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Research Article

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Physicochemical Properties of Biochar Prepared from Guinea Corn Straw as a Function of Different Pyrolysis Temperatures

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ABSTRACT

The present study examined the effect of pyrolysis temperature on the physicochemical properties of biochar produced from guinea corn straws at different temperatures (300-600°C). However, the produced biochars were designated as BCG-300, BCG-400, BCG-500 and BCG-600, respectively. The produced biochar's were characterized by different analytical techniques. These include; the proximate and elemental analyses, morphological, crystallographic, functional groups and specific surface area analyses were investigated using elemental analyzer, scanning electron microscopy (SEM), X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy and Brunauer–Emmett–Teller (BET), respectively. The results indicated that. increasing pyrolysis temperature increased the content of fixed carbon (C), the C content, the total content of K, Ca, and Fe, as well as the cation exchange capacity (CEC), pH, and EC. While the yield, the content of volatile matter O and H, and the ratios of O/C and H/C decreased. Additionally, at higher pyrolysis temperature, the BET surface area and pore volume increased with increased in temperature, typically due to the increase of the micropore surface area and micropore volume. Furthermore, the data of Fourier transformation infrared showed that at higher pyrolysis temperature, the biochar aromaticity increased and decreased in polarity. Similarly, the crystalline mineral components increased with pyrolysis temperatures due to the cellulose loss, as indicated by X-ray diffraction analysis and scanning electron microscope images. Hence, pyrolysis temperature has a strong effect on biochar properties, and therefore biochars can be produced by changing pyrolysis temperature in order to meet their applications, such as carbon sequestration, greenhouse gas mitigation and as an adsorbent for organic and inorganic contaminants when applied to the environment etc.

Keywords: Guinea Corn Straw, Biochar, Pyrolysis Temperature, Physicochemical Properties

Introduction

Biomass production are mainly from the largest agricultural biological sectors [1]. However, with the recent global growing population, there is need for the higher demand of agricultural production, and this resulted in the surged of agricultural waste [2]. Agricultural wastes are often obtained from crops, tuber and root, fruit and vegetable, seed, and animal etc. [3]. It is estimated that, the annual world agricultural wastes reached over 140 billion tons [4].

Through thermochemical conversion process such as: pyrolysis, combustion, gasification, and liquefication and/or biochemical process, this abundance of waste can be converted to an enormous. amount of energy and raw materials [2,5]. Pyrolysis is one of the oldest thermochemical methods of processing different biomass using higher temperatures and pressures into solid (biochar), liquid (bio-oil), and non-condensable gaseous materials [6].

Pyrolysis is a thermal degradation process that is commonly carried out at temperature ranging from 300 to 800ºC or more in the absence or presence of insufficient oxygen [7]. Pyrolysis process is used to make carbonaceous materials by decomposing

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organic materials and extracting non-carbon fractions [8]. Generally, pyrolysis process is divided into two main stages; the primary stage, which removes moisture from the biomass particle during the pre-pyrolysis and the main pyrolysis process, which start with the transfer of heat to the particles surface through radiation or convection, and then into the particle. The volatiles produced during this process flow through the pores of the particles and engage in heat transfer [9]. Subsequently, resulting in the formation of thermally stable solid char and liquids, followed by secondary decomposition (cracking and depolymerization) reactions that converts the unstable volatile compounds to gaseous products [10]. Variations in the pyrolysis process and its conditions including temperature and residence time, which are considered the most important parameters, greatly affect the quality and properties of biochar. The properties of the final product are also dependent upon the nature of the feedstock [11].

Biochar is a carbonaceous product generated by thermal decomposition of organic material (biomass) under a limited supply of oxygen, at relatively low temperatures (<700 ºC) [12]. Agricultural crop residues, agricultural by-products/ wastes, and forestry biomass are lignocellulosic materials, which are readily available and abundant, resulting in them being the most common feedstocks for biochar [13]. Meanwhile, the physicochemical properties of biochar such as higher surface area, pH, EC, CEC and functional groups are highly associated with changes in its structure during pyrolysis, and this has an impact on the material's functions [14]. Numerous studies have demonstrated that the temperature during pyrolysis affects the physicochemical characteristics of biochar [15]. The presence work is aim at producing the biochar from guinea corn straws, and to evaluate the effect of pyrolysis temperatures on the physicochemical characteristics of the biochar.

