



## EVALUATION OF PULP MAKING PROPERTIES AND KRAFT LIGNIN PRECIPITATION FROM *Ficus exasperata* (Vahl.)

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### ABSTRACT

This research aimed to evaluate pulp making properties and kraft lignin precipitation from *F. exasperata*. Fibre morphology, Kraft pulp yield, kappa number, isolation and precipitation of Kraft lignin were carried out to check the suitability of the raw material. The chips were obtained from the base, mid-stem and stem-top of the tree while cooking liquor was prepared by dissolving 40g NaOH: 13g Na<sub>2</sub>S<sub>2</sub> in 1000mL of distilled water to obtain 17 and 20% effective alkaline. The ratio of NaOH to Na<sub>2</sub>S<sub>2</sub> was 3:1, Liquor to chip ratio was 5:1 and at a varied temperature of 140, 160 and 180°C. The highest pulp yield and kappa number were obtained at 140°C and the lowest was recorded at 180°C. Pulp yield decreases from base to the stem-top along the sampling height from 53.38 – 47.87% at the base, 51.31 – 47.64% at the mid-stem and 50.31 – 45.67% at the stem-top. While the pulp at the base (34.67 -24.00) gave the highest kappa number, followed by (32.44 -30.67) at the mid-stem while the lowest kappa number of 31.89 -29.22 was recorded at the stem-top. Isolated and precipitated Kraft lignin recorded highest yield of 21.3% at 160°C cooking liquor. This study concluded that *F. exasperata* could be easily pulped using Kraft pulping to acceptable yield with moderate Kappa number. Also, kraft lignin was successfully recovered from spent liquor of *F. exasperata*.

**Keywords:** *Ficus exasperata*, kappa number, pulp yield, kraft pulping, lignin yield.

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### INTRODUCTION

*Ficus exasperata* is a tree species generally known as sand paper tree due to its rough leaves. It is said to be one of the best tree species for air filtration (Lucas, 2017). The various parts of the tree were reported to have several curative effect and industrial uses. Other parts of the tree give an abortifacient, oxytocic, analgesic, antidote, diuretic and stomachic effect (PROTA, 2012). It serves in lipid lowering and anti-fungal activities (Sonibare et al. 2008) and many more. It has been reported that *F. exasperata* has a low lignin content that leads to its weak bonding strength,

this suggest that the species can be pulped under mild condition. Moreover, the wood can be stored for a long time with less degradation due to the moderate content of other soluble extracts embedded in it (Anguruwa et al., 2021).

The pulp and paper industry is the 5<sup>th</sup> largest consumer of energy globally. In 2022, the global usage of paper and cardboard was estimated to be 414.19 million metric tons, a rise when compared to the year before (SRD, 2024). Paper production begins with pulping and the aim of wood pulping is to remove lignin from wood

fibre in order to use the fibre in paper production. Approximately 50 million dry tons of lignin was isolated by the pulp and paper industry in 2002 (Anonymous, 2005). There are several methods used for removing lignin, but the most common is Kraft pulping which produces strong pulps for use. Currently, kraft pulping is the leading pulping technology and accounts for 90 % of chemical pulping (Drebot *et al.*, 2021). The Kraft process utilizes a solution of sodium hydroxide and sodium sulphide to treat wood chips at an elevated temperature around 170°C to produce pulp and spent liquor. The spent liquor is a mixture of the removed lignin, chemicals and water used in the extracting process. Kraft pulping utilizes less than half of the tree while the remnants ends up being burnt or spread out on land field. The Kraft process has several advantages over other pulping methods, including a high strength of the Kraft yield, the capacity to handle various wood species, and favorable economics because of its high chemical recovery efficiency (about 97%). According to the role of the Kraft pulping process, it is expected to get high yields of pulp (Drebot *et al.*, 2021).

