PRODUCTION OF PULP FROM NON-WOODY MATERIALS OF AGRICULTURAL WASTE FOR HANDMADE AND TISSUE PAPERS IN AKWA IBOM STATE

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ABSTRACT

Production of pulps from non-woody materials of agricultural waste available in Akwa Ibom State, Nigeria was the focal point to the study. The pulps were produced from corn maize (Zea mays) and sponge gourd (Luffa cylindrical) straws. The chemical properties of these local raw materials and their suitability for production of pulps for the paper industry, especially in the production of handmade and tissue papers were evaluated. Thousands of tons of these agricultural materials are readily available in Nigeria annually, and presently are waste materials. The intermediate papermaking raw materials (pulps) were prepared by appropriate methods and used together with suitable chemical additives in preparing the paper stock for producing handmade paper and the tissue. The digester for pulping, hand mold and other papermaking accessories were designed and fabricated locally, and finally different hand sheets and tissues of different grammage were made using the hand mold and evaluated for their physical and mechanical properties. The results indicated that the pulps derived from corn maize (Zea mays) and sponge gourd (Luffa cylindrical) straws possess suitable fiber qualities for paper production. The physical tests, including tensile strength, burst strength, and tear resistance, demonstrated that the handmade paper and tissue produced meet industry standards for basic applications. The study concludes that by embracing this innovative approach, Akwa Ibom State can position itself as a leader in sustainable pulp production, driving long-term benefits for its people, economy, and environment. One of the recommendation was that more research should be carried out on the sample to find out how this non-wood material can be used to replace woody material since the process and the cost of afforestation is high.

Keyword: Production, Pulp, Non-woody materials, Agricultural waste, Handmade, Tissue papers, Akwa Ibom State.

Introduction

Straws are agricultural by-products consisting of the dry stalk of cereal plants after the grain and chaff have been removed. They make up to half of the yield of cereal crops. The fusion of straw can improve upon soil quality through improved nutrient cycling and soil organic carbon sequestration (Dobermann and Fairhurst, 2020; Ponnamperuma 2017). Straw is the main organic material that is available for most farmers and serves as an important source of potassium. Ponnamperuma (2017) indicated that at harvest, the straw contains 0.57% N, 0.07% P_2O_5, 1.5% K_2O, 0.1% sulphur (S) and 5% silicon (Si). Sponge gaurd (*Luffa cylindrica*) and corn maize (*Zea mays)* straws were used in the study.

Luffa cylindrica commonly called sponge gourd, vegetable sponge, bath sponge or dish cloth gaurd is a member of *cucurbitaceaese* family. *Luffa cylindrica* mainly composed of cellulose, hemicelluloses and lignin and the fibers are obtained from a subtropical *cucurbitaceaes* plant which produces a fruit with a fibrous vascular system. The plant size varies in relation to location, ranging from 15 cm to 1 m or even more than 1m in certain areas. Usually, the skin of the gourd is peeled off when it is used as a vegetable

(Udo *et al*, 2016a).

Zea mays is a significant crop grown all around the world. Its annual production is about 69 kg to 70 kg (Kim and Dale, 2014). The ratio of *Zea mays* straw to grain is typically assumed to be 1:1 (Wu *et al.*, 2024). In China, there are over 600 million tons of crop straw residues, including 250 million tons from maize straw alone, are produced every year (Udo *et al.*, 2016b). *Zea mays* straw is initially composed of leaves, husk fractions, and stalks (Shinners and Binversie, 2021).

Justification of the Project

The fibrous agro waste materials for pulp and papermaking are readily available in Akwa Ibom State, Nigeria and West African countries where there is sufficient rainfall throughout the year. Most of the plant sources that generate the waste take less than one year to mature and are easily regenerated unlike wood while the perennial ones can produce agro waste materials many times in a year.

Significance of study

The production of pulps from non-woody agricultural waste materials presents a transformative opportunity for Akwa Ibom State, Nigeria. This approach has not only tapped into the region's abundant agricultural residues, but also aligns with sustainable development goals, environmental conservation, and economic empowerment. The significance of this practice extends across several domains, including environmental sustainability, economic development, waste management, and rural industrialization.

