CHEMICAL COMPOSITION OF *Spathodea campanulata* WOOD AND ITS IMPACT ON PULPING AND PAPERMAKING

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Abstract: There are over five hundred wood species in Nigeria, half of which are yet to be studied and utilized to their full potentials and as such referred to as lesser-used species. One species that belongs to this category is the African tulip tree wood, *Spathodea campanulata*, mostly grown for its aesthetic benefits than other uses. Whether in its native regions or where introduced, its use as a medicinal plant is well documented. Although it is regarded as an inferior wood, used for carving, charcoal and as firewood; it is solely traded as plywood in certain parts. Its fibrous nature however, calls for consideration as a fibre source for pulp and papermaking.
For any lignocellulosic material to be accepted for pulp or papermaking, there must be an adequate information on its chemical properties evaluated through standard procedures. Hence, there is need to study the chemical composition and soluble contents of *S. campanulata* as it affects its pulpability. A stand was harvested from a natural forest in Ibadan, Nigeria and its stem converted to billets for milled samples at the 10%, 50% and 90% positions of the total heights. Chemical composition such as lignin, extractives, hemicelluloses and alpha cellulose were considered as well as its solubility in water and 1% NaOH solution and the data was subjected to analysis of variance. Sampling height was found to significantly affect only the extractive content and alpha cellulose at α=0.05. The values obtained for chemical contents indicate that the species may be suitable for pulp and papermaking.

Keywords: Pulping, Wood, Composition, Papermaking, ***Spathodea campanulata***

Introduction

Wood has been known to be versatile in nature to meet majority of the needs of man especially in shelter, aesthetics, education, record keeping as well as currency for trade. The digital revolution of the world has yet to affect the use of pulp and paper especially for the education of young children who should not be exposed to electronic screen radiations (UNSCEAR, 2013). In order to meet up the ever growing demand of the increasing population for paper and paper products, the quest for sustainable fibre source for pulp and papermaking continues with the focus on lesser-used wood species. Generally, wood and other lignocellulosic materials consists of lignin, cellulose, extractives, hemicelluloses and some inorganic compounds, the presence and proportion of whom affects their choice in the pulp and paper industry. Once the fibre morphology of a wood has been concluded to be useful foe pulp and papermaking, there is need to study further its chemical composition which may affect its pulpability, paper properties, and techno-commercial suitability (Ververis *et al*., 2004; Sotannde, 2014; Riki *et al.*, 2019).

Lignin is the most prevalent and significant component of a lignocellulosic material that is thought to be detrimental to pulping and bleaching (Kock, 2006). The factors affecting delignification rate include the lignin content of a fibrous material, maturity, as well as the number of syringyl units in the lignin (Marques *et al*., 2010). Since lignin makes up about 30% of aromatic biopolymers on Earth, it has the potential to be a sustainable feedstock for the synthesis of aromatic compounds and energy supplements (Upton and Kasko, 2016). The kraft paper and pulp industries and sulfite pulping methods yield the most abundant industrial lignins, sometimes known as "black liquor." Moreover, the lignin produced by pulping companies can be used by other industries interested in aromatic resources as raw materials.

Various tissues and age groups within the same lignocellulosic biomass individual have different lignin structures (Healey *et al*., 2016). Hardwoods typically contain 19–28% lignin, softwoods 24–33%, and grasses 15–25% lignin. Methoxyl, carbonyl, carboxyl, and hydroxyl functional groups attach to aromatic or aliphatic moieties in lignin in varying amounts and proportions, giving lignin a variety of compositions and structures (Kai *et al*., 2016).

