CHEMICAL CHARACTERIZATION AND THE PRODUCTION OF KRAFT PULP FROM *Bambusa vulgaris*

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Abstract: The use of wood pulp for paper making and other varieties of products for industrial applications has been on the increase in recent times. Non-wood materials and other agricultural residues are being exploited as alternative sources in the production of pulp to reduce deforestation arising from wood pulp. The chemical characterization and Kraft pulping of *Bambusa vulgaris* have been investigated in this study. Bamboo culms obtained from the University of Lagos were analyzed for their chemical properties using both TAPPI and ASTM standards. The results of the analyses showed that *B. vulgaris* contains 1.90% ash, 5.83% extractives, 49.59% alpha cellulose, 21.99% hemicellulose, 20.39% lignin content. 1% sodium hydroxide solubility was found to be 26.50%and hot water solubility was found to be 4.70%. The bamboo chips with the moisture content of 14% were subjected to Kraft pulping at 25% sulphidity and 20% effective alkali for 5 h at temperature range of 140 to 160^{o}C. These pulping conditions gave Kraft pulp of 51.19% with kappa number of 14.5. Treatment of the pulp with hydrogen peroxide at a pulp consistency of 4.30% at 50 to 60^{o}C for 3 h gave a bleached pulp yield of 64.33%.
Keywords: Cellulose, chemical characterization, non-wood fibers, paper making, pulp

Introduction

The limitation in the availability of wood resources for the production of pulp has resulted in the utilization of fast growing non-wood cellulosic biomass materials such as corn stalks, bamboo, sawdust, wheat stalks, sugar cane bagasse, grasses and other agricultural waste materials for pulp and paper production (Azeez *et al*., 2016; Przybysz *et al.*, 2018; Aklilu, 2020). A report from Food and Agricultural Organization (FAO) of the United Nations on the economic contribution of the forestry products showed that this sector generates over 354 billion dollars annually with the pulpand industry accruing half of the total amount (FAO, 2014). With the continued rapid development of the global economy and increase in world population, the overall demand for wood and wood-based products will continue to increase in the future. A global outlook study in 1997 on the trends of demand for wood products, predicted an increase in the demand of the order of 20 % by 2010 (FAO, 1997). The current concern is whether this future demand for forest products can be met sustainably with increased industrialization.

Paper is made from pulp derived from lignocellulosic biomass and as such consists of cellulose, hemicelluose and lignin components. Most paper products are produced from pulp extracted from cellulose fibres (Hietala *et al*., 2018). The versatility of paper products in our daily activities cannot be underestimated as they have wide range of applications in surgical wraps, paper money, cheques, ticket, toilet paper, handkerchiefs, paper towel, litmus paper, electrical insulation paper, packaging and wrapping papers (EPN, 2018). In addition, paper is majorly used as writing and printing material, for the purpose of storing information and communication. Hence,paper and paper products have today become indispensable component in almost all the finished consumer products in the market. To attainfuture sustainability of global pulp and paper industry, efforts should be made to replace about 4 billion trees worldwide that are lost annually to paper production. This represents 35% in fraction of all harvested trees (FAO, 2004). Furthermore, the present practice has led to deforestation and other severe environmental outcomes like drought and desertification. Unfortunately, deforestation due tounguarded harvesting of wood resources is still ongoing particularly in the developing countries like Nigeria (Olagunju, 2015).

With the increasing population and industrialization leading to greater demand for pulp, non-edible lignocellulosic biomass such as bamboo present a renewablealternative raw material for the production of pulp. Bamboo is a naturally occurring composite material which grows abundantly in most of the tropical countries. The cell walls consist primarily of three polymeric materials that include cellulose, hemicellulose and lignin. They also contain ash and extractives (Li, 2004). The cellulose, hemicellulose and lignin are composed of large molecules that make up 90-98% of the cell wall. Compared with wood, bamboo has the advantage of a short growth cycle and self-reproduction. Furthermore, bamboo species are invasive and spread very fast. Uncared bamboo species may cause environmental problems.

A clear understanding of the chemistry of bamboo and the properties of the pulp is important in determining its utilization potential in pulp and paper industry. This was the motivation for carrying out this study in finding an alternative non-wood material for paper production.

