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Evaluation of Biopolyethylene from Corn Cob

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ABSTRACT

In this study, biopolyethylene was generated from corn cobs obtained locally. The cobs were cleaned, then dried and ground to a particle size of 0.35 microns. It was treated to extract cellulose acetate, which was converted into a biopolymer by adding a plasticizer (polyethylene glycol - PEG 600) to a chitosan solution and mixing for 1 hour to create a polyethylene blend. Characterization of the biopolyethylene was conducted using Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), and Fourier Transform Infrared (FTIR) Spectrophotometer analyses. SEM results indicated an irregular structure with ridges and cracks, with pore sizes measuring 8.65 m. The TGA analysis revealed a gradual decomposition of biopolyethylene, whereas FTIR results indicate the existence of a functional group (-$CONH_2$) associated with the stretching vibrations of the amide group. The observations in the spectra suggest beneficial interactive effects during the blending procedure. Distinct peaks were identified in the untreated corn cob, with significant peaks observed at 3200.71 cm-1, corresponding to the –OH stretching vibration of hydroxyl groups and a stretching vibration related to C–H vibrations of alkanes. The hydrolyzed corn cob exhibited no new peaks. This research demonstrated that inexpensive biopolyethylene can be produced from polymer-based materials obtained from agricultural waste.

Keywords: Biopolyethylene, characterization, corn cobs, plastics

INTRODUCTION

Researchers have found over the past several decades that it is possible to substitute petrochemical-based plastics with biologically derived polymers that possess similar characteristics to traditional plastics and can naturally decompose after disposal [1]. Cellulose acetate is an example of such biologically derived polymers. Its biodegradability and biocompatibility, along with physical properties that resemble those of polypropylene, make it a viable alternative in the packaging and biomedical sectors. However, there are some disadvantages to the commercial production of cellulose acetate, such as its high manufacturing

costs and inferior mechanical properties, specifically, cellulose acetate tends to be brittle and has limited thermal stability, which restricts its competitiveness against petroleum-based plastics. As a result, researchers have been focused on enhancing the mechanical properties of cellulose acetate to make it more competitive with petroleum-based plastics. A significant challenge in the fermentation process for producing cellulose acetate is the high expense of raw materials and ineffective product recovery [2]. Corn cob constitutes a substantial portion of our agricultural waste, particularly in the northern regions of Nigeria, where maize is primarily cultivated through subsistence farming. This waste has limited applications, primarily being used as a heat source, due to its lack of nutrients and other beneficial vitamins. Nevertheless, studies have shown that corn cob contains approximately 45% cellulose. Consequently, utilizing corn cob as a source of refined cellulose presents a promising opportunity [3].

Petrochemical-derived polymers do not biodegrade and are often discarded improperly. The manufacturing of plastics from petrochemical sources accounts for 12% of the global demand for petroleum. The exhaustion of non-renewable resources, such as petroleum, along with greenhouse gas emissions and a lack of effective disposal technologies for non-biodegradable plastics, contributes to a severe environmental crisis, including rising global temperatures and the destruction of terrestrial and aquatic ecosystems [4]. The rapid growth of material science industries has resulted in various plastic products with outstanding durability and mechanical strength at a very low cost. Generally, the plastics utilized are of the single-use variety, particularly in food packaging and medical supplies. From 1950 to 2019, the synthetic polymer industry experienced an annual growth rate of 9%, and the market is projected to expand at a compound annual growth rate of 3.4% from 2020 to 2028, with the largest share located in developing Asian nations [5]. It is difficult for mankind to give up plastic products because of their versatility. A corn cob is an agricultural residue (waste) that is generated from corn (maize) and remains part of the ear on which the corn kernels grow.

Polyethylene has been a vital part of our life. However, disposal of these non-degradable petroleum-derived polyethylene threaten the ecosystem [4]. Also, the cost of obtaining this synthesizing petroleum-based polyethylene is on the increase; hence, extensive research has been conducted to find the best substitute to solve this problem. Prior to this research work, much interest has been gained in developing biopolyethylene from agricultural waste such as corn cob, sugar cane bagasse and bamboo calms [5]. Finding ways to convert agricultural waste, such as corncob, into profitable engineering uses have become essential. In 2011, global maize production reached 885 million metric tons, resulting in approximately 142 million metric tons of corncob waste. Remarkably, this figure grew by 116% from 2011 to

2022, with maize production hitting 2 billion metric tons and corncob waste rising to 306 million metric tons. It is estimated that 16% of the annual maize production worldwide leads to corncob waste.