Since the cellulose content of a fibrous substance determines the strength of paper, cellulose is the primary plant component used in pulping. It has been discovered that pulp output and alpha-cellulose concentration are correlated, whereas hemicelluloses are in charge of the swelling action of the pulp as well as the mechanical strength characteristics of the finished paper. Furthermore, the age of a plant and the content of its cell walls have an impact on cellulose (Pahkala, 2001, Sotannde, 2014). The majority of biomasses typically contain glucose molecules during hydrolysis of the cellulose polymer. To form fibres, the crystalline and non-crystalline structure of cellulose is encircled by microfibrils. Cellulose has a 7–15% absorption capacity and is insoluble in water and diluted acid at room temperature. Elevating the temperature can cause an increase in solubility due to acid concentration, while an alkali solution separates, reduces the degree of polymerization, and causes the crystalline structure of cellulose to swell.

Conversely, extractives are dependent upon species, age, and the orientation of a tree with respect to sapwood and heartwood (Rowell *et al*., 2000). While some of the extractives in a plant can be partially removed by using different organic solvents, a combination of ethanol and benzene offers almost total removal. Water solubility, on the other hand, provides an estimated quantity of starches, tannins, gums, sugars, colouring material, and inorganic compounds. Although it gives wood endurance, it also increases chemical use in the pulp and paper sector and generates pitch problems.

Spathodea campanulata also known as the African Tulip, is an indigenous species found in the tropical dry forests of Africa but has been introduced to many other places such as Phillipines, Australia, Fiji, Brazil among many others (USDA, 2015). The tree grows to a height of 7–25 meters (23–82 feet) and is widely planted in tropical landscapes for aesthetic purposes due to its spectacular campanulate flowers that are usually orange-red or fiery red. Extracts from the bark, stem, and leaves of *S. campanulata* have been used as a molluscicide and to treat diabetes, malaria, and schistosomiasis (Consoli *et al*., 1988; Makinde *et al*., 1988, 1990). Although it is regarded as an inferior wood, *S. campanulata* wood is used for carving in West Africa. Ethiopia uses it to make charcoal and as firewood (Bekele-Tesemma *et al*., 1993). The sole typical commercial use of the wood is that it is traded as African tulip (in England) or tulipier or plywood (in France). Given the aforementioned, it can be seen that this species is not adequately valued, particularly in Nigeria, which supports this research work on its utilization as a fibrous raw material for pulp and papermaking through the knowledge of its chemical composition. Therefore, it is important to study the chemical composition of *Spathodea campanulata*, as an integral determinant of its use for pulp and papermaking.

Materials and Methods

Sample Preparation

A stand of *Spathodea campanulata* was harvested from a forest as there are no existing plantations of the species. This was converted to billets with special attention to sampling heights of top, middle and base (90%, 50% and 10% of total height). The wood from each of these heights were debarked, chipped and milled for the determination of the chemical components of the wood species.

Determination of Extractive Content

Ethanol-benzene extraction of the milled samples was used to determine the amount of extractives in *S. campanulata* wood in accordance with ASTM designation D1107-56. A soxhlet apparatus was used to extract approximately 5g of the milled sample, whose moisture content had been determined, using 200cm^3 of ethanol-benzene at a ratio of 1:2 for 8 hours. Suction was used to filter the extracted sample before it was extracted again for four hours using 95% ethanol. After washing the solvent from the sample with hot distilled water, it was allowed to air dry for three days before being weighed. Using the following formula, the percentage extractive content was determined:

$$\% \text{ Extractive} = \frac{Wu - We}{Wu} \frac{x\ 100}{1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 1$$

Where,

Wu = Weight of un-extracted sawdust (g)

We = Weight of extracted sawdust (g)

Determination of Lignin Content

The ASTM designation, D1106-56, was used to calculate the amount of lignin present in the species under study. In order to perform this procedure, 1g of extractive-free oven-dried sawdust was dissolved for two hours at room temperature in 15cm^3 of 72% cold sulphuric acid. Hot distilled water was added to the content and left to boil at a steady volume for four hours; to which 475 cm^3 of water was later added. After settling for a full night, the resulting insoluble lignin was then filtered and its residue was washed with hot distilled water until it was neutral to litmus. The residue was dried in an oven at 85ºC to constant weight. It was determined that the percentage of insoluble lignin was:

$$\%\text{Lignin} = \frac{\omega L}{\omega o}\ x\ \frac{100}{1} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2$$

