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### RESEARCH ARTICLE

## PHYSICO, CHEMICAL AND THERMAL PROPERTIES OF PINEAPPLE FIBERS (ANANAS COMOSUS) LEAVES, STEMS AND ROOTS FROM THE CENTRE REGION OF CAMEROON FOR THE REINFORCEMENT OF ROOFING SHINGLES (TILES)

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### Abstract

Pineapple waste represents an average of 75% of the dry mass of giant grass, and because of its composition of plant fibers and others components, reveals a potential to exploit. Destroyed and abandoned in nature as agricultural wastes, it is not valued. This motivated us to research on pineapple wastes. The objective of this research work was to extract and characterize Pineapple waste (leaves, stems and roots), for the reinforcement of roofing shingles. To achieve this objective, we have studied the extraction techniques of plant fibers and selected four main processes that are suitable for the extraction of pineapple fibers; namely scraping, beaten, retting and chemical extraction using caustic soda. Then, we presented the state of the art on the Physico-Chemical extraction using caustic soda. Furthermore, we presented the physical characterization of plant fibers such as density, the section, the water content and diameter. With regard to chemical characterization. The chemical test we had hemicellulose, cellulose, lignin, ash, pectin and extractive content. The physical characterisation allowed us to note that the diameter of pineapple fibers ranges from 0.02 to 0.09mm. With a maximum density of 1.01g/cm<sup>3</sup> and a water absorption of 8.11%.

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The chemical characterization allowed us to note that the celluloses content of pineapple fiber is 72.14, 53.09 and 57.04%, holocellulose as 76.9, 74.76 and 74.8% lignin of 13.55, 15.16 and 13.22%, pectin of 1.46, 3 and 2.2 and finally ash content of 6.72, 6.61 and 6.67%. The TGA curves for these similar samples, the stem's fibers showed a somewhat stronger stability at elevated temperatures but an increasingly prolonged weight loss process between 200°C and 400°C. The phenomena might be brought on by the higher concentration of cellulose in stem fibers. Weight (%) fell more quickly with respect to increase in temperature from 400°C to 900°C, indicating a lower thermal stability in root fibers. This might be as a result of the higher lignin content of root fibers. In line with the patterns observed in stem and root fibers, leaf fibers had a declining weight pattern and a moderate degree of heat resilience. The fibers resulting from these processes are therefore best suited for the preparation of composite materials most especially for the reinforcement of roofing shingles.

### **Introduction: -**

The pineapple (*Ananas comosus*) is one of the most essential fruits in Cameroon and is the leading edible member of the Bromeliaceae family. It is an important food crop that is planted extensively in the tropical and sub-tropical regions (Rosnah Shamsudin et al. 2009). Commercially, it is mostly sold as canned fruit and is consumed worldwide. The plant can grow up to a height of 750 to 1500 mm with a spread of 900 to 1200 mm. It is short, having a stout stump with narrow, fibrous, and spiny leaves. The plant develops into a cone-shaped juicy and fleshy fruit with a crown at the top (Tran 2006). According to the FAO online data base, the area of pineapple plantations in 2012 around the world was almost 996,000 hectares with an estimated production of more than 23 million tons of pineapple fruit (FAO 2014).

According to the Agro food Statistics (2010 to present), one hectare of pineapple field can produce about 17,400 fruits, which is approximately 25 metric tonnes of pineapple fruit. Based on a rough estimate made in 2002, there are 40 leaves per plant with each leaf weighing about 0.065 kg and 2% fiber per leaf. The total fiber production based on 1.22 ton/hectare would be about 74,528.16 tons; considering that the fiber price is US\$ 0.36/kg, the market value of fiber is US\$ 434 per hectare (Satyanarayana et al. 2007). There have been very few studies on the fibers of the pineapple plant (leaves stems and roots). There are many varieties of pineapple plant, and each variety is different in terms of characteristics with respect to the geographical area in Cameroon and the World. The pineapple plant is considered not profitable after the fruit has been harvested because there is a lack of knowledge about the potential of the plants after the fruit is discarded.

After harvesting activities, most of the pineapple residue is disposed and either serves as fertilizer or is burnt in an open field. However, these methods are not only ineffective, but also contribute to air pollution (Wan and Zainuddin 2013). Thus, one of the possible ways to handle pineapple residue without jeopardizing or sacrificing the quality of the environment is by converting this residue into a value-added product. An innovative approach will not only help generate additional income, but will also create job opportunities (Ahmed et al. 2002). The plantation sector in Cameroon has generated large amounts of waste plant, thus creating problems for the environment and affecting the next cultivation of plants. Plant waste fibers, or agro-wastes, can be described as lignocellulosic materials comprising cellulose, hemicellulose, and lignin. Woods, agricultural wastes, water plants, grasses, and other plant substances are the example of the lignocellulosic materials (Abdul Khalil et al. 2006). Properties, composition, and structure of plant waste fibers make them appropriate for uses such as composite, textiles, pulp and paper manufacture. Furthermore, plant fibers can be used to produce fuel, chemicals, enzymes, and food. Biomass, including agricultural crops and residue, forest resources, animal and municipal wastes, is the largest source of cellulose in the world. Organic plant wastes such as palm oil, pineapple, banana, and coconut fiber are annually renewable, available in abundance, and cheap. These lignocellulosic byproducts could be a principal source of fibers, chemicals, and other industrial products (Reddy and Yang 2005).

After the harvesting period, these waste materials create significant environmental problems. Therefore, the economic utilization of these fiber wastes would be beneficial. Although a lot of work and research in agriculture has been undertaken, attention to agricultural technology in fiber applications has been limited and inadequate. Researchers should conduct more studies to assess the potential of agricultural wastes (Reddy and Yang 2005). In view of the above context and problem statement, the main objectives of this work are to extract fibers, determine the physicochemical and thermal properties of pineapple plant waste (leaves, stems and roots) from pineapple plant varieties that exist in Cameroon. The properties to be studied should include cellulose, hemicellulose, lignin, pectin, ash, fat, moisture content, density, water absorption and thermal properties. These research findings can lead to a

better understanding and knowledge of the physico-chemical and thermal properties of the fibers themselves. Information is important in order to minimize the environmental and health risk associated with disposal of pineapple plant in the field. Hence, findings from this study can be used by the polymer chemist, scientist, and food technologist for further applied research.

**Experimental method: -**

**Materials used in this study: -**

The pineapple (*Ananas comosus* L.) is one of the most essential fruits in Cameroon and is the leading edible member of the Bromeliaceae family (figure. 1a and 1b). The plant samples (leaves, stems and roots) were collected in a plantation from a village called Awae in the Center Region of Cameroon which is 42km from Nkoabang (Yaounde). The parts(leaves, stems and roots) were randomly selected and cut from mature plants directly after harvesting pineapple fruits from the plant and was transported by a vehicle to the National Advanced School of Engineering(NASE) Yaounde for extraction in the ECM (Laboratoire Engineering Civil et Mecanique).



Figure 1a. Pineapple field



Figure 1b. Fruit of a pineapple plant with parts [IJPS 2015]

The natural fiber extracted in this work is from the three different parts which are leaves, stems and roots of pineapple plant as shown in (table 1) below.

**Fiber Extraction equipment: -**

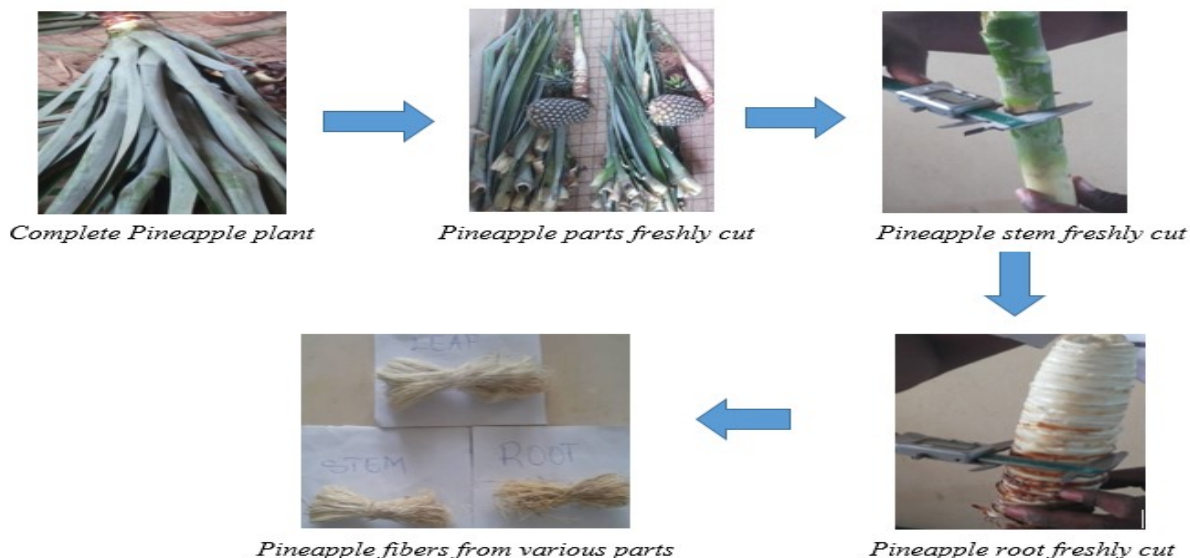
The main materials used for the extraction of pineapple fibers are illustrated on table 1, namely: Distillatory for water, hoven for drying of the fibers, digital scale for measurement, a blunt knife, wooden stick and fiber comb, a heating pot with adjustable temperature, and a thermometer to measure the temperature of the solution, vernier caliper, and containers.