Corncob, a biomass by-product from maize processing, holds significant scientific promise due to its large availability in both tropical and temperate regions. Composed of cellulose, hemicellulose, and lignin, corncobs are categorized as lignocellulosic materials. They are applicable in the cultivation of edible mushrooms, the generation of electricity, and various bio-synthesis processes that require intricate machinery and skilled personnel. The reported average contents of cellulose, hemicellulose, and lignin in corncob were 38.8%, 44.4%, and 11.9%, respectively. Lignin is recognized as the most prevalent aromatic polymer found in nature [6].

Following comprehensive research, a novel approach has been identified to reduce production costs by using corn cob waste as a raw material instead of petroleum-based resources. The maize cob served as the input for this process, leading to the creation of polyethylene. The aim of this research is to produce bio polyethylene from cellulose acetate derived from corn cobs.

MATERIALS AND METHODS

Sample Collection and Preparation

Corn cob (Figure 1) was collected from a local source, Uchi Market, Auchi Edo State. It was washed with water at room temperature for three times in order to remove impurities (dust and aerosol materials) (Plate 1). Thereafter, it was sun-dried for five days at a temperature of about 30°C (+/or -2°C). After wards, the size of the corn cob was manually reduced to about 1cm so as to enable easy mechanical crushing (Plate 2). It was further crushed in order to reduce the particle size to about 0.35 μm (Plate 3).

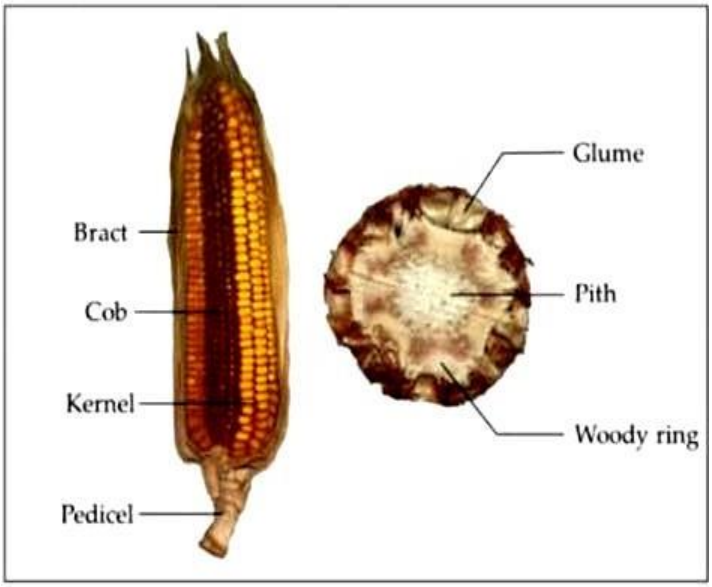

Figure 1: Layered structure of corn cob

Plate 1: Before Manual crushing 50mm size

Plate 2: After Manual crushing size 0.35mm

Plate 3: Particle size 0.063 mm

Pre-treatment and Extraction of cellulose acetate from Corn cob

The corn cob underwent treatment to isolate the lignin and hemicellulose components from the cellulose acetate required for production. The process of extracting cellulose acetate from corn cob includes a bleaching step and acid-base hydrolysis. Solutions of equal volumes (w/v) of 5% (6 g) sodium hydroxide and 5% sodium hypochlorite were prepared by diluting them in 100 mL and 120 mL of water, respectively. Subsequently, 50 g of crushed corn cob was bleached using 50 mL of the sodium hypochlorite solution and 50 mL of the sodium hydroxide solution, maintaining a powder to liquid ratio of 1:2 (g/ml) [7]. Thereafter, the residue recovered was dried in a laboratory DHG-9023A oven at minimum temperature of 110°C for 12 hours. After it was dried, the bleached corn cob was further pulverized and sieved in order to get a finer particle of less 0.00063 μm.

