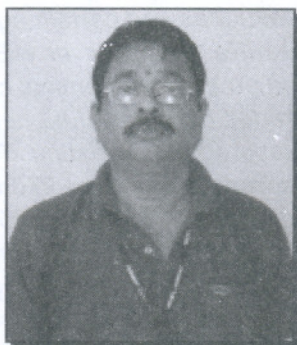
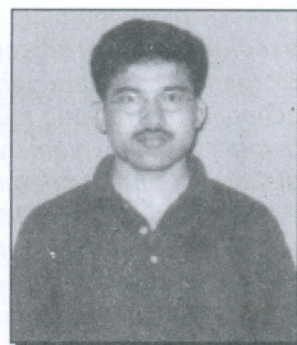


Utilization of certain forest weed and agro-waste materials for pulp and paper making



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About the Authors

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Abstract

Fibre qualities and pulp and paper making properties of certain forest weed species viz *Clynogynae dichotoma*, *Alpinea allughas* and certain agro waste material such as *Hibiscus esculentus* and *Hibiscus sabdariffa* available in NE region have been investigated. Morphological characteristics and proximate chemical constituents of the plants show their suitability for making suitable grades of paper of higher physical strength properties. The heights of all the plants were measured at harvesting. The height recorded for *Clynogynae dichotoma* and *Alpinea allughas* were 315cm and 270 cm. with stem diameter 3.5cm and 2.87cm respectively. The height and stem diameter recorded for *H. esculentus* were 250cm and 3.5cm and for *H. sabdariffa* were 275cm and 1.8 cm. The important plant constituents like cellulose contents were varied from 51.5-62.0%, lignin 13.5-20.2% & pentosan 15.3-17.6%. The pulping of the plant materials were conducted at a stainless steel rotary digester using 8 – 12 % cooking chemicals at $135 \pm 5^\circ\text{C}$ maintaining bath ratio at 1:6. A series of experiments were conducted to optimize the cooking chemicals to get optimum pulp yield.

The unbleached and bleached pulp yield were determined and found 48.3-54.8% and 39.4-43.8% respectively in all the four species. The paper properties made out of these plants showed higher physical strength properties with tensile index value varied from 39.8-48.7N/mg, burst index 3.35-4.2 Kpam²/g, tear index 6.4-8.8mNm²/g and double fold no. 120+ to 250+. Hence, these waste plant materials may be an alternative source of fibre for pulp paper and paper board industry.

Introduction:

In recent years, due to the rapid growth of population and industry, the forest areas of our country are gradually diminishing and also at the same time the supply of plant materials from the forest to the industry is decreasing at an alarming rate resulting in a huge shortage of raw materials for various forests and cellulosic based industries¹. Therefore, in recent years much attention has been given on utilization of alternate fibrous raw material mostly non woody plants to use in these industries². The plant resources of North Eastern region of India are enormous and unique owing to varied topography and wide climatic conditions. There are many varieties of wild plant available in the forests of this region having potentiality to use as raw material for different forest based industries³. Studies on possible utilization of some of these plants for making cordages and yarns have been made^{4, 5}. Among different weed species *Clynogynae dichotoma*, and *Alpinea allughas* are abundantly available weeds growing in the low marshy land of whole NE region and still not exploited for any commercial utilization. There are also some cultivated plant species available in this region contain good quality fibres. *Hibiscus esculentus* (Bhindi plant), and *Hibiscus sabdariffa* (Tengamora plant), are widely cultivated in this region for vegetables and fruits. These agricultural crops are seasonal and after harvesting the crops, the whole plant remains waste in the field. Studies in respect of bio-mass production of certain cultivated species of non-woody plants have been studied earlier at NEIST, Jorhat⁶. Considering the availability and high quality fibre bearing properties of such bio waste materials, an investigation was carried out in the laboratory to study the fibre characteristics and pulp and paper making properties of these plants in the laboratory scale and the results of the investigation are presented in this communication.

Materials and Methods:

Materials:

For the present study, two forest weed species viz. *Clynogynae dichotoma*, *Alpinea allughas*, and two cultivated plants viz. *Hibiscus esculentus* and *Hibiscus sabdariffa* were selected. Matured plants of all the four species were collected from different places near by Jorhat (26.47°N latitude, 94.12 °E longitude and 87m above the sea level), Assam, India. The plants materials were first cleaned by washing with fresh water and then removed the leaves, roots and the tip portion of the stem. The stem portion of the plants was only considered for the study.

