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Production and Characterization of Biochar from Rice Husk

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Abstract
Biochar was produced from Rice husk which is an agricultural waste obtained from rice milling process. The rice husk was pyrolized at a temperature of 500 ºC inside a drum pyrolyser for a period of 1 hr 30 min. A particle size of 450µm of the biochar was obtained using sieve analysis and the biochar was characterized. From the results obtained, the surface area of the biochar was 310.3m^2/g. The bulk density, pore volume and pore size were found to be 0.143g/cm^3, 0.143cm^3/g and 3.681 nm respectively. The yield was 49% and the pH is 8.4. The adsorptive capacity of the biochar was 0.894 mg/s. From the results, it is concluded that the biochar has good adsorption properties and can be used for, soil amendment/conditioning, wastewater treatment, gas separation and purification, water purification and vegetable oil refining processes. The biochar can also be considered for application in the production of chemical biochar based fertilizer. The production of biochar using rice husk is also a good solid waste management technique which helps in preventing environmental pollution.

Keywords: Electron, Production, Characterization, Biochar, Rice Husk

Introduction
Biochar is the carbon-rich product obtained from the pyrolysis of biomass such as such as wood, manure, or leaves, in oxygen depleted atmosphere. Extensive feedstock biomasses have been used in the production of Biochar (Angin, 2013), such as organic waste (green yard waste and animal manure) (Song and Guo, 2012); agricultural waste (Tsai *et al.* 2012); bioenergy crops (willows, miscanthus, and switch grass (Beeseley and Marmiroli, 2011); forest residues

(sawdust, grain crops, and nutshells) (Xu *et al.* 2012); kitchen waste, and sewage sludge (Lu *et al.* 2012). According to the International Biochar Initiative (IBI), biochar is primarily used for soil applications for both agricultural and environmental gains (Kajitani *et al.* 2013). The IBI definition differentiates biochar from charcoal, whose use is as a fuel for heat, as an absorbent material, or as a reducing agent in metallurgical processes (Aydın *et al.* 2008). Biochar contains porous carbonaceous structure and an array of functional groups (Lehmann and Joseph 2009). It has porous structure, charged surface, and surface functional groups (such as carboxyl, hydroxyl, phenolic hydroxyl, and carbonyl groups). These properties are the important factors that influence the migration, transformation, and bioavailability of contaminants in soil (Beeseley *et al.,* 2011).

In more technical terms, biochar is produced by so-called thermal decomposition of organic material under limited supply of oxygen (O_2) and at relatively low temperature (<700 °C) (Lehmann, 2009). Thermo-chemical processes include (i) slow pyrolysis (conventional carbonization); (ii) fast pyrolysis; (iii) flash carbonization; and (iv) gasification (Wardle *et al.,* 2008). Moreover, its molecular structure shows a high degree of chemical and microbial stability (Cheng *et al.,* 2010). The physical and chemical properties of biochar are highly dependent on pyrolysis temperature and process parameters, such as residence time and furnace temperature, as well as on the feedstock type (Abel *et al.,* 2013). A wide range of common raw materials are used as the feedstock, including wood chip, organic wastes, plant residues, poultry manure and agricultural wastes such as rice husk, corn cobs, groundnut shells etc. (Sohi *et al.,* 2010). The major elements in a biochar generally include Carbon, Nitrogen, Hydrogen, with some minute nutrient element, such as Pottasium (K), Calcium (Ca), Sodium (Na), and Magnesium (Mg) (Zhang *et al,.* 2013). The carbon Hydrogen and Nitrogen contents in biochar depend on pyrolysis temperature; and commonly, the carbon content increased with increasing pyrolysis temperature from 300 to 800 °C, while the contents of nitrogen and hydrogen decreased (Shinogi and Kanri, 2003). Biochar has a high specific surface area and a number of polar or nonpolar substances, which has a strong affinity to inorganic ions such as heavy metal ions, phosphate, and nitrate (Schmidt *et al.,* 2015).

