

## Comparative Study of Biochars from the Retort Co-Carbonization of Corn Cob and Polyethylene Wastes

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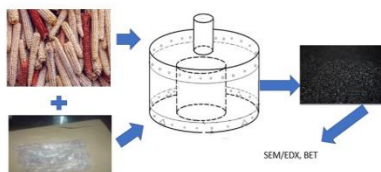
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### GRAPHICAL ABSTRACT



### ABSTRACT

Biochar is a pyrogenic, carbon-rich by-product of biomass valorization with multi-variant applications. The study focused on the retort carbonization of corn cob and co-carbonization of corn cob and polyethylene waste for comparative studies using a top-lit updraft conversion reactor. The properties of the biochar and hybrid were analyzed using Scanning Electron Microscopy - Energy Dispersive X-ray (SEM-EDX) and Brunauer-Emmet-Teller (BET). Results revealed the ordinary biochar had a homogeneous surface with repeating patterns forming a honeycomb-like structure with a 62% carbon composition while the hybrid biochar had a heterogeneous surface with a rough pattern and ridges with an 80% carbon composition. The heterogeneity and increase in carbon composition observed in the hybrid biochar were attributed to the presence of the polyethylene waste. Biochars with high carbon contents can find their applications in areas of carbon sequestrations. The biochar and hybrid biochar were found to be porous with specific surface areas of 392.5 m<sup>2</sup>/g and 889.9 m<sup>2</sup>/g respectively. The high specific surface area of the hybrid biochar produced highlights its potential as an adsorbent. The co-carbonization of corn cob and polyethylene waste has been established to be a means of conversion of both agricultural and plastic wastes into biochar with various potential applications.

**Keywords:** Co-carbonization, Corn cob, Polyethylene waste, Scanning Electron Microscopy, Biochar

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## 1. INTRODUCTION

Nigeria is considered the 7<sup>th</sup> most populous country in the world with an estimated population of 239 million people by 2025 [1]. By implication, food insecurity issues are met with reciprocating increase in agricultural activities to meet her rising population growth. One of the most cultivated food crops found in this part of West Africa is corn for both human and animal consumption. Food and Agriculture Organization of the United Nations reported increasing corn production in Nigeria between the year 2017-2020 [2]. A large proportion of this produced corn finds its use in ethanol and food processing plants, with emphasis placed on the grains rather than the cob. As a result, voluminous agricultural wastes generated from corn cultivation such as corn cobs are most times, left to rot on farm fields or burnt directly as a way of solid waste management. This practice is discouraged as more greenhouse gases are released into the atmosphere. Studies have shown various utilization of corn cobs such as biochar

production [3], pelletizing [4], carbon adsorbent [5], liquid smoke production [6], and soil remediation [7].

Recently, plastics have gained popularity for alternative uses such as packaging of edible products, water etc. Plastics like polyethylene, polystyrene and polypropylene find their applications in the food and beverage industries [8]. Plastics are long-chain organic polymers synthesized through chemical processes like polymerization and poly-condensation and are often preferred to other means of packaging due to their ease of production and versatility [9]. In Nigeria, one of the commonest plastic pollutants is polyethylene sachets (LDPE), which are used for packaging water for sale [10,11]. In a recent study which attempted to estimate packaged water consumption along with its associated generated plastic wastes in three West African countries (Ghana, Nigeria, and Liberia), Nigeria accounted for 63% (17,640 tonnes annually) of the overall total estimated plastic wastes annually [12]. Due to lack of effective waste management systems, LDPE sachets wastes have become a nuisance to the Nigerian environment because of their non-

biodegradability [10]. The need to find alternative means of reusing polyethylene wastes is sacrosanct to curb the lingering negative effects on the environment.

Thermal treatment of these wastes provides a sustainable means of solid waste management and production of other valuable products, with little environmental effects [13]. One of the products of the thermal treatment of biomass is biochar. Biochar is a pyrogenic black carbon solid, produced by heating biomass at high temperatures in a closed reactor with little or no oxygen [14]. Several works of literature have reported the areas in which biochar has found its applications; a source of heat and energy for various purposes [15], used to prevent CO<sub>2</sub> emissions during combustion of fossil fuels through co-firing [16], helps improve plant growth by providing elements such as calcium [17], provides a niche for the growth of essential soil microorganisms [18,19], increases the bioavailability of water [20], aids in promoting carbon sequestration [20], as a chelating agent [17], production of electrode materials used for super-capacitors [21].