Where

w_L= Weight of lignin (g).

w_o= Oven dry weight of extractive free milled sample (g)

Determination of Hemicellulose Content

One gram of moisture-free, extracted biomass was put into a 250 milliliter conical flask to which 150 ml of 500 mol/m^3 of NaOH was added and left to boil for 3.5 hours in distilled water. After cooling completely, it was filtered and washed. The residue was dried at 105ºC±2 until it reached a constant weight. The difference in sample weight before and after this treatment is known as the hemicellulose content (%w/w) of dry biomass (Blasi *et al*., 1999; Lin *et al*., 2010 and Ayeni *et al*., 2013).

$$\text{Hemicellulose} = \frac{\omega i - \omega 2}{\omega 1}\ x\ \frac{100}{1} \quad \ldots\ldots\ldots\ldots\ldots 3$$

Where,

W_1=weight of the original moisture free and extractive free milled sample (g)

W_2= weight of oven dried sample (g)

Determination of Alpha-cellulose Content

This test was conducted based on ASTM designation D1103-60 (1974). A 250 cm³ glass beaker was filled with 2g of the extractive free material to which 250 cm³ of 17.5% NaOH solution was added. The mixture was left for two minutes, before 10cm^3 of 17.5% NaOH was introduced, and the hemicellulose was gently agitated using a glass rod to achieve a uniformly distributed substance. Five minutes later, another 5cm^3 of 17.5% NaOH was added, stirred, and allowed to stand for thirty minutes. After this time, 33 cm^3 of cold distilled water was then added to the mixture to raise its percentage to 8.3%. For one hour, the entire content was left to stand before the addition of 100cm^3 of 8.3% NaOH. This last

caustic extraction was completed after an hour. After that, distilled water was used to wash the residue and a glass rod was used to distribute it. The whole procedure was repeated twice after which the residue was steeped in 15 cm^3 of glacial acetic acid for three minutes. After that, the residue was washed, put back into a crucible of known weight, and dried to a constant weight at 103ºC in an oven. The percentage alpha cellulose was then calculated as follows:

$$\% \text{ Alpha – Cellulose} = \frac{\omega i - \omega 2}{\omega 1} x \frac{100}{1} \quad \text{.....4}$$

Where,

W_2= weight of oven dried alpha cellulose (g)

W_1=weight of the original moisture free and extractive free milled sample (g)

Determination of 1% Sodium hydroxide solubility

The 1% NaOH solubility test for the milled *S. campanulata* sample was determined using ASTM designation D1109-56. A 200cm^3 beaker was filled with about 2g of the air-dried, moisture-free milled sample and 100cm^3 of 1% NaOH. The beaker containing the mixture was covered and submerged in a bath of boiling water for one hour, with stirring at intervals of ten, fifteen, and twenty-five minutes during the extraction process. After the allotted time had passed, the material was filtered by suction onto a tarred crucible and then thoroughly washed with hot water, 50ml of 10% acetic acid, and 100ml of hot distilled water respectively. After that, the crucible and its contents were dried at 100±2ºC in an oven until a constant weight was reached The 1% NaOH solubility was calculated as follows:

$$\% \text{ NaOH solubility} = \frac{W1 - W2}{W1} \frac{x100}{1} \quad \text{.....5}$$

Where,

W1= Weight of moisture free sample (g)

W2= Weight of dried sample after 1% NaOH extraction (g)