Methods:

Morphological Characters

The morphological characters of the plants such as plant height, diameter, weight etc. were determined in the field as well as in laboratory. The morphological characters of the plants are presented in Table 1.

Table 1: Morphological characters of the plants

| Particulars | Plant material | | | |
|--|-----------------------------|-------------------------|----------------------------|----------------------------|
| | <i>Clynogynae dichotoma</i> | <i>Alpinea allughas</i> | <i>Hibiscus esculentus</i> | <i>Hibiscus sabdariffa</i> |
| Height of the plant (Avg), cm | 315 | 270 | 250 | 275 |
| Diameter of the plant (Avg), cm | 3.5 | 2.87 | 3.5 | 1.8 |
| Length of the Pseudostem (m) | 3.15 | 2.70 | 2.30 | 3.20 |
| No. of leaves | 10-12 | 5-8 | 10-15 | Numerous |
| No. of sheath | - | 12 | - | - |
| Diameter of the Core, (cm) | 1.5 | - | 1.45 | 0.98 |
| Moisture content, Av % | | | | |
| Bark | 72.5 | - | 74.8 | 75.8 |
| Wood | - | - | 73.9 | 74.2 |
| Sheath | 83 | - | - | - |
| Pith / Core | 70.2 | 84.3 | 74.6 | 74.6 |
| Whole plant | 70.5 | 82.0 | 75.0 | 75.0 |
| Wood : Bark | - | - | 76 : 24 | 78 : 22 |
| Bark thickness | 0.68 | - | 0.85 | 0.72 |
| Specific gravity | - | - | - | - |
| Bark | 0.31 | 0.26 | 0.25 | 0.30 |
| Core | 0.34 | 0.28 | 0.29 | 0.34 |
| Average plant constituents (on OD basis) | | | | |
| Sheath | - | 45 | - | - |
| Central | 79.5 | 45 | - | 10 |
| Core/Pith | | | | |
| Leaves & twigs | 20.5 | 10 | 20 | 25 |
| Bark | - | - | 20 | 15 |
| Wood | - | - | 60 | 50 |

Proximate Chemical Analyses

The proximate chemical analyses of the plant materials were carried out as per TAPPI Standard methods⁷. The plants were converted to chips of 2 mm size and then oven dried. The chips were then powdered in a Wiley mill. The materials that pass through the 40 BS and retained on 60 BS mesh were considered for proximate chemical analysis. The results of the proximate chemical constituents of the plants are given in Table 2.

Table 2: Proximate chemical analysis of the plant

| Properties | C. dichotoma | A. allughas | H. esculentus | H. sabdariffa |
|-----------------------------|-----------------|-------------|------------------|------------------|
| Solubility in % | | | | |
| Cold water | 2.98 | 10.4 | 4.7 | 3.95 |
| Hot water | 6.93 | 13.2 | 7.6 | 7.2 |
| 1 % NaOH | 33.1 | 39.4 | 22.6 | 23.75 |
| Alcohol benzene | 3.45 | 7.3 | 10.2 | 4.82 |
| Pantosan % | 15.8 | 15.3 | 17.6 | 16.5 |
| Lignin % | 20.2 | 13.5 | 14.6 | 17.5 |
| Cellulose (Cross & Bevan) % | 53.8 | 51.5 | 61.3 | 62.0 |
| Ash content % | 5.7 | 5.1 | 0.92 | 1.15 |
| Alpha cellulose % | 66 | 78 | 63.4 | 65.7 |

Pulping and Bleaching:

For preparation of pulp, plant materials were converted to chips of 3 cm size and digested by kraft pulping. The pulping was carried out in an electrically heated rotary stainless steel digester having temperature control device. The chips (1000g) were charged in to the digester in each batch with required amount of chemical maintaining bath ratio fixed at 1:4. The cooking was conducted by varying chemical concentration from 10-16% on OD basic of raw material keeping sulphidity at 20 %. The chemical used for cooking was NaOH and Na₂S. Separate experiments were also conducted for optimization of temperature and time of cooking for each individual plant species. After cooking the pulps were washed thoroughly with deionised water and air dried and the unbleached pulp yield was determined. The cooking chemical requirement to get higher pulp yield for each individual is represented in Figure 1.