Researchers have reported the production of biochar through several methods such as pyrolysis [22-24], carbonization [25-27], co-carbonization [22], co-pyrolysis [28-32], co-gasification [33-36]. Among the methods reported, carbonization is very efficient and optimized for biochar production [37, 38]. It is considered a slow pyrolysis process that converts biomass into biochar [14]. For carbonization to occur, the biomass is heated in a chamber with limited air supply at low temperatures of 300-400°C over a period of time and produces a higher yield of biochar when compared to other thermal treatment methods [20]. Besides the high yield of biochar, another merit of carbonization is found in its ease of implementation and requiring less technical skills [39]. Co-carbonization is achieved when two different materials are carbonized together to obtain a product with improved properties. Several pieces of research have been done on thermochemical conversion of two blends of materials, usually done to improve the qualities of the products. Recently, the preparation of porous carbon using co-carbonization of mesophase pitch and sawdust was investigated [22]. Co-pyrolysis of sawdust and LDPE to produce bio-oil was previously reported [31]. Another report also studied the thermal and co-pyrolysis of rubber seed cake and polystyrene using a semi-batch reactor [40]. Lang et al. [41] co-hydrothermally carbonized corn stalk and animal manure (swine) to produce hydro-char. The potential of Pb (II) removal from aqueous solution by biochar derived from corn stalk and polyethylene co-pyrolysis was also reported [29]. Retort co-carbonization process utilizes heat from the controlled combustion of some forest biomass to drive the co-carbonization process. The system consists of two symmetrical, and cylindrical reactors with one placed in the other. The larger one is the combustion chamber while the smaller is the carbonization reactor. Co-carbonization takes place in the carbonization chamber with heat required to drive it coming from the controlled combustion of

biomass (retort heating) which fills up the space between both reactors. One major advantage of the process is that it requires no electrical power, and it is easy to operate. Retort co-carbonization of LDPE has been investigated with oil palm [42], sugarcane bagasse [43], elephant grass [44] and almond leaves waste [45].

Despite the dearth of studies reported, no study has been reported on the retort co-carbonization of corn cob and LDPE to produce a useful product and a means of waste management. Therefore, the study is aimed at the production of biochar and hybrid from corn cob and co-carbonization of corn cob and LDPE and evaluate their characteristics for comparative studies and potential carbon related applications. The biochar and hybrid were characterized using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM/EDX) and Branauer-Emmet-Teller (BET) analyses to ascertain their properties for comparative studies and potential applications.

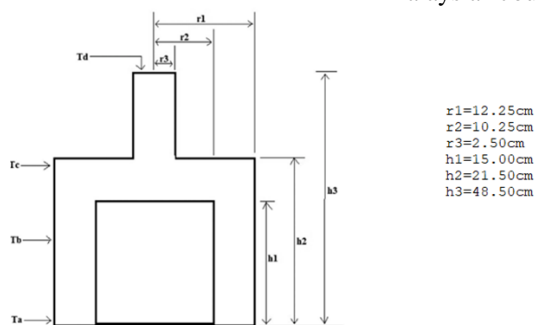
## 2. MATERIALS AND METHODS

### 2.1 Materials

The corn cobs were gathered after the shaft had been removed and the corn consumed. They were obtained from roadside sellers of roasted corn along the University of Ilorin Road, Tanke. The combustion fuels were Mimosa and neem stalk and stem. The corncob feed is readily available and easily found locally due to the absence of any form of competitive use. The polyethylene water sachets were handpicked within the vicinity of the university. They were easily sourced due to their large consumption level and absence of competitive alternative use.

### 2.2 Reactor description

A pre-constructed top-lit fixed-bed updraft biomass conversion reactor and the same methods previously reported in a similar study were used [43]. Both chambers were completely made up of stainless steel. T<sub>a</sub>, T<sub>b</sub>, T<sub>c</sub>, and T<sub>d</sub> are various points (as shown in Figure 1) at which the temperature was measured using a CASON CA380 infra-red Thermometer (Accuracy; ±0.1°C, Max; 380°C). The carbonization reactor consists of an outer and inner chamber, both cylindrical in shape. The outer chamber houses the combustion fuel, while the inner chamber holds the feed. The outer chamber consists of round and triangular holes at the bottom which allows the updraft of air through the heating gap. The inner chamber, which is the carbonization chamber consists of four small air-holes at the bottom to prevent pyrolysis of the feed. The reactor operates as a top-lit (the combustion fuel is ignited at the top) and the fuel burns gradually downwards till all the fuel is used up [17]. The heat from the combustion of the combustion fuel serves as a heat source for the carbonization of the feed. The outer chamber is fitted with a lid having a vertical exhaust pipe for removal of gases produced during combustion.



