SURFACE CHARACTERIZATION OF BIOCHAR FROM AGRICULTURAL RESIDUES FOR ENVIRONMENTAL APPLICATIONS

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ABSTRACT

Bio-waste materials banana (Musa ealbisiana) leaves, cassava (Manihot esculenta) peels, mango (Mangifera indica) leaves, plantain (Plantago lanceolata) leaves and peels were pyrolyzed at different temperatures; 300, 400, 450, 500 and 600 ^oC using a muffle furnace. The biochar samples obtained were characterized and properties evaluated for their potential application in soil for agronomic and environmental benefits. The measured physico-chemical properties; pH, bulk density, percentage yield and electrical conductivity varied with temperature of pyrolysis and somewhat with the source of the bio-waste. Surface morphology and elemental composition were determined from Scanning Electron Microscopic (SEM) and Energy Dispersive X-ray (EDX) measurements and showed variations in texture, porosity and elemental composition with pyrolysis temperature. The produced biochar yields were in the range 16.1 to 84.6% and highest for banana leaves (BL) pyrolysed at 300 ^oC. The scanned images showed a remarkable surface morphology for all the biochar samples obtained at temperatures \geq 450 °C. The pH obtained indicate the alkaline nature of *the biochars with mean values > 7.5 except for cassava peels (CP) pyrolysed at 300 °C having a value of 5.5. From the elemental analysis, the high carbon content (> 60%) at higher temperatures (> 450 ^oC) of the biochars and the molar O/C ratios may suggest the suitability of the biochars in applications for long-term carbon sequestration and also promising candidates for soil amendment due to increase in macro porosity.*

Keywords: Bio-waste materials, pyrolysis, biochar, surface morphology, elemental analysis

INTRODUCTION

Bio-wastes from agricultural residues which are abundant, inexpensive and readily available are a valuable resource used in the preparation of biochar, a carbon – rich product obtained from the pyrolysis of biomass in the absence or a limited supply of oxygen (O'Connor *et al*., 2018; Xiao e*t al*., 2018). Biomasses from agricultural residues, some of which are by-products of the agricultural

crops, raw materials from the forest, municipal solid waste, animal and human wastes, constitute an affordable source of energy and materials derived from them which include bio-oil, biochar, fuel and chemicals amongst others have been reported to have potential benefits to agriculture and the environment (Bardalai and Mahanta, 2018). Biochar has been reported to be an effective and environmentally friendly material in the agricultural sector for soil amendment and

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enhancement processes due to its large specific surface area, possession of surface functional groups such as hydroxyl, carboxyl, etc., microporous structure, high cation exchange capacity (CEC) amongst many others (Pattnik *et al*., 2018; Yang *et al*., 2017).

The use of biochar from different agricultural residues for environmental applications abound in literature (Adesemuyi*et al*., 2020; Wang *et al*., 2020; Safian*et al*., 2020) as biochar – related materials remain pillars for sustainable growth and development considering Nigeria's estimation of agricultural wastes production of approximately 998 million tons annually which has potential of producing biochar on commercial basis for environmental applications (Obi *et al*., 2016). Biochar from varieties of biomass such as bamboo leaves, rice husk, nutshells, pine wood, orchard pruning biomass, etc., have been exploited as feedstockand reported to produce efficient sorbents used in immobilizing heavy metals in aqueous environments, soils and sediments, remediate soil from pollutants such as petroleum hydrocarbons and pesticides, improve soil fertility and plant growth amongst many others (Guo*et al*., 2020; Xu *et al*., 2021; Kumar *et al*., 2023).

Agricultural residues (wastes) are being churned out in large quantities all over the world in general and Nigeria is not left out as population increases and some of these wastes are indiscriminately disposed in the environment which ultimately results in pollution with its attendant health effects on man. With the global call for sustainable practices which offer an eco – friendly approach to waste management, converting agricultural residues into valuable resource materials would no doubt enhance sustainability with the development of soil amendment materials for agronomic and environmental applications as well as reducing diverse health issues arising from the indiscriminate disposal of waste. Biochar have been reported to be prepared from different renewable resources and the general