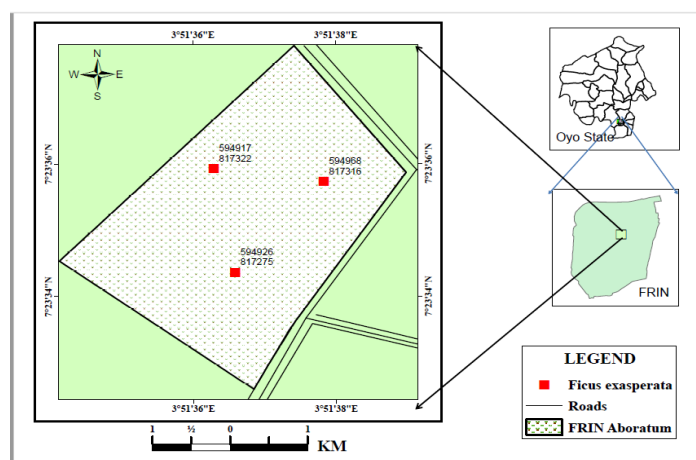
*Ficus exasperate* being a lignocellulosic plant is renewable and could be converted and processed into different feedstock, hence the need to discover possible and various uses of the species for producing chemicals, energy generation and paper production among others. Several works

have been reported on the effect of Kraft pulping process on different biomaterials but there is little information on the Kraft pulping process on *Ficus exasperata*, therefore, the objectives of this study are to investigate the effects of Kraft pulping on *Ficus exasperata* pulp yield and properties.

## MATERIALS AND METHOD

### Study Area and Sample Collection

Three stands of *Ficus exasperata* was felled from the Arboretum in Forestry Research Institute of Nigeria, Ibadan, Oyo State. The site is located along latitudes 7°23'34"N to 7°23'36"N and longitudes 3°51'36"E to 3°51'36"E (Figure 1). The dry season is from November to March with an average temperature range of 26°C to 32°C. The ages of the trees are 25, 20 and 18years with merchantable length of 3.60m, 3.50m and 3.78m and Diameter at breast height (DBH) 20.05, 18.46, and 15.8cm for T1, T2 and T3 respectively. The trees were felled for destructive sampling, delimbed and cross-cut into disc (5cm) and billets (60cm) at the base, mid-stem and stem-top along the sampling height resulting in 9 disc and billets, while Figure 2 shows that along the radial position, each disc was converted into 4 sections. The wood conversion into 2x2x2cm and chips was carried out at the FRM departmental wood workshop.



**Figure 1.** Map of the study area showing study location and tree stands. Survey: 2018

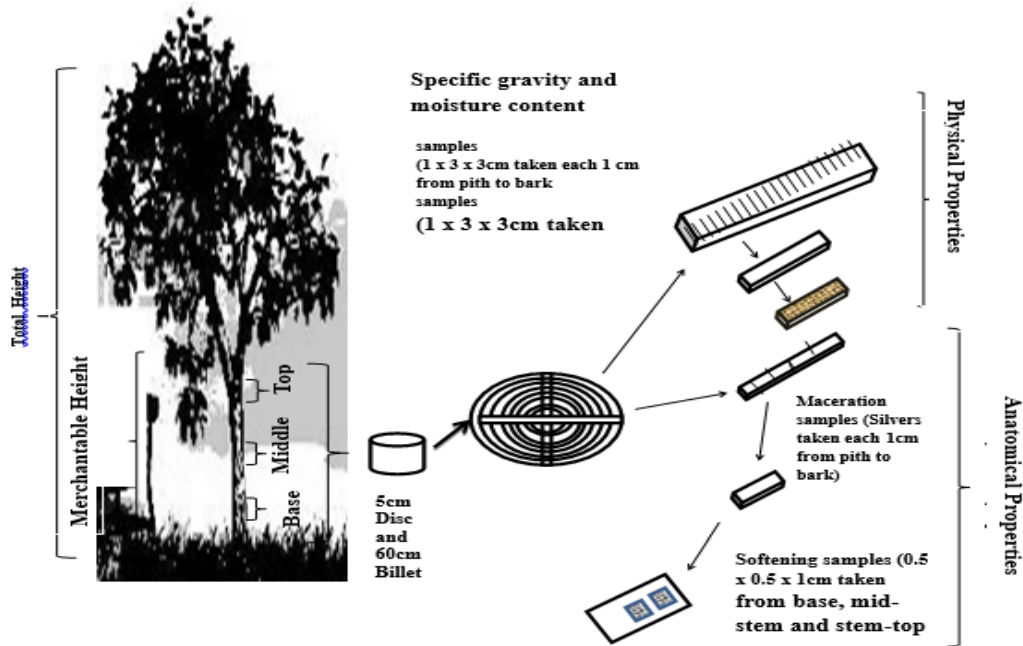


Figure 2: Mode of sample collection for physical and anatomical properties

**Pulping Experiment**

**Preparation of cooking Liquor (Kraft pulping)**

The cooking liquor was prepared by dissolving 40g NaOH: 13g NaS<sub>2</sub> in 1000 mL of distilled water

to obtain 17 and 20% effective alkaline. Below is the breakdown of the pulping condition.