**Figure 1** 2D sketch of top-lit updraft fixed-bed conversion reactors with dimensions.

### 2.3 Batch Experiment Procedure

The corn cobs were dried in the open air to further reduce its moisture content. The LDPE wastes were also dried in open air to remove any available moisture at atmospheric temperature. Combustion fuels were cut into relatively small sizes to fit into the space between the combustion and carbonization chambers. The carbonization chamber of the reactor was loaded with a known weight of corn cob evenly spread within the chamber. The carbonization chamber was then placed into the combustion chamber with the gaps between loaded with as much combustion fuel as possible. The system was ignited at the top and allowed to burn in the open air for about two minutes to allow homogeneity of the combustion front and afterwards, the system was closed by the lid. The entire experiment was carried out in open-air with initial temperature (ambient) recorded before ignition. Temperature measurements ( $T_a$ ,  $T_b$ ,  $T_c$ , and  $T_d$ ) was taken just as the experiment started and at intervals of 10 minutes till the temperatures of the system was at equilibrium with the ambient temperature. The combustion fuel burns completely to form ash and the biochar in the carbonization chamber was recovered, weighed, and stored in airtight containers before characterization. The entire process was repeated in another batch for the co-carbonization of the corncob and LDPE waste in a ratio of 5:1. Experiments were conducted in duplicates. The biochar yield was computed using Eqn. 1 [17].

$$Yield_{Bio-char} = \frac{m_{Bio-char}}{m_{raw}} \times 100\% \quad (1)$$

### 2.4 Biochar characterization

The biochar and hybrid produced from both the carbonization of corncob and co-carbonization of corncob and polyethylene waste were characterized to ascertain some of their properties using Scanning Electron Microscopy-Energy Dispersive X-ray (SEM/EDX) and Branueur-Emmet-Teller (BET) analysis. The surface structure of the particles and elemental composition of the biochar produced from both experimental runs were studied using Scanning

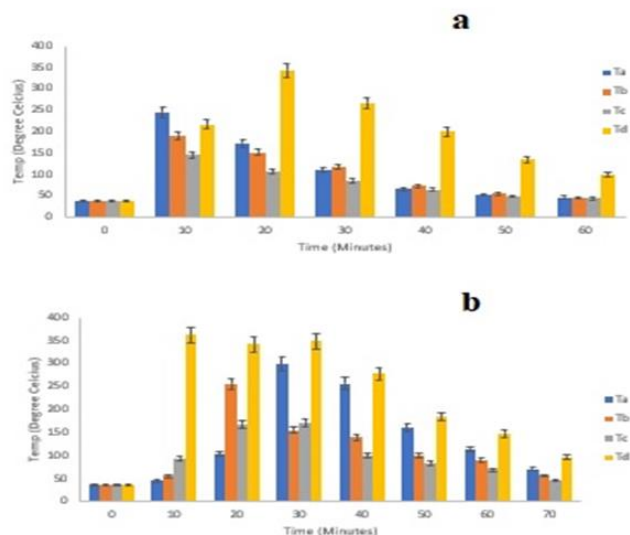
Electron Microscopy-Energy Dispersive X-ray (SEM Phenom ProX). The SEM analysis was done at a magnification of 500 to 1500 times with an acceleration voltage set at 15kV. Surface properties such as pore volume, surface area, and pore sizes were measured through the Branueur-Emmet-Teller (BET) analysis, using a NOVA Station BET analyzer. The Multipoint BET surface area and Dubinin-Radushkevich (DR) method for pore diameter and volume were used to determine the surface properties of the biochar by Nitrogen adsorption at a temperature of 77 K.

