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SOLUBLE EXTRACTS PRESENT IN *FICUS EXASPERATA* (VAHL.) SUITABLE FOR PULP MAKING

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Abstract:
This study was undertaken to determine the solublel extracts in Ficus exasperata (vahl.). Soluble extract is a vital information that should be considered in determining the suitability of plant for proper utilization. Soluble extract test (Moisture content, %NaOH solubility test, Hot and Cold water solubility test, Alpha-cellulose, Holocellulose and lignin) were carried out using ASTM methods. The result shows that moisture content of F. exasperata is 82.67% while *soluble extracts which includes; 1%NaOH solubility test, Hot and Cold water solubility test, Alpha-cellulose and Holocellulose had average values of 10.43, 12.71, 11.29, 16.63 and 51.94% respectively while Acid insoluble lignin (AIL) and Alkaline hydrolysis Lignin (AHL) were 18.27 and 1.08% respectively. It was observed that the low lignin content in F. exasperata is an indication that the species can be pulped under mild condition due to low bonding strength. The moderate content of other soluble extracts is an indication that the wood can last long in storage with less degradation.*

Key words*: holocellulose; moisture content; pulping; lignin; soluble extract; sodium hydroxide test.*

INTRODUCTION

Papermaking is an economic viable endeavour which has contributed immensely to the growth and development of industries and nations around the world. It is unfortunate today in Nigeria, that pulp and paper industries are in a state of disrepair and most of the papers being used are imported. Several wood species like *Gmelina arborea*, *Recinodendron eudelotii Eucalyptus camadulensis* amongst others have been identified and used for paper production around the world, probably for the chemical constituents of their wood, which is one of the many parameters that should be considered.

Soluble extracts in plant generally, determines or enhances the suitability of such plant (as raw material) for pulp and paper making (Keller *et al*. 2002). These extracts in wood include cellulose, hemicellulose, lignin and other organic matter (Ververis *et al.* 2004). Cellulose and hemicellulose are very abundant natural polymers that are found in wood fibre and usually very important at determining the tensile strength of paper produced. Nevell and Zenonian, (1985) opined that the higher the hemicellulose constituent in a plant, the more the mechanical strength properties, though the strength varies from one species to the other.

Ficus exasperata is one of the fast growing lesser used wood species mostly found in the rain forest, savannah and besides river and streams (Keay 1964, Odunbaku *et al.* 2008). Different parts of the plant has been reported to have been used for medicinal and industrial purposes (Cousins and Michael 2002; Buniyamin *et al.* 2007; Odunbanku *et al.* 2008; Sonibare *et al.* 2008), but information on other potentials like paper making has not been ascertained. Paper is a material that everyone uses, its demand is high and most of the desirable wood species for paper production are gradually becoming scarce.

In order to ease the stress and ensure sustainability, this study considers it reasonable to examine the percentage of soluble extracts present in the wood of *Ficus exasperata* as a viable alternative for paper production.

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MATERIALS AND METHODS

Ficus exasperata stand was felled from the Arboretum in Forestry Research Institute of Nigeria, Ibadan, Oyo State. The site is located along latitude 7°23'34''N to 7°23'36''N and longitude 3°51'36''E to 3°51'36''E. The dry season is from November to March with an average temperature range of 26°C to 32°C.

The tree was felled for destructive sampling, delimbed and cross-cut into billets (60cm) while samples were collected at the base (10%), middle (50%) and top (90%) along the sampling height.

The methods to determine soluble extract were as follows

Percentage Moisture Content

Since moisture is one of the main component of a plant material, which also serve as one of the determinant factor in the use of plant fibre for pulp production. Each of the fresh samples were cut from corewood, middle wood and outerwood into approximately 2x2x2cm and labelled. The labelled samples were dried in the oven at temperature of 100±2^{O}C until constant weight was obtained. The percentage moisture content (M.C) in relation to the fibre component was determined using ASTM D1348-94 (2008). The final moisture content was obtained using the formula below:

$$M.C = \frac{W_0 - W_1}{W_1} \times \frac{100}{1} \qquad (1)$$

where: M.C = Moisture content (%), W_O = Green weight (g), W_1= Oven dry weight (g).