The term biochar is a recent development, coming up in conjunction with soil management, carbon sequestration issues, mitigation of climate change and immobilization of pollutants (Kajitani *et al.,* 2013). Recently, it is reported that conversion of biomass into biochar not only result in the renewable energy (synthetic gas and bio oil), but also decrease the content of CO_2 in the atmosphere (Amutio *et al.,* 2013). Biochar was reported to improve not only soil chemical and physical properties but also soil microbial properties. Many studies indicated that the combination of biochar with soils could improve soil structure and properties of soil, such as the

water-holding capacity, organic matter content, aeration condition, pH value, porosity, bulk density, cationic exchange capacity (CEC), and the formation of aggregates of soil (Atkinson *et al.,*2010). Biochar has also been found to increase soil electrical conductivity by 124.6 % (Oguntunde *et al.,* 2004) and cation exchange capacity by 20 % (Laird *et al.,* 2010), while reduce soil acidity by 31.9 % (Oguntunde *et al.,* 2004). Biochar has also been reported to increase soil biological community composition (Chen *et al.,* 2010) and microbial biomass by 125 % (Liang *et al.,* 2010). The leaching losses of Nitrogen and Phosphorous in soil and the release of greenhouse gases (N_2O and CH_4) from soil could be decreased in the presence of biochar (Thies *et al.,* 2009).
In recent years, an increasing interest in applying biochar is focused on the amendment of nutrient-poor soil for soil ecological restoration including sequestering carbon (Jiang *et al.,* 2014; Liu *et al.*,2013). Various mechanisms have been suggested for the increase of plant nutrient availability in nutrient-limited agro-ecosystems such as (1) the initial addition of soluble nutrients contained in the biochar (Sohi, 2012) and the mineralization of the labile fraction of biochar containing organically bound nutrients (Lehmann *et al.,* 2009); (2) reduction of nutrient leaching due to biochar's physicochemical properties (Liang *et al.*, 2010); (3) lower escapable Nitrogen losses by ammonia volatilization and N_2 and N_2O from denitrification (Chen *et al.,* 2013); and (4) a retention of Nitrogen, Phosphorus, and Sulphur associated with the increase in biological activities or community shifts (Ibrahim *et al.,*2017).
Biochars are made from range of biomasses that have different chemical and physical properties. The properties of each biomass feedstock are important in thermal conversion processes, particularly the proximate analysis (ash and moisture content), caloric value, fractions of fixed carbon, and volatile components (Angin, 2013); percentage of lignin, cellulose, and hemicelluloses (Shivaram *et al.,* 2013); percentage and composition of inorganic substance, bulk, true density, particle size, and moisture content. It was again found that the structures of Biochar are temperature dependent and the following transition was proposed: (1) transition Biochar having crystalline nature of the biomass feedstock or material preserved; (2) amorphous Biochar that is a random mixture of thermally changed molecules and emerging aromatic polycondensates; (3) composite Biochars having poorly structured graphene stacks fixed in their amorphous phase; and (4) turbastic Biochar dominated by chaotic graphite crystallites (Zhou *et al.,* 2013). In general, high yields of Biochar derived from the biomass which has more lignin and less cellulose are always derived.
In this work, biochar was produced from rice husk and was characterized. The results of the characterization were also presented.

Materials and Methods

Material

The biomass used in this work is rice husk. It was obtained from The Rice mill Zil Merchandize at Ungwan Muazu in Kaduna South Local Government Area of Kaduna State.

The rice husk obtained was sorted to remove unwanted materials such as stones, wooden sticks, papers and polythene leather. It was then dried under sun to remove moisture content. The rice husk was pyrolized to form Biochar through the procedure bellow:

Pyrolysis

2.0 kg of rice husk was measured and placed inside a pyrolyser drum (see fig. 1) and the process was carried out at a temperature of 500 ^{0}C under limited supply of air for 1hr 30 min. The biochar produced was then removed and quenched. The biochar was then placed inside desiccators and allowed to cool; it was later packaged in air tight polythene containers for characterization.

Figure 1: Pyrolyser drum

Biochar Characterization

The produced biochar was characterized to determine the quality and its properties. The characterization process included both the physical characterization: (scanning Electron Microscopy (SEM) analysis, particle size distribution, moisture content, ash content, bulk density and pore volume), and the chemical characterization: (Brunauer Emmett Teller (BET) analysis, and X- ray Diffractometer (XRD) for elemental analysis and pH measurement).

Moisture Content

2g of the biochar was dried in an oven for 1 hour at 120 ^{0}C until weight of Sample becomes constant. The moisture was determined using the initial and final weight of sample.

$$Xo = \frac{W1 - W2}{W1} \times 100\ \%$$

Where:

Xo is moisture content in percentage.

W1, W2 are initial and final weight of sample respectively

Ash content

2g of the biochar sample was put into a crucible and then placed in muffle furnace and heated to a temperature of 500 ^{0}C for 4 hr. The ash was then removed and allowed to cool in a desiccator before weighing. The percentage ash content was calculated as follows:-

$$\text{Ash (\%)} = \frac{W1 - W2}{W1} \times 100$$

Where:

W1, is the weight of the biochar and W2 is the weight of ash.