Acid hydrolysis

A sulphuric acid solution with molarity of 18.4 mol/dm^3 and molar mass of 107 g/mol was dissolved into 1000 mL of distilled water. Bleached corn cob (16 g) was dissolved in 150 mL sulphuric acid solution. The mixture was stirred properly for 1 h to bring about the acetylation process. Then, it was filtered and washed severally with water until the pH of the fibers was above 6.51. The hydrolyzed corn cob was subsequently dried in an oven for 12 hours. This step produced the primary cellulose acetate for the production of biopolyethylene [8].

Biopolyethylene production

The concentrated acetic acid of 11.5 mL whose molarity is 17.4 mol/dm^3 was dissolved in 1000 mL of distilled water and 20 mL of the solution was used to dissolve 0.5 g of Chitosan which acts as a filler. An acetone of 40 mL was used as a solvent to dissolve 0.5 g of the cellulose

acetate (hydrolyzed corn cob) and it was stirred for 30 minutes. Also 30 mL of polyethylene glycol (PEG 600) was added as plasticizer to the Chitosan solution and it was stirred until a viscous liquid was formed. Thereafter, the Chitosan and glycerol mixture was added to the cellulose acetate mixture, and stirred for 1 hour to form a homogeneous blend (biopolyethylene). It was casted on a glass petri dish to form (mould) and dried for 4 days [8].

Characterization on untreated and hydrolyzed corn cob

Proximate analysis

The moisture content of the samples was assessed using the oven drying method. Ash content refers to the mass of the residue left after combusting a measured amount of the sample in an open crucible (i.e., in an air atmosphere) at 750 °C in a muffle furnace until a stable weight is reached. Crude fat was measured with a Soxhlet apparatus. The determination of crude protein was conducted following the micro Kjeldahl method as outlined by the Association of Official Analytical Chemists in 2000. Crude fiber was assessed by utilizing the fat-free extract obtained following the Ether Extract analysis and weighing a portion of it. The carbohydrate content percentage of the sample was calculated by adding the percentages of moisture, ash, crude protein, and fat (ether extract) and then subtracting this total from 100%. The analysis of a waste component typically involves the determination of the percentage of C (Carbon), H (Hydrogen), O (Oxygen), N (Nitrogen), S (Sulphur) [9].

Fourier Transform Infrared Spectrophotometer (FTIR)

The FTIR analysis was performed to identify alterations in the functional groups of both the raw and carbonized samples. Tablets were prepared by blending each sample with potassium bromide in a ratio of 1:100 prior to analysis. The spectra were collected over a frequency range of 4000 cm-1 to 500 cm-1 using an FT-IR spectrometer (Varian 660 MidIR Dual MCT/DTGS Bundle with ATR) at a detection resolution of 4 cm-1 and composed of 200 scans per sample. The refractogram derived from FT-IR spectroscopy, relating wave number to absorption, is presented in the table below. IR solution software was utilized to obtain the spectrum**.**

Scanning Electron Microscopy (SEM/EDX) Analysis

Scanning electron microscopy (SEM, Hitachi SU 3500 scanning microscope, Tokyo, Japan) was employed to determine the shape of the produce samples (untreated corn cob, hydrolyzed corn cob and produced biopolyethylene). Particles were coated with gold un-der vacuum before SEM.

Thermogravimetric Analysis (DTA/TGA)

The thermogravimetric analysis used in this laboratory is a Q50 TGA from Shimadzu

instruments (Shimadzu TGA - Q50 thermobalance) thermal analyzer. The temperature control system includes a circular environment chamber, a sample pan furnace, and a purge gas supply system.

RESULTS AND DISCUSSION

Proximate analysis reveals the makeup of biomass in terms of moisture content, volatile substances, ash content, fixed carbon, and other components. The classification of biomass based on its storage capacity and energy content is determined through this proximate analysis. Ultimate analysis focuses on the elemental composition, including carbon, hydrogen, nitrogen, sulfur, and oxygen. The objective of this study is to evaluate the impact of proximate and ultimate analyses on the physicochemical characteristics of corn cob during hydrolysis [10]. Tables 1 and 2 present the proximate and ultimate analyses of corn cob, respectively.