Materials and Methods

The guinea corn straw biomass was collected from the local farm. Prior to the experiments, the feedstocks were air dry for 30 days. However, for better exposure to heat during slow pyrolysis in the muffle furnace, the biomasses were cut into small pieces of approximately 5-6 cm in length. Subsequently, the biomass materials were washed with distilled water to remove any dirt and dust before drying in an oven (thermostat oven DHG-9023A) for 24 h at 105°C before pyrolysis [16].

Biochar Preparation

The guinea corn straws that had already been oven dried was put through a batch slow pyrolysis process in a muffle furnace (Carbolite Gero-30-3000oC, UK). Placed in a ceramic crucible, sealed with a tight-fitting lid, and coated with aluminum foil sheet, and then heated at 300, 400, 500, and 600°C for 30, 60, 90, and 105 minutes of residence time, respectively, at a steady heating rate of 10°C per minute. After reaching the desired temperature, the furnace was turned off and left to cool for a while before being removed and allowed to cool to room temperature in a desiccator. The pyrolysis experiment was repeated three times. The yield of biochar was calculated using the following equation

$$\text{Biochar Yeald\%} = \frac{\text{mass of Biochar(g)}}{\text{oven dry mass of feed stock}} \text{X100}$$

The produced biochar was crushed, and passed through a 100-mesh sieve and kept in airtight plastic containers for subsequent analysis. The produced biochars was designated as BCG300, BCG400, BCG500, and BCG600, respectively [17].

Characterization of Biochar

The ash content was analyzed according to the ASTM standard method D1762 – 84, 7582-10, approximately 1g of 105ºC oven-dried sample was heated at 750°C for 5 hours [18]. The moisture content was determined by taken exactly 1g of each of the dried samples into a ceramic crucible and heated for 2 hours at 105.

For the volatile matter, also an exact amount of 1g of the dried biochar samples each was heated at 950 ºC for 7 min in a muffle furnace. The Fixed carbon content (FC) was calculated by the following relationship: (Fixed carbon content) %FC =100-(VM + Ash + MC).

The C, N, and H elemental contents of biochar were determined by an elemental analyzer (Vario Macro cube, Elementa, Germany), while, the O content was calculated by subtracting with respect to the total sample [19]. The measurement of pH and EC of the biochar samples were determined using digital pH meter which was initially calibrated with standard solutions. A 1g each of biochars were mixed with 10 mil distilled water and Shaked for 1 h on a reciprocating shaker at room temperature [20].

The cation Exchange Capacity (CEC) was determined according to the procedure adopted by [21]. Briefly, 5g each of the biochar samples were mixed with 40 ml of 1.0 moldm3 ammonium acetate solution (pH 7) in 100mil plastic beaker and stirred with glass rod and left overnight. It was then suction-filtered with 55 mm Buchner funnel. The residue from filtration was leached with four 25ml portions of 1moldm3 NH_4Cl solution (pH 7). The solution was discarded and the electrolyte washed out of the sample with 150 ml ethanol. The samples were allowed to drain completely and leached gradually with acidified NaCl to 250 ml. 50 ml of 2% boric acid was measured into 250 ml comical flash and 3 drops of mixed indicator were added. The acidified NaCl leachate was poured into 500 ml Kjeldahl flask and 10 ml of 1.0 moldm3 NaOH and anti-bumping granules were added. The leachate was distillates over the boric. 1.5 ml of ammonium borate distillate was titrated with standard 0.1 moldm3 HCl.

BET surface area of biochars were measured using a NOVA 4200e analyzer (Quantachrome Instruments, USA) at liquid nitrogen temperature (77 K). The Brunauer–Emmett–Teller (BET) surface area, Pore Volume and Pore diameter of the biochars produced at different temperatures were determined using the BET equation.

The functional groups present in the biochars were characterized by FTIR spectrometer (PE-1710, USA). Dried biochar. Approximately 10mg of each of the samples were dispersed in 200mg of spectroscopic grade KBr to record the spectra using potassium bromide disks to prepare the biochar samples. The spectral range from 4000 – 1000 cm-1.

The mineral compositions of the straw biochar were analyzed using X-ray diffraction (XRD). The XRD was used to identify

the crystalline phases formed for the prepared biochars, using the diffractometer (Bruker D8 Advance), equipped with the Kα radiation of copper (λ =1.5406) produced at 45kV 15mA, the data were collected over 2θ range (0–80°) at 2θ scan rate of 10°min^{-1}.

