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ISOLATION AND CHARACTERIZATION OF CELLULOSE MICROFIBER FROM PLANTAIN PEELS

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ABSTRACT

This research is on the isolation of micro-fibers from agricultural waste (plantain peels) to produce cellulose. Chemical analysis for determination of the moisture content, ash content were assessed according to AOAC. Lignin and cellulose were determined according to ASTM D 1106e56 for the plantain peel fibre and the isolated cellulose micro fibre. Results showed a reduction in moisture content from 11.05± 0.42% in the plantain peel fibre to 5.85-±0.14% in Cellulose Micro Fibre (CMF), 1.76±0.08 to 1.46±0.26 and 4.35± 0.84 to 1.23±2.19 for ash content and lignin respectively. A notable increase in cellulose was observed from 44.10±3.68 in the plantain peel to 88.9±2.19 in the CMF. Also, the FTIR spectra of the plantain peel fibre showed a strong characteristic carbonyl absorption peak at 1733.44 cm^{-1}, attributed to the acetyl and uronic ester groups of the hemicelluloses or the carboxylic groups of the lignin and hemicellulose while the FTIR spectra of CMF showed the removal of pectins, lignin and hemicelluloses resulting from the vanishing of characteristic band at 1733.44cm^{-1} (carboxylate groups) and 1252-30 cm^{-1} (methyl ester groups), 1513.89 and 1426.65 cm^{-1} (aromatic c = c stretch). This indicated that the chemical treatment removed large amount of pectin, lignin and hemicellulose. This study will contribute to the field of nanotechnology and also will aid the Nigerian waste-to-wealth initiative.

KEYWORDS: FTIR, Hemicellulose, Lignin, Microfibres, Plantain Peel

INTRODUCTION

The growing demand for environmental sustainability has encouraged research into biodegradable polymers, to minimize the environmental impact of conventional polymers. In this

context, plant fibres, which are among the existing natural fibres are attractive materials. They constitute rich sources of cellulose, the main component of plant cell walls [1]. Recently, cellulose has been used to produce rigid nanosized particles from natural agricultural wastes like garlic skin, pineapple peels, plantain peels, etc [2].

Plantain is one of the most extensively consumed fruits in the world and represents 40% of world trade in fruits. Plantain peels can cause an environmental problem such as a bad smell and become a source of human disease. One way of reducing the problem is to convert the plantain peels into a more valuable product, cellulose, which can be more extensively used in the food industry [3]. The peel represents around 35% of the fruit weight (wet basis) [4]. To use the plantain peel, some attempts have been realized such as adsorption of heavy metals, biomass production, as an antioxidant source, and in cellulose nanofibres [5-7]. Cellulose fibres (CFs) offer many advantages: they are mechanically strong, stiff, and highly crystalline, so they find wide applications in the biomedical, pharmaceutical, and especially the paper industries. They can also function as reinforcing agents in polymer matrixes. The addition of CFs to polymer matrixes culminates in composites with improved mechanical, thermal, and permeability properties, providing both economic and strategic benefits [3]. Although various methods yield cellulose micro particles, their isolation usually involves three steps:

- Pretreatment of raw materials
- Partial hydrolysis
- Mechanical disintegration.

The process that lead to micro particles dimensions affect the morphological characteristics of the micro particles, thereby influencing how they perform as reinforcement materials in composites [8]. In this context, the current challenge is to develop new potential processing techniques that will produce CMFs with controlled size and morphology. The end use of banana and plantain peels depends on its chemical composition, which is affected by the fruit's ripeness. Peel from unripe fruit presents (on a dry basis) 6-10% protein, 6-12% ash, 2-6% lipids, 11-39% starch and 33-43% total dietary fiber (TDF): from the TDF, around 5-13% is soluble dietary fiber (SDF) and 7-36% is insoluble dietary fiber (IDF) [9]. Pectin and gums (Xanthan, Arabic, guar etc) are present in the SDF, whereas cellulose, hemicelluloses, and lignin are included in the IDF [10].

The aim of this study is to produce strong, recyclable and biocompactible cellulose microfibers from agricultural waste (plantain peels).

The justification of this study is as a result of the dramatic increase in recent years of the use of natural fibers as reinforcement in biocomposite processing.

MATERIALS AND METHODS

REAGENTS: Sodium Hydroxide (NaOH), Sodium hypochlorite, Distilled water

EQUIPMENT: Stirrer, Oven, Filter, Sieve, Beakers, Thermometer, FTIR model (Buck 530 Quickscan FTIR, USA).