1% Sodium Hydroxide Solubility Test (1%NaOH)

The solubility of the milled sampled wood in hot dilute alkali solution was determined using ASTM D1109-56 1972 in which 2g of air-dried moisture-free milled sample was placed in 200cm^3 beaker followed by addition of 100cm^3 1% NaOH. The beaker was covered and placed in boiling water bath for 1 hour. The content was stirred 3 times at 10, 15, and 25 minutes of the extraction period. After this, the content was filtered by suction on a tarred crucible and washed with 100ml hot distilled water, 50ml of 10% acetic acid and thoroughly with hot water in-turn. The crucible and the content were dried in an oven at 100±2°C to constant weight. The result was reported as percentage by weight of matter soluble in 1% NaOH solution as follow:

$$1\% NaOH = \frac{W_1 - W_2}{W_1} x \frac{100}{1} \qquad (2)$$

where: w_1 = Weight of moisture free milled sample;
w_2 = Weight of dried sample after 1% NaOH extraction.

Hot Water Solubility Test (%)

The hot water solubility test in addition provides a measure of amount of starches. The test was carried out based on ASTM D1110-56 (1977). 2g of milled sample of known moisture content was placed in Erlenmeer flask filled with 100mL of distilled water and attached to reflux condenser. The flask was placed in the boiling water bath with the solution in the flask just below the level of water in the bath. The sample was extracted by heating the water bath gently for 3 hours. Thereafter, the sample was filtered by suction, washed with hot distilled water and placed on tarred crucible of known weight. The crucible was dried with the sample in an oven at 100±2°C to constant weight; the result was reported as a percentage of matter soluble in hot water on the moisture free basis and calculated as follow:

$$\% HotWaterSolubility = \frac{W_1 - W_2}{W_1} x \frac{100}{1} \qquad (3)$$

where: w_1 = Weight of moisture free milled sample;
w_2 = Weight of dried sample after extraction with hot water.

Cold Water Solubility Test (%)

This test provides a measure of the contents of tannins, gum sugar and coloring matter in the milled sample of the wood of *Ficus exasperata*. The test was carried out based on ASTM D1110-56 (1977). 2g of milled sample of known moisture content was placed in 400mL beaker and covered with 300cm^3 of distilled water. The mixture was digested at a temperature of 27±2°C for 48 hours with frequent stirring. The sample was filtered by suction, washed with cold distilled water and placed on tarred crucible of known weight. The result was reported as a percentage of matter solution in cold water on the moisture free basis and calculated as follow:

$$\%ColdWaterSolubility = \frac{W_1 - W_2}{W_1} x \frac{100}{1} \quad (4)$$

where: w_1 = Weight of moisture free milled sample;
w_2 = Weight of dried sample after extraction with cold water.

Lignin Content (%)

This was determined by comparing two different extraction methods:

Method 1: ASTM Standard Test Method for Acid-Insoluble Lignin in Wood: D 1106 – 56 (1977)

Three grammes (3g) of the samples were weighed in tared glass-stoppered weighing bottles. It was dried in an oven for 2hrs at 100 to 105°C, and allowed to cool in a desiccator. The stopper was loosed to equalize the pressure and weighed. Drying and weighing continued for 1h period until the weight was constant.

In the extraction crucibles, two additional 10g test specimens were weighed for the lignin determination in duplicate. The extraction crucible containing the specimen was placed in a Soxhlet extraction apparatus. Extraction was done with 95% alcohol for 4 hours, and then with ethanol-toluene solution as described in Test Method D 1107. Solvent was removed by suction as possible and washed by suction with 50 ml of ethanol to remove the toluene. Also excess ethanol was removed, transferred to a beaker, and digest with 400mL of hot water in a hot-water bath at approximately 100°C for 3hrs. It was filtered, washed with 100mL of hot water, and finally with 50ml of ethanol to facilitate the removal of the test specimen from the crucible. After these preliminary extractions, the specimens were air dried.