To identify the structural morphology of the prepared biochar at different pyrolysis temperatures, scanning electronic microscopy (SEM) (JEOL JSM-6360LV, Tokyo, Japan) was used at 500x magnification and 15kv-image.

Statistical Analysis

The experimental data were expressed as means of three replicates. One-way analysis of variance (ANOVA) experimental data was conducted in this work using the MINITAB software, and the Turkey's HSD post-hoc tests ($p < 0.05$) were used to identify significant differences among different biochar [22].

Results and Discussions

Effect of Pyrolysis Temperature on Biochar Yield

The biochar yield at different pyrolysis temperature is presented in figure 1. A significant ($p < 0.05$) decreased in biochar yield with the corresponding increase in pyrolysis temperatures was observed. The percentage yield of BCG declined from 45.67 to 29.67 %, when the temperature raises from 300°C to 600oC respectively. This decreased in the yield is attributed to the fact that the corn straw biochar decomposition of hemicellulose, cellulose and lignin during the biomass pyrolysis process, which occurred within the temperature ranges 220−400 ◦C and >400 ◦C, respectively [19]. This resulted led to the release of more volatile chemicals and facilitate dehydration of hydroxyl groups [23]. This result is similar to the results obtained by previous researchers [20,24].

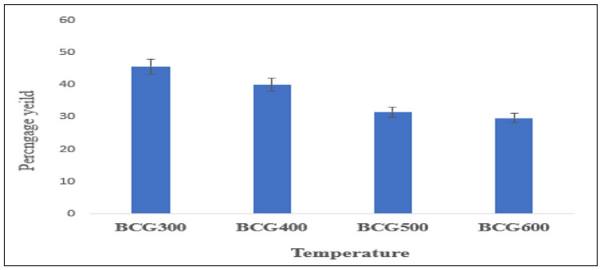

Figure 1: percentage yield of biochar at different temperatures (°C)

Effect of Pyrolysis Temperature on Biochar Physicochemical Properties

Proximate Analysis

The effect of pyrolysis temperatures of the biochar samples on volatile content (VC), moisture content (MC), ash content (AC), and fixed carbon (FC) are presented in Table 1. The moisture and volatile contents of the produced biochars decreased significantly ($p<0.05$) from 5.3 to 1.0% and 80.03 to 57.04% with the increased in pyrolysis temperature from 300 to 600°C, respectively. This result is in the same trend with the findings of [22,25] with millet straw biochar. This is due to the fact that the increasing temperature resulted in more destructive of the volatiles fractions of cellulose matters into low molecular weight liquids and gases [26,27]. However, the ash content increased with the raised in pyrolysis temperature table 1 [28]. This gradual increased in ash content can be ascribed by the increase in the inorganic substance [39]. Consequently, the biochar become more alkaline in nature due to the presence of alkaline substances and the progressive concentration of minerals with the upsurge of pyrolysis temperature [26,30]. In the meantime, the ash of biochar determines the value of biochar; a low ash indicates a high values biochar [31]. Similarly, the fixed carbon also increased significantly ($p<0.05$) from 13.97 to 33.96%, when the pyrolysis temperature increased respectively from 300 to 600°C, table 1. The result corresponds to the results of previous studies [16,32]. From the result it can be observed that BCG contain moderate fixed carbon content, when compared to the results of the previous literatures. Meanwhile, high fixed carbon content in biochar indicated a loss of volatile matter from the biochar, at the same time high fixed carbon in biochar is favorable for use as a solid fuel because of high energy content [32].

Table 1: Proximate Analysis of Guinea Corn Straw Biochar

Samples	Moisture content	Parameters (%) Volatile content	Ash content	Fixed carbon
BCG-300	5.3±0.15a	80.03±0.10a	0.7±0.06c	13.97±0.81d
BCG-400	4.8±0.10a	67.04±0.15b	6.8±0.06b	21.36±0.12c
BCG-500	2.7±0.02b	65..03±0.34c	7.8±0.10a	24.47±0.01b
BCG-600	1.0±0.09c	57.04±0.06d	8.0±0.03a	33.96±0.02a

Note: Each value represents the mean of three replicates (n=3), ± standard deviation. Small letters following the numerical values denote significant differences between each row data ($p < 0.05$). However, means that do not share a letter are significantly different.

