

**Preparation, Chemical composition, characterization and properties of paper sheets made from Napier grass**

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## **Abstract**

In this study, perennial fast growth Napier grass fibers were used for pulp and papermaking. Chlorination and alkaline process were carried using sodium chlorite and sodium hydroxide respectively for pulp extraction from Napier grass. Detailed chemical composition of the Napier grass fibers and the extracted pulp was carried out and a comparison with other perennial grasses made. The extracted fibers and pulp obtained from Napier grass were characterized using scanning electron microscopy and Fourier transform infrared spectroscopy. The physical, optical and mechanical properties of Napier grass pulp laboratory handsheets were investigated for opacity, brightness, tensile index, breaking length and burst index. Handsheets made from pulped Napier grass were compared to paper made from other perennial grasses. The superior properties of paper prepared from Napier grass pulp indicated the suitability of Napier grass as an alternative non-wood source for papermaking.

**Keywords:** Napier grass, Pulp, Chemical composition, Paper, Properties

## **1. Introduction**

Paper is a complex structure composed of a network of bonded fibers. The word paper is derived from the Latin word "papyrus" and Greek word "papuros", signifying the *Cyperus Papyrus* plant. Today, the paper applications of paper include a wide range of products and it is almost impossible to imagine a life without paper. World consumption of paper has grown by 400% in the last 40 years. Wood, the primary raw material for the pulp and paper industry, is a complex composite material, consisting mainly cellulose, hemicelluloses and lignin. The pulp and paper industry breaks down the wood to separate the cellulose from the non-cellulosic substances using mechanical and chemical methods. The pulp slurry is subsequently dried on a paper machine to produce a paper sheet.

The world consumes more than 400 million tons of paper annually. Today, nearly 35% (4 billion trees) of the total trees cut around the world are used in pulp and paper industries [1]. Vast majority (75%) of paper pulp is obtained from wood. While the world paper consumption is continuously increasing, the forest resources are diminishing globally. In many countries, the wood available will not last for long. Forest deficient countries are already looking for

alternative fibrous resources especially, non-wood [2]. Non-wood plants and agricultural residues are important raw materials in countries where wood is unavailable in sufficient quantities to meet the ever increasing demand of pulp and papermaking. Though cereal straw and bagasse are presently the leading non-wood plants, other sources such as flax, hemp, sisal, banana, jute etc. could also become important raw materials for papermaking in the future.

Non-wood fibers have been used for papermaking in since long. A major portion of the non-wood pulp is produced in Asia, Africa, Eastern Europe and Latin America. Of the world's total pulp production, non-wood pulp represents close to 10%. In developing countries, however, non-wood pulp production is often much higher, especially in China and India where it is nearly 70% [3]. Compared to wood sources, non-wood plants and agricultural residues offer several advantages including short growth cycles, moderate irrigation and fertilization requirements and low lignin content, resulting in reduced energy and use of milder chemicals during pulping [4]. Normally non-wood pulps are bleached more difficultly than wood pulp. However, non-wood sources have some serious drawbacks as compared with wood, including seasonal availability, collection, transportation and possible deterioration during storage [5]. The successful development of non-wood pulping processes is required to be able to exploit their full potential [6]. There is a need for new non-wood plants as alternative materials such as rapid growth plants [4], which can be processed by methods requiring modest investments and high yield at a low cost while preserving the environment [7, 8]. Many fast growing perennial plants (bamboo, switch and giant reed grasses) have been identified, cultivated and studied for their suitability for pulp and paper manufacture [9]. Napier grass is an important grass in the tropical regions and is considered to be among the most promising non-wood source owing to its easy cultivation, rapid growth and abundance.

Napier grass also called 'elephant grass' belongs to the Poaceae family of *Pennisetum purpurum schum* species. Napier grass has been a native of Africa and centuries back the plant was introduced to South America, Asia and Australia as forage for livestock. This is a perennial, robust, internodal fibrous grass. A single clump plant may produce up to 50 tillers and normally reach up to 4 m in height. Napier grass is a fast and wild growing species and requires very little supplement of nutrients. It can be harvested 3-4 months after planting and continues to be

harvested at an interval of 6–8 weeks for up to 5 years, yielding a dry biomass yield per hectare per annum of up to 40 tones [10]. Reddy et al. [11, 12] in earlier investigations studied the chemical composition, structural and thermal properties of Napier grass fibers. These studies were based on the characterization of ligno-cellulosic fibers from Napier grass and evaluation of its suitability for papermaking applications.