Two (2g) of the air-dried test specimen was transferred to a small beaker with a glass cover and 15mL of cold (12 to 15°C) H_2SO_4 (72%) was added slowly, while stirring. The specimen was mixed with the acid by stirring constantly for at least 1 min. It was allowed to stand for 2hrs, with frequent stirring, at a temperature of 18 to 20°C. A water bath was used to keep the temperature within these limits. The material was washed into a 1-L beaker, diluted to a 3% concentration of H_2SO_4 by adding 560mL of distilled water, and boiled for 4hrs, in a nearly constant volume condition maintained by the occasional addition of hot water to the flask. After allowing the insoluble material to settle**,** it was filtered into a filtering crucible that has been dried at 100±5°C and weighed in a glass-stopper weighing bottle. The residue was washed free of acid with 500mL of hot water and the crucible and contents was dried in an oven for 2h at 100±5°C, cool in a desiccator, and the contents of the crucible as lignin was weighed. The drying and weighing was repeated until the weight was constant.

Method 2: Alkaline Hydrolysis Procedure.

The first step involved drying the sawdust in an oven at 60°C for 16-24 hours. This was performed in line with alkaline hydrolysis procedure adopted by Sun *et al.* (1995) with some modifications. As a first step, the sawdust was dewaxed for 5hrs using toluene and ethanol (2:1, v/v) using a Goldfisch (soxhlet) apparatus. To obtain alkali soluble lignin, the dewaxed sawdust was subjected to hydrolysis with 1.25M of NaOH at 80°C for 5hrs (2g sample per 44ml extractant). The hydrolysate was filtered with a glass filter and washed with water (3×10mL), followed by ethanol (2×10mL), and acetone (1×10mL). This created two fractions, a filtrate and an alkaline treated residue. The pH of the filtrate was reduced to 5.5 by addition of 6M of HCl. The hemicellulose was separated from the filtrate by precipitation of the neutralized filtrate in 3 volumes of ethanol. After filtration with a glass filter, the precipitated material (i.e. hemicelluloses) was washed with 70% ethanol and allowed to air dry. Ethanol was evaporated from the filtrate, and the alkali soluble lignin was obtained from the filtrate by precipitation at pH of 1.5 by addition of 6 M of HCl. The alkali soluble lignin was washed with acidified water (pH 2), centrifuged and freeze dried. All extractions were conducted in triplicate.

$$\%LigninContent = \frac{Weight of Lignin}{Ovendry weight of extracted free milled sample} x \frac{100}{1} \quad (5)$$

Alpha-Cellulose (%)

This test was based on ASTM D1103-60 (1974). 2g of the sample was poured into a 250cm^3 glass beaker followed by addition of 250cm^{3}of 17.5% NaOH solution. After 2 minutes, 10 cm^3 of 17.5% NaOH was added and the holocellulose macerated lightly with a glass rod to get a well dispersed material. After 5 minutes, another 5cm^3 of 17.5% NaOH was added, stirred and left to stand for 30 minutes. After this, 33cm^3 of cold distilled water was added to bring the solution to 8%. The whole content was allowed to stand for 1 hour. The final caustic extraction was done after 1 hour. Thereafter, the residue was washed with distilled water and dispersed with glass rod. The above steps were repeated twice while the residue was steeped in 15cm^3 glacial acetic acid for 3 minutes. The residue was washed thoroughly with cold distilled water until it became neutr l to blue litmus paper.

The washed residue was transferred back into the crucible of known weight and dried in an oven for 15-20 hours at 103±2°C to constant weight. The percentage alpha cellulose was then calculated using the equation below:

$$\%Alpha-cellulose = \frac{W_2}{W_1} x \frac{100}{1} \quad (7)$$

Where: w_2 = Weight of oven dried alpha cellulose;
w_1 = Weight of the original moisture free and extractive free milled sample.

Holocellulose Content (%)

Holocellulose is a measure of all carbohydrate in the wood of *Ficus exasperata*. The holocellulose content was determined using ASTM D1104-56 (1978). In this method, 2g of extractive free sawdust was moisten with cold distilled water and the excess moisture removed by suction. The sample was chlorinated for 5 minutes followed by extraction with 50mL of 95% ethanol and hot ethanol-monoethanolamine solution. At the end of each extraction, the residue was washed thoroughly with distilled water followed by another round of extraction until the residue becomes white. The washing exercise was also repeated until the residue was neutral to litmus. The residue obtained was then oven-dried to constant weight. The percentage holocellulose based on moisture – free extractive – free milled sample was calculated as follow:

$$\%Holocellulose = \frac{W_0}{W_1} x \frac{100}{1} \quad (6)$$

where: W_0 = Weight of oven dried holocellulose residue (g);
W_1 = Weight of moisture free and extractive free milled sample (g).