Pore Volume

2g of the biochar sample was transferred into a measuring cylinder to get the total volume of particles. It was then transferred into a beaker and 5ml of distilled water was added. The biochar was then filtered and weighed. The pore volume was determined using the relation:

$$\text{Pore volume} = \frac{W2 - W1}{\rho w}$$

Where:

W1, W2 are the initial and final mass of the particle (g), and ρw is the density of water.

Bulk Density

20g of biochar was dried at 50 ^{0}C inside an electric oven for 6 hr. 10g of dried biochar was placed inside a 20ml measuring cylinder to determine the volume occupied. The cylinder was tapped for about 2 min to compact the biochar and the bulk density is calculated using the formula:-

$$\text{Bulk density (\%)} = \frac{\text{Weight of dry material (g)}}{\text{Volume of packed dry materials (ml)}} \times 100$$

Particle Size Analysis

The biochar sample was placed inside a ball milling machine and milled for 10min. The sample was then placed into a vibrator sieve trays to obtain particle size of 450 microns.

pH measurement

2g of biochar was placed in a beaker and 20ml of distilled water was added to form a suspension. It was then heated to 100 ^{0}C with stirring for 20 min. The suspension was then allowed to cool to room temperature and the pH was measured using pH meter.

Scanning Electron Microscope (SEM)

The surface and microstructure analysis of bio char produced from rice husk was carried out using a SEM. The bio char was mounted onto the SEM stubs (Layered with sticky carbon tape). The stub was then placed in a sputter coater for five minutes for coating with gold to provide high reflectively during the scanning process. The sample was then placed in an oven and heated for 10min at 40 ^{o}C before SEM analysis.

Percentage Yield

The mass of the raw biomass (rice husk) was taken before carbonization/Charring using weighing balance. After pyrolysis, the mass of biochar formed was also taken and the percentage yield was determined using the fglormula:

$$\text{Yield (\%)} = \frac{\text{mass of biochar}}{\text{mass of raw material used}} \times 100$$

Results and discussion

The results of the biochar characterization are presented in Tables.

Table 1: Physicochemical properties of rice husk biochar

1	**Yield**	**%**	**49**
2	Moisture Cont.	%	11
3	Ash Cont.	%	17
4	Pore Volume	cc/g	0.143

5	Bulk Density	g/cm^3	0.143
6	Particle Size	μm	350
7	pH		8.4
8	Surface Area	m^2/g	102.3
9	Pore Size	nm	3.681
10	Fixed Carbon	%	46
11	Adsorption Capacity	mg/g	0.894

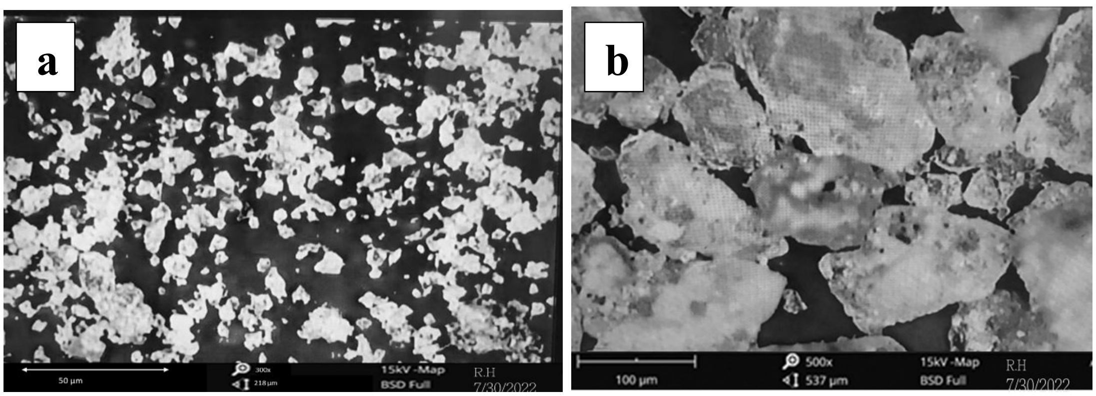

Figure 2: SEM images of the bio char (a) mag. ×300 (b) mag. ×500

Discussion of Results

The results of the physicochemical analysis of the biochar produced from rice husk carried out are presented in table 1.