## 3. RESULTS AND DISCUSSION

### 3.1 Temperature profiles

The process time, peak temperatures, and product yields were used to evaluate the performance of the reactor. The carbonization and the co-carbonization process lasted for 60 mins and 70 mins respectively, at peak temperatures of 343°C and 362°C respectively. The differences in carbonization time are due to the self-regulating nature of the process. The profiles are shown in Figures 2a-b. The process was run until the temperatures ( $T_a$ ,  $T_b$ ,  $T_c$ , and  $T_d$ ) were at ambient conditions. Under theoretical conditions, the peak temperature at the top of the reactor ( $T_c$ ) should be observed first, this should then be followed by  $T_b$  then  $T_a$ , demonstrating the downward flow of the combustion zone from top to bottom. During the carbonization process,  $T_a$ ,  $T_b$ , and  $T_c$  peaked 10 mins after the commencement of the experiment with  $T_d$  peaking after 20 minutes. This can be attributed to the speed at which the combustion fuel (Neem and Mimosa tree branches) combusted within the combustion chamber. The co-carbonization process had a different temperature profile with  $T_d$  peaking 10 minutes after the experiment commenced.  $T_b$  peaked shortly after with  $T_a$  and  $T_c$  peaking simultaneously afterwards. The temperature profile features several peaks highlighting variations in the intensity of combustion as the combustion zone moves downward. This can be attributed to the variation in sizes of the combustion fuels, with larger portions having higher heating values and combustion period. During the carbonization and co-carbonization process,  $T_d$  had the highest peak from the point of measurement because it represents the temperature of the surface of the inner chamber in which an exothermic reaction occurs. The temperature ( $T_d$ ) peaked at 343°C and 362°C for the carbonization and co-carbonization processes respectively. The difference in the peak values of  $T_d$  in both processes could be attributed to the inherently, self-controlling nature of the retort-heating process. In a recent study which investigated the co-carbonization of sugarcane bagasse and LDPE [46], the peak temperature for the co-carbonization process was lower (250 °C) than that of the carbonization process (349 °C). Another work which also utilized the same method employed in the current study reported a lower peak temperature (362 °C) for the co-carbonization process when compared with the carbonization process (494 °C). A higher peak temperature

(382 °C) was observed for the co-carbonization of almond leaves and LDPE like the observation in the current study (47). All these observations are pointers to the aforementioned reason that the system tend to adjust itself to suit the environmental situation of its immediate surroundings. The updraft was by natural air convection hence this could be considered. The process lasted for 60 minutes and 70 minutes for the carbonization and co-carbonization respectively.



**Figure 1** Temperature profiles (a) carbonization process (b) co-carbonization process

### 3.2 Product yield

This section presents the computation of the product yields from both the carbonization and co-carbonization processes. For the carbonization process, 136.1 g of biomass was loaded into the reactor to produce 90.7 grams of biochar. The co-carbonization process had a biomass feed of 226.8 g and LPDE feed of 45.36 g (accounting for about 16.7% of the total feed for co-carbonization process) to produce 90.72 g of hybrid. Biochar and hybrid yields were computed as 66.6% and 33.3% (Table 1) for the carbonization and co-carbonization processes respectively. The remaining portion of the biomass was converted to syn-gas. At 66.6% and 33.3%, the yields can be regarded as excellent. Adeniyi, Ighalo, & Onifade [17] converted elephant grass using a top-lit fixed-bed updraft biomass carbonization reactor similar to the one used for this study to obtain a biochar yield of 14.29% (at 300°C and 120 mins). Mullen, Boateng [3] pyrolysed corn cobs at a temperature of 500°C in a fluidized bed reactor to obtain a biochar yield of 18.5%. Although, it is expected that the co-carbonization process should improve the yield of the hybrid as previously reported [46]. However, converse was the case in this study as the yield from the carbonization process resulted in a higher product yield than the co-carbonization process. The reason for this may not be far-fetched considering both peak temperatures and feed

