



EXTRACTION AND CHARACTERIZATION OF ETHIOPIAN PINEAPPLE LEAF FIBER

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Abstract

Natural fibers particularly pineapple leaf fiber (PALF) plays important role in the uprising world industry. This fiber is extracted from the leaves of pineapple plant. Pineapple is cultivated for local consumption in Ethiopia within the regions. The pineapple leaves produce agro waste that cause problems for its disposal. This waste can be used as a raw material for value added processing contributing to maintain an ecological balance in nature. This study investigates simple methods of extracting fibres from its leaves. The method of extraction is Scrapping, retting and decorticating methods. The leaves are processed to explore practical utilization of the fibres. The pineapple leaf fiber yielded between 2.5-3%. Under this study the fibers physical and mechanical structure (morphology and molecular structures) are analyzed through different instrument.

Keywords: Pineapple leaf fiber (PALF), Extraction, Retting, Cultivation

1. Introduction

Pineapple leaf fiber is one kind of fiber derived from plants (vegetable fiber) which is derived from the leaves of the pineapple plant. Pineapple which also has another name, that *Cosmosus Ananas*, (including the family Bromeliaceae); in general this is a crop plant season. Historically, this plant comes from Brazilian and brought to Indonesia by the Spanish and Portuguese sailors around 1599(Adam et al., 2014).

The pineapple leaf shape resembles a sword that taper at the ends with black and green colors on the edges of the leaves are sharp thorns. Depending on the species or type of plant, pineapple leaf length is between 55cm to 75cm by 3.1cm to 5.3 cm wide and 0.18cm thick leaves of up to 0.27cm. In addition pineapple species, spacing and distribution of sunlight will affect the growth of leaf length and strength properties of the resulting fiber. Distribution of sunlight is not too much (partly hidden) generally will produce a strong fiber, refined, and similar to silk (Adam et al., 2016).

The pineapple leaves produce agro waste that cause problems for its disposal. This waste can be used as a raw material for value added processing contributing to maintain an ecological balance in nature(Nadirah et al., 2012). The present study investigates simple methods of extracting fibres from its leaves. The leaves are processed to explore practical utilization of the fibres (Bhattacharyya, 1998). While separation process or pineapple fiber from the leaves can be done in three ways, namely the manual, retting and mechanical methods. The most common and effective is the manual method, the process is done by immersion. In this process, micro-organisms play an important role to separate or remove Gummy substance which surrounds the pineapple leaves

and this process will cause fiber and decompose easily separated from each other (Hidayat, 2008b). This process is done by soaking the leaves of the pineapple into the water in a certain period of time such as a week.



Figure 1: Pineapple plant

Each pineapple fruit has equal number of hexagonal sections on outer shell and does not depend on the size or shape. Now Malaysia is one of large producers in Asia as much as Hawaii. It produces a huge amount of waste material, about 384,673 metric tonnes in year 2008. Productions of Pineapple leaf fibres are plentiful for industrial purpose without any supplementary addition and annually renewable and of easy availability. Pineapple is known as Nanas in Malaysia; basically they use different varieties for different purpose; for commercial purpose they use red pineapple and green pineapple; for edible purpose, they prefer Sarawak pineapple and Morris pineapple. Pineapple fruits contain many major and minor elements (Asim et al., 2015).

This research is basically depends on the characterization of pineapple leaf fibers under different testing methods like FTIR, DSC, TGA etc and testing of its properties. The research done based on our country for reduction of pineapple leaf wastes after the collection of pineapple fruits. When pineapple fruits are collected, its leafs and other wastes are thrown on the farming land, road and in the town. Then these wastes are damaged to cause pollution and environmental impacts. Therefore, the research helps to convert the thorny pineapple leaves which are mostly discarded as waste, may soon become valuable raw materials(fiber) products, also reduction of wastes in the town and rural places (pineapple planters). The research contributes the idea of fiber production from pineapple leaf and this helps the farmers how they easily produce the pineapple leaf fibers. Efforts are on to use these discards to produce natural fibres which have wide range of applications.