RESULTS AND DISCUSSION

Percentage Moisture Content (MC)

Table 1 shows that mean moisture content of *F. exasperata*. Effect of variation along the sampling height shows that moisture content increased from 78.10% at the base to 98.49% at the middle and slightly decreased to 93.49% at the top. Moisture content of *F. exasperata* averaged 82.67% (Fig. 1).

The moisture content of 82% obtained along the sampling height and radial position is high. This is an indication that moisture is one of the major components of the wood. It was also discovered that the moisture content was higher at the outerwood than corewood, and higher at the top of the wood than the base. As the moisture content increased, the overall size of the wood fibres also increased (Gao 2017). The high moisture content obtained in this wood suggests that efficient bailer will be required to densify the wood due to its volume for the purpose of handling, transportation and storage.

Table 1

Individual Mean Values along Sampling Height

Wood portion	Moisture content	1% NaOH	Hot Water (%)	Cold Water (%)
Base	78.10±26.79	10.44±1.36	12.69±1.36	11.18±0.39
Middle	98.49±25.04	9.77±0.49	11.63±0.58	10.52±1.02
Top	93.49±10.01	11.08±2.76	13.81±0.58	12.16±2.89

*Means± Standard error of mean of 3 replicate samples.

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Sodium Hydroxide Solubility (1%NaOH)

The Mean values of sodium hydroxide test as presented in Table 1 shows that there in no specific pattern of variation along the sampling height. The highest content of 11.08% was obtained at the top, closely followed by 10.44% at the base while lowest percentage NaOH of 9.77% was obtained at the middle. On average, 10.43% of Sodium Hydroxide Solubility was obtained in *F. exasperata* (Fig. 1).

The low sodium hydroxide soluble content of *F. exasperata* could be due to the occurrence of low molecular weight carbohydrates and other alkali soluble materials compared to other wood species. Sodium hydroxide solubility of the wood varied along the sampling height. The increase in sodium hydroxide solubility from 9.77% at the middle to 11.08% at the top is an indication that degradation of the wood due to fungal decay, heat and light will be more at the top than middle and the base. The sodium hydroxide soluble extracts 10.43% in the wood of *F. exasperata* is within the range of 8-15% in soft and hardwoods, respectively (As *et al*. 2002). This finding also agreed to decrease in sodium hydroxide solubility of milled wood of bamboo (non-wood) with maturity reported by Ogunsile and Uwajeh (2009). The low alkali soluble extracts of the wood of *F. exasperata* is more favourable for good pulping since it has been shown to be negatively correlated with pulp yield (Singh *et al*. 2011). This is more advantageous since extra cost on biotechnical and enzymatic pre-treatment commonly used for woody plants to prevent bio-deterioration will be low, thereby reducing cost of pulping as much as possible.

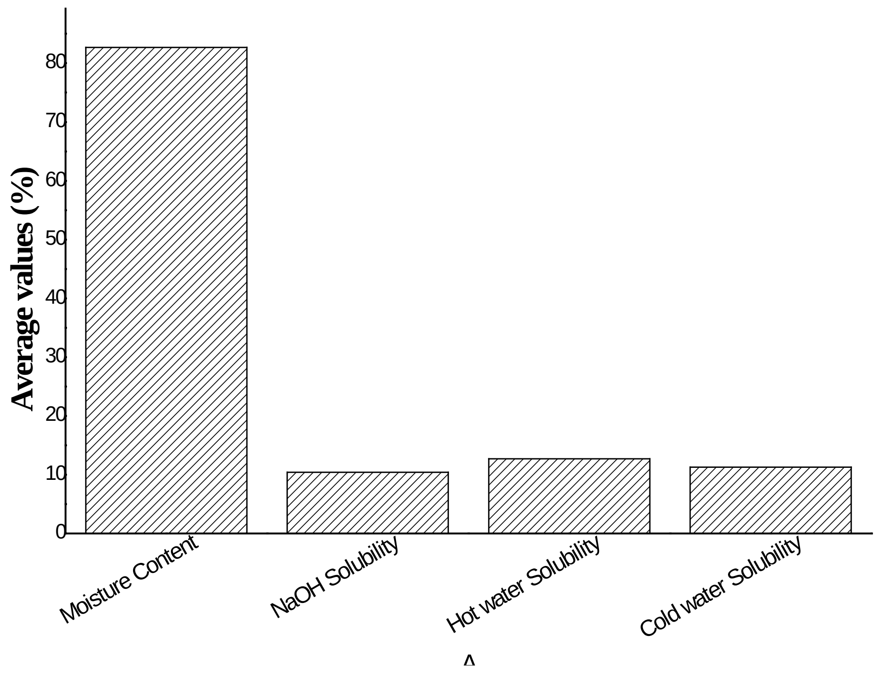

Fig. 1.
Average Moisture Content, NaOH and Water Solubility.

