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Investigating Sugarcane Bagasse Based Biochar for Supercapacitor Electrodes

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ABSTRACT
Nigeria has substantial biomass potential of about 144 million tonnes per year; Biomass-derived chars are stable solid materials with high carbon content, low density, high porosity and large surface area. Biochars are recently receiving particular attention in energy storage and conversion applications such as biochar for supercapacitors electrodes. Sugercane bagasse was pyrolyzed at temperature of 200 oC in a modified microwave oven to produce a biochar in which its properties were investigated for possible use in supercapacitor electrodes. SEM, EDX and BET methods were adopted for characterization. The biochar obtained is approximately 17% of the biomass weight. The SEM-EDX results revealed the presence of various similar surface elements the elements with higher percentage in the biochar are carbon and nitrogen with percentage of 81.45% and 78.59% respectively. The BET results shows that the biochar surface area, pore volume and pore radius found to be 5.42 m2g-1, 3.33 cc g-1 and 2.07nm respectively. The study shows that the produced biochar has some qualities which may found its applications in soil fertility improvement, enhanced soil structure, carbon sequestration, pH regulation, pollution mitigation, microbial activity enhancement, etc. This is due to its elemental composition and structure possessed at this pyrolysis temperature. These findings suggest that electrodes in supercapacitor application should be pyrolyzed at temperature greater than 200oC for the biochar to be effective in supercapacitor applications.

Keywords:
Biochar,
Biomass,
Electrodes,
Pyrolysis,
Supercapacitor.

INTRODUCTION

The demand for renewable energy sources worldwide has gained tremendous research attention over the past decades. Technologies such as wind and solar have been widely researched and reported in a literature. However, economical use of these technologies has not been widespread due partly to cost and the inability for service during off-source periods. To make these technologies more competitive, research into energy storage systems has intensified over the last few decades. The idea is to devise an energy storage system that allows for storage of electricity during lean hours at a relatively cheaper value and delivery later (Kwadwo et al., 2019).

Energy storage and delivery technologies such as supercapacitors can store and deliver energy at a very fast rate, offering high current in a short duration. The past decade has witnessed a rapid growth in research and development in supercapacitor technology. Several electrochemical properties of the electrode material and electrolyte have been reported in a literature (Jemima et al., 2012).

Supercapacitor electrode materials such as carbon and carbon-based materials have received increasing attention because of their high specific surface area, good electrical conductivity and excellent stability in harsh environment etc. In recent years, there has been an increasing interest in biomass-derived activated carbons as an electrode material for supercapacitor applications. The development of an alternative supercapacitor electrode material from biowaste serves two main purposes (Kwadwo et al., 2019). It helps with waste disposal; converting waste to a useful product, (Juliet et al., 2016) and it provides an economic argument for the substantiality of supercapacitor technology (Kwadwo et al., 2019).

Biomass is abundant in nature and broadly dispersed globally with its distribution being dependent on geographical area. Country such as Nigeria has significant natural resources to produce transportation biofuels, biopower and bioproducts from biomass. Nigeria has substantial biomass potential of about 144 million tonnes per year, most Nigerians, especially rural dwellers, use biomass and waste from wood, charcoal., and animal dung, to meet their energy needs. Biomass (comprising crop residues, manure, charcoal., and wood) accounts for about 80% of the total primary energy consumed in Nigeria, oil (13%), natural gas (6%) and hydro (1%). This large percentage represents biomass used to meet off-grid heating and cooking needs in the rural areas (Juliet et al., 2016).

Various researches have been conducted and would continue for the struggle to find the best materials for supercapacitor electrodes. Some of the researches published in literature are the conversion of Banana fibers, Neem leaves, Rice husk, and Sugarcane bagasse into biochar electrodes for supercapacitor having the surface area of 1097, 1230, 1442 and 1788 m^2g^{-1} respectively (Talam et al., 2017). The theory adopted in this research is to involve microwave heating at highest temperature of 200°C to decompose organic material or compound due to the applied heat, in the absence of oxygen or other reagent.

In this research, biochar from sugarcane bagasse available in northern Nigeria (obtained locally from sugarcane sellers in Gayawa quarters, Ungogo local government area, Kano state.) was produced at 200°C pyrolysis temperature using microwave pyrolysis machine for investigating suitable biomass precursor materials with better qualities for supercapacitor electrodes application.