The main objective of the research is extraction and characterization of Ethiopian pineapple Leaf fiber. Under this objective some specific parameters are studied as referred below.

- Reduction of pineapple leaf waste (use for fiber purpose) to make safe environment for the farmers.
- Contributing idea methods of pineapple leaf fiber production for the farmers and small scale sectors present around pineapple producers' area.
- Effective use of waste of natural plant leaf.
- Import substitution of cotton fiber with pineapple fiber.

1.1. Pineapple Cultivation in Ethiopia

Farmers and farming systems in the once extremely fertile midland region here are stressed by an increasing human population. A high proportion of children here are malnourished and the farming systems are still more subsistence- than market-oriented. With the rise of Ethiopian markets for cash crops and dairy products, farmers here are specializing in such crops as coffee and chat, which are replacing the region's traditional food crops like Inset (*Ensetvetricosum*) (Mengesha et al., 2013, Lemma et al., 2016).

In Ethiopia Sidama region is the only region which produces the high amount of pineapple fruit for local markets unlikely by vehicles directly from farm land, pineapple was introduced here about 50 years ago. Pineapples should do well here, where the warm climate and soils suit the plant (Debasis and Sanjoy, 2007). Although pineapples can fruit throughout the year, in Sidama, the peak harvests are from April to May and October to November. Because pineapple farming is limited to just a few districts in Ethiopia and is grown by just a few producers, the pineapple value chain is underdeveloped in the country. Among constraints faced by pineapple farmers are lack of planting materials, little knowledge of optimal production practices and inadequate marketing systems.

Sidama farmers use suckers costing 3 to 5 Ethiopian birr (ETB) each to propagate their pineapples, although planting can also be done using ratoons, slips, crowns, seeds and tissue-cultured plantlets. Plants propagated with suckers produce their first fruits ready for harvesting in less than sixteen months while plants propagated by other methods require more than two years for a first harvest (Mengesha et al., 2013). For this reason, suckers are commonly disseminated farmer to farmer in Sidama and other parts Ethiopia.

Sidama's pineapple production system is rain-fed, with growers harvesting less than 10 tons per hectare per year, a mere tenth of the 100 tons of pineapple produced per hectare per year elsewhere in the world. The Ethiopian farmers can increase their yields, and the quality of their fruit, by applying organic and inorganic fertilizers, regulating plant stands through sucker management, providing supplemental irrigation, and weeding and mulching.

2. Methods and Materials

2.1. Raw Materials

The sample of pineapple (*Ananas Comosus*) leaf is collected from Hawassa around rural village in the farmers land. This raw material was used as an alternative to produce fibers in this study. After collecting the leaf, fiber is produced using manual, retting chemicals and decortication methods. When fiber is produced use a clamshell (for removal of any remaining wastes on the fiber surface), water (running water so it will not knot the fiber) and any kind of soap to wash the fiber to become white. Finally dry the fiber using dryer machine or/and sun light.

The extraction process had to be carried out within 3 and 4 days of harvest. If kept longer, the leaves become dry and the fibres are difficult to extract. The leaves were tested for their dimensions such as weight and length. Various techniques for extracting fibres that are embedded in the leaves were experimented. Simple tools and methods such as scrapping with clamshell and knife were used to remove the fibres from the leaves which were covered with a protective thick hydrophobic wax layer.

2.2. Methods of pineapple leaf fiber Extraction:

2.2.1. Scrapping method of extraction

This process is carried out by preparation of a plate having different shapes (may be broken plate) and knife. This plate is used for scratching the pineapple leaf on a flat surface, for extracting fibers. Washout the fiber by the use of a clamshell for removal of remaining gummy substances on the fiber surface and whitening the fiber soaping.

