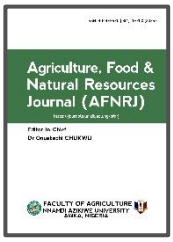
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Original Article

Trace element profiling in wood residue and lignin extracts of *Ficus exasperata* (Vahl.) using atomic adsorption spectrometer technique

OPEN ACCESS

Gloria Titi ANGURUWA

[1]*Department of Forest Products Development and Utilisation. Forestry Research Institute of Nigeria, Ibadan, Oyo State, Nigeria.*

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ABSTRACT

Trace element profiling analyzes the concentration and distribution of essential minerals and micronutrients within plant biomass. This study examined the inorganic constituents of Ficus exasperata by profiling its Milled-Wood Residue (MWR) and Extracted Lignin (EL). Three trees aged 18, 20, and 25 years were felled from the Arboretum of the Forestry Research Institute of Nigeria, Ibadan. Their merchantable lengths were 3.78 m, 3.50 m, and 3.60 m, with diameters at breast height of 15.8, 18.46, and 20.05 cm, respectively. Discs were collected at the base (10%), middle (50%), and top (90%) along the sampling height. Lignin extraction and silica (Si) content were determined using standard procedures. Atomic Absorption Spectrophotometry (AAS) was employed to quantify manganese (Mn), aluminum (Al), calcium (Ca), magnesium (Mg), zinc (Zn), lead (Pb), iron (Fe), and copper (Cu). Data were analyzed using Analysis of Variance. Results revealed that Si, Mn, and Pb were absent in EL but Si was 6.11% in MWR at the top wood. Calcium exhibited the highest concentration (68.89%) at the mid-wood in MWR, while EL recorded 10% at the top. Al, Mg, and Pb had the lowest contents, ranging from 0.01–0.05%. These findings establish a baseline elemental profile of Ficus exasperata MWR and EL, indicating its potential for applications in pulp production, bioenergy generation, and sustainable material technologies.

KEYWORDS: *Aluminum, Calcium, Lead, Sampling height, Silica content*

INTRODUCTION

The increasing demand for sustainable bioresources has intensified research into the chemical and elemental composition of lignocellulosic biomass (Wang *et al.*, 2023). *Ficus exasperata*, a widely distributed tropical plant known for its medicinal and ecological significance, has recently gained attention for its potential in bioenergy and biochemical applications (Manikandan *et al*., 2024). Profiling trace elements in its wood residue and lignin extracts is essential for evaluating its suitability in industrial processes and environmental safety.

Among the diverse plant species available, *Ficus exasperata* has emerged as a promising candidate for bio-based applications (Manikandan *et al*., 2024; Bello et al., 2024). While its biological and chemical properties have been explored, the mineralogical composition of its wood residue and lignin extracts remains largely uncharacterized (Adeniyi *et al.*, 2024).

*Corresponding author: gloriaanguruwa@yahoo.com

Trace elements such as iron (Fe), zinc (Zn), copper (Cu), manganese (Mn), and lead (Pb) play critical roles in plant metabolism, catalytic activity, and environmental interactions. However, their accumulation in biomass can pose toxicity risks or enhance functional properties depending on concentration and context (Akinloye & Ugbaja, 2024). Atomic Absorption Spectroscopy (AAS) remains a robust and sensitive technique for quantifying these elements in complex matrices, offering high precision and reliability in trace-level detection.

Recent studies have demonstrated the utility of AAS in profiling trace metals in various plant species. For instance, Anguruwa & Oluwadare (2020) investigated the chemical composition of *Ficus exasperata* residues using complementary techniques such as CHNOS analysis and FT-IR, revealing the presence of bioactive compounds like oxalic acid and vanillin, which suggest potential for biochemical valorization. Although their study did not focus on trace metals, it laid the groundwork for further elemental profiling using AAS.

Moreover, Alaimo & Varrica (2020) employed biomonitoring techniques to assess trace element contamination in *Ficus macrophylla* leaves, identifying significant concentrations of geogenic and anthropogenic elements such as Si, Cu, Mn, Zn, and Pb in urban environments. Their findings underscore the capacity of *Ficus* species to accumulate trace metals, reinforcing the relevance of profiling *Ficus exasperata* for environmental and industrial applications.

The study aimed to quantify trace elements in the wood residue and lignin extracts of *Ficus exasperata*, thereby contributing to the understanding of its elemental profile and potential utility in sustainable technologies.

MATERIALS AND METHODS

Study Area

Three stands of *Ficus exasperata* were 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 (FRIN Report, 2012).