MATERIALS AND METHODS

Agricultural sugarcane bagasse was obtained locally from sugarcane sellers in Gayawa quarters, Ungogo local government area, Kano state, Nigeria. The sugarcane bagasse used was that of black stem sugarcane with scientific recognition as Saccharum officinarum. The selection of this feedstock for biochar production was based on their availability as waste materials.

Experimental set-up

A microwave-assisted pyrolysis system, as shown in Figure 1, was constructed for the experiments. The microwave oven modified was Hisense product with model number (H20MOW). The Microwave frequency and the maximum output power of the system were 2.45 GHz and 700 W, respectively. The microwave oven was equipped with a process controller which enables accurate timing control of the process. The process controller was linked to a RKC temperature controller with model C100FD00-M*AN and temperature range 0-400°C to allow easier programming and data processing. Temperature was measured by inserting the tip of an ungrounded K-type thermocouple (model WRNT-02, Chanpin Hegezheng, DGDR) to the center of the biomass sample held in a crucible. To create and maintain an inert atmosphere required for pyrolysis, Nitrogen gas of high purity (99%) was purged into the reactor at 0.5 mL/min starting at least 5 min before the experiment and lasting 5 min after the completion of the experiment (Kwadwo et al., 2019).

Figure 1: Picture of Modified Microwave Oven for Pyrolysis

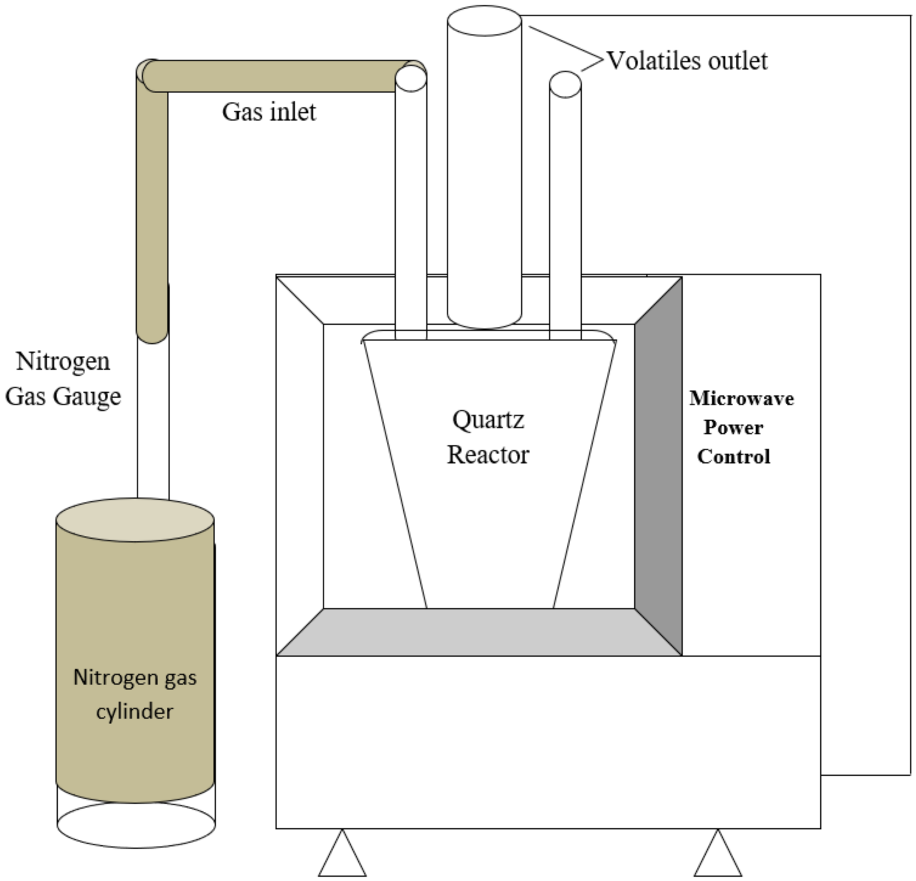

Figure 2: Experimental Set-up for Microwave Oven Pyrolysis

Pyrolysis Procedure

30.0g of sugarcane bagasse biomass sample was placed in crucible. The thermocouple was inserted to the center of the biomass sample; the pyrolysis temperature was set to 300^{o}C after which the oven was switch-on with maximum operating power of 700W. The temperature was ramped from room temperature to the temperature of 200^{o}C at pyrolysis time of 60minutes with residence time of 10minutes. After the experiment, the crucible and its contents was allowed to cool. Thereon, the sample was measured to be 5.0g.