Elemental Characteristics

Table 2 provides the chemical composition of BCG produced at different pyrolysis temperatures. The result showed that pyrolysis temperature has significant influence on elemental composition of biochar. The percentage carbon fraction increased significantly ($p< 0.05$) from 42.83 to 51.16%, indicating that straw biochars had good carbon sequestration ability. However, the H, O and N decreased. The increase in carbon content with increased in pyrolysis temperature is attributed to the increasing degree of carbonization. But, the decrease in O and H elements may be ascribed to breaking of weak bonds in biochar structure due to the dehydration of organic compounds and the loss of volatile components [30,33]. Resulting in the formation of highly carbonaceous materials [34]. This result is agreed with results of the previous literatures [26,35,36]. It has been reported that carbon-rich biochar has good potential for soil amendment, it immobilizes heavy metals in soil and/or wastewater, and served as bio-catalysts in microbial fuel cell technology to produce electricity [32]. Meanwhile, changes in the pyrolysis temperature have no significant effect on the amount of S. This finding followed the same trend with the results from previous literatures [26,37,38]. The H/C and O/C molar ratio which describe the degree of aromaticity, polarity and bonding arrangement of the biochar [39], Table 2 shows a gradual decreased with the increased in pyrolysis temperatures. The result is in same trend with the results obtained by previous scholars [12,16,40-42]. The increased in C/N molar ratio was observed table 2, with increased in pyrolysis temperatures, which indicates carbonization and the conversion of organic

nitrogen [39]. Therefore, it can be concluded that increased in pyrolysis temperature resulted in an increase in aromaticity and decrease in polarity for the biochars. hence, these changes could be ascribed to the formation of aromatic structures and removal of polar functional groups for biochars produced at a higher pyrolysis temperature [30]. Furthermore, the results of the present study showed that, the essential elements such as (Ca, K and Fe) increased with pyrolysis temperature, mainly due to increase in concentration of these elements in biochars with increase in pyrolysis temperature. And, these elements might not be lost by volatilization during pyrolysis [34].

Table 2: Elementals Composition of Guinea Corn Straws Biochar

Parameters	BCG-300	BCG-400	BCG-500	BCG600
C (%)	42.83±0.08a	43.48±0.2b	45.18±0.05c	51.16±0.04c
H (%)	5.59±0.07a	5.49±0.01ab	5.36±0.03b	5.22±0.02c
O (%)	48.28±0.09a	41.88±0.06b	39.31±0.25c	33.44±0.01d
N (%)	2.60±0.02a	2.35±0.02b	2.35±0.02b	2.18±0.02c
S (%)	8.35±0.09a	10.86±0.02b	4.36±0.09c	7.54±0.01d
H /C	1.56	1.50	1.41	1.22
O/C	0.85	0.72	0.65	0.49
C/N	19.20	21.56	22.41	32.45
Ca (mg/kg)	24.40 ±0.043a	26.24 ±0.096a	29.18 ±0.012a	29.28±0.062a
K (mg/kg)	33.55 ±0.168b	36.57±1.018b	42.20±0.478b	53.91±0.455b
Fe (mg/kg)	0.381±0.0135c	1.028±0.0216c	1.150±0.0051c	3.699±0.0035c
Cr (mg/kg)	ND	ND	ND	ND
Cd (mg/kg)	0.007±0.0010d	0.007±0.0011	0.008±0.0001d	0.009±0.0017d
Pb (mg/kg)	ND	ND	ND	ND

Each value represents the mean of three replicates (n=3), ± standard deviation. Small letters following the numerical values denote significant differences between each row data ($p < 0.05$). Though, means that do not share a letter are significantly different.

Table 3: Chemical Properties of Guinea Corn Straws Biochar

Samples	Parameters		CEC (cmol/kg)
	pH	EC (dS/m)	
BCG-300	7.03±0.05d	8.33±0.01d	47.00±0.00d
BCG-400	8.41±0.06c	8.38±0.01c	68.80±0.10c
BCG-500	9.58±0.15b	8.72±0.01b	70.00±0.00b
BCG-600	10.20±0.10a	10.02±0.02a	78.00±0.00a