Table 1: Equipment used for extraction

<p>(a) Water distillator</p>	<p>(b) Specimen and beakers</p>	<p>(c) Digital scale for measurement</p>	<p>(d) Oven for drying</p>	<p>(e) Boiling for extraction</p>

**Method of extraction fiber: -**

The leaves were manually separated from each other (leaves, stem and root) and the surface cleaned with tap water (Figure 2). The fiber-extraction methods have a major impact on yield and quality of fiber. It influences the structure, chemical composition, and physical properties of fibers. For getting good quality of fibers, suitable mechanical methods have to be adopted. Different types of machines were designed and developed for the extraction of fiber with increased productivity (R. Kannojiya et al, 2013).



**Figure 2. Preparation of pineapple plant raw materials**

Four methods of extractions were used here for the determination of fibers from the pineapple plant.

**Table 2: Extraction Processes**

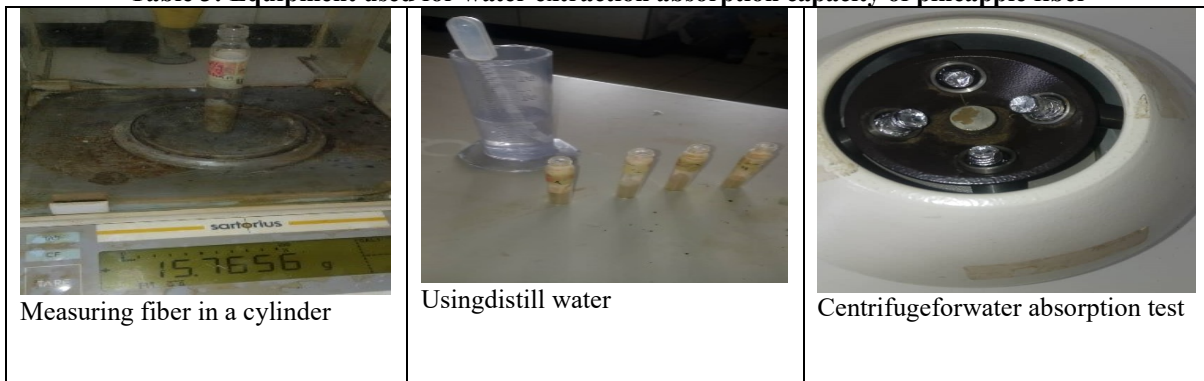
Extraction Processes	Description	Parameters	Values
Extraction by soaking (water retting)	The three different parts of the pineapple were strip off the plant and put into different plastic containers of distill water for extraction	Duration	30 days
		Temperature	Ambient
Extraction by scraping	Scrap only the leaves and put into a container mixed with distill water to remove wax and cholesterol	Temperature	60°C
		Duration	30 minutes
Extraction by beaten	Here the leaves and stem were beaten with w wooden stick and put into a container mixed with distill water to remove wax and cholesterol	Volume of water	800 ml
		Temperature	97°C
		Duration	1 hours
Extraction by sodium hydroxide (NaOH)	The leaves were dried into the hoven for 80 <sup>0</sup> C and then mixed with distill water and 5% caustic soda.	5%NaOH Distil water Heater Beaker Thermometer	The heating time varies, 30minutes to 4hours -Fibers eliminates linin -Fiber bundle

**Materiel of physical test: -****The water retention capacity of cellulose: -**

The water retention capacity is made in order to evaluate the amount of water absorbed by the cellulose produced. A strong affinity for water can be a limiting factor in its capacity vis-à-vis pollutants. The water retention capacity depends on several factors such as the treatment of fibers by the chemical and physical structure (Grigelmo-Miguel et al., 1999) and the size of the fibers. 2g of sample mixed with 38 ml of distilled water are placed in a centrifuge tube (table 3). The whole is stirred for few minutes then, put in a centrifuge for 30 minutes to acceleration with a rotational movement thus separating the two contents of the tube (Traynham et al., 2007). The retention capacity in water (CRE) is given by relation (1)

$$\text{CRE} = \frac{M_{t+r} - M_t}{M} \quad (1)$$

Where  $m_t + r$  is the mass of the centrifuge tube and the residue,  $m_t$  the mass of the centrifuge tube and  $m$  the mass of the weighed sample

**Table 3: Equipment used for water extraction absorption capacity of pineapple fiber****Moisture Content of Pineapple Fibers (% H): -**

Humidity affects many parameters such as mechanical properties, degradation, touch, etc. For this, it is necessary to compare fibers and applications with a similar moisture percentage. The determination of the moisture content is carried out by difference in weight, using an oven heated to 100°C for at least 4 hours. The moisture content of a material can be defined as the amount of moisture contained in 100 grams of wet material under specific climatic conditions (Norm NFP 94-054). The gravimetric method allows the measurement of the water content by determining the moisture content of the fibers by evaporation. To do this, the wet fibers are placed in a drying oven HERAEUS brand at a temperature of 100°C in the Macro molecular Chemistry laboratory of the University of Yaounde I. Firstly, it is necessary to note the mass of the fibers without them being put in the oven, then dry these fibers in an oven for 24 hours at a temperature of 100 °C and raise again its mass during N operation. It can be seen that the initial mass before introduction to the oven is lower than the other masses measured which remain constant. (Ahmed et al., 2012).

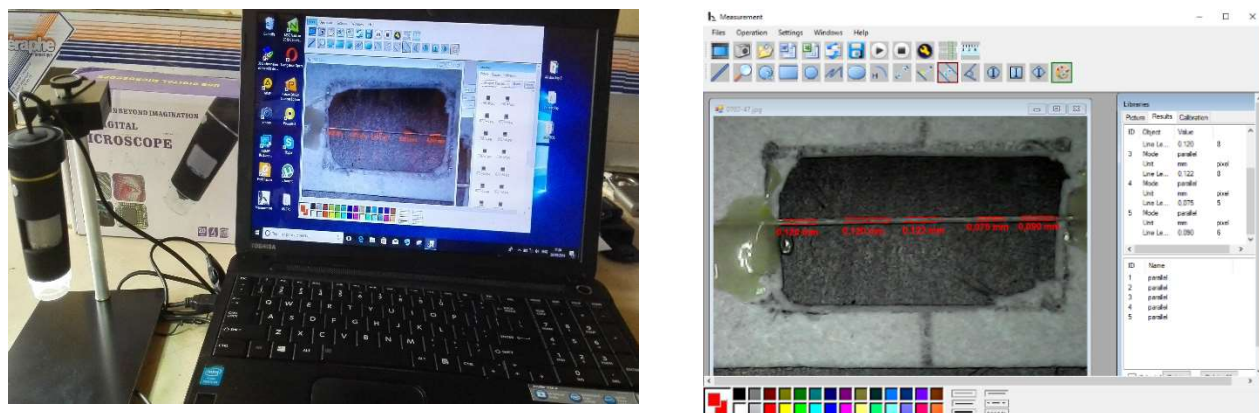
**The moisture content is determined by relation (2);**

$$\% H = \frac{M_w - M_d}{M_d} * 100 \quad (2)$$

Where  $M_w$  is the mass of the wet sample and  $M_d$  its mass after drying.

**Determination of fiber diameter: -**

The cross-sectional area of the pineapple fiber is not circular and that made us to use a digital micrometer and considered five (5) different sections of diameter and calculate the average diameter (figure 3a and 3b)



**Figure 3a. Determination of fiber diameter by a digital microscope**

**Figure 3b. Fiber's diameter determined with adigital microscope**

### Isolation of cellulose fibres :-

#### Chemical composition: deconstruction to short fibers of pineapples(leaves, stems and roots): -

The chemical deconstruction process used in his work was derived from those originally proposed by Neto et al. (1996) and Moine (2005), the fibers were pound to dust using a pounding mortar and further using a shift of 315 micrometer (0,315 millimeter) to separate the dust from particles. The detailed procedure is depicted in Figure4.