Materials and Methods

Sample collection and identification of bamboo chips

Three Bamboo culms were obtained from bamboo grooves on the University of Lagos, Akoka Campus, Lagos Nigeria (Geographical location - 6 30 59.99” N; 3 23 5.99” E). The culms were defect free and were taken to the herbarium at the University of Lagos for identification. The culms were cut into small discs and further hand chipped into smaller sizes. These chips were then reduced to meal using a pulveriser and made to pass through a 40 mm mesh according to TAPPI T257.

Determination of the sodium hydroxide soluble component of B. vulgaris

2 g of oven-dried bamboo meal was measured into a beaker; 100 mL of 1% NaOH solution was added and stirred with a glass rod. The beaker was covered with a watch glass and placed in a water bath maintained at 100°C for a period of 60 min. The solution was stirred with a rod for about 5 s at 10, 15, and 25 min intervals. At the end of 60 min, the material was transferred to a tarred filtering crucible and washed with 100 mL of hot water. 25 mL of 10% ethanoic acid was added and then washed with hot water until acid free. The crucible and its contentswere dried in an oven at 105°C to a constant weight and cooled in a desiccator. The loss in weight was determined and calculated as percent solubility.

Determination of water solubility of B. vulgaris

Thecold water solubilityof *B. vulgaris*was determined by weighing 2 g of oven-dried bamboo meal into a 400 mL beaker and 300 mL of distilled water was added. The extraction was carried out at 23°C for 48 h and transferred into a tared filtering crucible andwashed with 200 mL of cold distilled water and dried to constant weight at 105°C.

The hot water solubility test was carried outby digesting 2 g of the sample for 3 h in a reflux condenser. The contents of the flask was transferred to a tared filtering crucible and washed with 200 mL of hot distilled water. It was dried to a constant weight at 105°C.

Determination of the extractives of B. vulgaris

This was doneaccording to TAPPI 204. The determination was carried out in ethanol and acetone mixture of 1:2 ratio (50 and 100 mL) using a Soxhlet extraction apparatus. 4 g of air-dried bamboo meal was placed in the paper thimble and extracted with a mixture of ethanol and acetone for 6 h. A blank determinationwas carried out, evaporated to dryness, and the residue weighed to the nearest 0.1 mg and the extractive content of the sample of *B. vulgaris* was calculated.

Klason Lignin Determination

1 g of oven-dried sample of extractive-free bamboowas placed in a 150 mL beaker followed by the addition of 15 mL of cold sulphuric acid (72 per cent). The solution was placed in a water bath at 20ºC for 2 h with stirring and thereafter transferred into a 1,000 mL flask. The acid was diluted by adding 560 mL of distilled water after which the contents of the flask was refluxed for 4 h, allowed to cool and filtered. The residue was washed free of acid, then oven-dried at 103ºC.

Holocellulose content

2 g sample of oven-dried extractive-free bamboo was weighed and placed in a 250 mL flask followed by addition of 150 mL of distilled water, 0.2 mL of cold ethanoic acid, and 1 g of $NaClO_2$ and placed in a water bath set at 80ºC. The content of the flask shaken at interval of 1hour for five hours. The temperature was reduced to 10ºC and the content of the flask was filtered into a coarse porosity fritted-glass crucible of known weight. The crucible was then oven-dried at 103ºC, cooled in a desiccator and weighed to a constant weight.

Determination of alpha cellulose content of B. vulgaris

3 g oven-dried sample of holocellulose was placed in a 250 mL Erlenmeyer flask in a water bath set at 20ºC. The sample was treated with 50 mL of 17.5% NaOH and mixed. The reaction was allowed to continue for 30 min after which the mixture was filtered undervacuum into a fritted glass crucible of known weight. The residue was washed with 50 mL of 8.3% NaOH, then with 40 mL of 10 % ethanoic acid and then with distilled water. It was oven-dried at 103ºC, cooled in a desiccator, and weighed to a constant weight. The amount of non-sugar monomers present in the bamboo chips was determined by subtraction of the alpha cellulose content from the holocellulose content to give the hemicellulose content.

Kraft pulping of B. vulgaris

100 g of *B. vulgaris* chips (1.0 – 3.0 mm) was subjected to Kraft pulping in a 2000 mL flask using a hot plate and a reflux condenser. The white liquor used in pulping was prepared by dissolving NaOH (14.74 g) and Na_2S (4.9 g) in 344 mL of distilled water. The reaction was left to run for 5 h at a temperature of 160ºC. The resulting brown, fluffy pulp was separated from the black liquor, washed with deionized water and then air-dried. After drying and the yield calculated, the air-dried pulp was further delignified by treating the pulp (30 g) with 500 mL of hydrogen peroxide and 200 mL of distilled water to give a pulp consistency of 4.3% at 60ºC for 3 h.