This paper presents the chlorination and alkaline pulping process for pulp extraction from Napier grass. The chemical composition of the fiber and obtained pulp were also determined. In this study, we used a combination of different analytical techniques, i.e. scanning electron microscopy and Fourier transform infrared spectroscopy to investigate the morphology and structure of Napier grass fibers and the obtained pulp. In addition, the physical and mechanical properties of laboratory handsheets from Napier grass pulp were also investigated. The present work was aimed to investigate whether Napier grass is a potential fiber source for pulp and paper production.

## **2. Materials and Methods**

### ***2.1. Materials***

Extracted Napier grass fibers and analytical grade of acetic acid, sodium bisulphite, sodium hydroxide pellets (Merck Chemicals), ethanol, sodium chlorite and toluene (Sd-Fine Chemicals) were used.

### ***2.2. Fiber extraction***

The stems were collected from one year old Napier grass. Provenance of the Napier grass is Penakacherla village in Anantapur district (Geographical coordinates - 77°19' 09" E, 15°16' 36" N) of Andhra Pradesh State in India. The usual water retting process for extraction of fibers from the Napier grass internodes was adopted. Extracted grass fibers were washed with tap water several times, followed by distilled water and sun dried for a week. The fibers were then kept in a hot air oven for 24 h at 105 °C to remove the moisture. The yield was calculated as the percentage of the dried fiber over the initial oven dry Napier grass stem weight and then yield was found to be 40%. Fig. 1(a-d) presents the photographs of Napier grass, stems, stem cross-section and extracted fibers.

### **2.3. Pulping**

Napier grass fibers were used for pulp extraction using a two-step procedure. The first step involves the preparation of holocelluloses based on chlorination process. For this, the fibers were initially delignified with 0.7% sodium chlorite at 100 °C for 2 h at atmospheric pressure in an acidic solution (pH 4-4.2 adjusted by the addition of acetic acid-sodium acetate buffer), using a fiber/liquor ratio of 1:30. After filtering, the fibers were extensively washed with 2% sodium bisulphite and distilled water. In the second step, the delignified fibers were treated with 17.5% NaOH solution at 20 °C for 1 h, treatment after delignified fibers were taken out and washed with 8.3% NaOH solution at 20 °C, using a fiber/liquor ratio of 1:10. After this, the residue was filtered, washed with 10% acetic acid, tap water and subsequently with distilled water to neutralize the reaction. In this step rich content of cellulose were separated by eliminating hemicelluloses from delignified fibers. At the completion of chemical treatment process (pulping) the digested fibers obtained are called pulp. This pulp was dried in an oven at 105 °C until constant weight. The obtained pulp is shown in Fig. 1e.

### **2.4. Chemical composition analysis**

The chemical composition of the Napier grass fibers and obtained pulp was determined using the standard TAPPI (Technical Association of the Pulp and Paper Associations) and other methods for different components, namely: for T 204 cm-07 [extractives], T 203 cm-99 [ $\alpha$ -cellulose], T 222 om-06 [lignin], T 211 om-07 [ash]. The holocellulose was determined according to the method described by Wise et al. [13]. The hemicelluloses fraction was calculated as the difference between the holocellulose and  $\alpha$ -cellulose content. The % content of extractives,  $\alpha$ -cellulose, hemicelluloses, lignin and ash were determined and the average and standard deviation values based on five samples reported.

### **2.5. Pulp analysis**

The pulp yield was gravimetrically determined as the weight of the dried pulp to that of the dried initial fibers. The obtained pulp drainability was determined by measuring the Schopper Riegler degree ( $^{\circ}$ SR–ISO 5267-1), using a Drainage Freeness Retention Tester (UEC-2002 B, Universal Engineering Corporation, India). 10 g of oven dry pulp was dispersed in 1000 mL water and then poured into a tester and stirred at 700 rpm. The volumetric amount of filtrate collected in 10 s

was recorded. Using the TAPPI standard (T236 om-99) the Kappa number (which represents the amount of permanganate consumed by a sample) was determined to estimate the amount of residual lignin in the obtained pulp.

### ***2.6. Scanning electron microscopy***

Morphology of the Napier grass fibers and obtained pulp was examined using a scanning electron microscope. In this study, the samples were gold coated and their surface observed under a JEOL scanning electron microscope (model JSM 820) at 8 kV.