Hot and Cold Water Solubility (%)

Mean values presented in Table 1 showed Hot water solubility test of wood of *F. exasperate*. The trend along the sampling height for both hot and cold water solubility test shows no pattern of variation, hot water solubility of *F. exasperata* increased from 12.18% at the base to 13.41% at the middle and decreased at the top 12.54%. Also, the highest recorded cold-water content solubility of 12.16% was obtained at the top followed by 11.18% at the base while the least content of 10.52% was obtained at the middle.On average, hot and cold water soluble extract is 12.71% and 11.29% respectively (Fig. 1).

In this study, both hot and cold-water solubility of the wood were lowest at the mid-stem and highest at the stem top. The average value of 12.71 and 11.29% for hot water test and cold water solubility test respectively were higher than the values recorded by Agnihotri *et al*. (2010) and Dutt *et al*. (2010). The relative low values of both hot and cold water solubility of MWR made the species more flexible for good pulp (Uprenda and Shukla 2010).

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Lignin Content (%)

The mean values of lignin content extracted using the two methods is presented in Table 2. The lignin yield of the *F. exasperata* differ with respect to extraction methods. The content of AIL along the sampling height did not show any particular trend of variation, AIL yielded more with 18.98% obtained at the top closely followed by 18.07% at the base while the lowest yield of AIL content was obtained at the middle (17.78%). On the other hand, AHL showed a parttern of variation, it increased slightly from the base to the top with the highest AHL content of 1.07% at the top and lowest content of 1.00% at the base. However, average content of 18.27 and 1.08% were obtained for AIL and AHL respectively (Fig. 2).

The main function of lignin in plant tissues and fibres is to give comprehensive stiffens and strength to the cell-wall of the fibres, to protect from physical damage and the carbohydrates from chemical (Abdul Khalil *et al*. 2006). Most of the paper industry would desire low amount of lignin of about <15% because the fibres are more flexible, porous and they provide brighter colour in bleaching process (Syed *et al*. 2016). In this study, lignin in the wood of *F. exasperata* was isolated by comparing two methods of extraction in order to discover the best extraction method that can give high percentage lignin content. However, Acid Insoluble Lignin (AIL) gave higher lignin content averaged 18.27% while Alkaline Hydrolysis Lignin (AHL) is lower with lignin content of 1.02%. The percentage lignin content of *F. exasperata* was discovered to be low compare to the result of other wood species. High accumulation of lignin was obtained 28.0 (AIL) and 24.57% (AHL) for *Gmelina arborea* and 31.77 (AIL) and 31.70% (AHL) for *Tectona grandis* by Oluwadare *et al.* (2016). Also, the values reported in *F. exasperata* were found to be lower than, 23.4-34.5% in *Eucalyptus globules* (Anttonen *et al*. 2002), 20.59% for *Cramble tataria* (Tutus *et al*. 2010), 25-32% for softwoods, though AIL falls within 17-26% for hardwoods (Eroglu 1998). High yield of AIL when compared with AHL could probably be due to non-lignin components present in the wall of the wood that were dissolved by alkaline solution or due to the presence of some compounds in the wood that were not completely hydrolysed by the acid (Oluwadare *et al.* 2016). It is also reported that large amount of cellulose and low lignin quantity gives a greater paper making potential as well as boards and composite material production (Cordeiro *et al*. 2004).

Table 2

Individual Mean Values along the sampling height

Wood portion	AIL (%)	AHL (%)	Alpha-cellulose (%)	Holo-cellulose (%)
Base	18.07±3.62	1.00±0.21	16.20±2.89	51.74±3.89
Middle	17.78±0.69	1.01±0.13	20.67±4.83	55.03±6.38
Top	18.98±0.49	1.07±0.19	13.02±1.96	49.04±0.36

*Mean± Standard error of mean of 3 replicate samples.