The bleaching of the pulps was done by two steps hypochlorite with an intermediate alkali extraction (H-E-H) sequence. The brightness of the pulps were determined by using an EIL Reflectance spectrometer and the results were expressed in terms of MgO=100. The physical properties of both bleached and unbleached pulp were presented in Table 3.

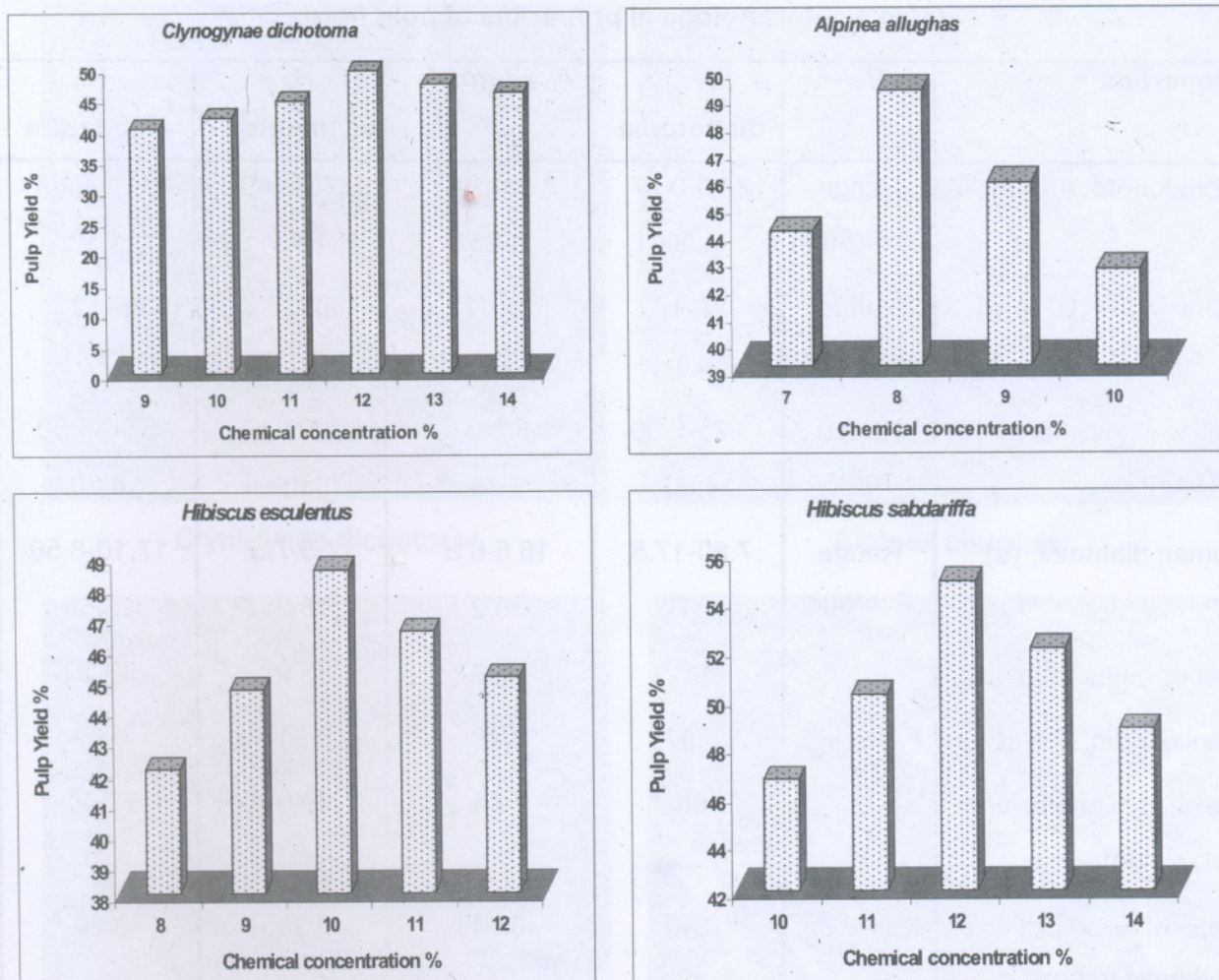


Fig 1: Effect of chemical concentrations on pulp yield (unbleached)