### ***2.7. Fourier Transform Infrared spectroscopy***

Fourier transform infrared spectroscopy studies on Napier grass fibers and obtained pulp was carried out dispersing the powdered fiber samples in KBr and using a Perkin Elmer 16PC FTIR spectrophotometer. All spectra were recorded in the 4000-500  $\text{cm}^{-1}$  region with 32 scans in each case at a resolution of 4  $\text{cm}^{-1}$ .

### ***2.8. Preparation and testing of laboratory handsheets***

The paper sheets were hand made in the laboratory according to the ISO standard 5369, using the SCA-model sheet former (Hand Sheet Former- UEC-2005 A). Circular sheets of 165 mm diameter were formed using 65  $\text{g/m}^2$  oven dry pulp. The pulp was disintegrated and dispersed in water to a dry content of 0.125  $\text{g/L}$ . 8L of the pulp suspension was poured into a mould and after 10 s the water was drained through the wire screen by applying pressure. The wet paper sheets obtained were pressed at 0.5 MPa pressure for 5 min. The blotter was then changed and the sheets pressed again for 2 min in a hydraulic press. The sheets were then dried in a dryer cabinet to obtain the final paper sheets (Fig. 1f). The sheets were immediately stored in polyethylene bags and placed at 23 °C and 50% RH for conditioning prior to further testing. The sheets were tested using the following instruments: brightness and opacity tester (model UEC-1017), burst strength tester (model UEC-1010 Bi) and tensile strength tester (model UEC-1005 B). Structural, optical and physical properties like basis weight, thickness, bulk, brightness, opacity, burst strength, tensile strength and breaking length were found out as per ISO 536, ISO 534, ISO 2470, ISO 2471, ISO 535, ISO 2758 and ISO 1924 standards respectively. Five samples from

different sheets were tested to determine the properties and the average and standard deviation values are reported here.

### **3. Results and Discussions**

The main aim of this work was to extract pulp from Napier grass fiber, convert it into paper and characterize it. Table 1 presents the chemical composition of extracted Napier grass fibers (3.1% extractives, 44.2%  $\alpha$ -cellulose, 30.5% hemicelluloses, 18.3% lignin and 3.7% ash). In Table 1, the dry matter yield and chemical composition of Napier grass fibers are compared with some other cellulosic perennial grasses [14-20]. From Table 1 it is evident that the chemical composition of most of the grasses is similar to Napier grass. However, extractives content of Alfa and Giant reed grasses is higher. For Alfa grass the cellulose content is higher while the hemicelluloses and lignin contents are similar. The ash content of the Alfa grass is higher than Napier grass. Further, it can also be observed from Table 1 that the dry matter yield of Napier grass is higher by 40 t/ha when compared to most of the other grasses. Thus, Napier grass is established to be more profitable for pulp and papermaking.

The yield of obtained Napier grass fiber pulp was found to be 58%. The acid-chlorite and alkali treatments are considered to be greatly affected by the composition of the Napier grass fiber biomass. However, this composition gets changed during the acid-chlorite and alkali treatment process due to the complex physical and chemical roles of acid-chlorite and alkali. The average pulp contained extractives, cellulose, hemicelluloses, lignin and ash contents were found to be, 1.01, 89.7, 3.12, 3.8 and 1.2% respectively. A comparative analysis of chemical composition reveals significant difference between the raw fiber and the obtained pulp. The obtained pulp had higher cellulose (89.74 vs 44.2%) content, while extractives (1.01 vs 3.14%), hemicelluloses (3.12 vs 30.5 %), lignin (3.8 vs 18.32%) and ash (1.22 vs 3.71%) contents were lower than the raw fiber. This indicates a large-scale removal of extractives, hemicelluloses, lignin and ash during the pulping process. This indicates that the lignin content of the Napier grass fibers was significantly reduced after the acid-chlorite and alkali treatment for a shorter time and hemicelluloses removal by disruption of lignin-carbohydrate complex (lignin-hemicelluloses) linkages may have resulted in the decrease of lignin and hemicelluloses content. However, many processes can be used for pulping from natural fibers such as like sodium sulphate, bisulfate,

kraft, and then further applying bleaching process for elimination of lignin. However, in this pulping method we did not apply any subsequent bleaching methods. Therefore, this method of a shorter time is favorable because the pulp yield is higher and the pulp making the process involves significantly lower energy.