Sample Collection

The estimated ages of the trees are 18, 20 and 25years with merchantable length of, 3.78m, 3.50m and 3.60m and Diameter at breast height, 15.8, 18.46 and 20.05cm for T1, T2 and T3 respectively. The trees were felled for destructive sampling, delimbed and cross-cut into disc at the base (10%), middle (50%) and top (90%) along the sampling height. The wood conversion was carried out at wood workshop of the Forest Production and Products Departmental, University of Ibadan, Nigeria and the logs were further processed into sawdust.

While lignin was extracted from sawdust using ASTM Standard Test Method for Acid-Insoluble Lignin in Wood: D 1106 - 96 (1977). Three 3g test specimens of each wood species was 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 1-h 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 50 ml 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 specimens 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 500 mL of hot water and the crucible and contents were 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 were repeated until the weight was constant.

$$\%Lignin = \frac{Weight\ of\ lignin}{Oven\ dry\ weight\ of\ extracted\ free\ milled\ sample} X\frac{100}{1} \quad (1)$$

Determination of Silica and Minerals Content

Silica content

This was carried out using TAPPI designation T244 0m-93 methods. 2g of ash was digested 3-times with 5 mL of 6M HCl and evaporated each time on a steam bath to dryness. Thereafter, hot distilled water was used to wash the residue free of chlorine on the ash-free filter paper. The filter paper together with the residue was then placed in the crucible of known weight and dried in the oven at 550°C for 3 hours after which crucible was allowed to cool in a desiccator and weighed. Also, same procedure was carried out for extracted lignin in order to compare the influence of pulping chemical on the mineral

content of the wood sample. The percentage of silica was then determined as follow;

$$\%Silica = \frac{w_0 - w1}{w_2} \times \frac{100}{1} \quad (2)$$

Where, w_o = Weight of crucible and silica, w_1 = Weight of empty crucible, w_2 = Weight of original sample.

Mineral elements

This was carried out using Atomic Absorption Spectroscopic (AAS) in accordance to ASTM D 4085-93 (2008). About 5g test specimen of milled-wood residue was obtained along the sampling height from each tree. The sample was saturated separately with a solution of 50% hydrogen peroxide, and added one piece at a time to about 10mL of concentrated sulphuric acid. The cellulose present was oxidized to carbon dioxide and water. The water was volatilized by the heat of the reactions involved. The solution was heated separately until dense fumes of the acid evolved. The remaining solution was allowed to cool and transferred into a 50ml volumetric flask and was made to mark with distilled water. Similar procedure was also followed for extracted lignin sample and also the make the blank regent. The metals investigated were; Calcium (Ca), Magnesium (Mg), Iron (Fe), Cupper (Cu), Zinc (Zn), Lead (Pb), Alluminium (Al) and Manganese (Mn).

Experimental Design

The experimental design adopted is 3 x 3 factorial experiment in a completely randomized design (CRD), the combination which gave 9 experimental treatment samples. However, the values presented are means of three replicates, thus, giving a total of 27 samples.

The following are the variables;

1. Tree- T1, T2 and T3
2. Sampling height- Base, Middle and Top

Statistical Analysis

The experimental data generated was subjected to both descriptive analyses. Analysis of Variance (ANOVA) was carried out to estimate relative importance of the various sources of variation. Follow-up test was conducted using Least Significant Difference (at 0.05 level of significant). This was carried out to compare the differences between the means.

Model specification

The statistical model is given as;

$$Y_{ijk} = \mu + A_i + B_j + (AB_{ij} + E_{ijk} \quad (3)$$

Where: Y_{ijkl} = Individual observation, μ= General mean, A_i= Effect of factor A_i (Tree), Effect of Factor B_j (Sampling Height), $AB)_{ij}$= Effect of interaction AB, E_{ijkl} = Experimental error

RESULT AND DISCUSSION

Silica Content (%)

The average percentage silica content was recorded as 5.86% (Table 1). Silica content deposition in milled-wood residue (MWR) did not follow a specific pattern of variation along the sampling height; it was highest at the top (6.11%) and lowest at the middle (5.00%). Figure 1 shows the absence of silica in extracted lignin (EL). Analysis of variance revealed that sampling height had no significant influence on silica content in MWR of *Ficus exasperata* ($p = 0.449$) (Table 2). Separation of the mean clearly proves that there is no significant difference in the percentage silica content of the milled wood at the base, middle, and top (Table 1).