characteristics which are somewhat similar have also been shown to be effective in the removal of pesticides and other contaminants in the environment (Singh *et al*., 2022). This study constitutes part of our efforts towards developing good biochar materials by selecting different biomasses, varying pyrolysis temperature conditions and determining the physicochemical characteristics to provide crucial information when designing selective biochar materials for remediation and/or improving specific soil properties. In this present study, the quality of biochar obtained from banana (*Musa balbisiana*) leaves, cassava (*Manihot esculenta*) peels, mango (*Mangifera indica*) leaves, plantain (*Plantago lanceolata*) leaves and peels over a range of pyrolytic temperatures in terms of pH, elemental analysis, bulk density, electrical conductivity, surface morphology and functional groups present was investigated so as to provide important baseline information for the production of biochars from agricultural wastes with desired properties for environmental applications and management.

MATERIALS AND METHODS

Five different biomasses were collected from nearby farmlands around the Ugbomoro village where the Federal University of Petroleum Resources, Effurun, Delta State, Nigeria, is situated. Prior to pyrolysis, the biomasses were cleaned and washed severally with deionized water to remove surface contamination, crushed $(> 1 \text{ cm})$, air – dried for approximately twenty – one (21) days and oven – dried at 60 \degree C for six (6) hours. The pyrolysis was carried out in a laboratory scale using muffle furnace coupled with a glass condenser and a nitrogen (N_2) gas cylinder using low and high temperatures at $300 - 450$ $\rm{^{\circ}C}$ and 500 – 600 $\rm{^{\circ}C}$, respectively. In a typical pyrolysis experiment, 60g of the biomass was placed in the muffle furnace, which had been heated to the desired temperature, in an inert atmosphere. Each sample was heated for one hour at a constant heating rate and biochar was the product obtained after pyrolysis which was

collected and stored in air – tight containers for further analysis. The percentage yield (%) for each biochar sample was obtained as a ratio of the mass of the dry biochar (M_2) to the mass of the dry biomass (M_1) using the equation:

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\% Yield = \frac{M_2}{M_1} x 100 \dots (1)
$$

Five biochar samples were obtained for each biomass: BL 300, BL 400, BL 450, BL 500 and BL 600 for biochars obtained from banana leaves; CP 300, CP 400, CP 450, CP 500 and CP 600 for cassava peels; ML 300, ML 400, ML 450, ML 500 and ML 600 for mango leaves; PL 300, PL 400, PL 450, PL 500 and PL 600 for plantain leaves; PP 300, PP 400, PP 450, PP 500 and PP 600 for plantain peels in addition to control samples for each biomass which did not undergo pyrolysis for comparison purposes.

Physico-chemical analyses and Surface Characterization of Biochar Samples

To assess the quality of the biochar obtained from the different biomasses, standard methods as described (Singh, *et al*., 2010; Adesemuyi*et al*., 2020) were used to determine the physico-chemical composition of each homogenized and ground $(> 1$ mm) biochar sample.The pH and electrical conductivity (E.C) of all the biochar samples was determined as a 1:10 biochar: deionized water ratio (w/v) slurry after vigorous shaking for two hours on a mechanical shaker using a Metrohm 744 pH meter and a Model S 213 compact conductimeter.The bulk density and exchangeable cations were determined as described previously (Singh, *et al*., 2010). All the experiments were carried out in triplicates and average values recorded.

The microstructure and elemental composition of the biochar samples were carried out using

a TESCAN Scanning Electron Microscope (TE-SEM) equipped with Energy Dispersive Spectroscope (EDS), Oxford Instruments, Czech Republic. The gold sputtering technique was used to coat the samples before analysis. To identify the functional groups, JASCO FTIR 4100 Fourier Transform Infrared Spectrophotometer was used to record the spectrum of the biochar samples over a scanning range of $400 - 4000$ cm⁻¹.

RESULTS AND DISCUSSIONS

Physicochemical analyses of biochar samples

The pyrolysis temperature has been reported to be strongly correlated with changes in the structure and physicochemical properties of biochar (Jindo*et al.* 2014). Data on these relationships are presented in Table 1. Generally, at the end of pyrolysis, the biochar yield decreased with an increase in temperature for all the biochar samples, however, the percentage decrease, $> 60\%$ was lower for BL and CP biochars produced at low temperatures $(300 - 400 \degree C)$ when compared with those produced at higher temperatures $(450 - 600 \degree C)$, ML, PL and PP biochars with \geq 80%. Biomasses have been reported to contain different proportions of lignocellulose components which vary and decompose differently due to the differences in the relative abundance of cellulose, hemicellulose and lignin present in them (Somparn*et al*., 2020; Yang, *et al.,* 2017). Plant biomasses such as BL, ML and PL used in this study have been reported to have main components as carbohydrates and lignin: the carbohydrates which are mainly cellulose or hemicellulose fibers gives strength to the plant structure and lignin holds the fibers together (Ucar and Ozkan, 2008).