Objectives of the Project

The main objective of the project is to use the abundantly available agricultural waste materials for local production of pulp for handmade papers and tissues

Specific objectives included to:

i) determine the amount of cellulose fibres present in the non woody fibrous agricultural waste materials;
(ii) determine the physicochemical properties and fibre characteristics of non-woody agricultural waste materials;
(iii) produce pulps from the non-woody agricultural waste materials;
(iv) develop dissolved pulp from non-woody agricultural waste materials to make available a relative cheap and pure source of cellulose as a raw material for the expansion of chemical industries;
(v) design and fabricate basic handmade paper equipment, namely hand mould, the press and the dryer.
(vi) produce handmade paper (HP) from bleached and unbleached pulps made from the agricultural waste pulps; and
(vii) evaluate the quality of handmade paper using standard equipment.

Review of Related Literature

Fibres are present in all plants and when commercially exploited, the plants become fibrous crops. Cellulose is a homoglycan constituted by ß-(1-4) linked D-glucopyranose units. Cellulose is a high molecular weight polysaccharide made up of repeating cellobiose units producing a linear chain in which both intra-chain and inter-chain molecular hydrogen bonds occur to link the chains (Donato *et al*., 2015), which in turn produce micro fibrils, matrices and multilayered cell walls. Plant fibres from different sources vary in length, colour, composition, strength, durability and resistance to water. Cellulosic fibres are more resilient than lingo-cellulosic fibres. Wood fibres are relatively large fibres. Cotton fibre is an elongation of seed coat cells and an example of one of the longest cells (Akgul, 2013).

Springer (2020) indicated that the typical components of plant biomass are moisture, cellulose, hemicelluloses, lignin, lipids, proteins, simple sugars, starches, water, hydrocarbon, ash and other compounds. The concentrations of these compounds depend on the plant species, type of tissue, growth stage and growing conditions. The sponge gourds (*Luffa cylindrica*) straw may contain triterpenoid, saponins, lucyosides A, B, C, D, E, F, G, H, I, J, K, L, M (Siqueira *et. al.*, 2024), ginsenosides Re, Rg1, etc. The leaf contains triterpenoid saponins, lucyin A, lucyosides G, N, O, P, Q, R (Takemoto *et al.*, 2014) 2-1-β-hydroxyoleanoic acid, 3-O-β-D-glucopyranosyl-maslinic acid (Mani *et al.*, 2016) . The fibres are composed of 60% cellulose, 30% hemicellulose and 10% lignin (Akgül *et al.*, 2013; Mani *et al.*, 2023). The fruits of *Luffa cylindrical* are smooth and have cylindrical shaped (Chen *et al.*, 2019). *Zea mays* straw is a lignocellulosic biomass which contains components such as cellulose (34.0%), hemicelluloses (37.5%) and lignin (22%). The carbon-nitrogen ratio (C/N ratio) for *Zea mays* straw is about 66.31%, while the proper C/N ratio for anaerobic digester should be within the range of 25–35%. Straw pre-treatment studies focus on adjusting the nutritional value of straw and improving upon its characteristics (Ajuzuigu and Ojuar, 2020).

Materials and Method

The *Zea mays* and *Luffa cylindrica* straws were obtained from swamp region of Ikpe Anang River in Ini Local Government Area, Akwa Ibom State, Nigeria. The samples were washed with water and cut into small length of about 2 cm and air dried for 3 days to reduce moisture before grinding to powder and stored in a container for further analysis.

Apparatus: 250 mL beaker, Filtering flask 100 mL, filtering crucible 30mL, reflux system, weighing balance, graduated cylinder 100 mL, watch glasses, stirring rod, thermometer, vacuum source, electric oven.

Reagents: sodium hydroxide (NaOH), Acetic acid (CH_3COOH), Hydrogen peroxide (H_2O_2)

Methods: The methods used were segmented into the following steps:

Characterization of samples

The three samples were characterized using the Association of Pulp and Paper Industries Standards (TAPPI) T 258 ^{o} s 76. Some of the parameters determined were:

Solubility in hot water

2.0 g of each of the air dried samples was place in a 250 mL flask and 100 mL of deionised water was added. The flask and its content were allowed to digest for 3 hours at a temperature of 120 ^{o}C using the reflux system after which it was filtered and dried in the oven before weighing and percentage solubility in hot water was determined using Equation 3.1

$$\%\ \text{solubility in hot water} = \frac{W_1 - W_2}{W_1} \times 100 \qquad \text{Equation 3.1}$$

where W_1 is the weight of air dried samples before solubility and W_2 is the weight of oven dried samples after solubility.