Fibre morphology

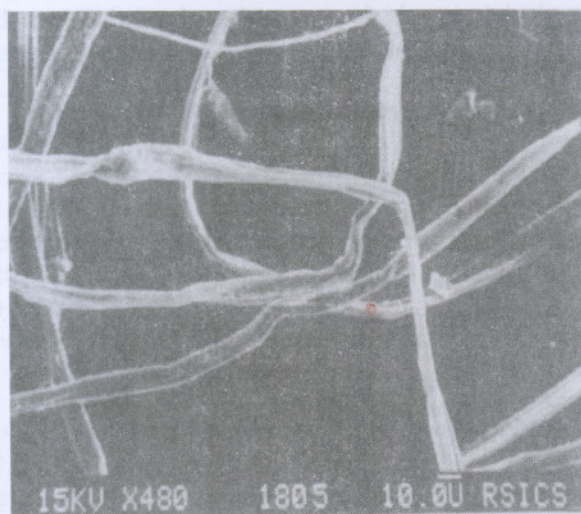
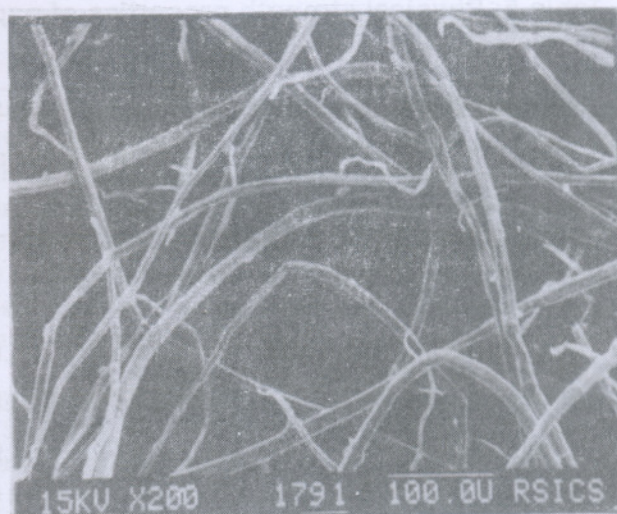
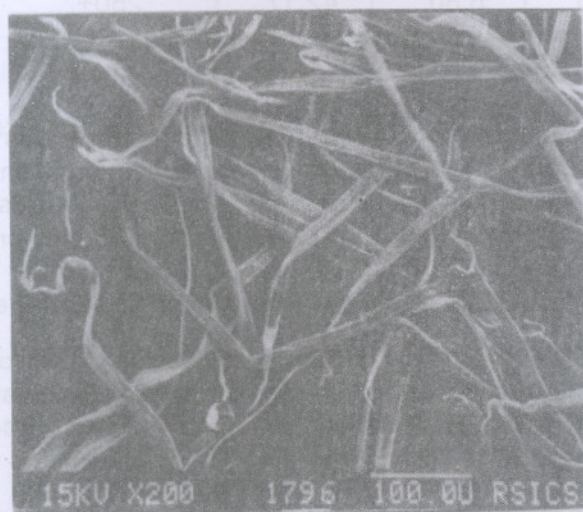
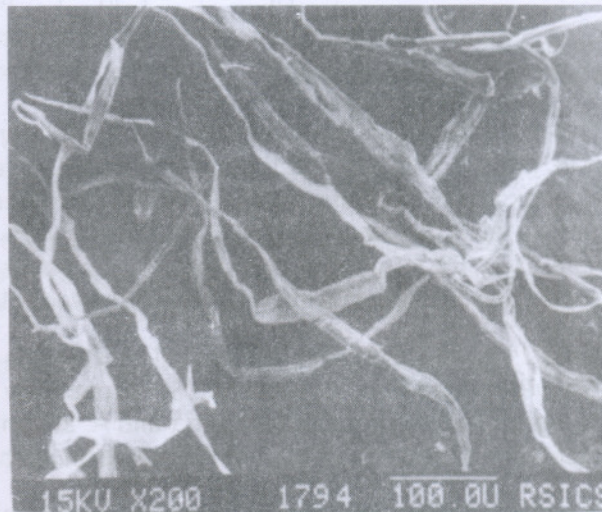
The morphological properties of the pulp fibre i.e. length (L), diameter (D), wall thickness (W) and lumen diameter (d) of well-digested pulp were determined by using a Docuval photomicroscope (Zeol Japan). For determination of fibre dimensions, well digested pulp fibres were taken and disintegrated for sometimes till the fibres free from bundles. These bundle free fibres were taken and observations were made under microscope at different magnifications and at two different microscopic fields. The average values of twenty five observations were recorded (Table 4).