Biochar	Temperature	Yield $(\%)$	pH	EC $(dSm-)$	Bulk density	CEC	
Sample	$({}^0C)$			$\mathbf{1})$	(g/cm ³)	(cmol/kg)	
Control	Banana Leaf	100.0	7.15	40.0	0.19	283.1	
BL	300	84.6	7.34	71.0	0.18	319.9	
	400	44.7	8.38	92.0	0.18	399.3	
	450	41.5	8.73	70.9	0.14	376.4	
	500	33.5	8.91	102.4	0.18	328.5	
	600	30.9	10.83	368.3	0.18	817.1	
Control	Cassava Peel	100.0	4.4	15.6	0.37	201.7	
CP	300	72.5	5.5	10.6	0.41	195.2	
	400	53.5	8.4	7.4	0.29	175.0	
	450	50.5	8.5	7.8	0.27	319.5	
	500	46.5	8.7	9.0	0.30	239.6	
	600	39.7	8.9	9.9	0.26	333.6	
Control	Mango Leaf	100.0	6.4	47.9	0.31	354.6	
ML	300	32.3	8.4	76.3	0.20	311.2	
	400	31.8	8.7	94.0	0.21	318.7	
	450	31.1	8.8	97.2	0.21	335.9	
	500	21.1	9.0	120.3	0.24	231.5	
	600	16.1	9.1	119.6	0.22	311.9	
Control	Plantain Leaf	100.0	6.5	10.1	0.30	272.9	
PL	300	54.1	7.5	55.4	0.14	240.8	
	400	38.8	8.1	51.1	0.13	230.5	
	450	38.0	9.2	128.3	0.13	260.5	
	500	30.1	9.5	106.2	0.15	274.1	
	600	22.0	10.1	122.6	0.11	339.4	
Control	Plantain Peel	100.0	6.8	26.4	0.46	217.3	
PP	300	61.2	7.4	27.4	0.27	360.1	
	400	45.5	7.7	52.1	0.23	258.1	
	450	37.0	9.7	54.4	0.21	220.3	
	500	36.9	9.7	50.8	0.22	332.6	
	600	22.1	10.0	67.1	0.22	272.1	

Table 1: **Physicochemical characteristics of biochars derived from different biomasses: banana leaf (BL), cassava peel (CP), mango leaf (ML), plantain leaf (PL) and plantain peel (PP)**

It has been reported that a good number of biochars from different biomasses used for soil amendment are alkaline as a result of carbonization during which acidic functional groups are removed and salts of alkali and alkaline earth metals become enriched (Singh, *et al*; 2010). The acidity/alkalinity of the biochars as determined by pH increased as the temperature increased for all the biochar samples as shown in Table 1. Except for BL, all the biomasses were acidic having pH values < 7 but after pyrolysis, the biochars became alkaline as a result of reduced acidic groups

and an increase in the salts of alkali metals which would make them suitable for improving acidic soils (Bardalai and Mahanta, 2018). Two effects of pyrolysis temperature on pH have been reported to be based on two aspects: an increase in pyrolytic temperature increases the formation of biochar ash which contains alkaline minerals and biochar produced at lower pyrolytic temperatures may contain greater densities of phenolic and carboxyl groups which are acidic thereby decreasing the pH (Zhao, *et al.,* 2018)

The electrical conductivity (EC) of a biochar sample is a measure of the quantity of soluble salts present and is an important property to consider when used for soil amendment purposes as it affects the salinity of the soil. Some authors have reported high EC values for biochars produced at higher pyrolysis temperatures (Kloss *et al*., 2012) and the values of EC obtained for the biochars in this study vary considerably with BL and ML having the highest values.