Table 1: Proximate analysis of untreated and hydrolyzed corn cob

Parameters	Untreated Corn (%)	Hydrolyzed Corn (%)
Moisture content	8.19	6.79
Ash content	0.67	0.62
Crude fats	0.4	0.28
Crude protein	5.03	7.89
Crude fiber	0.31	0.21
Carbohydrate	85.71	84.38

Table 2: Ultimate analysis of untreated and hydrolyzed corn cob

Parameters	Untreated Corn (%)	Hydrolyzed Corn (%)
C	49.02	40.44
H	4.47	0.69
N	0.6	0.7
S	0.14	0.2
O	45.76	57.87

The values obtained for moisture content, carbon content, organic, etc for untreated and hydrolyzed corn cob samples are shown in Table 1. Table 2 showed the elemental compositions of the untreated and hydrolyzed corn cob. It could be seen from Tables that there were changes in the values obtained during proximate and ultimate analyses for untreated and hydrolyzed corn cob, a confirmation that the corn cob hydrolyzed.

FTIR characterization

The FTIR spectra gave insight into the surface functional chemistry and structure of untreated corn cob, hydrolyzed corn cob and biopolyethylene (a blend of cellulose acetate from corn cob, polyethylene glycol and chitosan) over frequency range of 500 to 4000 cm^{-1}. The FTIR spectra of untreated corn cob,

hydrolyzed corn cob and bioplastic (biopolyethylene) are shown in Figs. 2-4, respectively.