This study was carried out in the Unit Operation Laboratory, Department of Chemical Engineering Technology, Auchi Polytechnic, Auchi, Nigeria.

Sample preparation

The sample (plantain peel) was prepared by drying and grinding to increase surface area. The raw material was analyzed to determine moisture content, ash content, lignin and cellulose.

Isolation of CMF from plantain Peel

About 400g of plantain peel was cut into small pieces, dried and ground. It was then hydrolyzed in 10% NaOH solution. It was heated for 30min at a temperature of 96^{o}C. The pulp was then poured into 1000ml sodium hypochlorite solution and left to stand for 2hours.It was then filtered, washed with distilled water and dried at room temperature for 2 days.

CHARACTERIZATION OF ISOLATED CELLULOSE MICROFIBRE

The plantain peel fibre and CMF were chemically analyzed for moisture and ash content by using the AOAC (2000) [11] standard method. The cellulose of the fibres was measured by using kursecher and Coffee method as explained above. It was also characterized in terms of lignin content and cellulose yield using standard method (ASTM D 1106e56) [12]

DETERMINATION OF ASH CONTENT

This is the non-volatile matter of compound which remains after subjecting it to high decomposition temperature. This was carried out in order to determine the amount of carbon present.

The sample (2 g) of CMF of plantain peel was weighed into a porcelain crucible. This was transferred into the muffled furnace set at 555 ℃ and left for about 4 hours. About this time it had turned to white ash. The crucible and its content were cooled to about 100 ℃ in air, then room temperature in a desiccator and weighted AOAC (2000) [5].

The percentage ash calculated from the formula below:

$$\%\ Ash\ content = \left[\frac{weight\ of\ ash}{weight\ of\ sample}\right] \times \frac{100}{1}$$

DETERMINATION OF MOISTURE CONTENT

Moisture content is to determine the percentage of water molecules present in the product.

About 4 g of plantain peel CMF was weighed in a dish and transferred to the oven heating at 100-105 ℃ for 2 hours. Then allowed to cool for 30-40 minutes in a desiccator and weighed again. Determination was done in triplicate.

Moisture content is expressed as a percentage by mass of the product;

$$moisture\ content\ \%\ by\ mass = (M_0 - M_1)/M_0 \times 100$$

Where Mo is the mass in grams of the test portion before drying, $M1$ is the mass in gram of the test portion after drying.

LIGNIN DETERMINATION

Lignin was determined according to standard method (ASTM D 1106e56). [12]. A gram, oven – dried sample was placed in a 150ml of dissolved NaOH (70%) was added slowly while stirring and mixed well. The reaction proceeded for 2hrs with frequent stirring in a water bath maintained at 20 ℃

The contents of the flasks were filtered after washing with distilled water into a glass crucible of known weight. The residue was washed free of chemical with 500 ml of hot water and oven – dried at 103±℃. Crucible were then cooled in a desiccator and weighed until a constant weight was obtained.

The following formula was used to obtain the lignin content of the sample; Lignin content in sample (%) $= \left(\frac{w4-w3}{100w2}\right) \times (100 - w1)$

Where:

W1 = weight of extractive free sample content.

W2 = weight of oven – dried extractive free sample (grams).

W3 =weight of oven – dried crucible (grams).

W4 = weight of oven dried residue and crucible (grams)

DETERMINATION OF THE CELLULOSE YIELD OF THE CMF

After each treatment, the fibres were filtered, washed with distilled water, dried in the oven and weighed. The pulp was bleached by pouring it into sodium hypochlorite solution and left 2hr.After which it was filtered, washed with distilled water and dried at room temperature for 2 days. The dried bleached material was ground in the mill until fine particulate fibres were obtained, sieved in a mesh size number 40 to get fine particle size that can be used in vast application. Cellulose fibres yield is expressed as percentage by mass of the initial material used:

Cellulose fibres yield, % by mass $= \frac{w1}{w0} \times 100$

Where, W_0 is the mass in grams of the initial material.

W_1 is the mass in grams of the yield after drying.

RESULTS AND DISCUSSION

Table 1: Comparative chemical composition for plantain peel fibre and CMF.

Principle composition	Plantain peel fibre %	CMF %
Moisture	11.05$\pm$*0.42*	5.85$\pm$*0.14*
Ash	1.76$\pm$*0.08*	1.46$\pm$*0.26*
Lignin	4.35$\pm$*0.84*	1.23$\pm$*2.19*
Cellulose	44.10$\pm$*3.68*	88.9$\pm$*2.19*

Table: Result means$\pm$SD from triplicate determination

Table 1 show that the cellulose content of the fibres was initially 44.10% in plantain fibres and it has been increased to 88.99% after chemical treatment. As it was expected the CMF showed higher cellulose content compared to the plantain fibres, because chemical treatment has removed most of the hemicellulose and lignin contents from the plantain fibre. This implies that the crytallinity regions of cellulose in the fibres increased after chemical treatment.