**Table1. Kraft Pulping Conditions**

Cooking variables	Cooking condition		
	1	2	3
Number of cooks	3	3	3
NaOH Concentration (%)	40	40	40
NaS <sub>2</sub> Concentration (%)	13.5	13.5	13.5
NaOH to NaS <sub>2</sub> ratio	3:1	3:1	3:1
Liquor to chip ratio	5:1	5:1	5:1
Cooking temperature (°C)	<b>140</b>	<b>160</b>	<b>180</b>
Cooking time (minutes)	90	90	90

**Kraft pulping procedure**

The pulping experiment was carried out using Kraft pulping process. Prior to pulping, the wood from each tree was chipped separately and labelled. For each cook, a 200g composite chip was pulped in a 2-litre electrically heated autoclave digester. The liquor to chip ratio was kept constant and the cooking condition was replicated 3 times.

After each cook, the digester was allowed to cool and the pulped chips withdrawn. The cooked chips were washed with distilled water, disintegrated with manually operated mixer for 30 seconds and screened on a flat screen (0.25mm slots). The pulp formed was thoroughly washed with distilled water until it became neutral to litmus paper. To avoid loss of fine material in the screening procedure, the filtrates was used as dilution water in screening (closed cycle screening). The pulp obtained was

spread on a wire to form a good mat and to drain excess moisture overnight. The pulp was dried in an oven at  $100 \pm 2^\circ\text{C}$  to constant weight.

#### **Determination of kraft pulp yield**

The result of the pulp yield was reported as a percentage of the bone dry weight of the original sample and calculated as follow:

$$\% \text{Pulp yield} = \frac{w_2}{w_1} \times \frac{100}{1} \dots\dots (1)$$

Where,

$W_2$  = Weight of oven dried pulp (g)

$W_1$  = Weight of the original chips (g)

#### **Determination of pulp kappa number**

Kappa number is used to describe the relative hardness, the degree of delignification obtained in a chemical pulping process, the bleachability of whitening aptitude of a pulp (ISO 302:2015). Kappa number was determined using TAPPI standard T 236 CM-99. About 0.5g of the oven dried pulp was disintegrated in 100ml of distilled water until free of fibre clots. Disintegration was done using magnetic stirrer. Disintegrated pulp was transferred into 500ml reaction beaker, 100ml distilled water was added to bring the total volume to 200ml. The beaker was place on a magnetic stirrer with continuously stirring. 25 ml of 2M of 94N sulphuric acid solution was measured and 25ml of 0.02M (0.1N) potassium permanganate solution was pipetted, they were added to the disintegrated test specimen simultaneously, starting a stopwatch. At the end of exactly 10.0 min, reaction was stopped with the addition of 5ml of 1M (1N) potassium iodide solution. Immediately after mixing, but without filtering out the fibres, it was titrated with 0.2M (0.2N) sodium thiosulphate solution, few drops of the starch indicator was added towards the end of the reaction. The blank determination using exactly the same method as above but omitting the pulp was carried out. Kappa number was calculated as follows:

$$\text{Kappa Number} = \frac{p \times f}{w} \dots (2)$$

$$p = \frac{(b - a)}{0.1} N \dots\dots (3)$$

Where;

f = factor for correction to a 50% permanganate consumption

w = weight of moisture free pulp in the specimen, g

p = amount of 0.1 N permanganate actually consumed by the test specimen, mL

b = amount of the thiosulfate consumed in the blank determination, mL

a = amount of the thiosulfate consumed by the test specimen, mL

N = normality of the thiosulfate.

#### **Precipitation and Purification of Kraft lignin from spent liquor**

Kraft spent liquor acquired from each cook at different temperature was isolated by precipitating the lignin from solution by acidification. The spent liquor was first filtered through a filter paper on a funnel. Approximately 100ml of Ethanol was added for every 300 mL of spent liquor. The solutions were then stirred vigorously for one hour. The spent liquor was further allowed to settle for 20hrs after which it was decanted. The precipitates were collected at a pH of 2 on a medium glass funnel (Li and Ge, 2011). It was washed twice with cold water by suspending the precipitates in the water and stirring vigorously Akpakpan *et al.*, 2023. The semi-solid precipitate obtained were air dried, and extracted for 8 hours in a soxhlet extractor using pentane to remove excess sulphur and other impurities. The Kraft Ficus lignin precipitates were further purified by suspending them in a 9:1  $\text{H}_2\text{SO}_4$ :  $\text{H}_2\text{O}$  solution and stirring for an hour. The precipitates were washed again with 9:1  $\text{H}_2\text{SO}_4$ :  $\text{H}_2\text{O}$  and stirred. The supernatants were filtered over on a medium glass funnel.  $\text{H}_2\text{SO}_4$  was removed by evaporation, the purified Kraft Ficus lignin freeze dried and the yield determined.