**Step 1: Determination of Ash Rate** The ash content gives information on the mineral matter present in a sample of lignocellulosic material. (Ahmed et al., 2012). It is determined by incineration of the sample. Different grams of material samples were dried in an oven at 60 ° C for 48 hours, weighed and placed in a ceramic crucible. The set is introduced in a Naberther brand furnace set at 900 ° C for 180 minutes. After this time, the crucible is cooled in a desiccator and reweighed.






#### The ash rate (% A) is calculated by the relation (3): -

$$\% \text{ Ash} = \frac{\text{FinalMass}}{\text{InitialMass}} \times 100 \text{ Which implies } \% \text{ C} = \frac{M_{df}}{M_{di}} \times 100 \quad (3)$$

Where  $M_{df}$  is the final dry mass of fiber after incineration and  $M_{di}$  initial dry mass before incineration

**Step2:**Determination of water-soluble extract (Ethanol-Benzene Extraction). A Soxhlet is mounted on a flask containing a 1: 2 V / V benzene / ethanol mixture and surmounted by a refrigerant. The solvent mixture is heated in a flask set to have 4 to 6 siphoning per hour. The extraction is performed for 7hrs. After this time, the capsule is recovered and dried in an oven at a temperature of 100 ° C overnight. The benzene / ethanol extractable content is calculated as percentage. These extractable materials are low molecular weight compounds contained in the cell walls of plant materials. These extractable materials include tannin, terpenes, alkaloids, steroids and fatty acids. These extractable have an effect on lignocellulosic materials and are soluble in various types of common organic solvents (hexanes, dichloromethane, acetone, toluene / ethanol, benzene / ethanol and water).

Table 4: Equipment used for extractives

				
Pounding Mortar	Shifting machine	Digital scale for measurement	Filter papers with fibres inside	Soxhlet for extraction

The extractable composition varies with the species of interest and influences color and odor (Bentley et al 1994). The extractable ethanol / benzene content was calculated as a percentage by the relation (4);

$$\%E_{EB} = \frac{M_0 - M_1}{M_1} \times 100 \quad (4)$$

**Step 3: Determination of Water Extraction.** The capsule recovered after extraction with the ethanol / benzene mixture is replaced in the Soxhlet and the solvent mixture of the balloon replaced by the distilled water (it allows to wash residuals of extractive materials still present in the sample obtained after extraction).

The extraction is carried out for 16 hours using the same protocol as above. The capsule is recovered and dried in an oven overnight, then weighed. The rate of water extraction is determined by the relation (5)

$$\%E_e = \frac{M_2 - M_1}{M_1} \times 100 - E_{EB} \quad (5)$$

$M_2$  is the solubilized dry mass after extraction with water and  $M_1$  that is calculated in relation (4)

#### Step 4: Determination of Pectin Content

The residue from the previous step is introduced into a 100 mL flask and 50 mL and a 2% hydrochloric acid solution is added. The whole is heated to reflux in a water bath at 80°C with magnetic stirring. At the end of this time, the solid residue is filtered, washed abundantly with water, dried at 105 ° C overnight and weighed (Le Goff et al., 2001) The pectin content is calculated by the relation (6)

$$\%T_{pec} = \frac{M_2 - M_3}{M_2} \times (100 - (E_{EB} + E_e)) \quad (6)$$

Where  $M_3$  is the mass of the sample obtained after drying and  $M_2$  that calculated in the Relation (5).

#### Step 5: Determination of Holocellulose content

The principle here is to completely delignify the lignocellulosic material and recover the polysaccharide fraction (cellulose and hemicellulose). In a 250-mL flask, a mass of dry matter obtained in the previous step was contacted with 125 mL of distilled water, then 1.00 mL of acetic buffer and 1.00 g of sodium chlorite were added. The reaction mixture was placed in a water bath under constant stirring for two hours at 80°C. The experiment was repeated two to three times with the same products, then filtered and washed with demineralized water. The filtered was left in an oven for 24 h at 60°C and weighed. The content is determined by the relations (7)

$$\%Th = \frac{M' - M_c}{M_2} \times (100 - (E_{EB} + E_e + T_{pec})) \quad (7)$$

Where  $M_c$  is the mass of the crucible,  $M_2$  the value calculated in relation (6) and  $M'$  the mass of the sample + crucible.

#### Step 6: Determination of Cellulose Content

In a 100 ml flask, a dry mass of holocellulose obtained at the previous step and 50 ml of a 17.5% NaOH solution (m / m). The mixture is maintained with magnetic stirring at 25°C for 30 minutes. The solution is then diluted to a NaOH concentration of 8.75% (w / w) by adding 50 mL of distilled water. The environment the reaction mixture is once again stirred magnetically at 25 degree. Minutes, then filtered. The solid residue (cellulose) is washed with 1%

acetic acid and then with distilled water and dried overnight under a hood and weigh. Content in cellulose is determined by the relation (8)

$$\%Cell = \frac{M''-m}{M_0} \times 100 \quad (8)$$

Where M'' is the mass of the crucible + holocellulose, m the mass of the hollow and Mo the mass of holocellulose.

#### Step 7: Determination of hemicellulose Content

The mass of hemicellulose is obtained following a mass subtraction of cellulose on holocellulose. Its content is obtained by subtraction of the yield of cellulose on holocellulose.

$$\text{Mass of hemicellulose} = \text{mass of holocellulose} - \text{mass of cellulose} \quad (9)$$

$$\% \text{ Hemicellulose} = \% \text{ holocellulose} - \% \text{ Cellulose.} \quad (10)$$

#### Step 8: Determination of Klason Lignin

The lignin level is determined by the Klason method which consists of solubilizing the polysaccharides (cellulose and hemicellulose) by treating the plant material with a sulfuric acid solution at 72% (by mass). In a 100-mL flask, 1.40g of material dried after extraction of the pectin's was treated with a solution of 20 mL of acid 72% sulfuric for 1 h in a water bath set at 30°C to completely hydrolyze polysaccharides. The mixture is then diluted to 3% H<sub>2</sub>SO<sub>4</sub> (by adding 345 mL of water distilled) and boiled for two hours. The mixture is then cooled, filtered and the residue washed thoroughly with distilled water until the wash solution is practically neutral. (Mounguengui S., et al. 2016) The residue (lignin) is dried overnight and weighed. The lignin level is calculated by the relation (11)

$$\%Lig = \frac{M_t - M_{cf}}{M_e} (100 \times E_{EB}) \quad (11)$$

#### Fourier Transform Infrared spectroscopy (FTIR): -

Fourier Transform Infrared spectroscopy (FTIR) is a rapid method for the characterization of functional groupings and major components of different samples (Silverstein et al., 1991). The infrared spectra are recorded on a spectrophotometer ALPHA-P a transformed Fourier brand "BRUKER" directly on small samples of compounds in the range of 400 to 4000 cm<sup>-1</sup>. Indeed, a small amount of the mixture (the sample + purified KBr salt) of the micrometer order is deposited on a surface crystalline made of diamond and previously cleaned. These analyzes are done in the laboratory of Analytical Chemistry in the University of Yaounde 1.

#### XRD Analysis on the three Samples: -

X-ray diffraction (XRD) analysis was used to determine the crystalline structure and composition of materials. This technique was used for analysing roofing tile reinforcement, XRD provide crucial information about the crystalline structure and orientation of pineapple fibers. The findings of the XRD analysis conducted on the pineapple fibre samples (S3, R3, and L3) are discussed.(Berzin et al 2017)

#### TGA Analysis of samples S3, R3 and L3: -

Thermogravimetric analysis (TGA) is used to examine a material's thermal characteristics, such as residual mass, decomposition patterns, and thermal stability. TGA can offer important information about the thermodynamic properties of pineapple fiber reinforcement. This technique was used to influence the effectiveness of roofing materials manufactured with these fibers with regards to samples S3, R3, and L3 as they were subjected to TGA analysis. (Sarangi, P.K et al 2022)