The amount of ash in wood consists of various metal salts such as carbonates, silicates, oxalates, calcium, magnesium, iron, manganese, and silicon (Shakhes *et al*., 2011). These minerals are typically deposited in cell walls, fibers, lumina of parenchyma cells, resin canals, and ray cells (Sjostrom, 1993).

The silica content accumulated in the wood of *F. exasperata* varied among trees, suggesting that environmental conditions may play a significant role in determining the amount of silica deposited in plant structures (Sawadogo *et al.*, 2024). High ash content in wood can adversely affect the recovery of cooking liquor and refining operations (Rodríguez-Angulo *et al*., 2008). However, the absence of Silica in Extracted Lignin (EL) is of advantage because silica can interfere with chemical pulping and cause scaling in recovery boilers. Its absence in lignin means fewer complications during lignin extraction and processing.

The percentage ash accumulated in the wood of F. exasperata compares well with the 3.5% reported in matured bamboo (Ogunsile & Uwajeh, 2009). Similarly, the silica embedded in the wood implies that F. exasperata is an accumulator of silica salts (Kone et al., 2023). Although the lack of significant variation in silica across sampling heights supports consistent energy conversion efficiency and reduces the risk of operational disruptions. Also knowing that silica is absent in lignin and stable in MWR helps in designing combustion systems and pre-treatment strategies.

Mineral Content (%)

As a prerequisite to the meaningful evaluation of this wood species for pulp and paper production, mean mineral elements and their contents in milled wood residue (MWR) and extracted lignin (EL) were presented in Table 1. Magnesium (Mg), Calcium (Ca), Aluminium (Al), Iron (Fe), Lead (Pb), Copper (Cu), Zinc (Zn), and Manganese (Mn) were profiled. MWR had the highest mineral element concentration compared to EL. Generally, among all the mineral contents, calcium in both MWR and EL had the highest concentration with average values of 54.62% and 13.35%, respectively. This was followed by magnesium at 14.69% and 4.24%. The content of iron was 5.45% and 1.77%, copper was 0.49% and 0.12%, aluminium

gave average values of 0.05% and 0.02%, and zinc recorded values of 0.02% and 0.13%.

Least Significant Difference (LSD) test conducted at the 5% probability level for all the mineral contents in MWR and EL showed significant differences along the sampling height, except for Mn content in MWR, which showed no difference between the base and top but differed from the middle. Likewise, the content in EL differed only in Al and Cu content at the top and middle, respectively (Table 1).

Additionally, the results of analysis of variance presented in Table 2 showed that the mineral elements in the milled wood samples significantly influenced the mineral contents along the sampling height ($p < 0.001$), except for Pb content in MWR, which was not significant ($p = 0.569$). However, all the mineral contents in EL were significantly influenced by sampling height ($p < 0.001$), except for Mn and Pb, which were not detected (Oluwadare *et al.,* 2016). The least average concentration of 0.02% was recorded for manganese and lead in MWR, but these elements were not detected in the extracted lignin (Yahaya *et al*., 2022; Osowe *et al*., 2021).

Table 1. Individual Mean values of Silica and Mineral content in MWR and EL along Sampling Height.

Mineral Elements	Material Type	Base	Middle	Top
Silica	MWR	5.56±2.46* [a]	5.00±3.12[a]	6.11±2.52[a]
	EL	ND	ND	ND
Magnesium	MWR	14.69±0.78[b]	17.91±1.19[a]	13.27±0.92[c]
	EL	4.00±0.32 [b]	5.56±0.45[a]	3.38±0.33[c]
Calcium	MWR	51.67±3.54[b]	68.89±6.51[a]	43.33±6.61[c]
	EL	13.11±1.06[b]	16.07±0.89[a]	10.87±1.02[c]
Aluminium	MWR	0.04±0.01[c]	0.06±0.02[a]	0.05±0.01[b]
	EL	0.02±0.01[a]	0.03±0.01[a]	0.01±0.01[b]
Iron	MWR	5.91±0.49[a]	5.54±0.26[c]	4.90±0.18[b]
	EL	2.10±0.21[a]	1.76±0.15[b]	1.46±0.12[c]
Manganese	MWR	0.01±0.01[b]	0.02±0.00[a]	0.01±0.00[b]
	EL	ND	ND	ND
Copper	MWR	0.63±0.08[a]	0.40±0.05[b]	0.37±0.09[c]
	EL	0.12±0.01[b]	0.14±0.02[a]	0.11±0.01[b]
Zinc	MWR	0.63±0.11[a]	0.34±0.09[c]	0.52±0.08[b]
	EL	0.13±0.03[b]	0.10±0.02[c]	0.17±0.04[a]
Lead	MWR	0.02±0.01[a]	0.02±0.01[a]	0.02±0.01[a]
	EL	ND	ND	ND

MWR= Milled-Wood Residue, EL= Extracted Lignin, ND=Not Detected.