#### **Experimental Design**

The experimental design adopted for pulp yield and kappa number is 3 x 3 x 3 factorial experiment in a completely randomized design (CRD) with three replications. While Lignin yield determination from Kraft liquor adopted a 3 x 3 factorial experiment in a completely randomized design (CRD), the combination which gave 9 experimental treatment sample



units. However, each treatment combination was replicated three times.

The following are the variables;

- i. Tree stand
- ii. Sampling height- Base, Mid-stem and Stem-top
- iii. Temperature (pulping)- 140, 160 and 180°C

**Statistical Analysis**

The experimental data generated was subjected to descriptive. Analysis of Variance (ANOVA) was carried out to estimate relative importance of the various sources of variation in all the parameters determined.

Follow-up test was conducted using Duncan Multiple Range Test (at 0.05 level of significance). This was carried out to compare the differences between the means.

**Model specification**

The statistical model for 3 x 3 x 3 factorial experiment in a completely randomized design (CRD) is given as;

$$Y_{ijk} = \mu + A_i + B_j + (AB)_{ij} + C_k + (AC)_{jk} + (BC)_{jk} + (ABC)_{ijk} + E_{ijkl} \dots\dots (4)$$

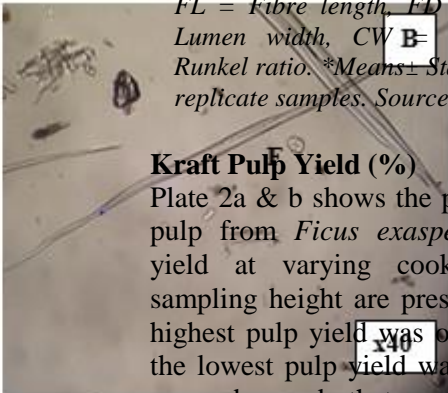
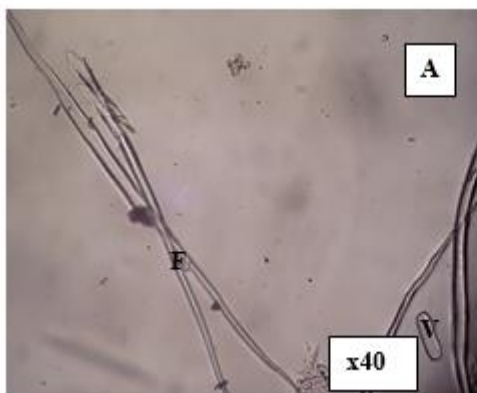
Where:

- $Y_{ijk}$  = Individual observation
- $\mu$  = General mean
- $A_i$  = Effect of factor A (Tree)
- $B_j$  = Effect of Factor B (Sampling height)
- $(AB)_{ij}$  = Effect of interaction AB
- $C_k$  = Effect of factor C (Temperature)
- $(AC)_{jk}$  = Effect of Interaction AC
- $(BC)_{jk}$  = Effect of Interaction BC
- $(ABC)_{ijk}$  = Effect of Interaction ABC
- $E_{ijkl}$  = Experimental error

**RESULT**

**Fibre Characteristic**

The fibres of *F. exasperata* in Plate 1 are narrow; most of the fibres are tapered in shape while others are bifurcated with lignified wall and provide support to the tree. The morphological characteristics of fibres like length and width and others are important parameter to estimate the pulp yield and quality (Table 2).