The yield of biochar obtained from the pyrolysis of the rice husk is 49%. The pyrolysis was done at 500 ^{0}C and the heating rate was 30 ^{0}C/min. temperature of pyrolysis has been reported to be main factor influencing the yield (Angin, 2013). He reported that the yield of the biochar decreases with increase in temperature of pyrolysis. The nature of feedstock has also been identified as a co-factor influencing the yield. Benavente *et al.,* (2018) suggested that the interplay of temperature and the composition of the feedstock: (hemicellulose, cellulose and lignin and moisture content) influences the yield of the biomass.

The bulk density of the biochars is 0.143g/cm^3. Shaaban *et al.,* (2014) reported that lower pyrolysis temperatures produce low bulk density. Bulk density of the biochar is significant in terms of its application. The slow pyrolysis of the rice husk to form the biochar at 500 ^{o}C can thus be said influence the bulk density obtained. When biochar is being used as soil conditioner, lower bulk density will have ramifications, increasing soil porosity and soil aeration, and potentially leading to a positive effect on microbial respiration. The bulk density of the biochar makes it suitable for soil conditioning.

The surface area of the biochar is 310.3 m^2/g surface area is also reported to increase with higher pyrolysis temperature (Abdul-Fattah *et al.,* 2015). The composition of the feedstock and their interaction with temperature are significant factors influencing the surface area. Researches have shown that the higher the surface area the better the adsorption capacity of the biochar.

The moisture content of the biochar is 11%. Research has shown that higher moisture levels are obtained at higher pyrolysis temperature. The more moisture at higher pyrolysis temperature was suggested to be absorbed from the atmosphere due to higher surface area of the biochar. Biochars with higher moisture levels are however an advantage for soil conditioning.

The ash content of the biochar was found to be 30%. Higher pyrolysis temperature has been reported for higher as content, (Hamid *et al*., 2014). Ash represents the largely inorganic fraction that cannot be burnt off, including potassium (K), calcium (Ca), magnesium (Mg), and heavy metals. Ash components hinder the formation of aromatic structures that contributes greatly to fixed carbon contents.

The fixed carbon content of the biochar is 46%. Feedstock has been found to be the main factor that determines the percentage fixed carbon of boichar, (Zhao et al., 2013). At higher pyrolysis temperature, more volatiles and gases are released which results into increase in fixed carbon. High carbon content makes the biochar have more capacity to adsorb toxins or waste particles across the pores (Aydin et al., 2008).

The pore volume and pore diameter of the biochar were found to be 0.143cm^3/g and 3.681nm respectively. The pore volume and pore size determines the kind of particles that can be adsorbed by the biochar and those that will just pass through based on their sizes. This is important consideration in the aspect of purification of gases and separations, water treatment and purification of liquids.

The pH of the biochar was found to be 8.4. This is slightly alkaline. There is a significant positive correlation between pyrolysis temperature and pH (Atkinson *et al.,* 2010). The separation of alkali salts (Shinogi & Kanri, 2003), and the loss of acidic functional groups (Jiang *et al.,* 2014) are suggested to be the reason for increase in the pH of biochar. Increasing the pyrolysis temperature has been reported to tend to increase alkalinity in biochar which helps in soil conditioning.

The pore characteristics of the biochar are shown in figure 2 as analyzed using scanning electron microscope (SEM). The porous structure of macropores and mesopores indicates the ability of the biochar to be utilized as a low-cost natural adsorbent for purification, separation, water treatment and soil remediation and conditioning processes.

From the BET results, the adsorption capacity of the biochar was evaluated to be 0.894mg/g. This shows that the biochar has a good adsorption capacity and can be used in various applications such as in purification of portable water, purification of oils, gas separation and waste water treatments.

CONCLUSION

The yield of the biochar obtained was 49% at pyrolysis temperature of 500 ºC, this is relatively good as compared to 51% yield from literature. The results of the biochar analysis shows that the surface area is 310.3m^2/g which indicates a large area to adsorb pigments, elements, nutrients or pollutants for purification, separation or soil conditioning processes. The pore volume and pore diameter of the biochar were found to be 0.143cm^3/g and 3.681 nm respectively, this shows that it can be used in the adsorption of very tiny elements such as heavy metals, gases, color and odor pigments. The pH of 8.4 shows that the biochar is slightly alkaline mwhich implies that it can be used for soil pH adjustment and conditioning. The adsorption capacity was found to be 0.894mg/s showing a high adsorption potential of the biochar. The results of the biochar analysis have shown that the biochar can be used as low cost adsorbent in gas separation, water purification, wastewater treatments, vegetable oil purification and soil amendment/conditioning processes. The biochar can also be a useful precursor in the production of the modern day chemical biochar based fertilizer.

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