residence times from both processes. As presented in Table 1, the co-carbonization process had a higher peak temperature (362 °C) than the carbonization process. At higher temperatures, the yield is expected to be lesser as devolatilization increases. Hence, there is an inverse correlation between temperature and biochar yield [48]. Furthermore, comparing the temperature profiles of both processes (Figure 2a-b), the carbonization process peaked 20 mins from the commencement of the experiment and the temperature dropped to below 250 °C after 10 mins from the peak. Thermal energy supplied during this period may not have been enough for complete thermal decomposition to occur, hence, a higher yield. The temperature profile for the co-carbonization process revealed a sustained period of about 30 mins exposure of the feed to temperature > 300 °C. The residence time at the elevated temperature exposed the feed to more thermal decomposition thereby resulting in the loss of more volatiles. The total residence time was eventually longer for the co-carbonization process (70 mins) than the carbonization process (60 mins). Cumulatively, the total carbonization time resulted in the loss of more volatiles from the hybrid than the biochar. By consequence, a lesser yield from the co-carbonization as compared to the carbonization process. A lesser product yield may have been obtained from the co-carbonization process, product quality was improved has elucidated by the EDS, and BET analyses.

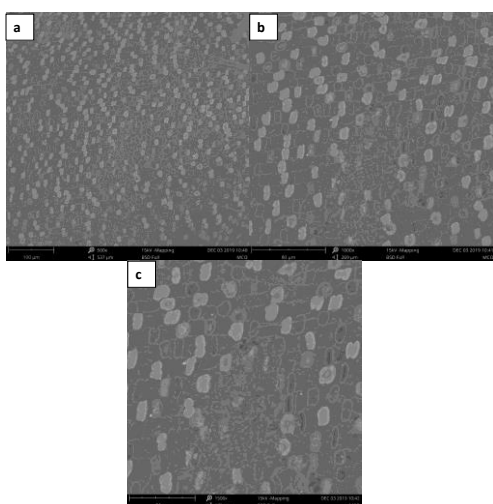
**Table 1** Summary of reactor performance

Index	Value	
	Carbonization	Co-carbonization
Process time	60 min	70 min
Peak temperature	343°C	362°C
Biochar yield	66.64%	33.33%

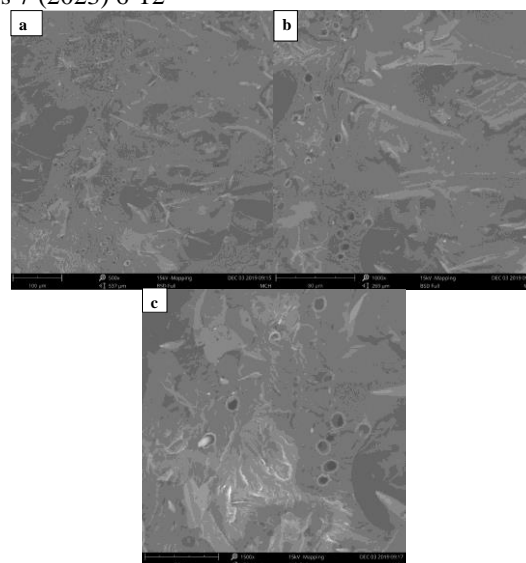
### 3.3 Biochar Morphology and Composition

The surface morphology of the particles and elemental composition of the biochar produced from both experimental runs were studied using a Phenom ProX SEM with its microscope set at an acceleration voltage of 15kV. The ordinary biochar was observed to have a homogenous surface with repeating patterns forming a honeycomb-like structure as depicted in Figure 3a. The particles were observed to be sphere-like in shape with what appears to be a smooth patch at higher magnification as shown in Figure 3c with the brighter zones representing elements with higher atomic number [49]. The homogenous surface of the ordinary biochar observed can be attributed to the presence of cellulose and hemicellulose which serve as binding agents in the corncob. The hybrid biochar had a heterogeneous surface compared to the ordinary biochar as depicted in Figure 4a. The honeycomb-like surface observed on the ordinary biochar was absent but instead had a rough surface with ridges as observed in Figures 4b-c. This can be attributed to the addition of the LDPE, which upon thermal