2.2.2. Retting of pineapple leaf

Pineapple leaves were immersed in a water container having a capacity of 10 liters, filled with tap water and kept for 30-35 days to ret. The leaves were not exposed to sunlight, rain and wind for uniform retting of all leaves. Water was added frequently to maintain its level. The leaves were observed regularly. The leaves were analyzed intermittently for testing the ease of fibre extraction. This process was carried out without chemicals. But, the second method is using chemical. Small bundles of scratched pineapple leaves are immersed in a water tank which contains substrate: liquor in 1: 20 ratio, urea 0.5%, or diammonium phosphate (DAP) for fast retting reactions. This method was taken about 10-15 days for retting.

2.3. Analysis of Fiber properties

Testing the fiber properties like fineness, Length, strength, counts etc and characterization of pineapple fiber under FTIR, DSC, TGA and its structures under microscope.

2.3.1. Leica Biological microscope

Leica biological microscope (Leica DM500) gives detailed micro structure of fibres. Application of Leica biological microscope is for High quality and accuracy microscope, longitudinal and sectional analysis of fibres.

2.3.2. Fourier Transform Infrared (FTIR)

The extracted fiber was identified using a Fourier transform infrared spectroscopy (FTIR, Nicolet impact 410).

2.3.3. Thermo gravimetric analysis (TGA)

Thermogravimetric analysis was carried out using a Perkin Elmer (Model TGA4000) analyzer. The samples weighing between 7-20 mg were placed in ceramic crucibles, while the tests were carried out in nitrogen atmosphere. The heating rate of the samples was $20^{\circ}\text{C min}^{-1}$. It measures the amount and rate (velocity) of change in the mass of a sample as a function of temperature or time in a controlled atmosphere.

2.3.4. Differential scanning calorimetry (DSC)

The preparation of the samples that were used for the differential calorimetric was similar to the TGA. Meanwhile, the analysis in the DSC was performed using the Perkin Elmer (Model DSC4000) analyzer. The temperature was programmed in the range of 25°C to 300°C , under nitrogen atmosphere. In a DSC the difference in heat flow to the sample and a reference at the same temperature, is recorded as a function of temperature. The temperature of both the sample and reference are increased at a constant rate.

3. Result and Discussion

The pineapple leaves collected from the planters were examined. The average length of the pineapple leaf is about 62.5cm weighing approximately 20.8 gm. The leaves were processed to extract fibres by various techniques.

3.1. Yield of Pineapple Leaf Fiber

For the extraction process of pineapple leaf were converted into a fiber either by retting, manual and decortication methods as described on methodology. For this study pineapple leaf were collected around Hawassa in southern region in Ethiopia and converted to fiber. Before fiber extractions the single leaf samples are measured by weighing instruments, initial sample weights have recorded. Then, fiber has been extracted from individual leafs which its initial weights measured and their weight of individual extracted fibers are recorded. Finally calculate percentage of fiber yield. The yield is calculated by the formula given below.

$$\text{Fiber Yield (\%)} = \frac{M_f}{M_i} * 100 \dots\dots\dots 4.1$$

Where M_i - original weight of pineapple leaf; M_f - Final weight of pineapple fiber extracts and dried.

Table 1: Percentage yield of pineapple leaf fiber

Sample	Fresh sample (grams)		Yield of fresh sample fiber (%)
	Initial Wt	Fiber Wt	
1	21.8	0.67	3.07
2	19.65	0.575	2.92
3	20.92	0.56	2.67
4	36.5	0.91	2.49
5	27.4	0.77	2.81
6	23.31	0.65	2.79

Based on the above formula the yield of pineapple leaf fiber is calculated in percent. As explained in the table the percentage weight of pineapple fiber yield is varying between 2.49 to 3.07% because water content for a fresh pineapple leaf would weigh around 80% of the leaf and the other remaining is gummy substance. The

result obtained in these study is related with the result of Adam et.al (Adam et al., 2016). If the fiber is extracted after drying the leaf a very small amount of fiber is obtained due to difficulty of fiber extraction after drying the pineapple leaf. This causes high breaking of fibers; fibers removed with gummy substances and resulted in short fiber.