FTIR CHARACTERISTICS OF PLANTAIN FIBRES AND CMF

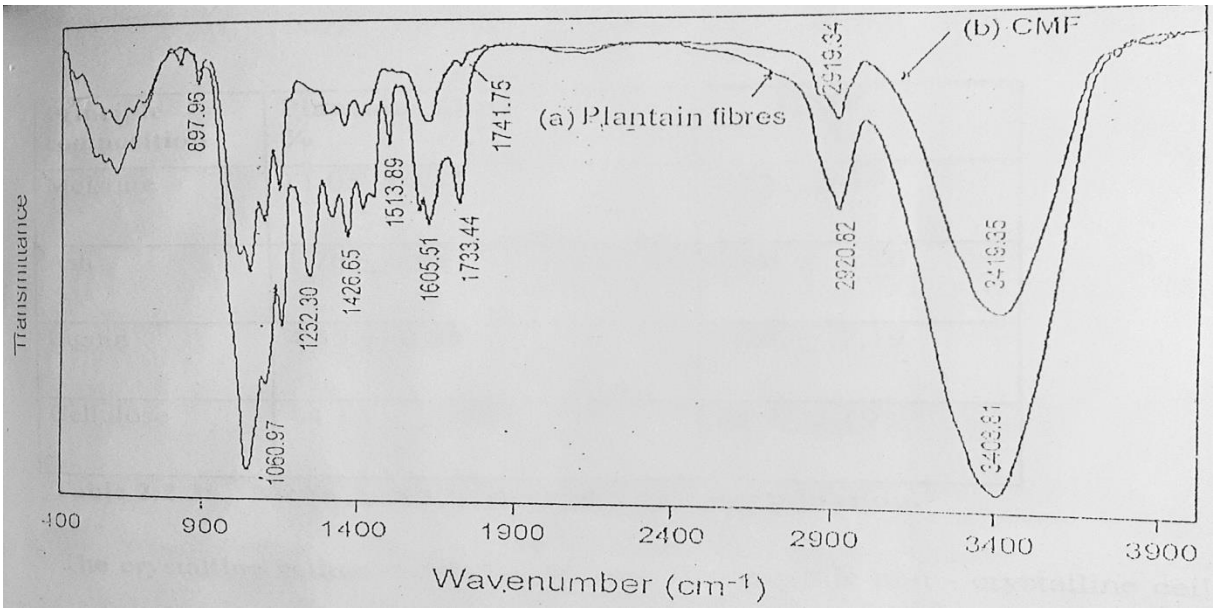

Figure 1: FTIR Spectra of (a) plantain fibres and (b) CMF

The chemical structure of plantain fibres and CMF components are shown in figure 1. The FTIR spectra of plantain fibres show a strong characteristic carbonyl absorption peak at 1733.44 cm^{-1}. This was attributed to the acetyl and uronicester groups of the hemicelluloses or the carboxylate group of the ferulic and p-coumeric acid of lignin and hemicellulose. The peaks at 1513.89 cm^{-1} and 1426.65 cm^{-1} in the plantain peel fibres show the aromatic C=C stretch of the aromatic rings of lignin. The FTIR spectra of CMF show the removal of pectins, lignin and hemicelluloses resulting from the vanishing characteristic band at 1733.44 cm^{-1} (carboxylate groups) and 1252.30 cm^{-1} (methyl ester groups), 1513.89 and 1426.65 cm^{-1} (aromatic C=C stretch), indicating that the chemical treatment had removed large amount of pectin, lignin and hemicelluloses.

The crystalline cellulose consists of long chains while non-crystalline cellulose has shorter chains. As chemical treatment has effects on the crystallinity of the cellulose fibres. It is considered desirable to retain pure cellulose, whose crystallinity forms a high packing density that would result in a stronger composite.

CONCLUSION

Many properties of the CMF showed that it could be of use in many applications. Chemical analyses as well as FTIR measurements of the fibres were performed. These revealed that some chemical components of the fibres, such as hemicelluloses, lignin and amorphous regions of cellulose were removed by the chemical treatment. The cellulose content of the CMF increased from 44.10±3.68% to 88.9±2.19%.

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