Table 4 : Morphological properties of pulp fibre

| Properties | C. | dichotoma | A. allughas | H. esculentus | H. sabdariffa |
|--|---------|-----------|-------------|---------------|---------------|
| Fibre length, (L) mm | Range | 2.10-0.27 | 2.80-0.42 | 3.25-0.40 | 3.70-0.45 |
| | Average | 1.20 | 1.25 | 1.45 | 1.58 |
| Fibre width, (D) (µm) | Range | 21-17 | 22-17 | 22-18 | 25-12.5 |
| | Average | 20 | 22 | 22 | 22 |
| Cell wall thickness, (W), µm | Range | 3.25-5.00 | 5.2-3 | 5.25-3.50 | 5.00-3.50 |
| | Average | 4.90 | 3.75 | 3.85 | 4.20 |
| Lumen diameter, (d) µm | Range | 7.80-17.5 | 16.5-6.8 | 17.5-7.5 | 17.10-8.50 |
| | Average | 17 | 15.2 | 15.0 | 15.50 |
| Slenderness ratio, L/D | | 60 | 56.8 | 65.9 | 63.20 |
| Runkel ratio, 2 W/d | | 0.47 | 0.49 | 0.51 | 0.54 |
| Flexibility Co-efficient, (d/D x 100) | | 85 | 69.1 | 69.0 | 70.45 |
| Ratio of twice cell wall thickness to fibre diameter | | 0.40 | 0.34 | 0.35 | 0.38 |
| Ratio of twice cell wall thickness to fibre width | | 0.40 | 0.34 | 0.27 | 0.27 |
| Shape factor | | 0.16 | 0.35 | 0.53 | 0.33 |

Scanning electron microscopic study

Disintegrated pulp fibres samples were mounted on specimen holder with the help of electro conductive tap. The fibre samples were coated with gold in an ion sputter (ZFC 100 Zeol Japan) in low vacuum with a layer 150 to 200 nm. The observations were made in the electron microscope at an accelerating potential of 15 K V. Micrographs were taken at this potential. (Figure 2).

*Clyniogynae dichotoma**Alpinea allughas**Hibiscus esculentus**Hibiscus sabdariffa***Fig. 2: SEM micrograph of different pulp fibres**

Paper sheet making and testing

Both unbleached and bleached pulps obtained from the plants were beaten in a laboratory valley beater at 1.2% consistency. The pulps were beaten up to 45 oSR (Schoper Reiglar) freeness. Using this pulp stock standard hand sheet of 60±1 gm-2 was made in the British Standard laboratory hand sheet-making machine. The sheets were pressed in the hydraulic press and dried under pressure. The dried paper sheet were conditioned at 65±2% relative humidity at 27 °C for 2 h and tested the various physical strength properties as per TAPPI Standard Method 7.

Table-5: Physical strength properties of paper sheets made from unbleached (UB) and Bleached (BI) pulps

| Plant material | Pulp used | Bulk density Cc/g | Burst index kPam ² /g | Tear index mNm ² /g | Tensile index N/mg | DF nos (double fold) |
|----------------|-----------|----------------------|-------------------------------------|-----------------------------------|-----------------------|-------------------------|
| C. dichotoma | UB | 1.55 | 3.95 | 11.5 | 58.7 | 340+ |
| | BI | 1.43 | 3.35 | 7.8 | 47.6 | 125+ |
| A. allughas | UB | 1.45 | 4.81 | 10.3 | 64.2 | 450+ |
| | BI | 1.37 | 3.80 | 6.4 | 39.8 | 150+ |
| H. esculentus | UB | 1.47 | 4.90 | 11.77 | 63.26 | 325+ |
| | BI | 1.39 | 3.73 | 6.37 | 48.7 | 120+ |
| H. sabdariffa | UB | 1.52 | 5.09 | 10.5 | 62.03 | 425+ |
| | BI | 1.38 | 4.20 | 8.80 | 42.17 | 250+ |