Determination of Hot Water Solubility Test

The hot water solubility test yields starch content of the material. This test was conducted using ASTM D1110-56 standard. A reflux condenser was connected to a conical flask that held 2g of milled sample of known moisture content and 100ml of distilled water. With the solution inside the flask slightly below the bath's water level, the flask was submerged in the boiling water bath. The water bath was gradually heated for three hours in order to extract the sample. The sample was then filtered by suction, washed in hot distilled water, and put on a tarred crucible for weighing. The result was calculated and reported as a percentage of matter soluble in hot water on a moisture-free basis after the sample and crucible were dried to constant weight at 100±2ºC in an oven:

$$\%\text{Hot water solubility} = \frac{\omega i - \omega 2}{\omega 1} x \frac{100}{1} \quad \text{.....6}$$

Where,

W1 = Weight of moisture free milled sample (g)

W2 = Weight of dried sample after extraction with hot water (g)

Determination of Cold Water Solubility Test

The tannins, gums, sugar, and colouring matter in the milled sample of *Spathodea campanulata* wood were measured by this test. The test was conducted using ASTM D1110-56 as a guide. A 400 ml beaker containing 2g of a milled sample of known moisture content was filled with 300 cm^3 of distilled water. The mixture was constantly stirred for 48 hours at a temperature of 27±2ºC to aid its digestion. After that, the sample was suction-filtered, washed in cold distilled water, and set on a crucible and weighed. In an oven set to 100±2ºC, the sample and crucible were dried to a constant weight. The result was calculated as follows and presented as a percentage of matter soluble in cold water:

$$\% \text{ Cold water solubility} = \frac{\omega i - \omega 2}{\omega 1} x \frac{100}{1} \quad \text{.....7}$$

Where,

W1 = Weight of moisture free milled sample (g)

W2 = Weight of dried sample after extraction with cold water (g)

Results and Discussion

Chemical Analysis of Spathodea campanulata Wood

Extractive Content

The height of the tree from which samples were collected greatly affected the extractive content as evident in Table 2 ($p < 0.05$). In Table 1, it was observed that the wood at the base had the least extractive content (2.94±0.20%), closely followed by the middle (3.41±0.24%), and the wood at the top had the highest extractives (4.62±0.32%). This low level of extractives at the basal region was due to the presence of reaction wood that extends a little to the middle of the tree. Meanwhile, the average ethanol-benzene extractive content of *S. campanulate* as deduced here was 3.66±0.20%. This trend of values corroborates the findings of Oliveira *et al* (2023) who found no direct relationship between extractive content and age; therefore supporting the claim of Mascarenhas *et al*. (2021) that extractive contents of wood rather depends on factors such as environmental, genetics, silvicultural techniques and management choices rather than age.

Ethanol-benzene extractives of wood consist of waxes, fats, resins, photosterols, non-volatile hydrocarbons, low molecular weight carbohydrates, salts and water soluble substances. Its contents varied with sampling height as deduced from this study showing an increase with maturity age. Meanwhile, the average extractive content of *S. campanulata*as determined from this study was 3.66. The value obtained for this study is lower when compared with the range of 5.3–7.8 % reported for *Acacia melanoxylon* (Santos *et al* 2012).. This value is also within the 1-8% extractive contents of most hardwood and softwoods (As *et al*., 2002) as well as the 3.1% for *Eucalyptus spp* (Pereira *et al*., 2013) but lower than 5.0% submitted for *Populus spp* (Kacik *et al*., 2012), so it is expected that liquor consumption will be low during pulping.

Lignin Content

Sampling height has no effect on the lignin content of *S. campanulata* wood as observed in Table 2 at $p > 0.05$. Meanwhile the lignin content appeared to increase axially in the wood The wood from the base produced the lowest

lignin content, 22.76±0.37% (Table 1) in this study. This is closely followed by 23.52±0.34% obtained in both middle and top woods respectively (Table 1). The average lignin content of *S. campanulata* as obtained from this study was 23.27±0.18%

Lignin is responsible for the strength of individual fibres and cell wall stiffness in order to protect the carbohydrates from chemical and physical damage (Khalil *et al*., 2006). However, it is an undesirable element in pulp and papermaking which is why pulping is carried out to remove it from a fibre.