**Means±SEM of 3 replicate samples. Values with the same alphabet in each column are not significantly different at α = 0.05 using Least Significant Difference test. SEM = Standard error of mean.*

Table 2. Significance (p-values) of Silica and Minerals content in MWR and EL along the Sampling Height

Sources	df	Si	Mg	Ca	Al	Mn	Cu	Zn	Fe	Pb
Milled-Wood Residue										
Trees (T)	2	1.311*	<0.001	0.003	<0.001	0.194	0.622	0.252	0.022	0.029
Sampling H(SH)	2	0.449	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.569
T x SH	4	0.137	0.480	0.401	0.151	0.667	0.006	<0.001	0.939	0.016
Error	18									
Total	26									
Extracted Lignin										
Trees (T)	2	ND	0.063	<0.001	0.197	ND	0.005	<0.001	0.005	ND
Sampling H(SH)	2	ND	<0.001	<0.001	<0.001	ND	<0.001	<0.001	<0.001	ND
T x SH	4	ND	<0.001	0.788	0.382	ND	0.198	0.112	0.006	ND
Error	18									
Total	26									

**p-values > 0.05 are not significant. ND=Not Detected*

Among the nutrient elements evaluated, calcium was the most abundant in the wood of *Ficus exasperata*, while manganese and lead were the least. The high content of magnesium in fuel material is known to cause low volatile yield and demethoxylation of lignin units during pyrolysis (Fahmi *et al*., 2008; Bridgwater, 2012; Akinrinola *et al*., 2014). Therefore, the low concentration of nutrient elements in the wood of *Ficus exasperata* offers a great advantage due to the un-prolonged thermochemical conversion process during pulping and recovery of cooking liquor. The results also show that the proportion of elements in extracted lignin is not dependent on those in the milled wood residue. This could be attributed to the loss of mineral nutrients during both alkaline and acid hydrolysis, which are part of the lignin extraction process (Oluwadare *et al*., 2016). Therefore, the mill-wood residues and their lignin exhibit different behavior. The mineral matter obtained from the ash of the mill-wood residues is much lower than those found in major energy crops and is therefore not expected to cause fouling problems during combustion or processing (Akinrinola *et al*., 2014). It implies, High mineral content, especially calcium and magnesium, can affect combustion efficiency and ash behavior in thermochemical conversion processes like pyrolysis or gasification. Therefore, MWR may require pretreatment to reduce mineral load before being used for bioenergy. Also, lower mineral content in EL makes it more suitable for biochemical pathways, such as fermentation or enzymatic hydrolysis, where inorganic interference must be minimized.

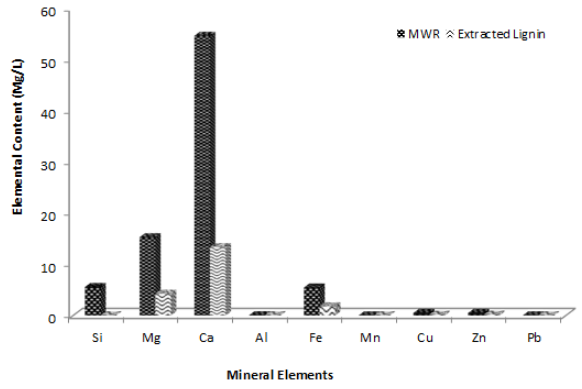


Figure 1. Variation in Silica and Element content of Milled-Wood Residue and Extracted Lignin of *Ficus exasperate*

CONCLUSION AND RECOMMENDATION

One major deduction from this result is that the proportion of element in extracted lignin is not dependent on those in the Milled-wood residue. The results obtained brought about the conclusion that there is no major difference in the concentration of elements in the wood of *F. exasperata* although an increasing possibility was observed for some heavy metals, due to their higher concentrations in the MWR. However, selective use of MWR and EL based on mineral content could optimize their application in specific biomass valorization routes.

It could be recommended that future research should explore chemical modification of extracted ligninbto enhance its industrial Utilisation in pulp and paper, biofiber, bioenergy and food processing. Strengthened collaboration between industry and research institutions will also be essential for expanding applications and market awareness of lignin-based products

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Author Contribution

GTA: Conceptualization, Methodology, resources, investigation and data analysis, manuscript review, wrote the first and final draft.

Ethical statement

Not applicable

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