In Table 2 the yield, Kappa number and freeness of the Napier grass fiber pulp is compared with those obtained from other perennial grasses [14, 17, 18, 21-23]. From Table 2, it is evident that the yield of pulp from Napier grass fibers was higher when compared to other fibers. The Kappa number and freeness ( $^{\circ}$ SR) of the pulp were found to be 26 and 20 respectively. The Kappa number of Napier grass pulp was close to Giant reed and Bamboo pulp; higher than the remaining grasses' pulp. The freeness of Napier grass pulp was lower than Reed cannery pulp but higher than the remaining grasses' pulp. The higher yield, lower Kappa number and good drainage property of Napier grass fiber pulp are very positive features, when considering the valorization of this perennial Napier grass for papermaking.

The morphology of Napier grass fiber and obtained pulp was studied using scanning electron microscope. Figs. 2 and 3 reveal the morphological changes between the raw Napier grass fibers and obtained pulp. The micrographs of Napier grass fiber surface shown in Figs. 2a, b and c, reveal that the impurities are localized on the surface. Generally most of natural cellulose fibers are multi cellular structure and then composed of cellulose surrounded and cemented together with lignin and hemicelluloses. In our previous studies, the chemical composition and structural characterization of Napier grass fibers have already been reported [12]. During the pulping process, most of the lignin and hemicelluloses were removed and fibrils separated from raw fibers (Fig. 3a). The micrograph also indicates a highly fibrous network-like structure consisting of cellulose microfibrils. At higher magnification (Fig. 3b), microfibrils appear to have a clean and rough surface. The morphological changes in the fiber surface were attributed to the removal of the hemicelluloses and lignin by chemical treatments. Consequently, rough surface area appears which could result in an increased adhesion at the fiber-fiber interface and water absorption in the papermaking process.

FTIR spectra of the raw Napier grass fibers and obtained pulp are shown in Fig. 4. The spectra exhibited a strong absorption band at around  $3420\text{ cm}^{-1}$  corresponding to the OH-stretching vibration. The intense and broad absorption is a clear indication of the presence of many hydroxyl groups in the fiber. The absorption bands at  $2923$  and  $2853\text{ cm}^{-1}$  correspond to the asymmetric and symmetric stretching vibrations of methylene group of all the three (cellulose, hemicelluloses and lignin) constituents [24]. The absorption band at  $1727\text{ cm}^{-1}$  was attributed to C=O stretching of the carbonyl and acetyl groups in hemicelluloses [24]. The intensity of this band significantly decreased in the obtained pulp because of removal of most of the hemicelluloses during pulping process. The band at  $1634\text{ cm}^{-1}$  corresponds to the bending mode of the absorbed water [25]. The absorption bands at  $1605$ ,  $1514$  and  $1428\text{ cm}^{-1}$  represent aromatic ring vibrations [26]. The intensity of these bands significantly decreased in the obtained pulp due to the removal of most of lignin during pulping process. The absorption band at  $1455\text{ cm}^{-1}$  corresponds to the C–H deformation methyl, methylene and methoxyl groups combined with aromatic ring vibration of lignin [27]. The absorption bands at  $1377$  and  $1319\text{ cm}^{-1}$  correspond to C–H asymmetric deformation and –OH bending vibration of cellulose [28]. An intense absorption band at  $1260\text{ cm}^{-1}$  corresponds to –COO vibration of acetyl groups in hemicelluloses [24]. The intensity of this band also significantly decreased in the obtained pulp indicating removal of most of the hemicelluloses during the pulping process. The absorption band at  $1164\text{ cm}^{-1}$  corresponds to C–O antisymmetric bridge stretching of cellulose. The absorption band at  $1110\text{ cm}^{-1}$  belongs to C–C and C–O asymmetric in-phase ring stretching of cellulose. A strong absorption band at  $1054\text{ cm}^{-1}$  corresponds to C–O–C symmetric stretching dialkyl ether linkages and C–O stretching vibration in cellulose [29]. The absorption band at  $897\text{ cm}^{-1}$  corresponds to C–H rocking vibrations of cellulose [25]. FTIR spectra thus confirm the large-scale removal of hemicelluloses and lignin from the Napier grass fiber during the pulping process. These observations are largely consistent with the results of chemical analysis.