Results and Discussion:

The morphological characters such as plant height, diameter, core diameter etc. of different plants as given in Table 1 showed that though the plants were of different height there was not much variation in diameter except H. sabdariffa plant (1.8cm). The different constituents of plants were varied from species to species. A. allughas constitute 45% of sheath, 45% of core and 10% of leaves. So also, C. dichotoma consists of 79.5% of central core and 20.5% leaves and twigs. The morphological data recorded for H. esculentus and H. sabdariffa showed 50-60% of wood, 15-20% barks and 20-25% leaves and twigs respectively.

Table 2 shows the proximate chemical constituents of the plants. Important plant constituents like cellulose content were recorded 61.0% and 62.0% respectively for H. esculentus and H. sabdariffa while 53.8% and 51.5% were recorded for C. dichotoma and A. allughas respectively. The pentosan content was recorded between 15.3-17.6%. The chemical composition indicates their suitability for producing good quality pulp.

The optimum Kraft pulping conditions were determined after carrying out a series of cooking experiments varying chemical concentration, time of cooking and temperature. The unbleached pulp obtained from all the plants were found between 48.3%-54.8% and bleached pulp between 39.4%-43.8%. The brightness of the bleached pulps were varied between 72-78% (Table 3). The physical properties of the pulps indicate their suitability for making good quality paper.

Fig 1 represents the optimum chemical requirement to get higher yield of pulp for each individual species. The chemical concentrations were varied from 8-14% and respective pulp yields were recorded between 35.4-54.8% for unbleached pulp.

The characteristics morphological properties of the bleached pulp fibres are given in Table 4. The average fibre length (L), varied from 1.20 mm (C. dichotoma) to 1.58 (H. sabdariffa) and the average fibre width (D) did not show much variation among the species, the average cell wall thickness (W) varied from 3.75 µm (A. allughas) to 4.90 µm (C. dichotoma), lumen diameter (d) from 15.0 µm (H. esculentus) to 17.0µm (C. dichotoma). Thus runkel ratio (2W/d) and shape factor (D²-d²/D²+d²) varies from 0.47 (C. dichotoma) to 0.54 (H. esculentus) and 0.16 (C. dichotoma) to 0.53 (H. esculentus) respectively. The fibres characteristics are collectively represented by runkel ratio and shape factor. The paper formation and its strength properties are

depend upon these properties. The results of the morphological characteristics show that all the four plants species are suitable for paper making.

Fig 2 shows the scanning electron micrograph of the pulp fibres of *Clynotogynae dichotoma*, *Alpinea allughas*, *Hibiscus esculentus* and *Hibiscus sabdariffa*. The fibres of *C. dichotoma* are flat with occasional cracks. Two types of fibres viz. matured fibres where fibrils are seen and the immatured fibres where fibrils are not visible. *A. allughas* fibres are solid rod like with some nodes and internodes. Two types of fibres i.e. thin and thick fibres where fibrils are not distinctly visible. *H. esculentus* fibres are flat, rosette like, pointed at both the end. All the fibres are of similar types where no fibrils are distinctly visible. A few occasional pores are also seen in the fibres. Fibres of *H. sabdariffa* are flat ribbon shaped and larger in diameter. Fibres are wavy with distinct pores and occasional cracks in the entire length of fibres.

The physical strength properties of paper sheets made from unbleached and bleached pulp are shown in Table 5. The data obtained for bulk densities (1.45-1.55 Cc/g), burst index (3.95-5.09 KPa m²/g), tear index (10.3-11.77 mNm²/g), tensile index 58.7-64.2 N/mg) and double fold no.s (325+ - 450+) of unbleached paper sheets made from all the four plant species are found to be more or less comparable. So also, the properties of paper sheets made from the bleached pulps showed adequate physical strength properties.

Conclusion:

From the above study it can be concluded that all the plant species under the present investigation would be ideal source of supplementary raw material for pulp and paper industry.

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