Biochar Yield Method

The biochar yield after pyrolysis was estimated through percentage ratio of biochar mass to feedstock mass (Juliet et al., 2016).

$$\text{Yield } \% = \frac{M_{biochar}}{M_{biomass}} \times 100 \qquad (1)$$

Where, $M_{biochar}$ and $M_{biomass}$ is the initial mass of biomass and final mass of biochar respectively.

Chemical and Structural Analysis Methods

The biochar and biomass samples were taking for determination of chemical and structural composition. Both samples were subjected to Scanning Electron Microscopy (SEM) in combination with Energy Dispersive X-ray (EDX) and Brunauer-Emmett-Teller (BET). Different BET methods were applied to the samples including; Single point, multi point, Barret-Joyner-Halenda (BJH), Dollimore-Heal (DH), Dubinin-Radushkevich (DR), t-Plot method, Density Functional Theory (DFT) and Langmuir method, Harkins-Kura (HK), Saito-Foley (SF) and Dubinin-Astakhov (DA) method for the determination of surface area, pore volume and pore diameter of the samples. The machine used for SEM analysis was Quorum Technologies Model Q150R while the machine employed for BET was a made in United State of America NOVA 4200E-series.

RESULTS AND DISCUSSION

The Biochar Yield

As 30g of biomass was used for the conversion, a 5.0g of the final product was obtained. The mass loss of the biomass is due to the released of various volatile compounds from the bagasse. The specific volatile compounds typically lost during the pyrolysis process include: water (H_2O), Acetic acid (CH_3COOH), Methanol (CH_3OH), Aldehydes (CH_2O), Phenolic compounds, Volatile fatty acid, Light hydrocarbons, Ammonia (NH_3) and Carbon Dioxide (CO_2) (Talm 2017; Mohammed 2023). This gives the yield of 16.67% char product according to (Kwadwo et al., 2019). This yield indicates that a relatively small portion of the biomass is converted into stable biochar possibly due to pyrolysis temperature impact, as the pyrolysis temperature of 200^{o}C is relatively low for the optimal production of biochar. This implies that raising the temperature to a certain threshold could potentially increase the biochar yield and improve the quality of the product (Juiet et al., 2016).

Chemical and Structural Analysis

Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX)

Two samples SB000 and SB200 were taken for characterizations, the SB000 (assigned as sample B) denotes Sugarcane Bagasse produced at zero pyrolysis temperature which is simply refers to the biomass materials while the SB200 (assigned as sample A) denotes the Sugarcane Bagasse produced at 200^{o}C pyrolysis temperature. SEM, in conjunction with EDX analysis, can be utilized to investigate the composition and properties of biochar (Juiet et al., 2016). SEM–EDX analysis is valuable for identifying the different components present on the surface of biochar. This technique has been frequently utilized in research on biochar applications to assess the biochars surface characteristics. The SEM-EDX analysis revealed the presence of various compounds on the surface of produced biochar, including carbon (C) and Nitrogen (N) as major compounds with little amount of Silicon (Si), Aluminium (Al), calcium (Ca), potassium (K), Sodium (Na), Sulphur (S) and Magnesium (Mg) in sample A (i.e. SB200), (Fig. 2,Table 1). For sample B (i.e. SB000), the elemental compositions includes; carbon (C) and Nitrogen (N) as major compounds with trace amount of Silicon (Si), Aluminium (Al), potassium (K), Sodium (Na), Chlorine (Cl) Magnesium (Mg) and Phosphorus (P) as shown in figure 3, and Table 1.