3.2. Pineapple Fiber Extraction

The appropriate time for Pineapple leaf fiber extraction is generally done at the age of 1 to 1.5 years according to Hidayat's report (Hidayat, 2008a). A fiber derived from the leaves of the young pineapple generally is not long and strong. For fiber produced from pineapple that is too old, exposed to sunlight without protection will produce short fibers, coarse, and brittle. Therefore, to obtain a strong fiber, soft and smooth, the selection should be done in pineapple leaves enough and protected from the sun, this result is related with Anbia Adam et.al (Adam et al., 2016).

Pineapple fibers are extracted from pineapple leaves by manual means hand (figure 7); the common method in the practice is a combination of water retting and scrapping. The fibers are thoroughly washed and dried the total yield of the fiber is 2.5 to 3% of the weight of green leaves. Pineapple leaves steeped in to water for 18 days yield good spinnable fibers. If steeped in water for more than 18 days microorganisms were feed fibers with gum, as well the strength of the fiber is very weak due to microbial.

Manual Scrapping the hydrophobic waxy layer on the leaf was removed by scrapping with either broken plate or dull knife. It demanded a lot of patience and caution so that the fibres do not get damaged and the leaves are not cut. The clamshell and soft wire brush could be used to clean the residue wax layer on the fiber extracted. It was cumbersome and time consuming. Some of the fibres obtained were broken due to use of the clamshell which may result in some wastage. The leaves could be scrapped to remove the fibres with some amount of ease. The combination of tools for extraction was an effective way to extract fibres.



Figure 2: Pineapple leaf fiber extraction by scrapping method

Fibers are extracted by means of machine/manual (hand) processing. The extracted fibers in the form of long strands with slightly yellowish in colour. This fiber are then washed and dried followed by gentle combing in the wet condition with fine pins moving slowly to separate the ultimate coarser bundles and gives fine fibers of considerable length.

The process of extracting long vegetable fiber is one of the great importance since the quality as well as the quantity of extracted fiber is strongly influenced by method of extraction employed.

3.2.1. Retting of PALF (Degumming of PALF)

Pineapple leaves were kept immersed in pure water (not waste) for 15-30 days. The outer layer on the leaves had changed colour and softened to some extent. Scrapping of the leaves was required to extract fibres. As the retting period increased the ease with which the fibres could be extracted improved, this were depends on the

quality of water otherwise dirty water increase the microorganisms extremely and lead to damaging of fiber and loss of fiber strength during extraction and after extraction. It was noted that if the leaves were scraped before retting, the fibres were easier to extract. Figure 8 shows water retted leaves and fibres under the outer skin of the pineapple leaf.



Figure 3: Pineapple leaf fiber extraction by retting methods

Retting is done by means of water and microbial activity. PALF contains about 25 to 30% non-fibrous materials visually indicated by its greenish-beige. The Water retting is done for a period of 5 days reducing non-fibrous content in a PALF by about 25% and the resultant fiber is found to have a linear density of 2.48tex and an average linear density of about 3.75tex after microbiological retting. The fineness depends on variety of pineapple and its prosperity of the leave. The retted fibers, being aggregates of elongated cells are still cemented together by non-cellulosic materials, composed mostly of hemicelluloses, lignin and other constituents, not of pectin nature (majority of the pectin's are removed during retting processes). Further, removal of these non-cellulosic materials by chemical degumming could be expected to yield more opened fibers of much lower density and increased fiber tenacity.

Chemical degumming is usually accompanied by subjecting PALF leaves to a solution of urea. The materials liquor ratio is 1:20 (prepare 20ml of urea solution in 1gr of Pineapple leaf) and its prepared in a tank contains 10 liters of solution. The pineapple leafs are added in to the water tank contains urea. The retting process is fast in compared with water and taking about 10-15 days for damaging the gummy parts of leafs but it needs continuous checking.