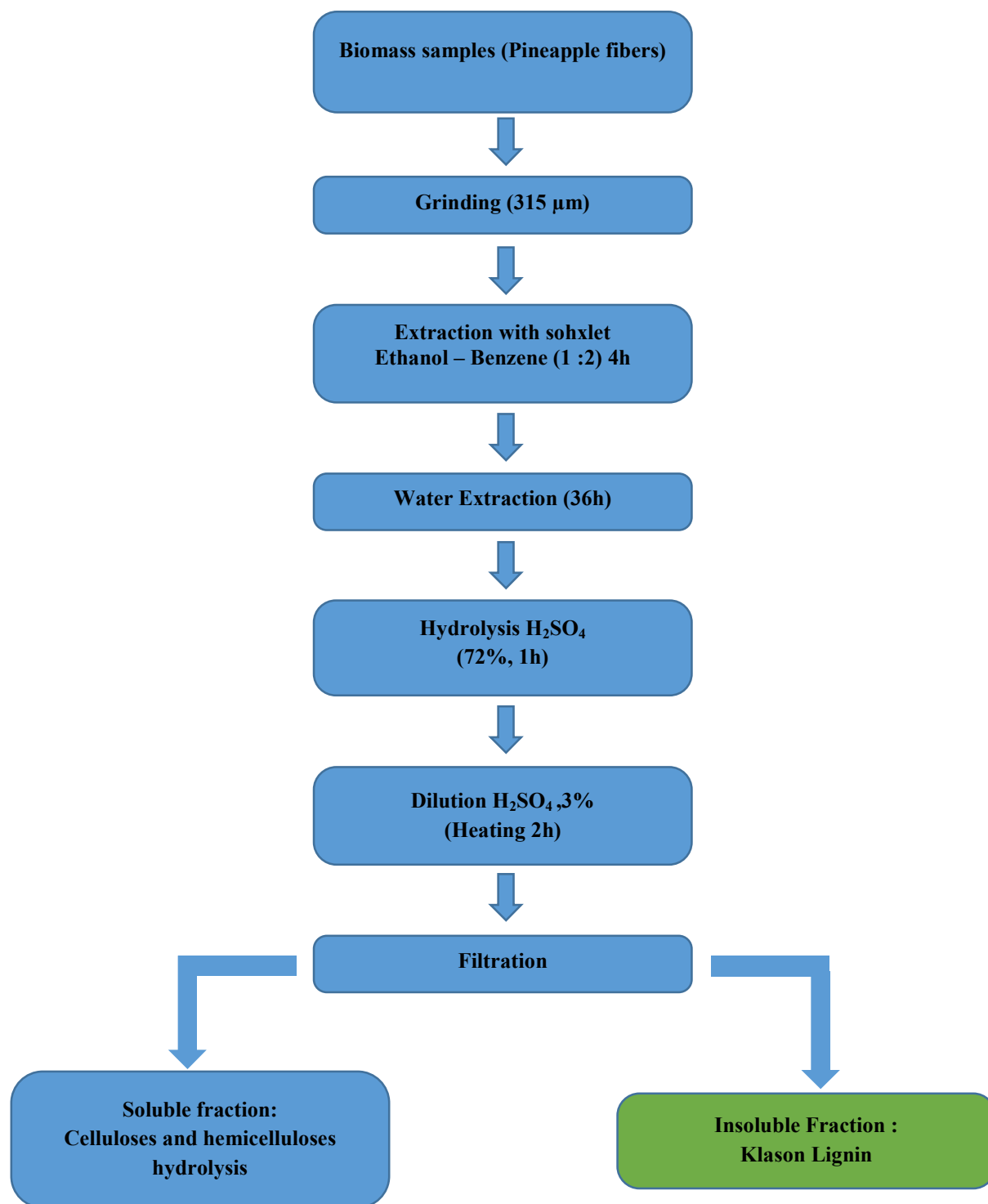


Figure 4: Schematic of the Klason lignin extraction protocol (Mounguengui S., et al. 20116).

**Results and Discussion: -**

Yield of extraction method: -

**Table 5: Efficiency of different extraction processes**

Extraction Process	Soaking solution	Period/ Days	Temperature	% of fiber yield
Manual extraction by soaking	Distill water	4weeks in distill water	Ambient temperature	70 to 80%
Extraction by scraping	Distill water	7days	Ambient temperature	60 to 70%
Extraction by beaten	Distill water	7days	Ambient temperature	40 to 60%
Extraction by Water retting	Distill water	7days	Ambient temperature	70 to 80%
Extraction sodium hydroxide (NaOH) for 1h30mins	Distill water + caustic soda	1hour 30minutes	100 °C	45%
Extraction with sodium hydroxide (NaOH) for 2hrs	Distill water + caustic soda	2 hours	100 °C	20 to 45%
Extraction with sodium hydroxide (NaOH) for 3hrs	Distill water + caustic soda	3 hours	100 °C	30 to 60%
Extraction with sodium hydroxide (NaOH) for 4hrs	Distill water + caustic soda	4 hours	100 °C	30 to 60%

**Presentation of fibers obtained: -**

The images of some fibers obtained with the help of different extraction processes are presented in Figure5 below.

**Figure 5: fibers obtained****Table 6: Average length and diameter of pineapple fiber**

Fibers	Average length	Average diameter
Leaves	500mm long	0.0403mm
Stems	250mm long	0.0423mm
Roots	80mm long	0.051mm

**Average length and diameter of pineapple fiber: -**

The evolution of average diameter in function of the extraction process. It is difficult to estimate the process to be used and conclude on the average diameter. Knowing that the standard deviation varies in a non-uniform manner as shown on fig 7.

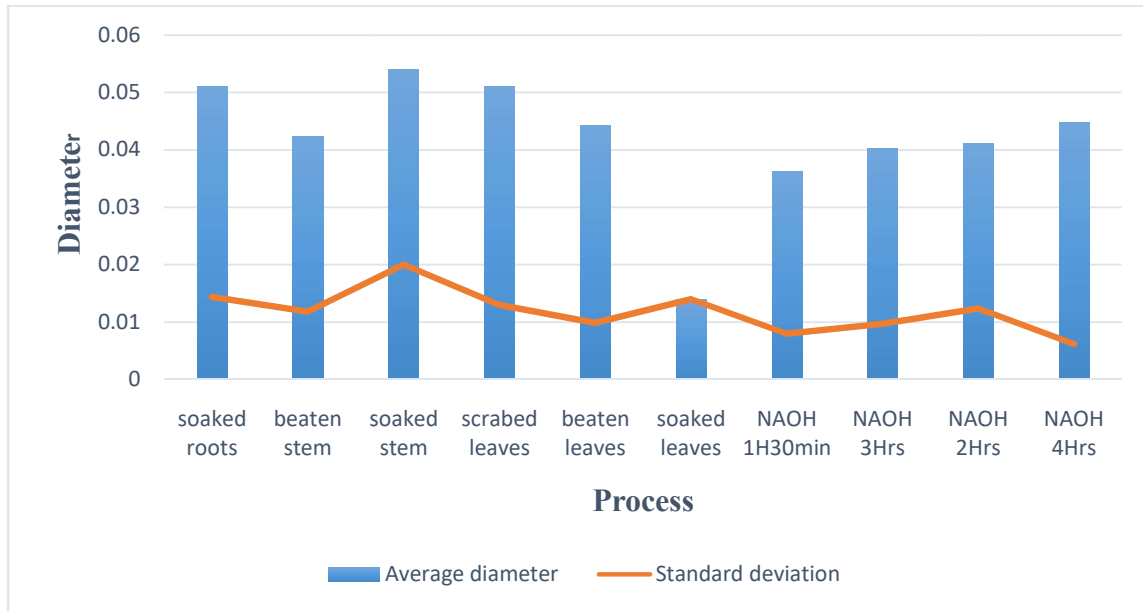


Figure 6: Evolution of diameter with respect to standard deviation

**Density: -**

The density of the pineapple fibers were determined using the: (Norm NFP 94-053)

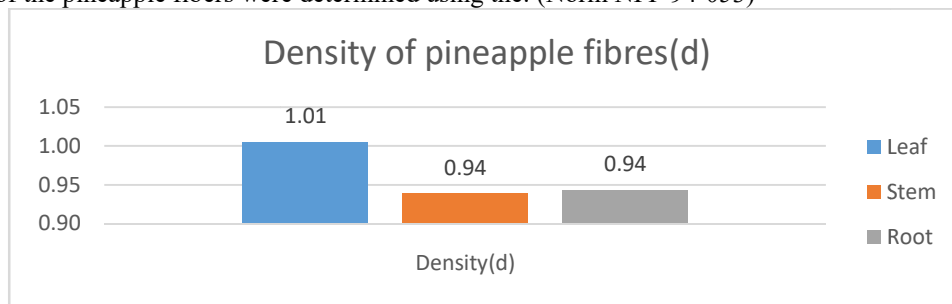


Figure 7: Density of pineapple fiber

**Moisture content: -**

The moisture content of the pineapple fibers by taking three different parts of the pineapple plant. The gravimetric method allows the measurement of the water content figure 8 by determining the moisture content of the fibers by evaporation using the Norm NFP 94-054.