The average lignin content of *Spathodea campanulata* used for this study, 23.28%. is slightly higher compared to the range of 19.8 to 22.4 % for Klason lignin in *Acacia melanoxylon* (Santos *et al* 2012). It is however lower than the values of 28.0-31.7 and 24.57 and 31.77 reported for acid insoluble and alkaline insoluble lignin of Gmelina and teak mill wood residues respectively (Oluwadare *et al*., 2016). This value implied that there will be mild consumption of active alkali to pulp the *S. campanulata* wood. Ndukwe *et al* (2009) opined that the amount and type of lignin present in wood species have an effect on the pulp yield of such woods.

Hemicellulose Content

Hemicellulose content was highest at the base, 26.82±0.37% closely followed by the top with a value of 26.48±0.17% while the middle wood had the lowest (26.11±0.13%) as evident in Table 1. However, *S. campanulata* had an average hemicellulose content of 26.47±0.14. Sampling height does not affect the hemicellulose content of *S, campanulata* as seen from Table 2 ($p > 0.05$). Not much difference was observed in the values from the top to the base.

The hemicellulose determined from this study is slightly higher than the 17-25 postulated for hardwood but it is a desirable advantage as it has been known to increase the strength of papers. The average obtained here is however higher compared to the 24.46- 25.09 reported for silver birch (Lachowicz *et al*., 2019) but within the range of 15-35% opined for hardwoods (Abik *et al*., 2023)

Alpha-cellulose Content

The alpha cellulose content of *Spathodea campanulata* was greatly influenced by the height at which each sample was collected ($p < 0.05$) (Table 2). Also, the wood at the top had the highest alpha-cellulose content of 44.50±0.12% (Table 1). This value is closely followed by 44.40±0.28% at the base while wood from the middle had the lowest, 43.73±0.12% (Table 1). This trend corresponds to what was observed in *Acacia mangium* where the highest alpha cellulose content was found in the top wood. The average alpha cellulose content obtained from this study was 44.21±0.12% which is lower than the 47.00-49.84% reported for *Acacia mangium* wood (Wahab *et al*., 2017) but higher than the 41.09, 39.82 and 42.91 submitted for *Nauclea diderrichii, Prosopis africana* and *Brachystegia eurycoma* (Duruaku et al.,2023).

In order to establish the suitability of *S. campanulata* for pulp and papermaking, it must possess high quality fibres. Cellulose content is one of the major qualities to be determined. However, the average alpha cellulose content of *S. campanulata* obtained from the result of this study was 44. 21. This value correlates with the 40-45% reported for softwoods (As *et al*., 2002). It also falls in line with the Nieschlag *et al* (1960) classification in which it was reported that alpha cellulose contents as high as 34% and above are better suited for making pulp and paper from any lignocellulosic material. Hence, *S. campanulata* is highly suitable for pulp and paper making.

Table 1: Effect of Sampling Height on the Chemical composition of *S. campanulata* Wood

Sampling Height	Lignin	Extractive	Hemicellulose	Alpha cellulose
Base	22.76±0.37	2.94±0.20^{b}	26.82±0.37	44.40±0.28^{b}
Middle	23.52±0.09	3.41±0.24^{b}	26.11±0.13	43.73±0.12^{a}
Top	23.52±0.34	4.62±0.32^{a}	26.48±0.17	44.50±0.12^{b}
Mean	23.27±0.18	3.66±0.20	26.47±0.14	44.21±0.12

*Means ± Standard error of mean of four replicate samples. Values with the same alphabet in each column are not significantly different at $\alpha = 0.05$ using Duncan multiple range test.