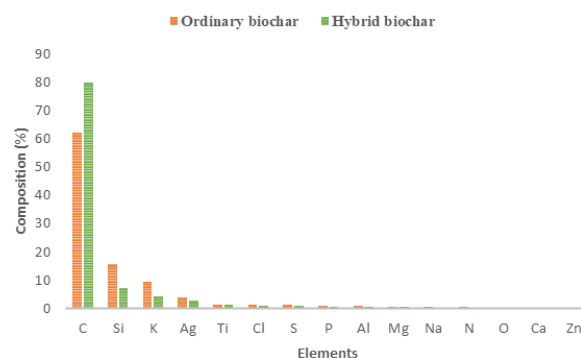
decomposition produces grease-like solid products and oily liquid products resulting in a form of a coating on the surface of the biochar [50]. This feedstock change resulted in the increase in the carbon content of the hybrid biochar compared to the ordinary biochar as depicted in Figure 5. The rougher and irregular surface of the hybrid biochar resulted in higher porosity and larger surface area compared to the ordinary biochar. The ordinary biochar produced had a lower carbon content (62.11%) when compared to the hybrid biochar (80.09%) (Figure 5). LDPE is a polymer of ethylene with a general formula  $(-CH_2-CH_2)_n$ , and consists of coordinated arrangements of C-H structures. At elevated temperatures, the C-H bonds are broken, leading to an increased carbon composition in the system, hence, higher carbon content observed in the hybrid when compared with the biochar. The available hydrogen atoms from the decomposition of LDPE may have gone into reaction to form metal hydrides with potassium, and silane/disilane with silicon [51-53]. This may have been responsible for the reduction in percentage composition of potassium and silicon observed with the hybrid products. Both products are found to be sources of carbon-rich materials for carbon related applications. Mullen et. al. [3], Nanda et. al. [20] discussed the potential of using carbon-rich biochar to extenuate the negative effects of extensive crop farming on land thereby improving soil quality, which in result helps sustain food production with the rising world population and food demand associated. Nanda et. al. [20] further discussed the potential use of biochar as a carbon storehouse in soil due to the high stability of carbon present in biochar. The higher carbon content present in the hybrid biochar produced reveals a potential to remove carbon from the atmosphere through using materials ordinarily regarded as waste. Nanda et. al. [20] also highlighted the potential to reduce emissions of CO<sub>2</sub>, N<sub>2</sub>O and CH<sub>4</sub> using biochar, thereby establishing the potential of adopting the use of the hybrid biochar to mitigate the production of greenhouse gases.



**Figure 3** SEM micrograph of biochar produced from carbonization, at (a) 500× (b) 1000× and (c) 1500×



**Figure 4** SEM micrograph of biochar produced from co-carbonization, at (a) 500× (b) 1000× and (c) 1500×



**Figure 5** Elemental composition of ordinary biochar and hybrid biochar

### 3.4 Biochar Textural Properties

ANOVA station A BET analyser was used to measure the surface areas, pore-volumes, and pore diameters of the biochar and hybrid. Isotherms and pore size distributions of both products are presented in Figures 6a-b and 7a-b respectively with a general summary of the textural properties presented in Table 2. Using the Multipoint BET surface area method to determine the surface area, the biochar and hybrid biochar produced were shown to have had specific surface areas of 392.508 m<sup>2</sup>/g and 889.905 m<sup>2</sup>/g respectively. The increase in surface area can be attributed to alteration of the pore distribution due to the presence of the LDPE [30]. Some studies have attributed the low surface area of biochar to the incomplete decomposition of biomass within the process chamber resulting in the entrapment of compounds like tar within the pores, therefore it can be deduced that the co-carbonization process decomposed better due to the presence of the LDPE [54,55]. The biochar

and hybrid produced can both be considered to be mesoporous from their measured pore diameters and their abilities to be characterized by Nitrogen gas at 77K without resulting in diffusional problems (Table 2) [17]. Both products can find their applications in adsorption related processes. Although Mullen, Boateng [3] postulates that the ability to remove metal ions is a function of its surface properties rather than porosity, recent studies have shown that coating biochar with graphene can improve its adsorption ability of polycyclic aromatic hydrocarbons. The internal pores of the biochar can also serve as a niche for microorganisms for protection against predators or detrimental environmental factors, this improves the microbial activities within the soil which is beneficial to plant growth [20].