Kappa number determination of the Kraft pulp

3 g of Kraft pulp was weighed and the moisture content determined. The pulp was disintegrated using a blender in 500 mL of distilled water until it was free of fibre clots and undispersed fibre bundles. The disintegrated test specimen was transferred to a 2000 mL beaker and rinsed with distilled water to a volume of 795 mL. The beaker was placed in water bath at 25ºC and 100 mL of potassium permanganate solution and 100 mL of sulphuric acid solution were added into a 250 mL beaker, the temperature of the solution was brought quickly to 25°C. The reaction was terminated by adding 20 mL of the potassium iodide solution after which it wastitrated with the sodium thiosulphate solution. A few drops of starch indicator were added at the end of the reaction. The percentage ash content of the sample was determined by weighing 1 g sample of air-dried bamboo meal into a constant weight crucible and placed in the muffle furnace at 600ºC and ignited until all the carbon was eliminated.

TAPPI and ASTM standards for each analysis

S/N	PROPERTY	STANDARD
1	Moisture content of bamboo chips and meal	TAPPI T 257om-02
2	1% NaOH solubility	TAPPI T 212om-02
3	Water solubility	TAPPI T 207cm-99
4	Extractive content	TAPPI T 204cm-97
5	Klason lignin	ASTM D 1106-56 (Reapproved, 1997)
6	Holocellulose	ASTM D 1104-56 (Reapproved, 1978)
7	Alpha cellulose	ASTM D 1103-60 (Reapproved, 1978)

Results and Discussion

The efficiency of pulping can be significantly affectedby the composition of wood constituents that include the extractives and lignin (Silva *et al*., 2012). The results of the chemical analyses of *B. vulagris* and the pulp prepared from it are shown in Table 1.

Table 1: Chemical composition of *B. vulgaris* and the pulp

Parameter	Pulp
% Moisture Content	14.00 ± 0.01
1 % sodium hydroxide solubility	26.50 ± 0.03
Hot water solubility (%)	4.70 ± 0.05
Ash content (%)	1.90 ± 0.02
Extractive content (%)	5.83 ± 0.01
Alpha cellulose (%)	49.59 ± 0.02
Hemicellulose (%)	21.99 ± 0.01
Holocellulose (%)	71.58 ± 0.03
Lignin content (%)	20.39 ± 0.01
Yield of Kraft Pulp (%)	51.19 ± 0.023
Yield of delignified Pulp (%)	64.33 ± 0.021
Pulp Kappa Number	14.50 ± 0.012

It is seen in Table 1 that the moisture content of *B. vulagris* was found to be 14%. The bamboo was also found tocontain1.90% ash, 5.83% extractives, 49.59% alpha cellulose, 21.99% hemicellulose, 71.58% holocellulose and 20.39% lignin. With a high percentage of alpha cellulose (49.79%) *B. vulgaris* is a good material for pulp and paper production.

The hydrophilic extractive contents of *B. vulgaris* was determined by the extraction of the sample with hot water. Hot water removes the extraneous materials such as tannins, gums and colouring matter and sugars. The water solubility test gives an indication of the levels of these materials in the wood.In this hot water extraction, part of the hemicellulose may be removed alongside with the lipophilic extractives such as phenolic acids and flavonoids. The hot water solubility testwas found to be 4.70%. This result is comparable to 5% reported by Azeez *et al*., 2016 for *B. vulgaris.* These values fall within the acceptable range for paper production. Woods with low hydrophilic extractives content are more desirable during pulping as they require less cooking chemicals and shorter pulping times, compared to woods that have high hydrophilic extractives.

The ash content of *B. vulgaris* was determined to be 1.9% in this study. This result is comparable to the value reported by Hisham *et al*. (2006) for *G. scortechinii* which is about 1.90 - 3.50%. High levels of ash in wood is undesirable for pulping as they affect normal alkali consumption and pose problem at recovery of cooking liquor and operational problems in material handling, pulp washing and beating (Kristova and Karar, 1999) as well as interference with bleaching (Dutt *et al*., 2009).