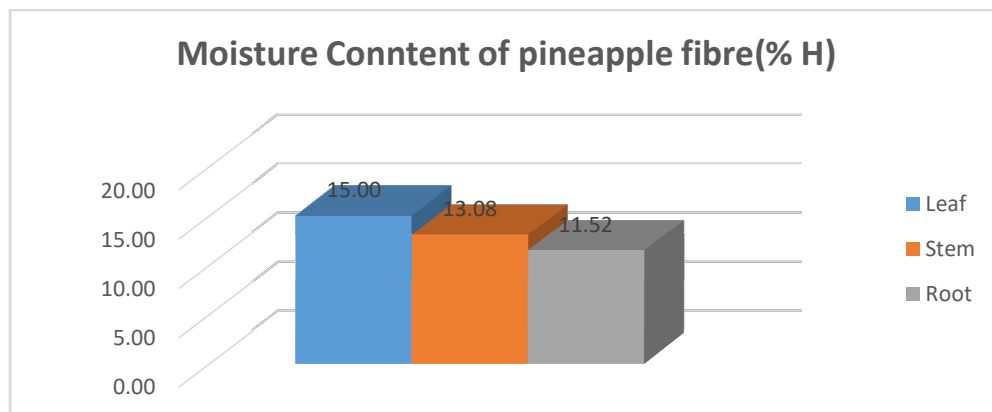


Figure 8: Evolution of moisture content of pineapple fiber parts

**Comparative study: -**

Comparative study of chemical properties of some natural fibers and that of pineapple fibers

**Table 7: chemical properties of natural fiber studied with that of literature revue.**

Fibers	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Ash (%)
Abaca	61-64	21	12	-	3
Bagasse	32-48	21	19,9-24	-	-
Banana	60-65	6-19	75-10	-	-
Bamboo	26-43	15-26	21-31	8	-
Coir	46	0,3	45	-	-
Cotton	82-96	2-6	0,5-1	-	-
Flax	60-81	14-19	2-3	-	-
Hemp	70-92	18-22	3-5	0,9	0,8
Jute	51-84	12-20	5-13	0,2	0,8
Kenaf	5144-57	21	15-19	-	3,3
Ramie	68-76	13-15	0,6-1	1,9	
Sisal	43-78	10-13	4-7	10	0,6
Wood	4545-50	23-30	27	-	1,3
Pineapple leaf	72,14	4,86	13,55	1,6	1
Pineapple stem	53,09	20,91	15,16	3	0,95
Pineapple root	57,04	17,96	13,22	2,2	0,86

**Fourier Transform Infrared spectroscopy (FTIR): -**

From the FTIR spectroscopy used to observe functional groups in natural fibers, table 8 the following table that shows that the lignocellulosic material is pure because the bands is between 3333, 80 to 871, 73cm<sup>-1</sup>.

**Table 1: Assignment of absorption bands of the functional groupments of pineapple**

Frequency (cm-1)			Assignment of functional groupments
leaves	stems	roots	
3600 to 3000			The broad band, observed in all spectra at 3600–3000 cm <sup>-1</sup> , represents the stretching vibration of hydroxyl groups (O-H)

2937 to 2851	Bands between 2937–2851 $\text{cm}^{-1}$ are attributed to the symmetrical and asymmetrical stretching vibrations of the C-H links.
1736 to 1732	This band was assigned to carbonyl functions (C=O) corresponding to their stretching vibrations in acetyl groups and uronic esters in hemicelluloses or in ester linkages between hydroxyl groups and of p-coumaric and ferulic carboxylic groups in lignins.
1648 to 1635	The band located at 1648 to 1635 $\text{cm}^{-1}$ corresponded to the bending-angular deformation of the structural water molecules (H–O–H) adsorbed in the structure.
1612 to 1519	The absorption bands, were attributed to aromatic skeleton carbon-carbon double bonds (C=C stretching) and aromatic skeletal vibration of polyphenolic groups of lignin.
1397 to 950	The symmetrical and asymmetrical stretching bands between 1397-950 $\text{cm}^{-1}$ are characteristic of C–O–C ether bond of anhydropyranosic cycle, C–C, C–OH, C–H ring and side group vibrations.
895 to 871	Finally, the band at 895 $\text{cm}^{-1}$ , in angular deformation, is typical of the $\beta$ –(1→4) glycosidic bonds between anhydroglucose units in cellulose.

The figure 9 to 11 shows the FTIR spectroscopy used to observe the functional groups in natural fibers of pineapple before and after the extractives.

#### The Fourier Transform Infrared Spectroscopy (FTIR) for Samples Leaf(L), Stem(S)and Root(R): -

The Fourier Transform Infrared Spectroscopy (FTIR) results on the mechanical and thermal conductivity characterization of a roofing tile reinforcement using pineapple fibers with the combination of Leaf (L) sample: Figure 9: shows the FTIR analysis of Sample Leaf(L)

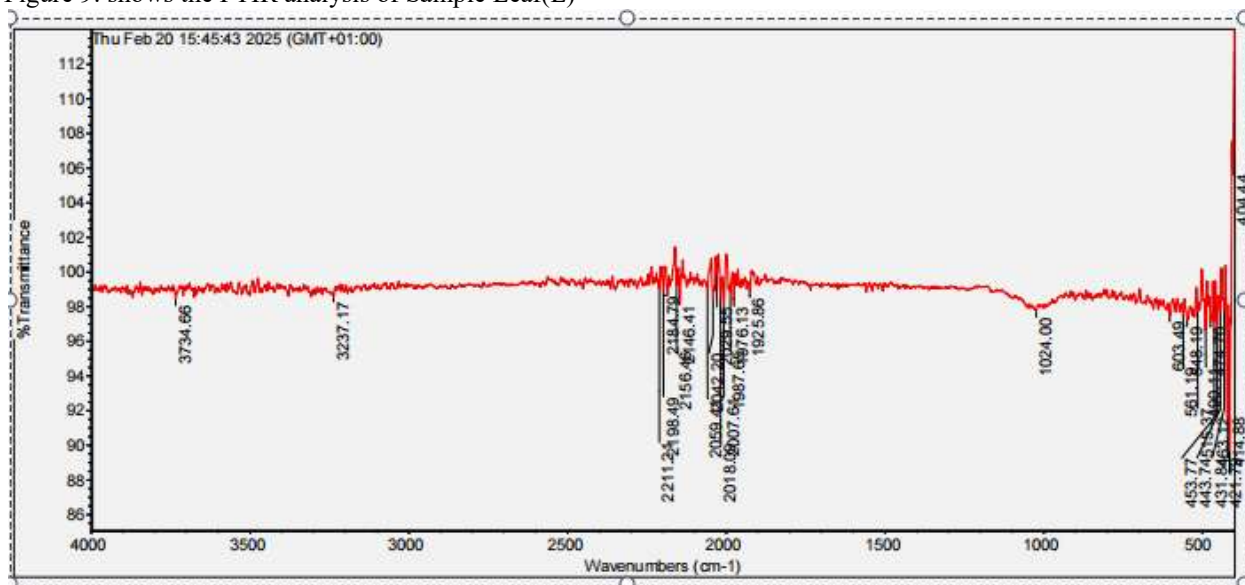


Figure 9: FTIR analysis of Sample Leaf(L)

**FTIR Analysis for Sample STEM(S): -**

FTIR analysis was performed to identify the functional groups present in the pineapple fiber-reinforced roofing tile. The FTIR spectrum showed several peaks, which can be assigned to specific functional groups. Figure 10 shows the FTIR Analysis with combined Stem(S).

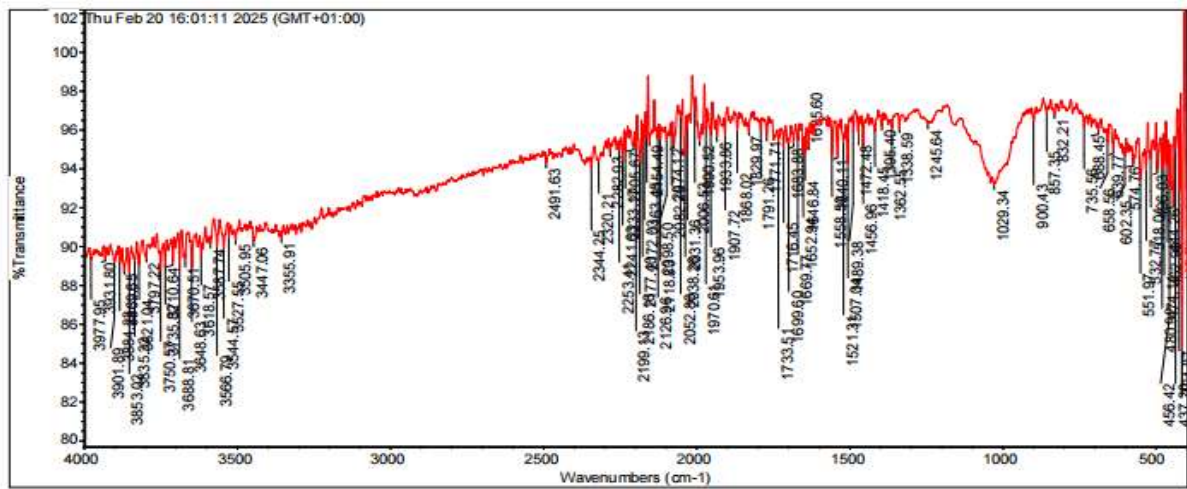


Figure 10: FTIR Analysis with combined Stem(S).