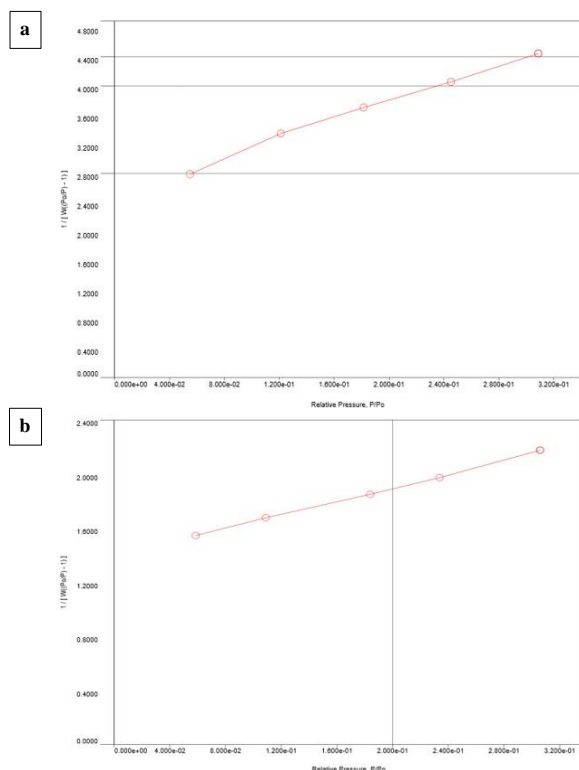


Figure 6 Isotherm Profiles (a) Ordinary Biochar (b) Hybrid.

Table 2 Summary of surface properties of biochar from corn cob and hybrid biochar from corn cob and LDPE.

Sample	BET surface area (m <sup>2</sup> /g)	Micro-pore volume (cc/g)	Pore diameter (nm)
Biomass biochar	392.508	0.1502	6.214
Hybrid biochar	889.905	0.3175	6.503

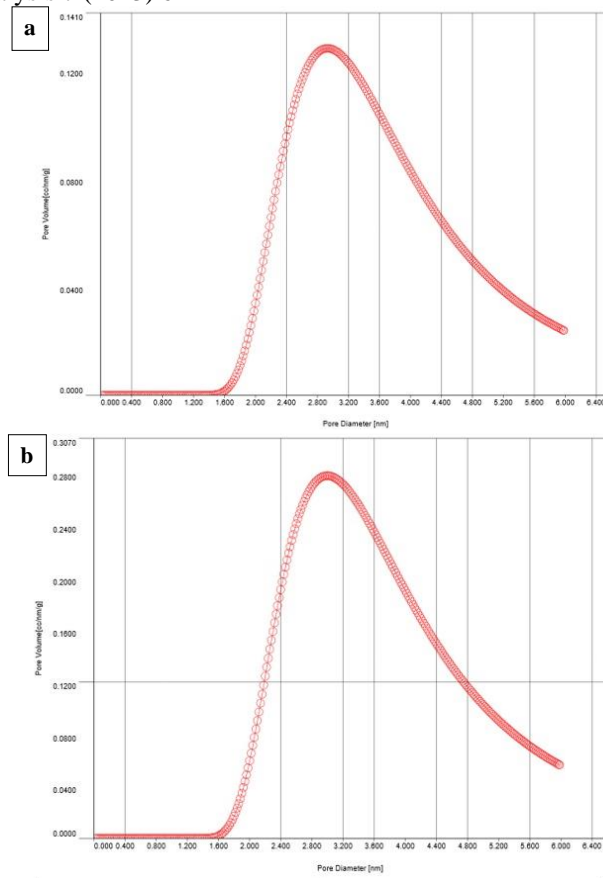


Figure 7 Pore Size Distribution Profile (a) Ordinary Biochar (b) Hybrid.

#### 4. CONCLUSION

The corncob feed was carbonized for 60 mins to give an ordinary biochar yield of 66.6 % with a peak temperature of 343°C. The corncob and LDPE feed were co-carbonized for 70 minutes to give a hybrid biochar yield of 33.3% at a peak temperature of 362°C. The SEM-EDX analysis of the biochar showed a homogenous surface with repeating patterns forming a honeycomb-like structure with a 62% carbon composition. The SEM-EDX analysis of the hybrid showed a heterogeneous surface compared to the ordinary biochar with a rough pattern and ridges with an 80% carbon composition. The BET analysis revealed the biochar and hybrid were found to have large specific surface areas of 392.5 m<sup>2</sup>/g and 889.9 m<sup>2</sup>/g respectively. The high specific surface area of the hybrid biochar produced highlights its potential as an adsorbent. The biochar and hybrid from both processes are both carbon-rich products for potential carbon related applications. The co-carbonization of corncob and LDPE has been established to be a means of conversion both agricultural and plastic waste into a more valuable product with various potential applications.

## DISCLOSURE STATEMENT

Conflict of Interest: The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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