Solubility in 1% NaOH

2.0 g of each of the air dried samples was macerated and transferred to a 250 mL conical flask. 100 mL of 1% NaOH was added. It was allowed to boil for 3 hrs in a water bath at the temperature of 120 ^{o}C. The samples were filtered, washed thoroughly with hot deionised water and dried in an oven before weighing. The solubility was then calculated in percentage based on oven dried sample (Anon, 2001; TAPPI, 2002). Equation 3.1 was used to evaluate the solubility in 1 % NaOH.

Bulk Density (BD)

An empty measuring cylinder 100mL was weighed using a digital weighing balance to the nearest 0.001 g. The measuring cylinder was filled with the sample and the material was slightly compacted to ensure absence of large void space. The container and sample were then weighed. Three replicates were carried out. The bulk density of the sample was calculated using the Equation 3.2, thus:

$$BD = \frac{W_2 - W_1}{V} \qquad \text{Equation 3.2}$$

where BD is the bulk density of the sample (gcm^{-3}), W_1 is the weight of the container (g), W_2 is the weight of the container and sample (g) and V is the volume of the container (cm^3)

Fibre Analysis

Wood silvers of the three samples were put into test tubes and macerated with an equal volume (1:1) of glacial acetic acid and 30% hydrogen peroxide following the work of Oluwadare, (2017). This was later heated in an oven for 4 hours at 80°C until bleached white and soft. The chemical mixture was decanted and macerated fibres were rinsed several times with distilled water and defiberised to separate the fibres. This process was conducted for each of the sample collected.

The fibres were viewed under electron microscope. Ten (10) fibres were randomly obtained from each of the sample collect fibres were obtained from each sample for this analysis. The fibre length, fibre width and fibre diameter was measured and recorded. Derived fibre properties were determined using the Equations 3.3 to 3.7 as follows:

Lumen Width, $LW = \frac{FD}{2}$ Equation 3.3

where FD is the fibre diameter.

Cell Wall Thickness, $CW = \frac{FD - LW}{2}$ Equation 3.4

The Runkel Ratio, $RR = \frac{2CW}{FD}$ Equation 3.5

where 2CW is the Fibre wall thickness and D is the Diameter coefficient.

Flexibility = $\frac{LW}{FD} x\ 100$ Equation 3.6

where LW is the Lumen Width and FD is the Fibre diameter.

Slenderness ratio, $SR = \frac{FL}{FD}$ Equation 3.7

Results

Characteristics of straws

The results of the experimental work are presented in Tables 1, 2, 3 and 4.

Solubility Test

The results of the soluble contents of *Zea mays* straw and *Luffa cylindrica* fibres are presented in Table 1. These include solubility in cold water, hot water, 1% sodium hydroxide and bulk density.

Table 1: Physical parameters of *Zea mays* straw and *Luffa cylindrica* fibres (%)

Characteristics	*Zea mays*	*Luffa cylindrica*
% Solubility in 1% NaOH	71.0	48
% Solubility in hot water	24.50	28.5
% Solubility in cold water	20.30	16.8
Bulk density (gcm^{-3})	0.25	0.88

Bulk Density

The results of bulk density determined on the samples are presented in Table 3. The values obtained were 0.25 gcm^{-3} for *Zea mays* straw and 0.14 gcm^{-3} for *Luffa cylindrica*.

Fibre Characteristics

Tables 2 and 3 show the variations in fibre characteristics in *Zea mays* straw and *Luffa cylindrica* fibres.

Table 2: Variations in fibre characteristics in *Zea mays* straws

Fibre length (mm)	Fibre diameter (µm)	Fibre width (µm)
1.12585	12.65	6.325
0.3795	12.65	6.325
0.2783	12.65	5.06
0.4048	12.65	6.325
0.7084	11.39	3.795
0.5313	43.01	25.3

0.9361	6.33	2.53
1.012	12.65	6.325
0.5313	6.33	2.53
0.31625	11.39	2.53

Table 3: Variations in Fibre Characteristics in *Luffa cylindrica* Fibres

Fibre length (mm)	Fibre diameter (µm)	Fibre width (µm)
1.265	25.30	12.65
2.138	25.30	18.98
1.265	25.30	18.98
0.860	25.30	12.65
0.860	25.30	12.65
1.252	37.95	25.30
1.771	25.30	18.98
0.912	37.95	25.30
1.518	24.04	12.65
0.949	25.30	18.98

The derived values for the characterization of *Zea mays* straw and *Luffa cylindrical* are presented in Table 4.

