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# INVESTIGATION OF THE CONCENTRATION OF GLUCOSE PRODUCED FROM PLANTAIN AND BITTER YAM PEELS USING HYDROCHLORIC ACID MEDIUM

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*Abstract:* This study contributed to ongoing research on the conversion of waste biomasses into energy and other useful products, by investigating the viability of unripe plantain and bitter yam peels in the production of glucose by acid hydrolysis, using hydrochloric acid. The glucose yields from the waste biomasses were analyzed and compared. The chemical constituents in both untreated and treated waste biomasses were determined using standard characterization techniques; after which the functional groups present in the samples were analyzed using FTIR spectra techniques. The results obtained revealed that the cellulose content of the samples of unripe plantain is higher. It also showed that the cellulose content of the waste biomasses increased after treatment while other constituents decreased; meaning more glucose can be synthesized by acid hydrolysis of the samples. The effect of substrate and acid concentrations were equally studied; which revealed that generally, the yield of glucose increased as the concentrations of the acid and the substrate are increased. Thus, unripe plantain and bitter yam peels are good sources of glucose by acid hydrolysis; though unripe plantain peel yielded more glucose than bitter yam peel.

Keywords: Glucose, Unripe plantain peel, Bitter yam peel, Cellulose, Acid hydrolysis.

#### 1. INTRODUCTION

Most agricultural wastes consist of cellulose, hemicelluloses, lignin and other materials called extractive (Aberuagba, 1997). Cellulose, being the main constituent of lignocellulosic biomass, is a viable renewable source of energy and chemicals. The estimated global annual production of biomass is  $1 \times 10^{11}$  tons (US National Petroleum Council 2007; *Smeets et al.*, 2007). There are four phases of converting lignocellulosic biomass into ethanol. They include Pretreatment, Hydrolysis, Fermentation and Distillation for recovering ethanol (Cardona and Sánchez, 2007). Lignocellulosic biomass has a complex and recalcitrant structure; making dilute acid pretreatment a necessary step for carbohydrate hydrolysis because it is capable of high solubilization of hemicellulose (Palmqvist and Hahn-Hägerdal, 2000). Furthermore, it permits hemicellulose hydrolysis of pentoses and hexoses, removes some of the lignin, and makes the cellulose structure more accessible, so that a fraction can be converted to glucose. According to Wu et al. (2010), cellulose and lignocellulosic biomass can be converted to glucose by two methods: enzyme and acid hydrolysis. Studies have shown that although enzymatic hydrolysis is the best (Benkun et al., 2009); the process is not always feasible due to high cost of enzymes and slow rate of de-polymerization. Acid hydrolysis is promising especially if the by-products can be controlled because it is relatively inexpensive and fast. This study will focus on production of glucose from unripe plantain and bitter yam peels by acid hydrolysis using dilute hydrochloric acid as a case study.

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#### 2. MATERIALSAND METHODS

#### 2.1 Materials:

Unripe plantain and bitter yam were purchased from Ikpoba hill market in Benin City Edo state and Olo in Ezeagu L.G.A., Enugu state respectively. Ethanol (absolute), Sodium hydroxide (analytical grade), Acetic acid, Sulphuric acid, Hydrochloric acid, distilled water. The equipment used are: beaker, measuring cylinder, electric digital weighing balance, heating mantle, soxhlet extractor, desiccator, water bath, oven, volumetric cylinder, conical flask, hot plate and stirrer, Petri dish, UV spectrometer, test tube, Isothermal shaker, Centrifuge, FTIR, Gas chromatography.

#### 2.2 METHODS:

#### 2.2.1 Substrate Preparation:

Unripe plantain and bitter yam were peeled. The peels were washed with water to remove all dirt particles and then sun dried for 8days. The washed peels were ground into fine particles using a grinding machine; after which they were stored in a polyethylene bag for further use.

#### 2.2.2 Pretreatment of Biomass

Pretreatment of agricultural waste is necessary in order to remove lignin and hemicelluloses, reduce the crystallinity of cellulose, and increase the porosity of the lignocellulosic material. The alkaline treatment using sodium hydroxide was used, following the method described by Muthurecayudham and Virthagiri (2010).20g of each sample were dissolved in 6w/v% NaOH, the solution was placed inside the water bath at 60°C for 1hr and was washed with water till neutral <sub>P</sub>H was obtained and then dried in an oven at 105°C until constant weight was obtained.

#### 2.3 Separation of Cellulose from Unripe Plantain Biomass:

Layokun (1981) described the modified procedure for separation of cellulose from each sample of agricultural waste. The method involves addition of 20ml of diethyl ether to 10g of the powdered sample of unripe plantain peels in a 250 ml Erlenmeyer conical flask so as to remove the extractives. The resultant residue (free of extractives) was filtered and washed thoroughly with distilled water. 20 ml of 14M sulfuric acid was added to the washed residue in order to dissolve the cellulose and hemi-cellulose, leaving lignin as a hard precipitate, which was later filtered off. Cellulose was then obtained from the filtrate by adding 8M of sodium hydroxide solution to; thereby obtaining a residue that was predominantly cellulose; while hemi cellulose remained in solution. The solution was filtered and the resultant cellulose residue was then washed thoroughly with distilled water until a neutral pH was obtained. The cellulose residue was then dried at 80°C in an oven until a constant weight was obtained for subsequent hydrolysis.