Carbonization through slow temperature have been reported to enrich the carbon content, create a porous structure and less dense biochar generally attributed to the decomposition of organic matter thereby leading to the transformation of biomass matrices into a lighter and porous structure (Amupam *et al*., 2016). The bulk density for all the biochar samples generally decreased with an increase in pyrolytic temperature indicating an increase in the porosity of the biochar samples. PL biochar samples had the lowest values for bulk density and may appear to a highly porous surface.

The cation exchange capacity (CEC) is an indication of carboxyl groups present and the ability of a soil or substrate to hold exchangeable cations and supply nutrients for plant uptake is of agronomic advantage. The application of biochar has been reported to increase the cation exchange capacity of soils thereby improving soil fertility (Wang, *et al*., 2020). The CEC obtained for the biochar samples from different biomasses varied: BL and PP biochar samples had CEC values increasing with an increase in temperature, CP and PL had higher CEC values at temperatures \geq 450 °C while the CEC for ML biochar samples decreased with an increase in pyrolysis temperature. High CEC values for BL, CP, PL and PP biochar samples indicate their potential application in soil to hold cationic nutrients and retain cationic fertilizer.

Elemental Composition using EDX measurements

Surface elemental analysis was performed using energy dispersive X-ray spectroscopy (EDX). For accuracy in data analysis, each EDX analysis was repeated three times for the biochar samples and average values obtained for the different elements present are given in Table 2. The major elements detected included C, O and K accompanied by small amounts of other elements such as Cl, Si, P, Mg amongst others as presented. The carbon content increased exponentially with an increase in pyrolytic temperature for most of the biochar samples which has been reported to be associated with the loss of –OH functional groups caused by dehydration (Song, *et al*., 2023). The oxygen (O) content in some cases, decreased with an increase in temperature. The decrease in oxygen concentration with increasing temperature can be explained by the fact that various oxygen functional groups are released as volatiles during carbonization (Wu *et al*., 2016).

Scanning Electron Microscopic (SEM) measurements

Scanning electron micrograph (SEM) images are very useful in obtaining accurate details about the surface structure of biochar and the comparison of the images at various temperatures might provide more insight into the morphological changes taking place during pyrolysis. The micrographs obtained for the different biochar samples pyrolysed at different temperatures are shown in Fig. 1. All the biochars at various temperatures possess porous structures. Each micrograph shows a porous structure of the biochar with different shapes and scales of pores with cylindrical crevices interconnected by some large tubes, seeming honeycomb-like at higher temperatures. Temperature strongly influences the structure of different biochars and previous studies (Devens *et al*., 2018) have shown that the porosity of biochar is mainly attributed to the intrinsic physical structure of the precursor biomass, and may be influenced by the sample pyrolysis temperature. The biochar samples

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contain several cracks and holes formed because of the evolution of volatile matter during carbonization The morphology of the biochar samples pyrolysed at different temperatures varied from each other and the SEM scans showed the complex surface structure of the biochars which are

heterogeneous. As the pyrolytic temperature increased, the biochar structures became more uniformed as the number of macropores increased and the number of micropores decreased thereby exhibiting a large degree of macro-porosity in the 1 to 10-micron scale.