Table 4: **Derived values for the characterization of *Zea mays* and *Luffa cylindrica***

Samples	*Zea mays*	*Luffa cylindrica*
FL (mm)	0.62	1.28
FD (µm)	14.17	27.70
FW (µm)	4.43	18.72
LW (mm)	7.09	13.85
CW (mm)	7.09	6.92
RR	1.00	0.50
FC (%)	6.03	50.00
SR	0.04	0.05

4.2 Discussion

1% sodium hydroxide solubility is used to determine the degree of fungus decay that can taken place in a given wood or non- wood sample.

The value obtained in 1% NaOH was 71.80% and 48% for the *Zea mays* straw and *Luffa cylindrical* fibres respectively, *Zea mays* straw recorded 71.80% solubility due to the fact that it soften more easily in 1% NaOH (Akpakan *et al*., 2023). The value obtained for the wood fibres was very high compared to those obtained for some non-woods raw materials reported by Akpakpan *et al.* (2012).

The value for hot water soluble were 24.50% and 28.5% and cold water soluble were 20.30% and 16.8% for the *Zea mays* straw and *Luffa cylindrical* fibres respectively. The value obtained for the *Zea mays* straw and *Luffa cylindrica* fibres was within the range for those reported for some non-woods (Usungurua *et al.*, 2024).

The density value obtained from *Zea mays* straw was closely related to the values obtained by Michelin *et al*. (2015). The bulk density value of *L. cylindrica* are within the value of 0.82 – 0.92 gcm^{-3} as reported by Michelin *et al*. (2015). The density of *L. cylindrica* is around 0.82 to 0.92 gcm^{-3}, which is lower than the density of some common natural fibres like sisal (1.26 to 1.45 gcm^{-3}), hemp (1.48 gcm^{-3}), coir (1.25 gcm^{-3}), ramie (1.50 gcm^{-3}) and cotton (1.51 to 1.60 gcm^{-3}) as reported by Boynard *et al*. (2013) and Siqueira *et al.* (2024).

Average fibre length was 0.62 mm and 1.28 mm for the *Zea mays* straw and *L. cylindrica* fibres respectively. *Luffa* has the highest value of 1.28 mm. It is stated that fibre length above 1.60 mm are said to be long and those below 1.60 mm as short (Anon, 2001). Pulping raw material with short fibre lengths are less suitable for paper making than the long fibre (Anon, 2001; Alma *et al*, 2021; Ademiluji and Okeke, 2020). In similar observation, Carvalho *et al.* (2017) and Copur *et al.* (2008) reported fibre lengths of less than 1.60 mm in some Nigerian hardwood species. Akpakpan *et al*. (2012) recorded 2.14 and 1.98 mm as fibre length of plantain pseudo stem waste and screw pine leaves, respectively. Oluwadare and Ashimiyu (2017) and Oluwadare (2017) recorded 0.65 mm as fibre length of *Leucaena leucocephala*. Also similar fibre length ranges of 1.50 to 2.9 mm and 1.46 to 2.91 mm were reported for bamboos in Sudan and Thailand respectively (Udo *et al*., 2016a). Though the fibre length of *Zea mays* straw and *Luffa cylindrica* are short, the samples can be use for preparation of cellulose derivatives.

The average fibre diameter was 14.17 and 27.70 µm for the *Zea mays* straw and *Luffa cylindrica* fibres respectively. In similar observation, Akpakpan, *et al*. (2023) recorded 12.0 µm as fibre diameter for *Nypa fruticans*. Also falls within the range recorded for coniferous and commercial pulp woods (Sen *et al.*, 2015; Oke *et al.*, 2016).