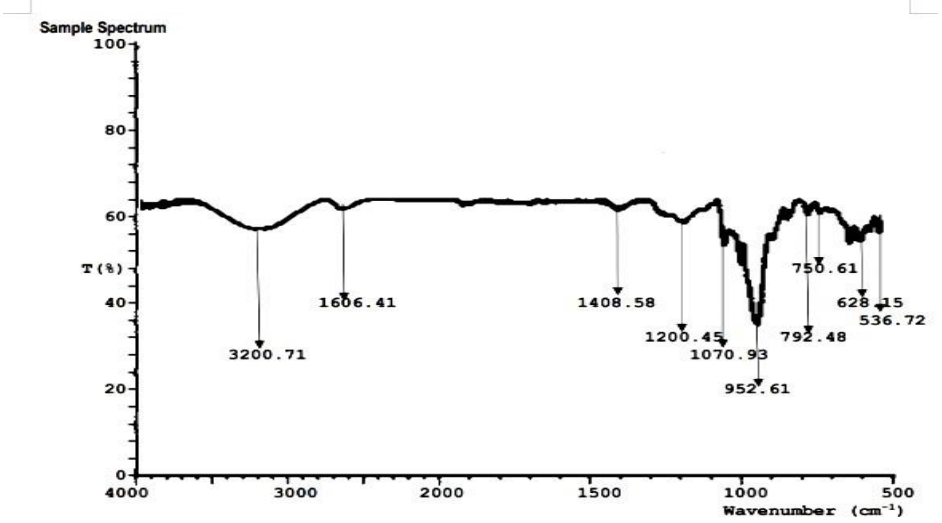

Fig. 2: FTIR monogram of untreated corn cob

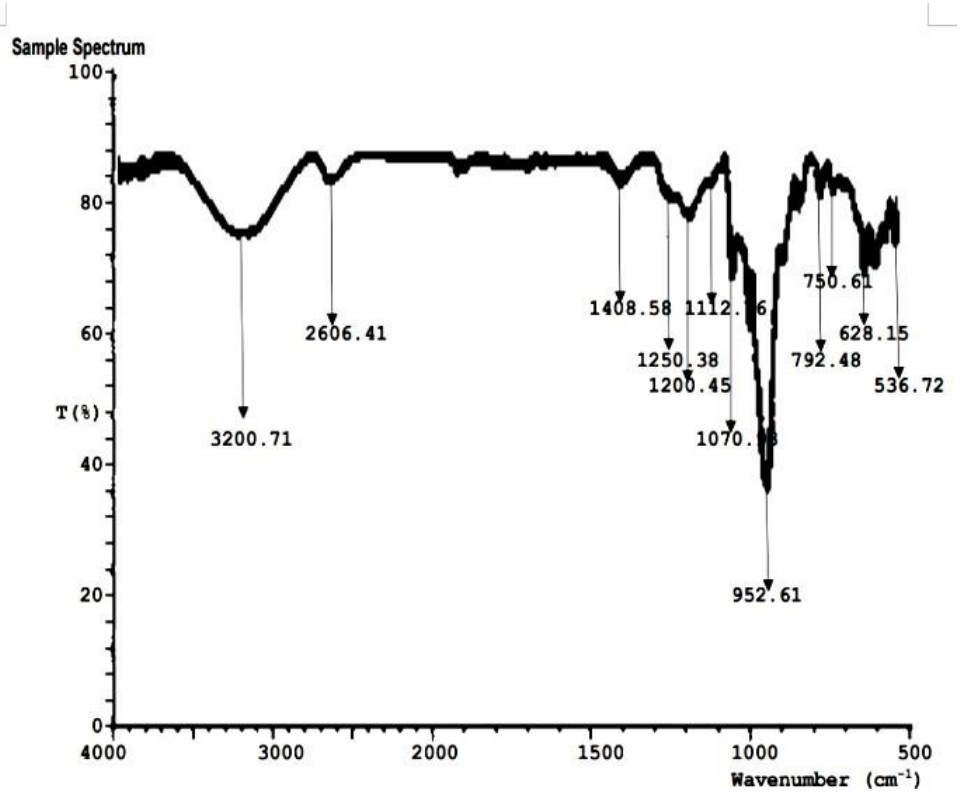

Fig. 3: FTIR monogram of Hydrolyzed corn cob

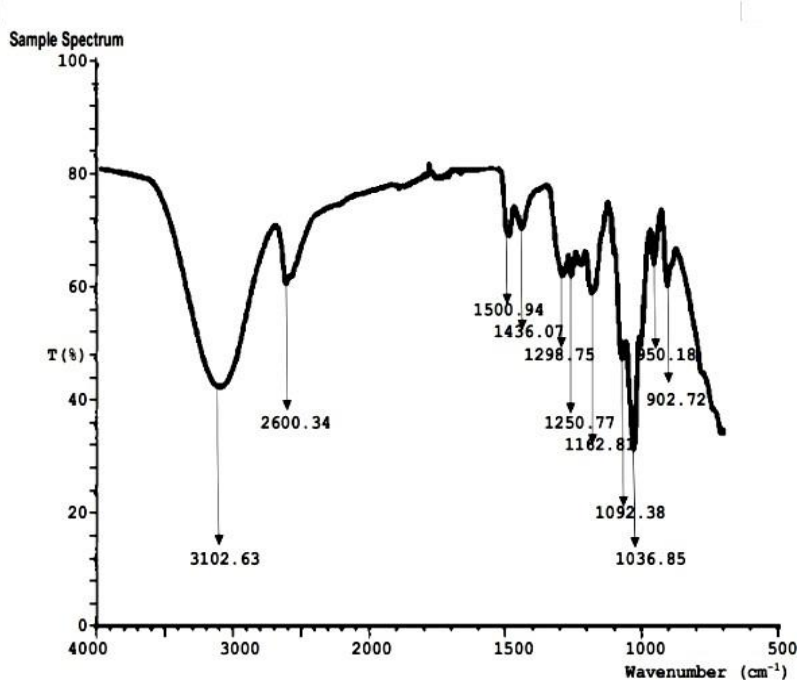

Fig. 4: FTIR monogram of biopolyethylene (a blend cellulose acetate from corn cob, PEG and chitosan)

The FTIR spectra of untreated corn cob, hydrolyzed corn cob and biopolyethylene are shown in Fig. 2, Fig.3 and Fig. 4, respectively which indicate the presence of different types of functional groups [11]. However, certain discernable peaks were observed in the untreated corn cob image (Fig. 2), with the prevalent peaks found to be specifically at 3200.71 cm-1 which corresponds to the –OH stretching vibration of hydroxyl group; 2606.41 cm^{-1} stretching vibrations attributed to C–H vibration of alkanes; 1408.58 cm^{-1} 1408.58 wave number dedicated to C-OH bending vibration of starch molecules of Hydroxyl group; 1070.93 cm^{-1} assigned to C–O stretching and C–O deformation of alcohol; 792.48 cm^{-1} which correspond to C-O-C bending vibration of Ether; 750.61 cm^{-1} bandwidth assigned to C–H stretching vibrations of aromatic carbon–hydrogen bond of hemicelluloses in Alkenes and 536.72 cm^{-1} C–H bending vibrations indicating the presence of alkyl groups. A close comparison of the FTIR results of the untreated corn cob (Fig. 2) and the hydrolyzed corn cob (Fig. 3) indicates that no new peaks were detected. However, there is increase in the intensity of transmittance [11].