FL = Fibre length, FD = Fibre diameter, LW= Lumen width, CW = Cell-wall thickness and Runkel ratio. \*Means± Standard error of mean of 4 replicate samples. Source: Anguruwa et al. 2019

**Plate 1: Fibre of *Ficus exasperata***

**Kraft Pulp Yield (%)**

Plate 2a & b shows the picture of the chips and pulp from *Ficus exasperata*. The mean pulp yield at varying cooking temperature and sampling height are presented in Figure 3. The highest pulp yield was obtained at 140°C while the lowest pulp yield was obtained at 180°C. It was observed that an increase in cooking temperature from 140°C to 180°C brought about slight decrease in average yield of Kraft pulp, while pulp yield decreases from base to the stem-top along the sampling height from 53.38 – 47.64% at the base to 45.67% at the stem-top. ANOVA of variance at 5% level of significance showed that influence of temperature (p=0.007) and sampling height (p=0.012) on pulp yield was significant (Table 3). Further test of

**Table 2: Mean values of Fibre Morphology along Sampling Height**

Variables	FL(mm)	FD (µm)	LW(µm)	CW(µm)	RR
<b>Sampling Height</b>					
Base	1.03±0.13 <sup>a</sup>	23.06±14.43 <sup>a</sup>	13.74±2.42 <sup>a</sup>	4.66±6.69 <sup>a</sup>	0.65±0.72 <sup>a</sup>
Mid-stem	1.07±0.12 <sup>a</sup>	26.62±17.54 <sup>a</sup>	13.81±2.12 <sup>a</sup>	6.4±8.38 <sup>a</sup>	0.90±1.05 <sup>a</sup>
Stem-top	1.11±0.59 <sup>a</sup>	23.89±13.29 <sup>a</sup>	13.81±1.86 <sup>a</sup>	5.35±6.57 <sup>a</sup>	0.83±1.03 <sup>a</sup>
<b>Average</b>	<b>1.07±0.35</b>	<b>25.10±15.40</b>	<b>14.05±1.50</b>	<b>5.00±7.23</b>	<b>0.79±0.94</b>

significant using Duncan at 5% probability level recorded that pulp yield at different temperature

and along the sampling height did not differ from each other (Table 4).