Average fibre width was 4.43 and 18.72 µm for the *Zea mays* straw and *Luffa cylindrica* which was very short compared to those of wood and other common non-woods fibres (approximately 32.0 – 43.0 µm and 18.0 – 30.0 µm for softwood and hardwoods, respectively. Akpakpan *et al*. (2012) reported 12 µm as fibre width of *Nypa fruticans* frond and petiole.

Lumen width and length were 7.09 µm and 13.85 µm for the *Zea mays* straw and *Luffa cylindrica* which is within the range of 4.9 µm and 12 µm were recorded.

The cell wall thickness is one of the significant fibre dimensions that determine the choice of a fibrous raw material for pulp and paper production. The values obtained were 3.54 and 6.93 µm for the *Zea mays* straw and *Luffa cylindrica*. Lumen size and cell wall thickness affect the rigidity and strength of papers made from fibre.

The value of Runkel ratio obtained for *Zea mays* straw and *Luffa cylindrica* was 1 and 0.5, slenderness ratio was 0.04 and 0.05 while flexibility coefficient was 6.03 and 50 respectively. It is stated that if slenderness ratio of a material is lower than 70, it is invaluable for quality pulp and paper production (Usungurua *et. al.*, 2024). High Runkel ratio fibres (that is fibres with relatively thick walls, such as those in summerwood) are stiffer, less flexible and form bulkier paper of low bonding area than low Runkel ratio fibres. Fibres with ratio less than or equal to one are very good for paper making (Shwei *et al.*, 2020). However, the pulps obtained from *Zea mays* straw and *Luffa cylindrica* will give stiffer, less flexible and bulkier paper since its Runkel ratio is less than one. Therefore, *Zea mays* straw and *Luffa cylindrica* pulps may be suitable for producing paper boards. The higher the value of the flexibility coefficient, the greater the pliability of the fibres and the well–bonded the sheet formed. Although pulp mechanical strength also depends on other processing variables such as pulping conditions, bleaching and beating.

Conclusion and Suggestions

The investigation of solubility contents and fibre characteristics of *Zea mays* straw and *Luffa cylindrica* fibres reveal that the species have fairly long fibre length for paper making. The derived parameters (Lumen width, Cell wall thickness, Runkel ratio and the flexibility coefficients) showed that the pulp could be used to produce paper of different grades. The bulk density is low which is within the acceptable range of some non-wood fibres which makes it easier for transportation. The solubility contents are also within the acceptable range for non-wood pulping raw materials. Therefore, outcome of this investigation showed that the species is a potential material for pulp and paper production. In view above, thousands of tonnes of *Zea mays* straws and *Luffa cylindrical* waste allowed to rot away annually in our garden and in commercial farms should be utilized in the production of good cultural papers. The production of pulp from non-woody agricultural waste materials in Akwa Ibom State represents a significant opportunity to drive sustainable development, economic growth, and environmental stewardship. Leveraging locally abundant agricultural residues like palm fronds, rice straw, cassava peels, and plantain stems, this approach can provide a cost-effective and eco-friendly alternative to traditional wood-based pulp production. It addresses critical challenges such as deforestation, inefficient waste management, and rural unemployment while adding value to agricultural by-products that are often discarded or burned.

Developing a non-woody pulp industry in Akwa Ibom State could catalyze rural industrialization, create jobs, and improve livelihoods, especially in rural communities. It aligns with global trends toward sustainability and offers the potential to attract investments and access new markets for environmentally friendly products. Moreover, this practice supports Nigeria's policy goals and international commitments to sustainable development by promoting responsible consumption, reducing carbon footprints, and contributing to a circular economy.

In conclusion, producing pulp from agricultural waste in Akwa Ibom State not only fosters economic diversification and industrial growth but also champion’s environmental conservation and resource efficiency. By embracing this innovative approach, Akwa Ibom State can position itself as a leader in sustainable pulp production, driving long-term benefits for its people, economy, and environment.

Recommendations

Based on the study, the following suggestions are made for further investigation.

i) Since the fibres are good pulping intermediate for pulp and paper production, it is therefore recommended that more research should be carried out on the sample to find out how this non-wood material can be used to replace woody material since the process and the cost of afforestation is high.

ii) More research should be done to investigate how this species can actually get the finishing product which is paper.

iii) The investigation on other non-woody agricultural by-product like pineapple leaves,raffia, musa, etc. for the production of paper.

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