The results obtained from the FTIR analysis shows the spectrum’s peaks that attributed to the functional groups. These functional groups are O-H stretching: 3447.06 cm-1 (intensity: 90.276), 3355.91 cm-1 (intensity: 90.470). Stretching from C to H: 3505.95 cm-1 (intensity: 90.332), 3527.55 cm-1 (intensity: 90.479) 1652.94 cm-1 (intensity: 94.814) and 1733.51 cm-1 (intensity: 95.403) exhibit C=O stretching. The C-O stretching was 1032.34 cm-1 with an intensity of 93.216 and 1160.13 cm-1 with an intensity of 91.706. C-C stretching: 1489.38 cm-1 (intensity: 95.916), 1456.96 cm-1 (intensity: 95.208) - Si-O stretching: 548.84cm-1 and 540.53cm-1 (intensity: 86.462 and 86.788, respectively).

**FTIR Analysis for the sample root: -**

The FTIR analysis was used to determine the functional groups that were present in the roofing tile reinforced with pineapple fibre. The FTIR analysis was conducted and the results show the number of peaks in the FTIR spectrum which were attributed to particular functional groups. Fourier Transform Infrared Spectroscopy (FTIR) results on the and thermal characterization of a roofing tile reinforcement using pineapple fibers with the combination of roots(R) sample. Figure 11 shows the FTIR analysis of Sample Root (R).

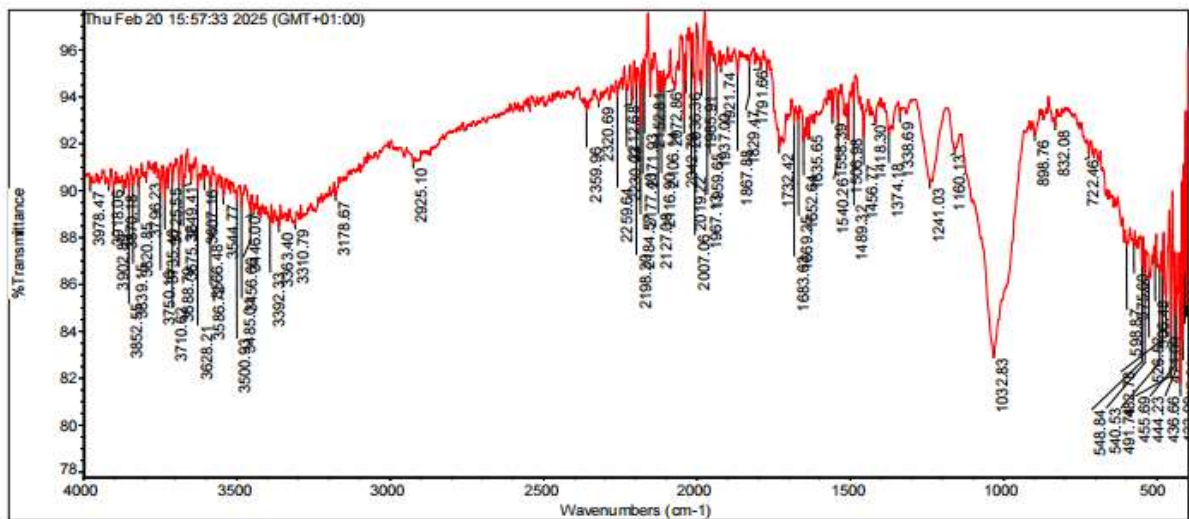


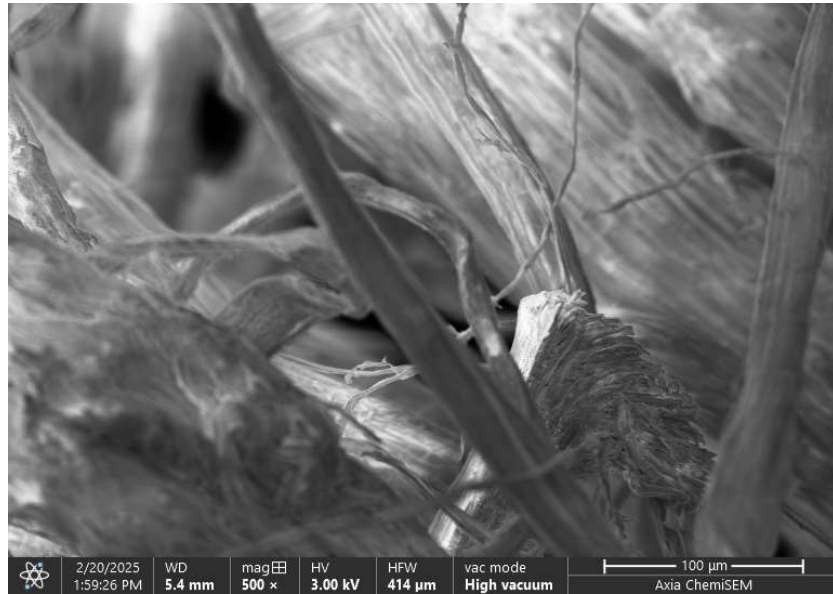
Figure 11: FTIR analysis of Sample Root (R).

The FTIR spectrum's peaks can be attributed to the functional groups listed below: O-H stretching distances: 2925.10 cm<sup>-1</sup> (intensity: 91.194), 3178.67 cm<sup>-1</sup> (intensity: 89.766) - C-H stretching: 3310.79 cm<sup>-1</sup> (78.553 intensity), 3363.40 cm<sup>-1</sup> (88.488) cm<sup>-1</sup> - C=O stretching: 1652.64cm<sup>-1</sup> for 92.086 intensity and 1732.42 cm<sup>-1</sup> for 91.831 intensity Tension of C-O stretching: 82.920 at 1032.83 cm<sup>-1</sup> and 91.706 at 1160.13 cm<sup>-1</sup> The C-C stretching measures 1456.77 cm<sup>-1</sup> with an intensity of 92.299 and 1489.32 cm<sup>-1</sup> with anintensity of 93.820. - Si-O stretching: 548.84 cm<sup>-1</sup> and 540.53 cm<sup>-1</sup> (intensity: 86.462 and 86.788, respectively). Several functional groups are present in the pineapple fiber-reinforced roofing tile combined with the use of the Root (R). According to the FTIR data obtained, the results revealed the existence of hydroxyl groups: the O-H stretching peaks at 2925.10 cm<sup>-1</sup> and 3178.67 cm<sup>-1</sup>, which could be related to the cellulose and lignin components of pineapple fibres.

**SEM Analysis on Sample Leaf(L), Stem(S)and Root(R): -**

**SEM Analysis on Sample Leaf(L): -**

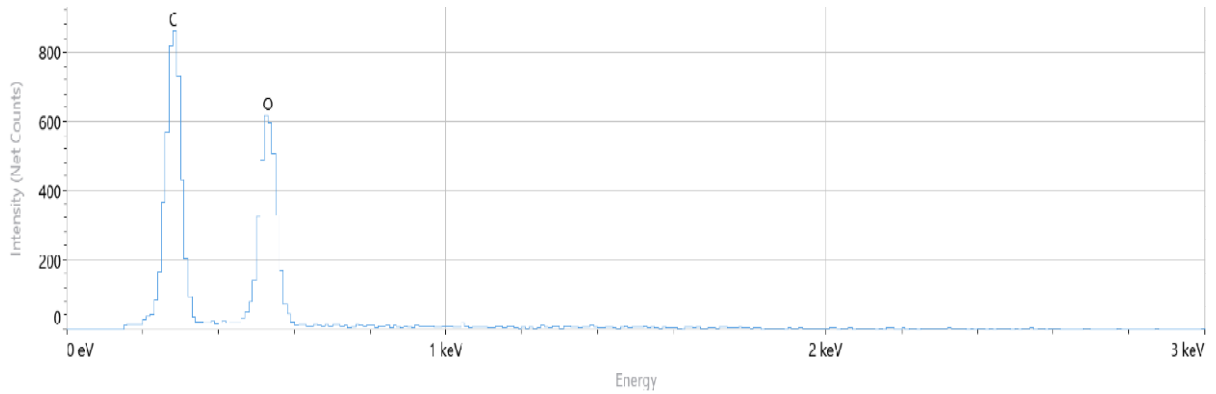
Interpretation of the SEM results on characterization of a roofing tile reinforcement using pineapple fibers with the combination of Leaf (L) and EDX (Energy-Dispersive X-ray spectroscopy) analysis Figure 12: shows the SEM Image with combined Leaf (L) as reinforcement, Table 9 shows the EDX results with the elemental weight composition. Figure 14 shows the intensity net count for carbon above 800 and O above 600.