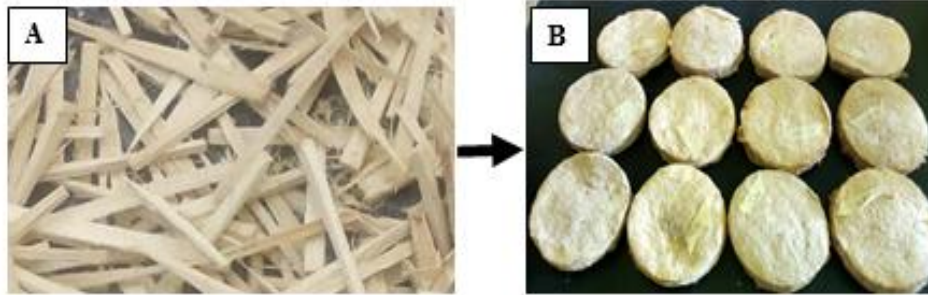


Plate 2: A-Wood chips and B-Pulp made from *Ficus exasperata*

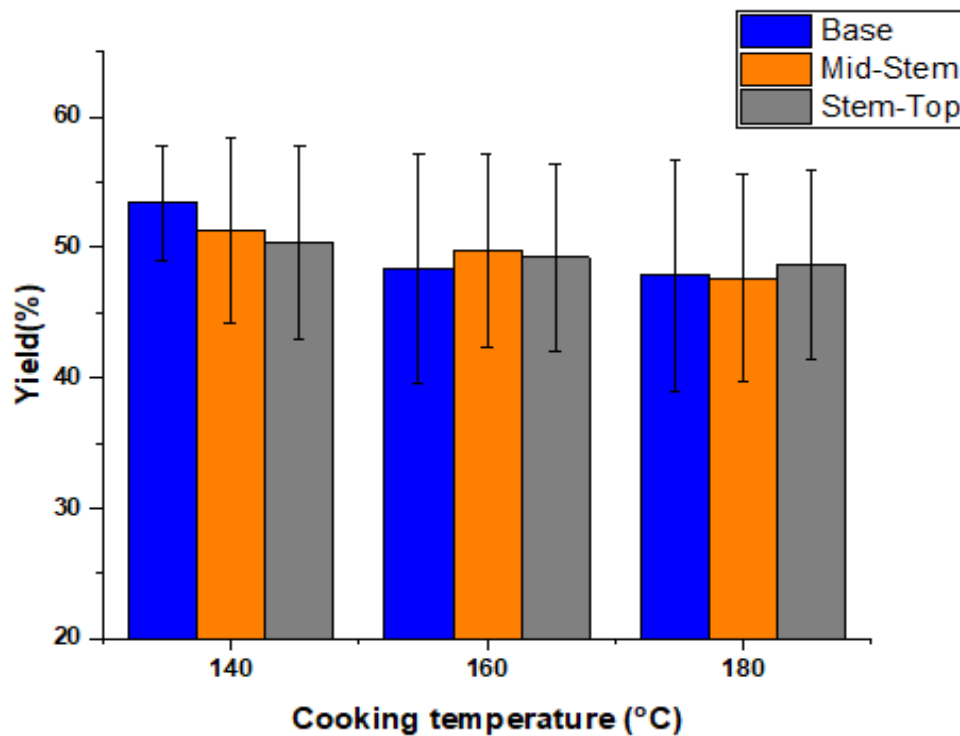


Figure 3: Mean pulp yield along the tree sampling height at varied temperature

Table 3: Analysis of variance for Pulp yield.

Sources Variation	Df	Pulp yield	Kappa number
<b>Pulp yield (%)</b>			
Tree	2	<0.001	0.007
Sampling height	2	0.007	0.020
Temperature	2	0.012	0.015
Tree*Sampling height	4	0.036	0.690
Tree *Temperature	4	0.0223	0.534
Sampling height*Temperature	4	0.013	0.217
Tree*Sampling height*Temperature	8	0.002	0.571
Error	54		
Total	80		

*p-values > 0.05 are not significant*

**Table 4: Variation in Sampling height and Temperature on Pulp Yield and Kappa Number.**

Variables	Pulp Yield (%)	Kappa Number
<b>Sampling height</b>		
Base	49.86±7.76 <sup>a</sup>	31.37±7.73 <sup>a</sup>
Mid-stem	49.73±6.99 <sup>a</sup>	29.56±8.75 <sup>a</sup>
Stem-top	49.24±7.98 <sup>a</sup>	29.56±8.32 <sup>b</sup>
<b>Temperature (°C)</b>		
140	49.61±7.50 <sup>a</sup>	31.44±8.58 <sup>a</sup>
160	49.30±7.58 <sup>a</sup>	30.30±8.03 <sup>a</sup>
180	48.75±7.58 <sup>a</sup>	27.74±7.79 <sup>a</sup>

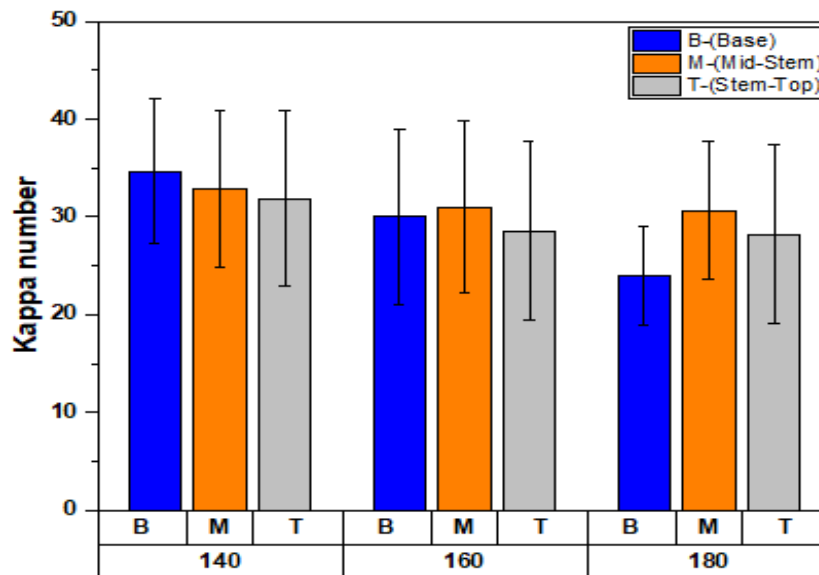
\*Means± Standard error of mean of 3 replicate samples. Values with the same alphabet in each column are not significantly different at  $\alpha = 0.05$  using Duncan multiple range test.

