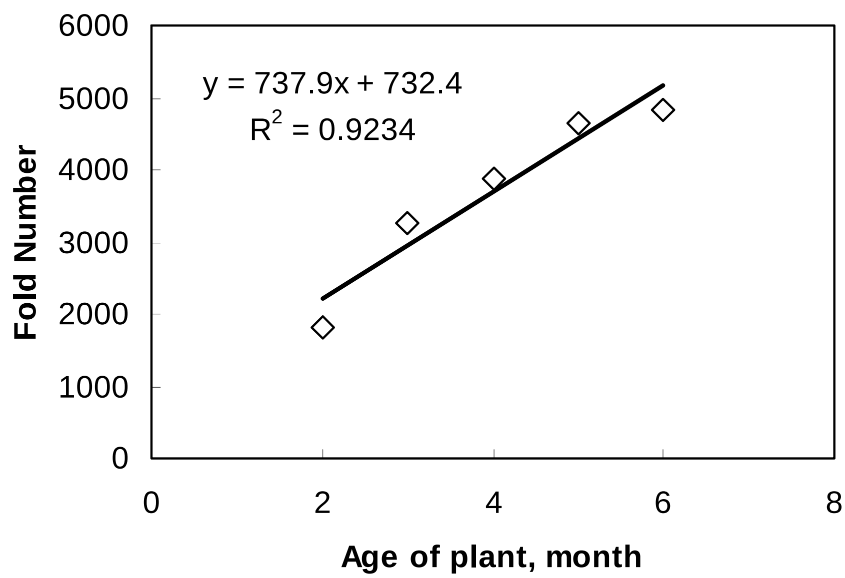

Fig. 6. Effect of harvesting age on double fold number of dhaincha pulp

Bleaching

The dhaincha pulps were bleached by $D_oE_pD_1$ bleaching sequences, and results for bleached pulp are shown in Table 5. Unbleached pulp from 5 and 6 months old dhaincha was easier to bleach than that of younger plants. After the final bleaching stage, the 5 and 6 month plants displayed similar brightness (79 %), while 2, 3, and 4 month plants showed 77 % brightness (Table 5). In CEHH bleaching, Dutt et al. (2004) obtained only 71% brightness in kraft pulp from *Sesbania aculeate*, while *Sesbania sesban* gave 69.7% brightness. In our earlier investigation we obtained almost similar bleaching potential in ODED bleaching sequences (Jahan et al. 2007). Bleached pulps were beaten in a PFI mill with 3000 revolutions, and handsheets were prepared for physical properties. Dhaincha of all ages plants showed excellent physical properties. There was no direct relationship between physical properties (except tear index) and plant age. The 3 month old plants gave slightly better tensile and burst index as compared to other aged plants. Tear index showed a slightly increasing trend with plant age. It increased from 9.4 $mN.m^2/g$ at 2 months to 10.5 $mN.m^2/g$ for 6 months plants.

Table 5. Effect of Plant Age on the Physical Properties of Bleached Dhaincha Pulp

Plant Age	^oSR	Tensile index $N{\cdot}mg^{-1}$	Tear index $mN{\cdot}m^2g^{-1}$	Burst index $kPa{\cdot}m^2g^{-1}$	Fold Number	Density gml^{-1}	Brightness TAPPI %
2	48	68.9	9.4	5.5	2187	0.513	76.8
3	49	72.5	9.8	6.6	3120	0.533	77.3
4	47	70.2	10.3	5.2	3390	0.532	77.2
5	49	70.8	10.2	5.5	3198	0.549	79.3
6	48	69.4	10.5	5.6	2726	0.499	79.7

CONCLUSIONS

Extractives content in dhaincha decreased and α-cellulose increased with increasing harvesting age. Alkaline nitrobenzene oxidation products showed that the dhaincha lignin was composed of syringyl and guaiacyl units. Total aldehyde yield increased with harvesting age, but syringyl to guaiacyl ratio remained constant. Pulp yield did not follow any relationship with harvesting age, but kappa numbers of pulp decreased with the increase of age. The physical properties of unbleached pulp showed a linear relationship with harvesting age. The bleachability of dhaincha pulp was improved with harvesting age. The best pulping results were obtained when harvesting the dhaincha at the age of 5 months.

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