#### 2.4 Acid Hydrolysis of Cellulose

Each sample of the cellulose (5g) obtained was added to 25ml of 0.5Mol/dm<sup>3</sup> hydrochloric acid in a 250ml conical flask, which served as a batch reactor and place in an isothermal shaker set at a temperature of 45°C with an agitation speed of 150rpm, and was allowed to operate for 4hrs and at intervals of 1hr. Samples were withdrawn to determine the glucose concentration. The experiment was repeated at other temperature of 60, 75, 90, 105°C and HCL and nitric acid concentration of 1.0, 1.5, 2.0 and 2.5mol/dm<sup>3</sup>, respectively.

#### **2.5 Determination of concentration of glucose**

The reducing sugar content (glucose) was determined by the DNS method with glucose as standard (Miller, 1959; Marsden et al., 1982). Absorbance was measured at 550nm. However, the DNS reagent was modified according to Mwesigye (1988). The dinitrosalicylic acid reagent for the determination of reducing sugar is composed of dinitrosalicylic acid, Rochelle salt, phenol and sodium hydroxide. 20g of potassium sodium tartarate (Rochelle salt) was dissolved in 20ml of distilled water in a beaker. 1g of sodium hydroxide was also dissolved separately in 20ml of water in a beaker. To the sodium hydroxide solution was added 1g of DNS and 0.3ml of phenol. The mixture was stirred using a magnetic stirrer until the DNS was completely dissolved. The mixture was added to the 20g of potassium tartarate and stirred properly till uniform mixture. The resultant solution was then made up to one litre distilled water. The mixture gave the stock of the modified DNS reagent containing 1% w/v DNS acid, 0.03% w/v phenol, 1% w/v sodium hydroxide and 20% w/v Rochelle salt (Mwesigye, 1988). The DNS reagent was then stored under refrigeration in an amber colored bottle.

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#### 3. RESULTS AND DISCUSSION

#### 3.1 Characterization of unripe plantain and bitter yam peels

Table1: Percentage composition of treated and untreated unripe plantain (UP) and bitter yam (BY) peels respectively

Properties	Untreated Unripe Plantain Peel		Untreated Bitter Yam Peel	·Treated Bitter Yam Peel
CELLULOSE	46.1	91.9	39.8	84.2
HEMICELLULOSE	45.7	40.5	22.5	21.9
LIGNIN	27.7	0.94	29.4	1.65
EXTRACTIVES	6.8	0.64	5.0	2.4
MOISTURE	2.5	2.8	1.1	2.1

Table 1 revealed the percentage composition of the chemical constituents present in the untreated and treated biomass samples. Generally, for both plantain and bitter yam peels, only the cellulose and moisture contents increased after treatment. This could be because the pretreatment process alters the chemical structure of the biomasses; thereby by reducing the amounts of hemi-cellulose, lignin and extractives (Sun and Cheng, 2002). Also, the percentage composition of cellulose is higher both in untreated and treated unripe plantain than in both samples of bitter yam peels. The percentage composition of cellulose increased from 46.1% to 91.9% and from 39.8% to 84.2% after treatment of unripe plantain and bitter yam peels respectively.

## **3.2** Effect of substrate dosage and acid concentration on glucose production from unripe plantain and bitter yam peels

Substrate dosage(g)	Glucose concentration from unripe plantain	Glucose concentration from bitter yam
0.5	0.5754	0.285
1.0	0.7541	0.545
1.5	0.8742	1.034
2.0	0.9367	1.031
2.5	1.1256	1.012

As the concentration of the substrate increased from 0.5 to 2.5g, the quantity of glucose produced from each substrate also increases. This could attributed to the fact that the higher the substrate concentration, the higher the cellulose content; hence the higher the amount of glucose that can be synthesized. However, at any given concentration of the substrate, the amount of glucose synthesized from unripe plantain substrate is higher than that obtained from bitter yam peel.

Acid Concentration(M)	Glucose concentration from unripe plantain	Glucose concentration from bitter yam
0.5	0.384	0.296
1.0	0.575	0.454
1.5	0.854	0.712
2.0	0.937	0.810
2.5	0.968	0.92

The results revealed that the yield of glucose from each biomass increased with increase in the concentration of the acid during acid hydrolysis. This result was also in agreement with Talebnia et al. (2007). The increase in glucose concentration could mean that the effectiveness of the dilute acid in removing lignin and splitting down cellulose bonds, at any given temperature and pressure, is higher when the concentration of the acid is increased to right amount.