Chemical Characteristics

Table 3 summarized the chemical characteristics of BCG. The result showed that increase in pyrolysis temperature significantly ($p < 0.05$) increased the pH, EC, and CEC values of BCG. The significant increase in pH at higher temperatures is attributed to the increased relative concentration of non-pyrolyzed inorganic elements in the feedstocks and the formation of basic surface oxides under high pyrolysis temperature [43]. However, the increased in EC from 8.33 to 10.02 dS/m with increased in pyrolysis temperature from 300 to 600℃, respectively, might be due to higher degree of volatilization, decomposition of surface oxygen groups and dehydroxylation which contribute to increased ash residue portion particularly K+ ion in the biochar at higher temperature during the conversion process [42,44]. Furthermore, the higher CEC of BCG as shown in table 3 at higher pyrolysis temperature, may be attributed to increase in inorganic elements such as Ca, Na and K, and formation of oxygen-containing functional groups on the surface of biochars which raised with increased pyrolysis temperature. These findings are in total agreement with result previous researchers [45]. CEC is one of the important characteristics of biochar that influences the soil fertility and the adsorption of mineral nutrients and ions such as sodium (Na+), Ca2+, Mg2+, K+, and NH4+, when amended to soils [46].

Functional Group Analysis

Changes in the FTIR spectrum can show the effects of pyrolysis temperature on the functional groups of BCG. As shown in figure 2. The decrease in the O-H stretching vibration (3755–3563 cm^{-1}) indicated that the bound water of straw biochar's disappeared when the pyrolysis temperature exceeded 500 ℃ [15,25]. The absorption peak in BCG-300 at 2918cm^{-1} is attributed to the C-H stretching vibration in the aliphatic group, indicating the existence of cellulose and hemicellulose in the guinea corn straw. However, this peak was lost at temperatures increased over 500 °C [47-49]. This indicated a decrease in the aliphatic group and the formation of a dense ring structure in BCG at higher temperature [15]. The peaks at 1420cm^{-1} is attributed to the symmetric stretching vibrations of C=O in ionic carboxylic groups (–COO−), which disappeared at temperature increased from 500-600℃ [47]. Additionally, increase in pyrolysis temperature above 500℃ lost the peaks at 1065 and 1066 cm^{-1} (stretching vibration of ether, C–O–C and/or the stretching vibration of C–OH of alcoholic groups and carboxylic acids [47,50]. The band at 875 cm^{-1} to 689cm^{-1} designed for the aromatic C–H out-of-plane vibration and/or the Si-O band [51]. From the present study it can be clearly understand that increase in pyrolysis temperature may increase the decomposition of polymers in the biochars. additionally, the biochar derived at 600 °C lost some of the absorption peaks, except for bands in

689–875 cm^{-1}, which was related to the Si–O–Si groups. This may imply that increasing the pyrolysis temperature, increase the stability of the biochar's.

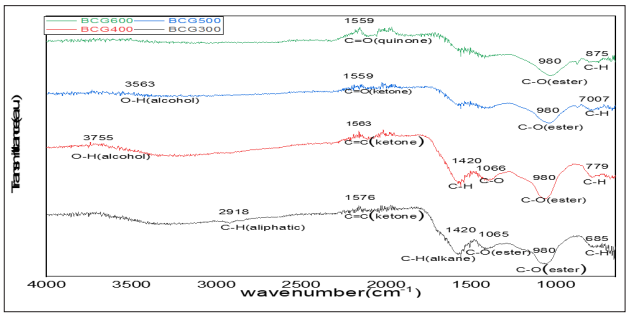

Figure 2: FT-IR Spectrum for BCG-300, BCG-400, BCG-500 and BCG-600

Surface Area

The BET analysis of the biochar produced at four different temperatures is presented in Table 4. The result revealed that increased in pyrolysis temperature from 300 to 600°C, raised the BET surface area and Pore Volume of the biochar from 438.573 to 530.442 m^2/g, and 0.225 to 0.270 cc/g, respectively. The effects of pyrolysis temperature on the biochar surface areas, is due to the progressive degradation of organic material (cellulose and lignin), which increased considerably with increasing temperature [15,18]. Which caused thermally unstable compounds in biochars to convert to a gaseous state [39]. Higher specific area biochar is crucial to biochar adsorption capacity for contaminants [52,53].

Table 4: BET Analysis of Guinea Corn Straws Biochar

Samples	BET Surface Area (m^2/g)	Pore Volume (cc/g)	Pore diameter (nm)
BCG-300	438.573	0.225	2.153
BCG-400	477.203	0.314	1.853
BCG-500	487.664	0.299	2.101
BCG-600	530.442	0.270	2.113

Surface Morphology

Figure 3 shows the scanning electron microscope (SEM) images (1000x) of biochars produced at four different temperatures (300, 400, 500, and 600°C). From the images it can be clearly observed that varying the temperature of pyrolysis played a significant role in changing the surface structural morphology with the change in temperatures. The physical structure of BCG looked to show little damage at an initial pyrolysis temperature of 300 °C [15]. However, with the increasing temperature of pyrolysis to 400 °C, the surface of the biochar become more destructive. Again, as temperatures increased from 400 to 600 °C, the surface structures appeared cracked and collapsed resulting in the destruction of its pore structure, surface deformation increases in pore quantity, and decreases in pore volume were noticed as the pyrolysis temperature increases, which may be due to the collapse of the internal pore structure [6].