3.2.2. Fourier Transform Infrared (FTIR)

Chemical functional groups of the fibres were determined by Fourier transform infrared spectroscopy (FTIR) using a Spectrum 1000 spectrophotometer (Perkin Elmer) with 2 cm⁻¹ resolution in the range of 500–4000 cm⁻¹. The samples were prepared using tongs and hand technique. FTIR spectra in Fig. 9 show the response of pineapple leaf fibers. The strong bands at 2923.2 and 2854.6 cm⁻¹ of pineapple leaf fiber are attributed to C-Hn asymmetric stretching present mainly in cellulose, which was the major component of the lignocellulosic fibers. The band near 1735 cm⁻¹ assigned to unconjugated stretching vibrations of the C=O of the carbonyl and acetyl groups in the xylan component of hemicellulose was clearly seen on the fibers. The bands at the region from 1000 to 1600 cm⁻¹ are assigned to the aromatic region related to the lignin: the bands at 1455 and 1220 cm⁻¹ are characteristic of C-H and C-O deformation, bending or stretching vibrations of many groups in lignin and other cellulosic parts of fiber.

Cellulose show bands characteristics at 1034.5 and 1156 cm⁻¹ attributed to anti-symmetric bridge C-OR-C stretching of cellulose and C-OR stretching or secondary alcohol (C-OH) ring breathing, respectively. The peak at 1320 cm⁻¹ and 1240.2 cm⁻¹ is attributed to the O-H of alcohol groups and C-O stretching of aryl groups respectively and the band at 1420-1430 cm⁻¹ is assigned to aromatic skeletal vibrations associated to C-H in plane deformation of cellulose.

FTIR of the lignocellulosic materials identifies the presence of components such as hemicellulose which giving them a hydrophilic character. This hydrophilic character could reduce the compatibility of the fibers with polymer matrices.

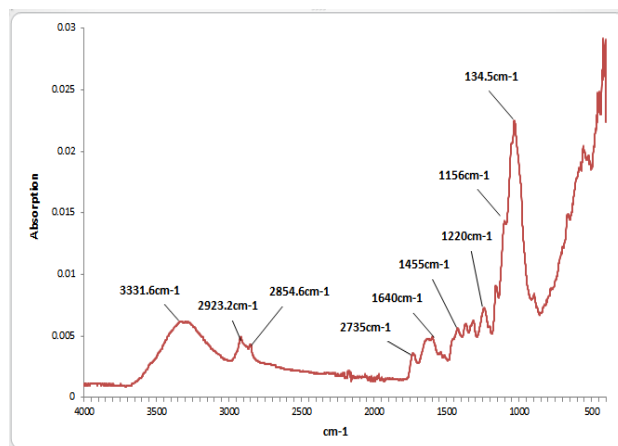


Figure 4: Result of pineapple fiber under FTIR

3.2.3. Differential scanning calorimetry (DSC)

DSC analyses were used to measure the thermal characteristics and investigation of the heating response of PALF. As the result shown in Figure 5 the DSC curves of pineapple leaf fiber does not appear obvious endothermic peak and exothermic peak during the heating process from room temperature to 210-220 °C, which indicates that the pineapple leaf fiber structure does not change significantly in this temperature range, mainly due to the heat changes caused by evaporation. The result obtained is the same with Dong et.al report (Dong et al., 2014).