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#### 3.3 Fourier Transform Infra-Red Spectroscopy (FTIR)

#### 3.3.1 FTIR spectra of untreated plantain peel

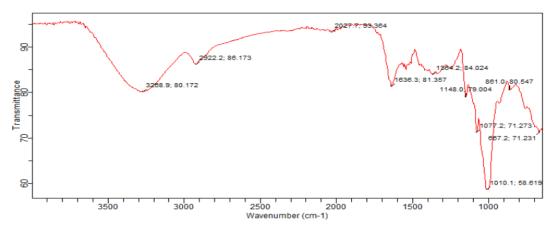


Fig 1: FTIR spectral analysis of developed sample of untreated plantain peel.

#### 3.3.2 FTIR spectra of untreated bitter yam peel

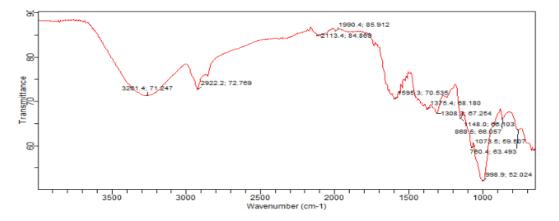


Fig 2: FTIR spectral analysis of developed sample of untreated bitter yam peel.

#### 3.3.3 FTIR spectra of treated plantain peel

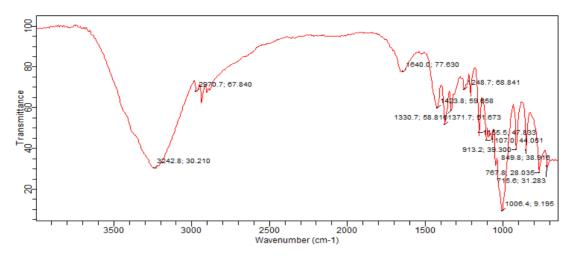


Fig 3: FTIR spectral analysis of developed sample of Treated plantain peel

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#### 3.3.4 FTIR spectra of treated bitter yam peel

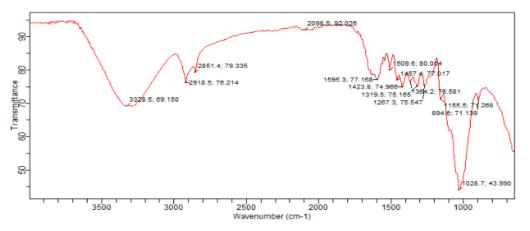


Fig 4: FTIR spectral analysis of developed sample of Treated bitter yam peel

The FTIR spectra shown in Figures 1 to 4 indicated the presence of carboxyl compounds which is one of the strongest IR absorption and is very useful in structure determination of hydroxyl groups. FTIR spectra of developed untreated and treated sample wave number increased from 667.2-3268.9 (cm<sup>-1</sup>) for untreated plantain peel, 715.6- 3242.8 for treated plantain peel, 780.4- 3281.4 for untreated bitter yam and 894.8- 3328.5 for treated bitter yam. The peak around 667.2cm<sup>-1</sup> for untreated plantain peel (C-Br stretch) shifted to higher frequency 3268.9cm<sup>-1</sup> (N-H stretch), while the peak around 780.4cm<sup>-1</sup> for untreated bitter peel (C-CL stretch) shifted to higher frequency of 3281.4cm<sup>-1</sup> (-C=C-H: C-H stretch). In a similar way, the peak around 715.6cm<sup>-1</sup> for treated plantain peel (C-H rock) shifted to higher frequency of 3242.8cm<sup>-1</sup> (N-H stretch) while the peak for treated bitter yam peel shifted from 894.8cm<sup>-1</sup> (C-H "oop", N-H wag) to higher frequency of 3328.5cm<sup>-1</sup> (-C=C-H: C-H stretch). The shifts in peaks and intensities of the functional groups can be attributed to reduction in the amount of lignin and hemicelluloses and increase in the porosity of the lignocellulosic material after treatment.

#### 4. CONCLUSIONS

The results revealed that the chemical composition of both unripe plantain and bitter yam peels make them good sources of glucose. The quantity of glucose that can be synthesized from the biomass raw materials can be greatly increased through chemical pre-treatment, as indicated by the significant rise in the cellulose content and reduction of other constituents after treatment. The results also indicated that glucose concentration is affected by the concentration of the substrates and dilute hydrochloric acid. Generally, the concentration of glucose increases with the increase in both the acid and substrate concentrations. This means that acid hydrolysis is effective at the required concentration of the acid because the cellulose bond is easily broken, while the amount of other constituents is significantly reduced. The functional groups revealed by FTIR analysis of the hydrolysed sample indicated the presence of glucose. Finally, all the analyses carried out revealed that more glucose was produced from unripe plantain peel. This is easily understood from the fact unripe plantain peel has more cellulose content both before and after treatment than bitter yam peel samples. Thus unripe plantain peel is better source of glucose than bitter yam peel.

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