The physical and optical properties of the laboratory handsheets fabricated from Napier grass fiber pulp are summarized in Table 3. The sheets made from Napier grass fiber pulp showed promising papermaking characteristics as follows: basis weight ( $64.62\text{ g/m}^2$ ), thickness ( $138.4\text{ }\mu\text{m}$ ), bulk ( $2.14\text{ g/cm}^3$ ), brightness ( $74.6\%$ ), opacity ( $96.16\%$ ), burst index ( $4.98\text{ kPa m}^2/\text{g}$ ), tensile index ( $78.2\text{ Nm/g}$ ) and breaking length ( $8237\text{ m}$ ). The physical and optical properties of

paper sheets are compared with those with other perennial grasses [17, 18, 22, 23, 30, 31] and presented in Table 3. From this table, it is evident that the basis weight, thickness and bulk properties of the laboratory handsheets from Napier grass fiber are similar to Alfa grass and higher than those other grasses. The brightness of Napier grass pulp based paper sheets was higher than other grasses (Table 3), while opacity was relatively close to other grasses. Generally, conventional pulping methods without applying bleaching process cannot acquire the brightness of pulp to higher level (beyond 80%) without sacrificing the mechanical properties of pulp. However, in this method we could achieve higher level brightness of the pulp without bleaching process. The burst index was relatively close to Bamboo, lower than Switch but higher than other grasses (Table 3). The tensile index was found to be lower than Switch but better than Reed canary, Miscanthus and Bamboo. The breaking length of Napier pulp paper is superior to Bamboo and Miscanthus. This comparison indicates that Napier pulp is an extremely promising raw material for papermaking.

The yield, kappa number and freeness from Napier grass is compared with those pulping methods in Table 4. From this table, it can be observed that the higher yield and kappa number of the chlorination with alkaline pulp compared to the kraft pulp was due to the higher residual lignin content. While lower freeness of the chlorination with alkaline pulp compared to the kraft pulp was due to more chemical damages of cellulose structure by using chlorite on the pulping process. However, lower temperature is used in chlorination with alkaline pulping process compared to the kraft pulping process. Also paper sheets from Napier grass based papers were made and their physical properties are reported in same table (Table 4). From this table, it appears that the basis weight, thickness and bulk property of the chlorination with alkaline pulp compared to the kraft pulp may partially result from an overestimation of the thickness of the sheet due to the presence of impurities not fully eliminated by the screening operation. Brightness of chlorination with alkaline pulp was higher than the kraft pulp due to depolymerization reactions of lignin during elemental chlorine causes further removal of chromophoric groups than the soda and sulfate groups. But there are no significant differences between opacity of two different pulping methods. This is because of the presence of fiber particles (fines) in pulps or more bonding between fibers. The strength properties of Napier grass obtained in this work were comparable to those produced by kraft pulping [32]. Under this

process (acid-chlorite and soda) we have the advantage of higher pulp yield, brightness and lower ash content, while retaining good strength properties than kraft process.

#### **4. Conclusion**

The perennial Napier grass is successfully demonstrated to be an effective alternative source for producing paper pulp. In the present study Napier grass fibers were successfully pulped applying the chlorination-alkaline process. The pulp was obtained with higher yield, lower kappa number and moderate freeness. Chemical analysis indicated that obtained pulp had higher cellulose content and lower hemicelluloses and lignin contents than the raw Napier grass fiber. FTIR analysis also confirmed the removal of lignin as well as most of the hemicellulose during the pulping process. The properties of laboratory handsheets from Napier grass pulp were found to be far superior to those made from most of the other grasses. The abundance and fast growth of Napier grass are an added advantage as an alternative non-wood source for papermaking.

#### **ACKNOWLEDGMENTS**

K.O.R. and C.U.M. sincerely acknowledge the Faculty of Engineering and Built Environment, University of Johannesburg, South Africa, for the award of Postdoctoral Research Fellowship. M.S. and E.M. acknowledge the financial assistance provided by the Research Committee University of Johannesburg, for carrying out this research.

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### **Captions for the Figures**

Figure 1: Photographs of (a) Napier grass (b) stems (c) stem cross-section (d) extracted fibers (e) obtained pulp and (f) laboratory handsheet

Figure 2: Scanning electron micrographs of Napier grass fiber surface at (a) lower and (b-c) higher magnifications

Figure 3: Scanning electron micrographs of obtained pulp fibrils surface at (a) lower and (b) higher magnifications

Figure 4: FTIR spectra of Napier grass fibers and obtained pulp

### **Captions for the Tables**

Table 1: Comparison of chemical composition of Napier grass fiber with other perennial grasses

Table 2: Comparison of Pulping method, yield, Kappa number and freeness of Napier grass fiber with other perennial grasses

Table 3: Comparison of properties of laboratory handsheets from Napier grass fiber with those made from other perennial grasses

Table 4: Paper properties made from Napier grass fibers - comparison with data by Madakadze1, et al.