The results for the extractive content of *B. vulgaris* is shown in Table 1. This was found to be 5.83%. The extractives are the non-structural components of wood. The extractives in bamboo consists of the soluble materials not generally considered part of the bamboo substance, which are primarily the waxes, phenolics, fats, resins, and some gums, as well as some water-soluble substances that include inorganics. Althoughthe extractives are a minor component, often constituting less than 10% of the wood, they contribute disproportionately to the characteristics of the wood by giving it the colour and odour. The extractive content of *G. brand*, *G. levis*, *G. scortechinii* and *G. wrayi* have been reported as 8.30, 9.23, 8.00 and 8.62% (Wahab *et al*., 2013).

The 1% NaOH soluble extractive content of *B. vulgaris* was determined to be 16.50% for the bamboo meal. The value of the NaOH solubility of pulp indicates cellulose degradation during pulping and bleaching processes and this has been related to strength and other properties of pulp (TAPPI, 2002). Alkali charge must be kept low in order to preserve the cellulose content and enhance good pulp yield (Sadiku *et al.*, 2016). High concentration of NaOH can lead to peeling reactions as acetals are very unstable to bases at the temperature of pulping.

The holocellulose is the water-insoluble carbohydrate fraction of wood from which the extractives and lignin have been removed. It can be extracted by the chlorination method using sodium chlorite ($NaClO_2$) for the degradation of the lignin. The holocellulose content of *B. vulgaris* was 71.58%. The result of this determination compares favourably with those reported by Wahab *et al.* (2013) for *Gigantochloalevis* (85.08%), *G. wrayi* (84.53%), *G. brang* (79.94%) and (74.62%) for *G. scortechinii*. Hisham *et al*. (2006) reported a range of 78.60 - 82.30% for *G. scortechinii*. However, lower values have been reported and these include 63.14 - 69.94% for *P. pubescens* (Li *et al*., 2007), *Eucalyptus camadulensis* 55.6% for Eucalyptus hybrid (Ashori, 2006). The holocellulose content of *B. vulgaris* obtained in this study further supports an earlier report that *B. vulgaris* has potential as an excellent material for pulp and papermaking (Sadiku *et al*, 2016).

The alpha cellulose content (49.59%) of *B. vulgaris* obtained in this study exceeds the acceptable alpha cellulose content of 40 % required for pulp production. The value (49.59%) obtained in this study is above the average alpha cellulose content of softwood (45 ± 2%). Paper mechanical strength is directly proportional to cellulose content (Madakadse *et al*., 1999). Therefore, the high content of alpha cellulosein bamboo makes it a good material for pulp production.

The hemicelluloses content of *B. vulgaris* obtained in this study was 21.99%. This value is higher than the range of 8.66 - 18.88% reported for *Eucalyptus spp* (Dutt and Tyagi, 2011) but close to the value reported by Li (2004). Hemicelluloses exhibit considerable diversity. The dominant hemicellulose in bamboo is glucuronoarabinoxylan that has xylose as backbone sugar residues with arabinose, glucuronic acid and acetyl groups as branches. Bamboo typically contains about 20% xylose, 1% arabinose, 3% acetyl groups and 1% uronic acid.

The high lignin content of 20.39% indicates a more intense delignification reaction, high liquor consumption and long cooking cycle (Ogunsile and Uwajeh, 2009) as compared to wood (Cao *et al*., 2014). The Kraft pulping process of *B. vulgaris* gave a yield value of 51.19%. This shows that the pulping conditions were favourable and after bleaching, the resulting yield is 64.33%.

Conclusion

Results of the chemical composition of *Bambusa vulgaris* showed that this specie of Bamboo is a good alternative to the use of wood for the production of pulp and paper as a result of its high holocellulose and alpha cellulose content. The holocellulose and alpha cellulose content are desirable; they predict good pulp yield and good paper strength. However, high lignin, ash and extractive content may pose problem when considering it for pulp making.

Conflict of Interest

Authors have declared that there is no conflict of interest reported in this work.

References

Aklilu EG 2020. Optimization and modeling of ethanol–alkali pulping process of bamboo (*Yushaniaalpina*) by response surface methodology. *Wood Sci. Techn.*, 54: 1319–1347 doi.org/10.1007/s00226-020-01188-z

Ashori A 2006. Pulp and paper from Kenafbast fibers. *Fiber. Polym.*, 7(1): 26-29.