**Pulp Kappa number**

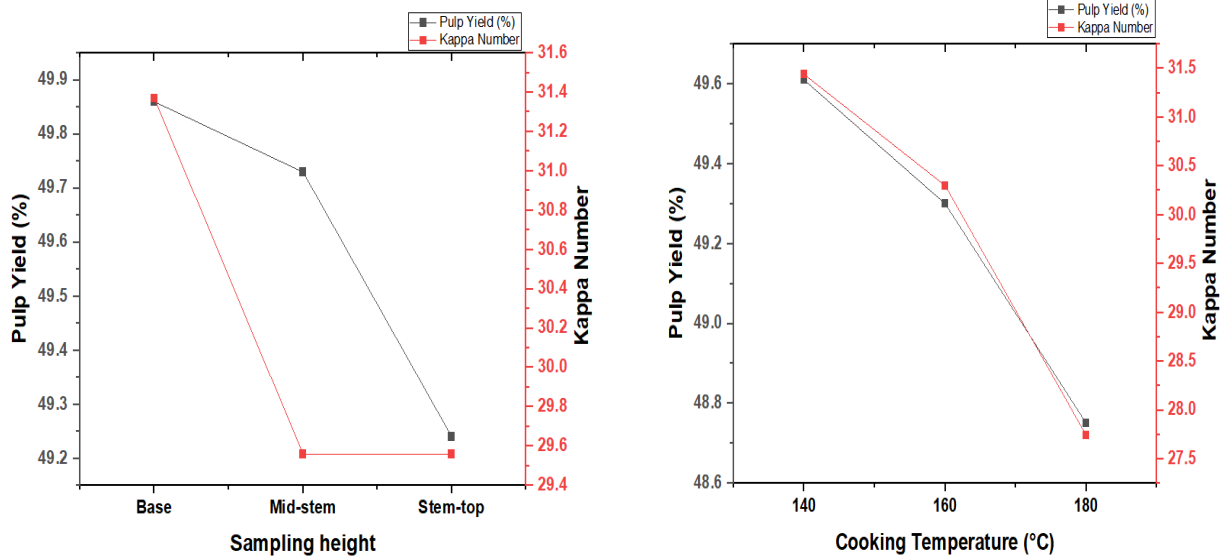
The mean Kappa number is presented in Figure 4. Just like the pulp yield, the highest kappa number was recorded at temperature of 140°C while the lowest kappa number was obtained at 180°C also the wood chips at the base (34.67 - 24.00) produced the highest kappa number, followed by (32.44 -30.67) at the mid-stem while the lowest kappa number of (31.89 -29.22) at the stem-top. It was observed that an increase in cooking temperature from 140°C to 160°C brings about slight decrease in the average kappa number of Kraft pulping from 31.44 to 30.74. Further increasing the temperature to 180°C decreased the kappa number to 27.74. Analysis

of variance carried out shows that temperature and sampling height also had significant influence on the Kappa number of Kraft pulp at  $p=0.015$  and  $p=0.020$  respectively (Table 3).

Duncan Multiple Range Test was used to test the level of significance at 5% probability level shows that kappa number at different temperature did not differ from each other while along the sampling height, the kappa number at the base and mid-stem differ from the kappa number at the stem-top (Table 4). Figure 5 shows the correlation between total pulp yield versus kappa number in Kraft pulping base on sampling height and cooking temperature.



**Figure 4: Mean Kappa Number of *Ficus exasperata* along the sampling height at varied temperature**



**Figure 5. Correlation between total pulp yield versus kappa number in Kraft pulping base on sampling height and cooking temperature**

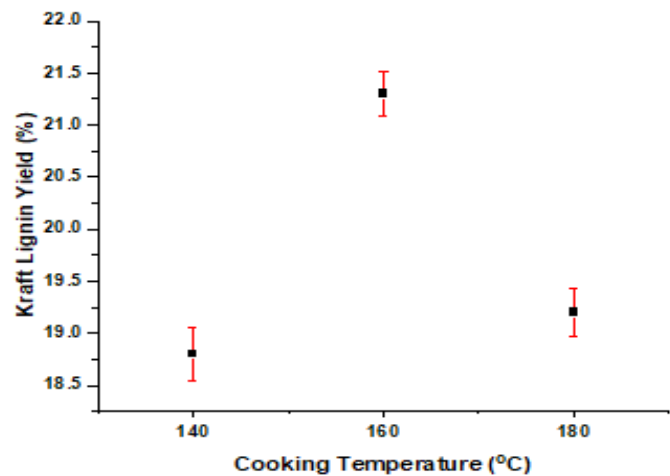
**Kraft Ficus Lignin Yield**

The lignin precipitated from *Ficus exasperata* is presented in Plate 3. The mean values of precipitated kraft lignin were presented in Figure 6. It gave highest yield of 21.3% at 160°C

cooking liquor; it was followed by 19.2% at 180°C and lowest yield of 18.8 at 140°C. The result of Analysis of variance in Table 5 shows that pulping temperature had remarkable influence on Kraft lignin yield ( $p = 0.038$ ).