**Figure 13: SEM Image with combined Leaf (L) as reinforcement.**

**Table 9: EDX Elemental composition for Sample Leaf (L)**

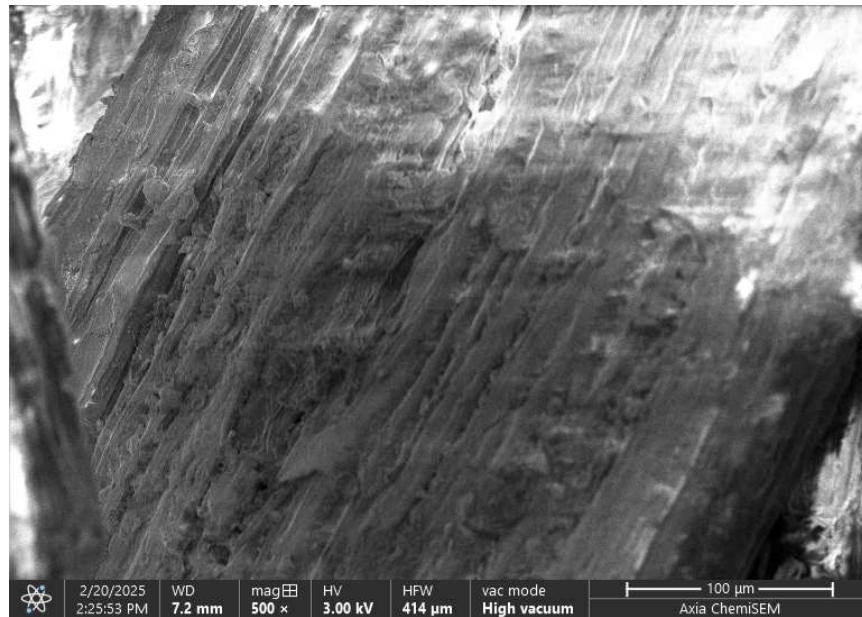
Element	Line	At. %	Wt. %	Net Counts	At. % Error	Wt. % Error
C	K	42.1	35.3	4 447	0.6	0.5
O	K	57.9	64.7	3 114	1.2	1.3



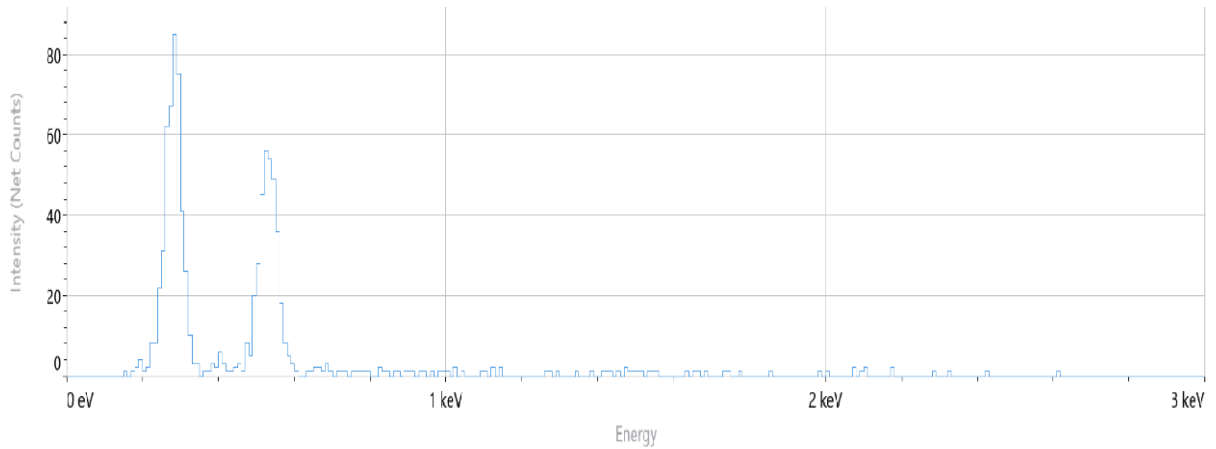
**Figure 13: Intensity net count for carbon above 800 and O above 600**

**SEM Analysis on Sample Stem(S): -**

SEM examination: To investigate the morphology and microstructure of the roofing tile reinforced with pineapple fibre, SEM (Scanning Electron Microscopy) examination was carried out. The SEM pictures showed: Interpretation and discussion of the SEM results on mechanical and thermal conductivity characterization of a roofing tile reinforcement using pineapple fibers with the combination of stem (S): Figure 14 show the SEM Image with combined Root (R) as reinforcement. Figure 9 shows the intensity net count for carbon above 80 and O above 40



**Figure 14: SEM Image with combined Stem (S) as reinforcement**

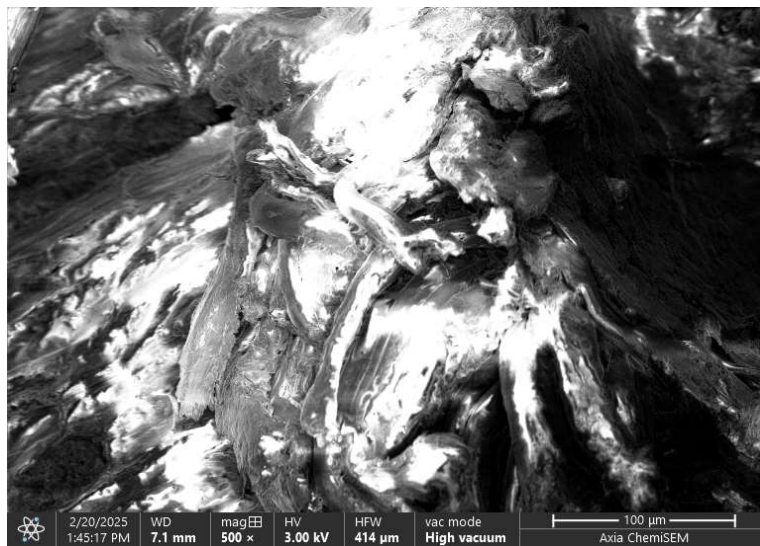


**Figure 15: Intensity net count for carbon above 80 and O above 40**

SEM Analysis revealed the morphology and microstructure of the pineapple fibre-reinforced roofing tile in conjunction with Stem (S). The SEM images showed: The roofing tile matrix contains a skewed dispersion of pineapple fibres. The pineapple fibres and matrix have a poor interfacial connection, and the composite material's existence of certain flaws and gaps.

**SEM Analysis on Sample Root(R): -**

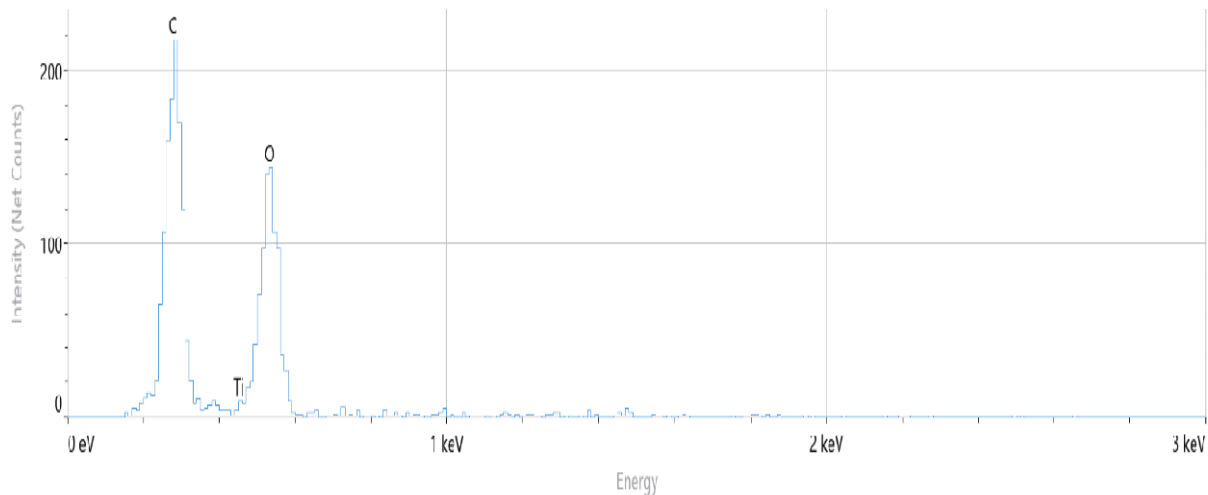
Interpretation of the SEM results on thermal characterization of a roofing tile reinforcement using pineapple fibers with the combination of Root(R) and EDX (Energy-Dispersive X-ray spectroscopy) analysis: Figure 16 shows the SEM Image with combined Root (R) as reinforcement. Table 10 shows the EDX results with the elemental weight composition. Figure 18 shows the intensity net count for carbon above 200 and O above 100