However, a close comparison of the FTIR results of the untreated corn cob (Fig.2) and biopolyethylene (Fig. 4) indicates a shift in peaks and appearance of new peaks. Notably, the 3200.71 to 3102.63 cm^{-1} band; 2606.41 to 2600.34 cm^{-1}; appearance of 1500.94 cm^{-1} assigned to -$CONH_2$ stretching vibrations of amide group, etc. These observable changes in the spectra indicate a favorable interactive effect during the blending process.

SEM characterization of biopolyethylene

SEM images (Fig. 5) was used to depict the morphology and structure of the biopolyethylene.

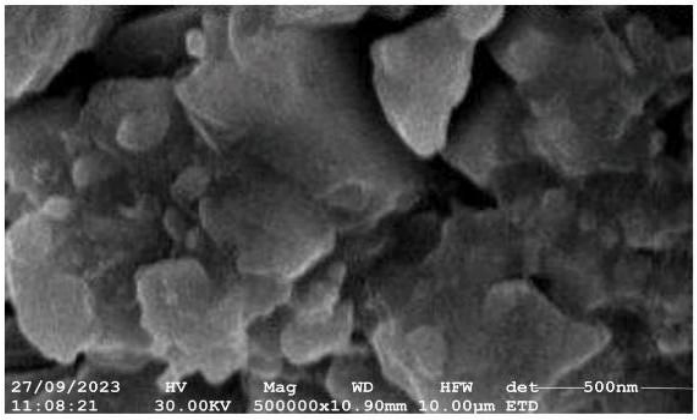

Fig. 5: SEM microgram of the Biopolyethylene

It was observed that the SEM image (Fig. 5) was characterized by irregular structure,

with ridges, grooves and visible cracks with Particle size of 8.65 μm.

The TGA result for the bioplastic, which is a characteristic of its thermal analysis at regulated temperature, is shown in Fig. 6.

TGA/DTA characterization of Biopolyethylene

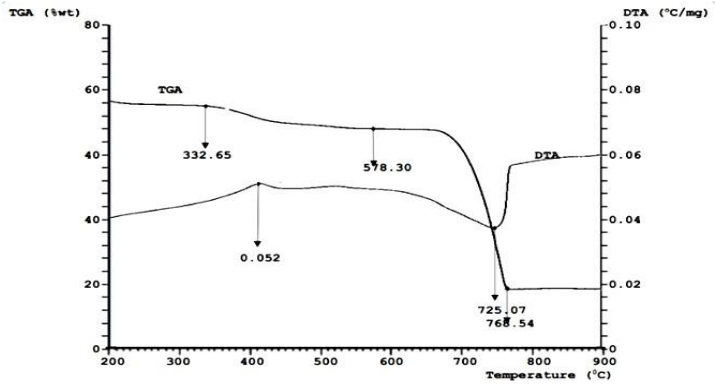

Fig. 6: TGA/DTA thermogram of biopolyethylene

From Fig. 6, the TGA thermogram of biopolyethylene showed a slow decomposition (weight loss) of biopolyethylene in four steps from 200 to 900 ^{0}C owing to the homogeneity of the blend. The first weight loss within temperature range of 200-250 ^{0}C (56.1%) could be due to the eradication of water vapour and volatile matter. The second weight loss occurred within temperature range of 300 – 450 ^{0}C (54.48%) which could be attributed to the initial degradation of the bioethylene. Third loss in weight occurred within the range of 500 – 700 ^{0}C (50.06%) when char was formed and 750 – 800 0C (29.54%) when ashes were formed [12].

CONCLUSION

Biopolyethylene can be made from polymeristic materials derived from biological sources, one of which is agricultural waste (corn cob) used for this work. The biopolyethylene was characterized by various techniques which analyzed the potential of corn cob cellulose acetate on biopolyethylene production. The variation in values obtained from proximate and ultimate analysis results proves that the corn cob was hydrolyzed and that corn cob contains an appreciable amount of carbohydrate that could be converted into polymers by acid hydrolysis. From the ultimate analysis, the reactive component of corn cob was majorly oxygen which contains functional groups that may play important roles during polymerization. The study also established the thermal decomposition (DTA/TGA) of polyethylene to occurs at temperature of 200°C with complete decomposition at 900°C. Generally, few commercial applications exist for bioplastics; the biobased could replace many applications for petroleum-

derived plastics, however cost and performance remain problematic.

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