Crystallographic Characteristics

The crystallographic characteristics of the biochars was conducted using XRD analysis. The results are presented in Figure 4. The samples were analyzed in the region of 2 Theta (θ) and angle range of 0˚- 80˚ at room temperature. According to XRD spectra analysis, similarities were noted among the biochars at four different pyrolysis temperature. The XRD pattern for all the biochars generated indicated that, they are predominantly of amorphous carbon mineral phases due to the absence of peak carbon [54]. However, a high XRD background halo between 2θ of 19.5° and 21.37° can be noted [51]. Indicating the presence of inorganic minerals of SiO_2 (quartz), KCl (sylvite at 43.2°). other minerals phase present in all the BCGs are $CaCO_3$ (calcite at peaks of 50.6° and 80.2°) [55]. The XRD results showed that the surface of the biochars could be heterogeneous [56]. Meanwhile, the XRD patterns showed no significant differences with the raised in temperatures [57-64].

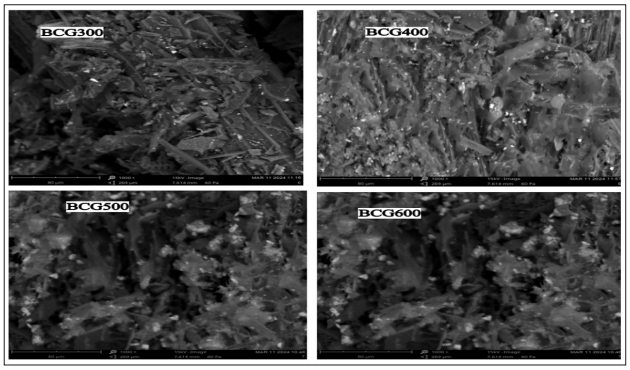

Figure 3: SEM Images of guinea corn straw biochar pyrolyzed at 300, 400, 500, and 600°C at 500x magnifications.

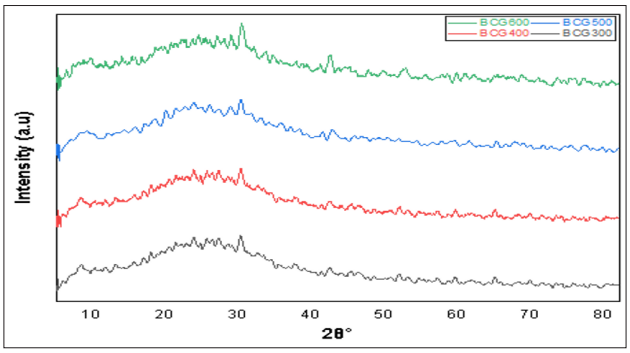

Figure 4: XRD Analysis of guinea corn straw biochar produced at different pyrolysis temperature.

Conclusion

This study covers the production of biochar from guinea corn straw feedstock by slow batch pyrolysis process, aimed at assessing the effect of the pyrolysis process on the production and physicochemical characterization. The pyrolysis temperature has significant effect on the structural and physicochemical properties of biochar. The results show that yield, VM, and MC, H and O were decreased with increase in pyrolysis temperature. Meanwhile, ash content, FC, pH. EC, CEC BET surface area of the produced biochar have positive correlation with the increase in pyrolysis temperature. However, based on the physicochemical properties of the BCG, such as higher specific surface area, abundant functional groups, and higher CEC at elevated temperature, the biochar could be used as an adsorbent for the immobilization of heavy metals from soils and wastewater contaminants and/or as cation-nutrient adsorption and retention due to amphiphilic properties. And/or used as

source of amendment to soil, due to its higher pH, EC, as well as in carbon sequestration process due to higher carbon content.

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Author Contributions
All authors contributed to the study conceptual design. I Abubakar: conducting experimental research processes, statistical and mathematical computation of the data. MD Saeed: responsibility for the management and coordinating the research activity and planning and executing. AM Ayuba: conceptualization and supervision

Competing Interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The corresponding author is submitting the paper on behalf of all the co-authors.

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