**Figure 16: SEM Image with combined Root (R) as reinforcement**

**Table 10: EDX Elemental composition for Sample Root (R)**

Element	Line	At. %	Wt. %	Net Counts	At. % Error	Wt. % Error
C	K	38.4	25.9	1 174	1.1	0.8
O	K	51.2	46.0	732	2.1	1.9
Ti	L	10.4	28.1	107	6.8	18.4

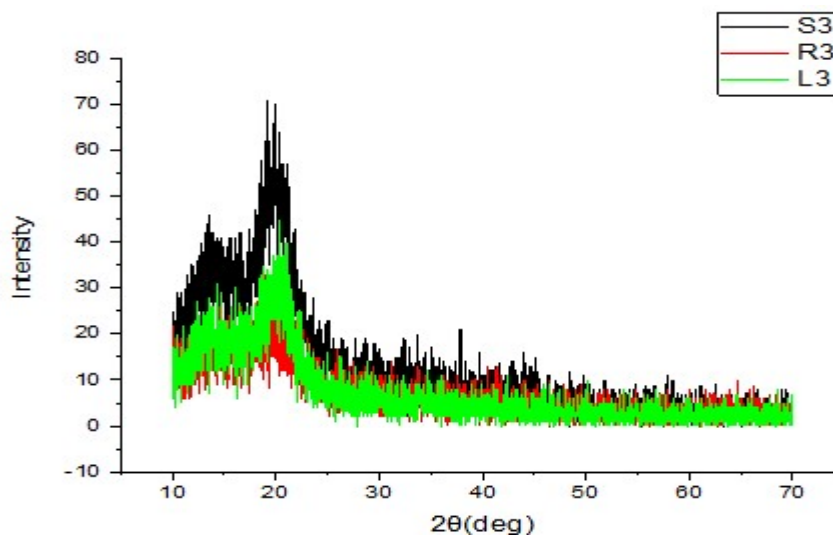


**Figure 17: Intensity net count for carbon above 200 and O above 100**

Sample Stem (S) was examined by utilising SEM, which was used to analyze the morphology and microstructure of the roofing tile reinforced with pineapple fibre. The results revealed that the Pineapple fibres were evenly distributed throughout the roofing tile matrix, and there was strong interfacial adhesion between the matrix and the pineapple fibres. Pineapple fibres are evenly distributed throughout the roofing tile matrix, and there is strong interfacial adhesion between the matrix and the pineapple fibres. - the composite material's existence of certain flaws and gaps.

#### **XRD Analysis on the three Samples: -**

X-ray diffraction (XRD) analysis was used to determine the crystalline structure and composition of materials. This technique was used for analysing roofing tile reinforcement, XRD provide crucial information about the crystalline structure and orientation of pineapple fibers. The findings of the XRD analysis conducted on the pineapple fibre samples (S3, R3, and L3) are discussed. According to the XRD patterns, Stem (S3) fibers had a higher crystallinity index (CI) of 63.2%, indicating a better arranged crystalline structure. The peak at  $2\theta = 25^\circ$  with respect to 70 intensity represents the cellulose's (002) plane. The root (R3) fibers showed a more amorphous structure with a lower CI of 55.1%. The peak at  $2\theta = 20^\circ$  with respect to 30 intensities represents the cellulose's (101) plane. Lastly, leaf (L3) fibers showed an intermediate CI of 59.5%, indicating a mixture of crystalline and amorphous structures. The peak at  $2\theta = 22^\circ$  with respect to 45 intensity represents the cellulose (101) plane.



**Figure 18: XRD for Stem, Roots and Leaf Samples**

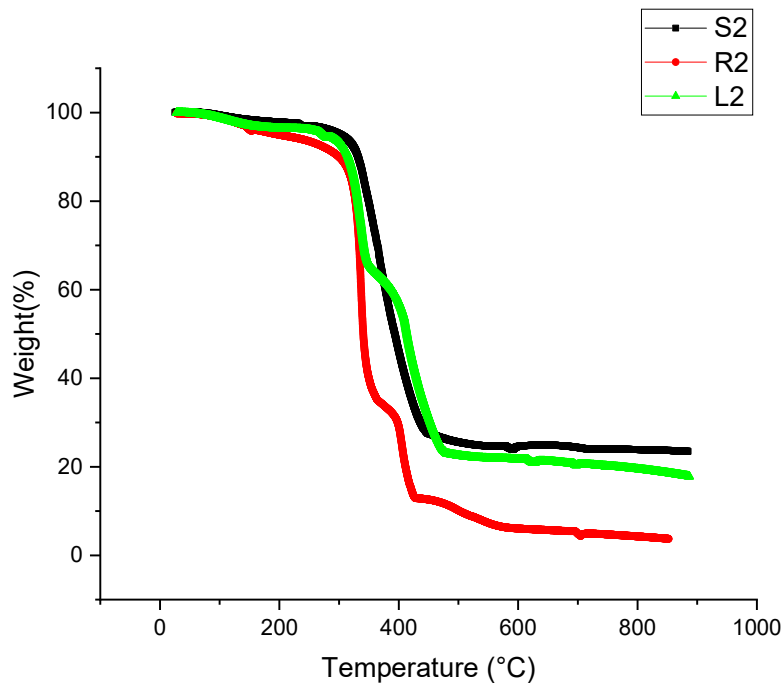
**Comparing Results: Comparing the XRD data of the fiber samples (S3, R3, and L3), a few differences were observed:**

(a) Crystallinity Index (CI): The stem fibers (S3) had the highest CI, followed by the leaf fibers (L3) and the root fibers (R3).

(b) Peak Intensity: The stem (S3) fibers have the highest strength peak which revealed a better structured crystal arrangement.

#### TGA Analysis of samples S3, R3 and L3: -

Thermogravimetric analysis (TGA) is used to examine a material's thermal characteristics, such as residual mass, decomposition patterns, and thermal stability. TGA can offer important information about the thermodynamic properties of pineapple fiber reinforcement. This technique was used to influence the effectiveness of roofing materials manufactured with these fibers with regards to samples S3, R3, and L3 as they were subjected to TGA analysis. (Neto, A.R.S et al 2013)



**Figure 19: TGA for Stem, Roots and Leaf Samples**

The fibers are impacted by a chemical reaction because the temperature in the surrounding area increases leading to a considerable reduction in weight (%) between 400°C and 900°C. This degradation resulted from the breakdown of the cellulose, hemicellulose, and lignin components of the fibers. The residual mass at 900°C indicates the quantity of carbon and mineral compounds that are in the fibers.

#### Comparative Results:

When analysing the TGA curves for these similar samples (S3, R3, and L3), certain distinctions can be observed: The stem's (S3) fibers showed a somewhat stronger stability at elevated temperatures but an increasingly prolonged weight loss process between 200°C and 400°C. The phenomena might be brought on by the higher concentration of cellulose in stem fibers. Weight(%) fell more quickly with respect to increase in temperature from 400°C to 900°C, indicating a lower thermal stability in root fibers (R3). This might be as a result of the higher lignin content of root fibers. In line with the patterns observed in stem and root fibers, leaf (L3) fibers had a declining weight pattern and a moderate degree of heat resilience.

**Conclusion: -**

Studies have shown that natural defects including elbows have different behavior from the rest of the fiber which also influences the behavior of it. The proportion of natural flaw statistic for a tested fiber length is not the same from one fiber to another due to its nature, A comparison of these results with other plant fibers revealed that pineapple fibers have better physico- chemical properties than several fibers encountered in literature like coconut fiber, flax, ramie, jute etc. The stem's (S3) fibers showed a somewhat stronger stability at elevated temperatures but an increasingly prolonged weight loss process between 200°C and 400°C. The phenomena might be brought on by the higher concentration of cellulose in stem fibers. Weight(%) fell more quickly with respect to increase in temperature from 400°C to 900°C, indicating a lower thermal stability in root fibers (R3). This might be as a result of the higher lignin content of root fibers. In line with the patterns observed in stem and root fibers, leaf (L3) fibers had a declining weight pattern and a moderate degree of heat resilience. For XRD, Crystallinity Index (CI): The stem fibers (S3) had the highest CI, followed by the leaf fibers (L3) and the root fibers (R3), Peak Intensity: The stem (S3) fibers have the highest strength peak which revealed a better structured crystal arrangement. According to the achieved level of physico- chemical and thermal properties of pineapple fibers appear to be quite suitable as reinforcement for roofing shingles materials. This work made it possible to identify some important points on which efforts must be granted.

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