**Plate 3: Kraft Lignin of *Ficus exasperate***



**Figure 6: Mean of Kraft Ficus Lignin**



**Table 5: Analysis of variance for Kraft lignin yield**

Sources Variation	Df	Sum Square	Mean Square	P-value
Tree	2	1.209	0.605	<0.001
Temp	2	0.001	0.001	0.038
T x Temp	4	0.017	0.004	0.610
Error	18	0.112	0.006	
Total	26	1.339		

*p-values* > 0.05 are not significant

## DISCUSSION

The fibre of *Ficus exasperata* could be classified as medium fibre with an average fibre length of 1.39 mm (Anguruwa et al. 2019; Ekhuemelo et al., 2018). Increase in the diameter of the fibre was said to be associated with molecular and physical changes that takes place in the vascular cambium and increase in cell walls in the process of tree growth processes (Plomion et al., 2001; Rogue et al., 2007). In terms of the cell-wall thickness, the fibres in *F. exasperata* have large lumen and thin wall. Papers made from wood of *F. exasperata* is expected to exhibit good contact between fibres and consequently good strength characteristics suitable for writing, printing, wrapping and packaging purposes (Anguruwa et al 2019). Large fibres with thin walls give a positive effect as they tend to form non-porous tightly bonded paper sheet that is easily collapse and flexible (Syed et al., 2016). Anguruwa et al 2019 reported that fibres of this wood species have average runkel ratios of 0.79 which makes it a thin walled fibre. This is expected to have positive effect on tensile and bursting strengths as well on folding endurance of the paper produced from the wood. Anguruwa and Oluwadare 2019 recorded a low specific gravity of *Ficus exasperate* 0.50 which is an indication that the wood can be chipped easily and liquor consumption during pulping would be low. With the findings recorded on the fibre morphology of this species, it is therefore necessary to subject it to further test in order to discover more potential (pulping).

However, Worku et al., 2023 listed kraft pulping as one of the most used chemical procedures for pulping. Kraft pulping amongst other chemical pulping is usually preferred due to its ability to leave the biomass fibers intact and increase the flexibility and conformability of the undried fibers (Fearon et al., 2020). Pulp yield in this

study is a product of the higher holocellulose content of the *F. exasperata*, this is because high holocellulose content brings about swelling behaviour of the pulp produced from it as reported by Singh et al. (2011). Megra et al., 2022 recorded a maximum pulp yield for *Melia azedarach* as 41.81% at a temperature of 170 °C. However, Rullifank et al., 2020 opined that high quality pulp is produced using temperature of 160 to 180 °C.

Kappa number is a standardised process, which is described in ISO 302:2015. The kappa number in this study was significantly influenced by sampling height. Kappa number values falls within the range of 26 to 36, this values are within the range of 17.5 to 39.8 obtained in Kraft pulping of *Leucaena diversifolia* (Feria et al., 2012). Also, value in this study is within the moderate kappa number favorable for pulp and paper, because high kappa number increases the bleaching chemical costs while low kappa number results in damaging the cellulose fibres that affects the strength and yield of pulp (Flavio et al., 2014). Santos et al., 2012 recorded that higher pulp yields were associated with lower Kappa numbers and alkali consumption, suggesting the important role of chemical composition of wood on Kraft cooking.

The lignin is a bio-based by-product and is thus considered a sustainable material with a great potential (Lawoko and Samec, 2023). The findings for kraft lignin precipitate in this study is in line with Cherif et al., 2020 who reported the physicochemical properties of Organosolv and Kraft lignins from selected hard and soft woods.

## CONCLUSION

The following conclusion were drawn;

- i. The analysis of the pulp properties showed that *F. exasperata* can be easily pulped using Kraft pulping to acceptable yield.
- ii. Kappa number of the pulp was moderate neither low nor high because high kappa number increases the costs of bleaching chemical while low kappa number damages the cellulose fibre which affect the strength and yield of the pulp.
- iii. Kraft lignin was successfully recovered from spent liquor of *F. exasperate*.

### RECONNENDATIONS

- i. Due to the similarity of most of the properties of the wood along the sampling

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- height, sorting and classification of the wood chips will not be required. Hence, they can be combined in the same digester operations.
- ii. Modification of the Kraft Ficus lignin and its composition would improve the utilization of lignin in several industries including paper and pulp, bio fibre, bioenergy and food Processing. The networking among the industry and research institutes would go a long way in increasing the application and awareness in the market for lignin
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