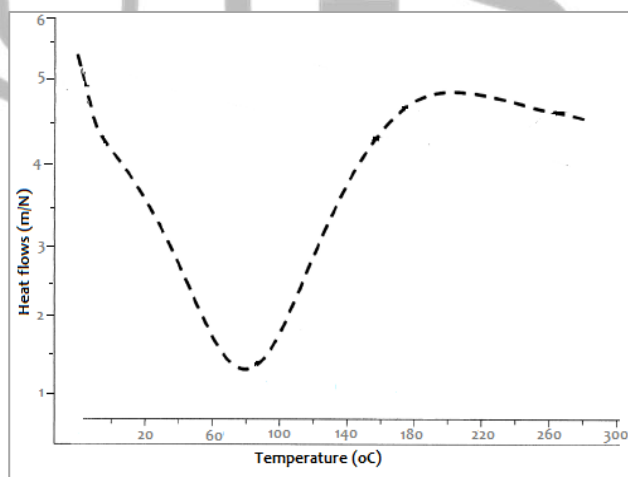
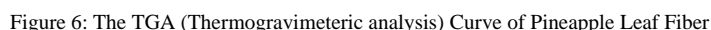


Figure 5: The DSC Curve of Pineapple Leaf Fiber

3.2.4. Thermogravimetric analysis (TGA)

It can be seen from the figure 6; the pyrolysis of the pineapple leaf fiber was carried out in three steps. The first weight loss step occurs below 100°C, maybe it is because of the adsorbed water or residual solvent; the second step is mainly due to loss of small molecules in the sample; the third step is the decomposition of specimen itself, which is the main weight loss stage. It can be seen that in the temperature range from room temperature to 100°C, the evaporation of moisture from the pineapple leaf fiber stopped at 95-100°C, the TG curve tends to balance. Then, from 236.8°C, the impurities or oil on the surface of fiber begins to evaporate or cracking. Pyrolysis of the fibers starts from 328.2°C, after that the fiber weight is of a sharp decline; when the temperature reaches 375.5°C, the change of weight losing is slow. Finally the whole fiber is heated and losses its weight. Also the result is reported by Dong et.al (Dong et al., 2014).



The fibers structure and coarseness under this microscope is shown in figure 7. From this fiber structure, pineapple leaf fibers have many matrix of fiber on the surface. Pineapple leaf gives high fiber content because of the arrangement of fiber. From figure 7, Leica microscopic longitudinal view of the fiber structure shows many bundle of packed fiber on the surface area of pineapple leaf fiber. The fiber surface contains waxes and other of entrusting substances like lignin, pectin and hemicelluloses. These substances form a thick layer to protect the substances of cellulose inside the matrix layer of fiber. Similar results are obtained by Rowell (Rowell et al., 2000).

3.2.6. Physical and Mechanical properties of Pineapple leaf fiber

Table 2: The physical properties of pineapple leaf fibers (PALF).

No.	Properties	Result obtained
1	Linear Density (g/cm ³)	1.35
2	Length (mm)	250-900
3	Average diameter (μm)	61.75
4	Moisture content (%)	12
5	Tensile strength (MPa)	228.3
6	Elongation at break (%)	2.45

The result in table 2 shows some physical and mechanical properties of pineapple leaf fiber. We can find that the average tensile strength of the pineapple leaf fiber is high and the linear density is low, so there is a high fiber spinnability and yarn quality can be obtained. Also the moisture content of pineapple leaf fiber is the same with moisture content of that of natural fibers. If PALF is in wet condition it loses its strength and elongation due to the penetration of water molecules in the multicellular lignocellulosic fiber.

4. Conclusion

In this study the extraction and characterization of PALF shows the percentage yield of pineapple leaf fiber is found between 2.5-3% since pineapple leaf has high amount of water content and gummy substances. The appropriate time for Pineapple leaf fiber extraction is generally done at the age of 1 to 1.5 years to obtain a strong fiber, soft and smooth, the selection should be done in pineapple leaves enough and protected from the sun. The optimised method of extracting pineapple leaf fiber is mechanical method and retting method which gives outstanding fibers. TGA result shows pyrolysis of the fibers starts from 328.2°C, and when the temperature reaches 375.5°C, the whole fiber is heated and losses its weight. Finally FTIR result obtained at maximum spectral band reveals the PALF fiber is a lignocellulosic fiber, due to these lignocellulosic, it is potentially able to act effectively as important fiber material in polymer composites production, besides avoiding the deposition of this waste in landfills.

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