Table 2: Variations in the Chemical composition of *S. campanulata* Wood

Chemical variables	Lignin	Extractive	Hemicellulose	Alpha cellulose
P-value	0.129	0.000	0.150	0.017

* P-value< 0.05 are significant at $\alpha = 0.05$

1% Sodium Hydroxide Solubility Test

Table 4 below showed that sampling height had no effect on the 1% sodium hydroxide solubility of *S. campanulata* ($p > 0.05$). In this study, the highest sugars were found at the middle (15.30±0.76) closely followed by the top (14.40±0.95) and lowest at the base (13.53±0.30) as observed in Table 3. The average sugar content of *S. campanulata* as measured by the 1% sodium hydroxide solubility test was 14.41±0.43%

This extractive is used to assess the liability of the wood fibres to fungal decay with time in storage and expected yield when subjected to alkaline pulping. The average polysaccharides that can be obtained from *S. campanulata* as deduced from this study were 13.53 to 15.30%. It is closer to the 8-14% postulated for softwoods and hardwoods respectively (As *et al*., 2002), but lower than 16.1 to 24.3% for alkaline solubility obtained for *Casuarina equisetifolia, Terminalia cattappa, Elaeocarpus robusta, Albizia richardiana, Khaya anthotheca, Albizia procera and Terminalia arjuna* (Hossain *et al*., 2023). This value will lead to better pulp yield, low cost of pulping and bleaching. This low value could be due to the presence of low molecular weight carbohydrates (Sotannde, 2014). The moderately low alkali soluble extracts of *S. campanulata* wood is favourable for good pulping since it has been reported to be negatively correlated with pulp yield.

Hot and Cold Water Solubilities of S. campaanulata Wood

There was an increasing trend from the top to the base in hot and cold water solubility test results. The highest value for hot water, 9.54±0.09, was found at the base while the lowest (8.97±0.18) was observed at the top (Table 3). For the cold water, the range of 7.67±0.23 – 8.23±0.10 was noticed from the top to the base respectively (Table 3). Water solubility is an indication of lower molecular weight components and polysaccharides present in a lignocellulosuic material. It gives the amount of tannins, gums, sugar, colouring matter and starches. In this study, both the hot and cold water solubility increased with maturity as there was a decrease from the base on the top. This trend supports the previous findings on wood species such as *Casuarina equisetifolia, Terminalia cattappa, Elaeocarpus robusta, Albizia richardiana* among many others. The range of 8.97 to 9.54%, and 7.91 to 8.23% for hot water and cold water solubilities were higher compared to 1.0 to 5.7% and 0.9 to 8.3% for cold and hot water of seven hardwood species assessed by Hossain *et al* (2022). According to Hossain *et al* (2022), high hot water solubility is an indication of easy degradation of polysaccharides and translates to less chemical consumption especially during pulping. The relative low values of both hot and cold water solubility of *S. campanulata* wood are more flexible for good pulp (Uprenda and Shukla, 2010).

Table 3: Solubility of *S. campanulata* Wood based on Sampling Height

Sampling Height	1%NaOH	Hot water	Cold water
Base	13.53±0.30	9.54±0.09	8.23±0.10
Middle	15.30±0.76	9.12±0.28	7.84±0.23
Top	14.40±0.95	8.97±0.18	7.67±0.23
Mean	14.41±0.43	9.21±0.12	7.91±0.12

*Means ± Standard error of mean of four replicate samples. Values with the same alphabet in each column are not significantly different at α = 0.05 using Duncan multiple range test.

Table 4: Variations in the Solubility of *S. campanulata* Wood

Chemical variables	1%NaOH	Hot water	Cold water
P-value	0.247	0.134	0.142

* P-value< 0.05 are significant at α = 0.05

Conclusion

The lower percentage of lignin showed that milder pulping conditions will be required for *S. campanulata* just as the low to moderate percentage of soluble contents and extractives in the wood implied that the wood of *S. campanulata* will be easily degraded for quick pulping and can stay long in storage with minimal degradation. However, the high percentage of alpha-cellulose and hemicelluloses gave an indication of high pulp yield of the wood.

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