Biochar	Temp	$\mathbf C$	$\mathbf 0$	$\mathbf K$	Si	Ca	Mg	Fe	Mn	${\bf P}$	Al	CI
Sample	(^oC)	Weight%										
BL	300	48.8	38.2	1.3	$\overline{}$	11.7	$\overline{}$	\overline{a}	$\overline{}$			
	400	75.4	20.5	1.5	0.5	1.0	0.8			0.2	0.3	
	450	75.8	18.8	2.5	0.8	2.0	$\overline{}$			$\overline{}$	-	
	500	66.9	18.4	4.5	$\overline{}$	7.8	1.4			0.9		
	600	72.5	15.9	6.6	0.7	2.5	1.7					
CP	300	56.5	37.1	0.6	2.2	$\overline{}$	$\qquad \qquad -$	0.9		$\qquad \qquad -$	2.6	$\qquad \qquad \blacksquare$
	400	69.1	22.0	3.1	1.8	1.1	0.4	0.8		0.5	1.3	
	450	71.7	23.0	1.7	0.7	0.7		1.0		\overline{a}	1.1	
	500	65.7	25.1	5.2	$\overline{}$	3.2	0.8					
	600	72.0	15.7	4.6	2.4	1.5	$\overline{}$	1.4	$\overline{}$	0.7	1.6	$\qquad \qquad \blacksquare$
ML	300	70.1	27.0	1.7	$\overline{}$	1.2						
	400	71.0	34.0	7.9	7.9	27.1	0.4		5.7			
	450	73.1	23.8	1.0	0.8	1.3						
	500	43.0	35.8	2.2	4.7	12.5	0.4			0.4	0.9	
	600	69.1	12.1	2.2	1.2	15.3						
PL	300	65.0	25.4	4.1	$\frac{1}{2}$	4.0						1.6
	400	71.8	13.4	3.7	11.1	$\overline{}$						$\overline{}$
	450	68.3	15.4	8.3	2.6	3.4	0.8					1.3
	500	64.5	21.5	5.2	1.0	6.7	1.0					
	600	69.9	18.3	5.6	0.9	3.4	1.4					0.6
PP	300	57.5	24.9	16.9	$\overline{}$	-						0.7
	400	61.3	20.8	15.1								2.7
	450	38.1	35.2	25.0	0.6							1.2
	500	69.7	19.0	9.2	0.5					0.4	\overline{a}	1.3
	600	62.5	21.7	13.6	0.4		0.3			0.6	$\overline{}$	0.8

Table 2: EDX analysis of biochars derived from different biomasses: banana leaf (BL), cassava peel (CP), mango leaf (ML), plantain leaf (PL) and plantain peel (PP)

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Fig. 1:Scanning electron micrographs (SEM) at 10 µm magnification for all biochar samples

Infrared (IR) Spectral Analysis

The biochar samples were subjected to FTIR analysis to determine the functional groups present.A typical FTIR spectra of CP at 300 °Cis shown in Fig. 2.

Fig. 2: TypicalFTIR spectra for CP biochar sample at 300 °C

For all the biochar samples, the FTIR spectra were characterized by three principal absorption bands between 3305 – 3505, 2920 -2927 and $1580 - 1630$ cm⁻¹ corresponding to the stretching vibration of $-OH$, $-CH₂$ and aromatic C=O ring respectively. It has been reported that biochar from biomass is dominated by functional groups typical of oxygenated hydrocarbons which reflect the carbohydrate structure of cellulose and hemicelluloses (Tomcyzk, *et al*., 2020). The production of biochar at various pyrolytic temperatures has been shown and reported to cause the disappearance of absorption bands characteristic of the raw material and the appearance of new bands typical of biochar samples (Ghani, *et al*., 2013; Tomcyzk, *et al*., 2020). Typical FTIR bands reported for biochars ((Claoston*etal*. 2014; Ghani, *et al*. 2013; Liu, *et al*. 2015; Tomcyzk, *et al*., 2020; Zhao, *et al*., 2017), were present in all the biochar samples at different pyrolytic temperatures.

CONCLUSION

This study produced biochars from banana (*Musa balbisiana*) leaves, cassava (*Manihot esculenta*) peels, mango (*Mangifera indica*) leaves, plantain (*Plantago lanceolata*) leaves and peels at different pyrolysis temperatures –

300, 400, 450°C, 500 and 600°C. They were characterized using energy dispersive spectroscopy, scanning electron microscopy and Fourier transform infraredspectroscopy. The biochar yield, pH, electrical conductivity, bulk density and CEC were also determined using standard methods. The pH, bulk density, percentage yield and electrical conductivity of all the biochar samples varied with temperature of pyrolysis. The produced biochar yields were in the range 16.1 to 84.6% and highest for banana leaves (BL) pyrolysed at 300 °C. The scanned images showed a remarkable surface morphology for all the biochar samples obtained at temperatures \geq 450 \degree C as the porosity of the biochar samples increased exhibiting a large degree of macroporosity in the 1 to 10-micron scale. The pH obtained indicate the alkaline nature of the biochars with mean values > 7.5 except for cassava peels (CP) pyrolysed at 300° C having a value of 5.5. The high carbon content $($ 60%) at higher temperatures $(> 450 \degree C)$ obtained for the biochars may suggest their suitability for applications in long-term carbon sequestration. In addition to their carbon sequestration potential, biochars prepared from CP, PP and PL at 450 $^{\circ}$ C are promising candidates for soil amendment applications.

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