Azeez MA, Andrew JE & Sithole BB 2016. A preliminary investigation of Nigerian *Gmelina arborea* and *Bambusa vulgaris* for pulp and paper production *Maderas. Ciencia y Tecnología,* 18(1): 65 – 78. http://dx.doi.org/10.4067/S0718-221X2016005000007.

Cao S, Ma X, Lin L, Huang F, Huang L & Chen L 2014. Morphological and chemical characterisation of Green bamboo (*Dendrocalamopsisoldhami* (Munro) Keng F.) for dissolving pulp production. *Bio. Resources,* 9(3): 4528-4539.

Dutt D & Tyagi CH 2011. Comparison of various Eucalyptus species for their morphological, chemical, pulp and paper making characteristics. *Indian J. Chem. Techn.,* 18: 145-151.

Dutt D, Upadhyaya JS, Singh B & Tyagi CH 2009. Studies on *Hibiscus cannabinus* and *Hibiscus sabdariffa* as an alternative pulp blend for softwood: An optimisation of delignification process. *Industrial Crops and Products,* 29: 16-26.

Environmental Paper Network (EPN): The State of the Global Paper Industry 2018. Available online: http://https://environmentalpaper.org/stateoftheindustry2018/ (accessed on 12 July, 2021).

FAO 1997. Provisional outlook for global forest products consumption, production and trade. Forestry Department, Policy and Planning Division, FAO, Rome.

FAO 2004. Trends and Current Status of the Contribution of the Forestry Sector to National Economies. Available online: http://www.fao.org/docrep/007/ad493e/ad493e00.htm#TopOfPage (accessed on 12 July, 2021).

Hietala M, Varrio K, Berglund L, Soini C & Oksman K 2018. Potential of municipal solid waste paper as raw material for production of cellulose nanofibers. *Waste Management,* 80: 319-326.

Hisham NH, Othman S, Rokiah H, Abd Latif M, Ani S & MohdTamizi M 2006. Characterisation of bamboo *Gigantochloascortechinii* at different ages. *J. Trop. Forest Sci.*, 18: 236–242.

Kristova P & Karar I 1999. Soda-anthraquinone pulp from three *Acacia nilotica* species. *Bioresource Technology,* 68: 209-213.

Li X 2004. Physical, chemical, and mechanical properties of bamboo and its utilization potential for fibre board manufacturing. *LSU Master's Theses*, 12: 866-889.

Li XB, Shape TF, Peter GF, Hse CY & Eberhardt TL 2007. Chemical changes with maturation of the bamboo species *Phyllostachyspubescens. J. Trop. Forest Sci*., 19(1): 6-12.

Madakadse IC, Radiotis T, Li J, Goel K & Smith DL 1999. Kraft pulping characteristics and pulp properties of warm season grasses. *Bioresource Technology,* 69: 75-85.

Ogunsile BO & Uwajeh CF 2009. Evaluation of the pulp and paper potential of a Nigerian grown *B. vulgaris*. *World Appl. Sci. J.,* 6(4): 536-541.

Olagunju TE 2015. Drought, desertification and the Nigerian environment: A review. *J. Ecol. and the Natural Envt.*, 7(7): 196-209.

Przybysz, KE, Małachowska DM, Boruszewski PD and Halina KP 2018. Yield of pulp, dimensional properties of fibers, and properties of paper produced from fast growing trees and grasses. *BioResources,* 13(1): 1372-1387.

Sadiku NA, Oluyege AO & Ajayi B 2016. Fibre dimension and chemical characterisation of naturally grown *Bambusa vulgaris* for pulp and paper production. *Journal of Bamboo and Rattan,* 15(1-4): 33-43.

Silva TCF, Gomide JL & Santos RB 2012. Evaluation of chemical composition and lignin structural features of Simarouba versicolor wood on its pulping performance. *Bioresources,* 7(3): 3910- 3920.

Wahab R, MohdTamizi M, Mahmud S, Aminuddin M, Shafiqur R, Hashim WS & Izyan K 2013. Extractives, holocellulose, α-cellulose, lignin and ash contents in cultivated tropical bamboo *Gigantochloabrang, G. levis, G. scortechinii* and *G. wrayi*. *Current Res. J. Biol. Sci.,* 5(6): 266-272.