Alpha-cellulose Content and Holocellulose (%)

Table 2 shows the mean values of Alpha-cellulose and holocellulose. Along the sampling height, the trend of variation for Alpha-cellulose content didn't show any distinct pattern of variation, the highest content of 20.67% was obtained at the middle, followed by 16.20% at the base while the lowest content of 13.02% was obtained at the top. Likewise, holocellulose content exhibited no pattern of variation; the samples collected middle had the highest percentage of 55.03%, followed by 51.74% obtained at the base while the top had the lowest content of 49.04%.

On average, 16.63% was obtained for percentage Alpha-cellulose content while Holocellulose content was 51.94%. Average value of in the MWR of *F. exasperata* (Fig. 2).

According to Agu *et al.* (2012), high cellulose content of wood will provide suitability and high tensile strength for pulp and paper produced. High fibre qualities are required in order to use the wood of *F. exasperata* for pulp and paper production. Holocellulose, which gives an indication of cellulose and hemicellulose content, is the chief constituent of plant fibres in paper making. It gives a qualitative indication of a fibre raw material for papermaking. Holocellulose and alpha-cellulose contents in the fibre of *F. exasperata* varied among trees and along sampling height. Alpha-cellulose and holocellulose contents averaged 16.63% and 51.94% respectively. The wood of *F. exasperata* could be classified as a fibrous plant as evidenced by its relatively high holocellulose content. Nevertheless, the relatively high holocellulose content of the wood is a qualitative indication of the suitability of the wood for paper making. Since high amount of holocellulose is said to be required for paper production due to its correlation with greater yield of pulp (Shakhes *et al*. 2011b). The greater the amount of holocellulose in a material, the better the swelling behaviour of the pulp produced from it (Singh *et al*. 2011). Similarly, Nevell and Zeronain (1985) established a positive relationship between holocellulose content and mechanical strength properties of handsheets.

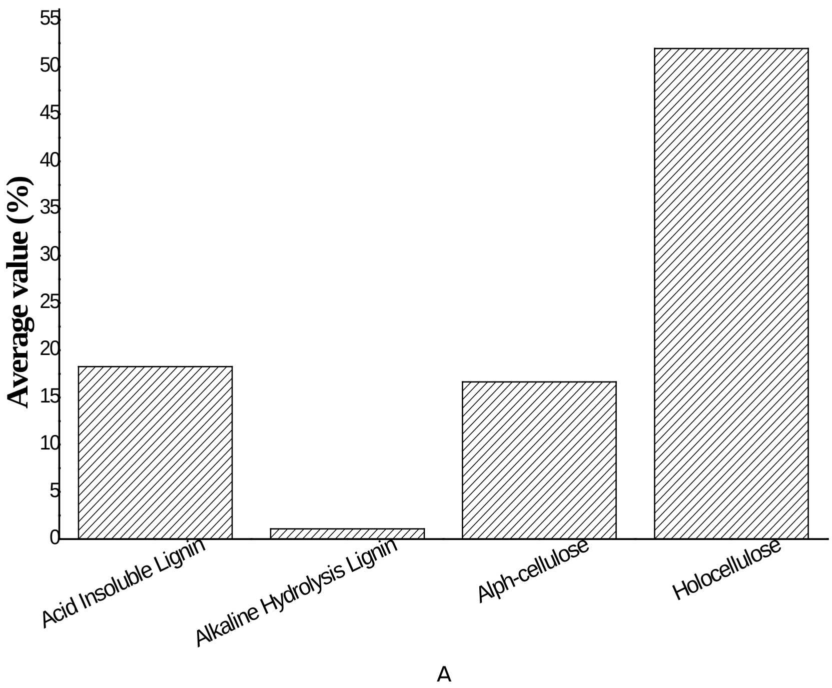

Fig. 2.
Average Lignin Content, Alpha-cellulose and Holocellulose.

CONCLUSIONS

1. The low lignin content is an indication that the *F. exasperata* can be pulped under mild condition without requiring high concentration of chemical during pulping.
2. Moderate percentage of soluble content is an indication that the wood can be stored for a long period of time without much degradation.
3. The holocellulose content of this wood species proved that it can generate high pulp yield.

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