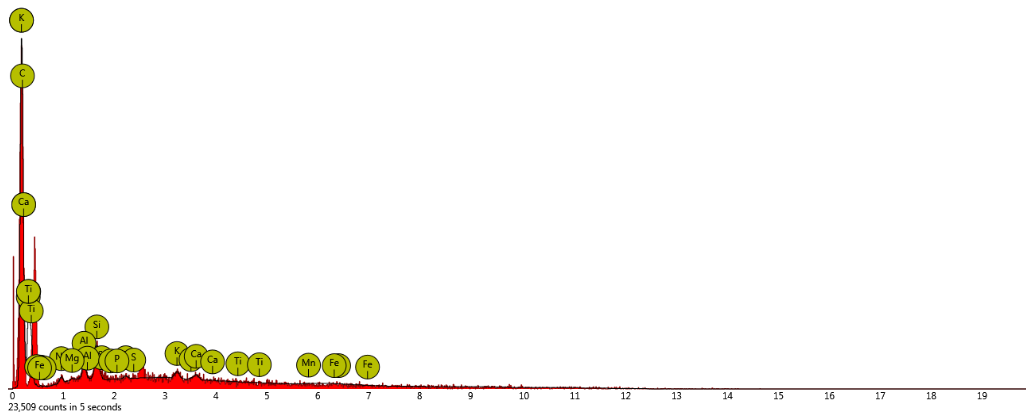

Figure 3: The plot of elemental composition for pyrolyzed sugarcane bagasse

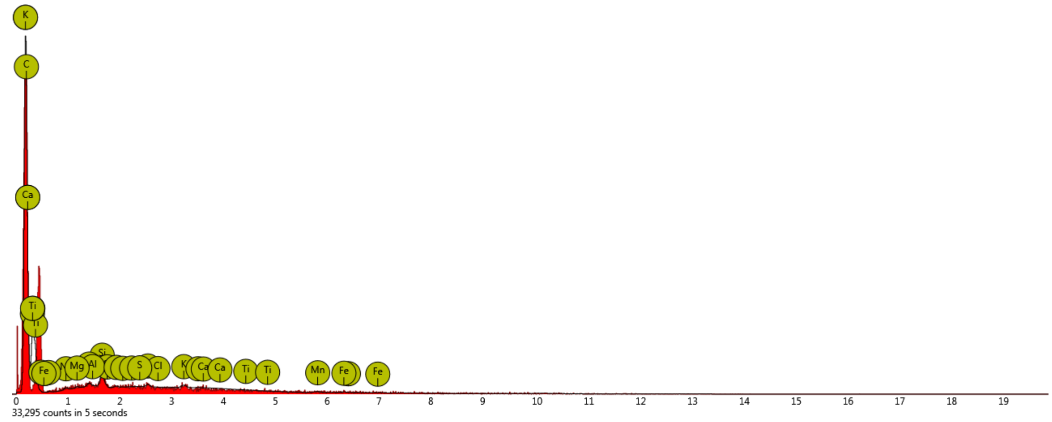

Figure 4: The plot of elemental composition for sugarcane bagasse biomass

Table 1: Elemental Composition (Weight Concentration %) of Biochar and Biomass of Sugarcane Bagasse

Element	SB000	SB200
C	81.45	78.59
N	14.11	13.84
Si	1.27	2.88
Al	0.34	1.47
K	0.45	1.14
Ca	---	1.04
Na	0.32	0.65
S	---	0.28
Mg	0.16	0.11
Ti	---	---
Mn	0.89	---
Fe	0.58	---
P	0.13	---
Cl	0.30	---

From the result of table 1, the concentration of C and N elements is typically higher in SB000 compared to SB200 due to several factors related to the pyrolysis process and the chemical composition of the biomass. For the chemical composition of the biomass, it contains a significant amount of organic compounds, including cellulose, hemicellulose and lignin, which are rich in carbon and nitrogen. These compounds contribute to the overall higher concentrations of these elements in the raw biomass compared to the produced biochar (Talam et al., 2017).

During pyrolysis, biomass undergoes thermal decomposition, which leads to the loss of volatile compound including gases and tars. This process results in a reduction of the overall mass and alters the chemical structure of the biomass, leading to a decrease in the concentration of nitrogen and other elements in the resulting biochar.

At lower pyrolysis temperatures, such as 200°C, the decomposition of organic matter is not complete, and some nitrogen-containing compounds may still be present in the biochar. However, the overall carbon content in biochar tends to be lower than in the original biomass due to the loss of volatile organic compounds during the pyrolysis process (Talam et al., 2017). The presence of some elements such as manganese, iron, and phosphorus in sample SB000 and not available in sample SB200 while the presence of calcium and sulfur in SB200 and not found in SB000 is due to their volatility, transformation processes and the incomplete conversion of biomass to biochar (Noor, 2017; Van Loo, 2018; Liu et al 2019)

In the case of surface morphology, it is observed (figure 4.) that both samples (SB000 and SB200) have cracks on them which suggest possession of brittleness properties, but the presence of larger cracks in SB000 is more pronounced than in SB200, which suggest differences in materials properties. The differences between the samples in terms of crack density and surface roughness indicates variations in composition, processing conditions or environmental exposure (Cha et al., 2016).

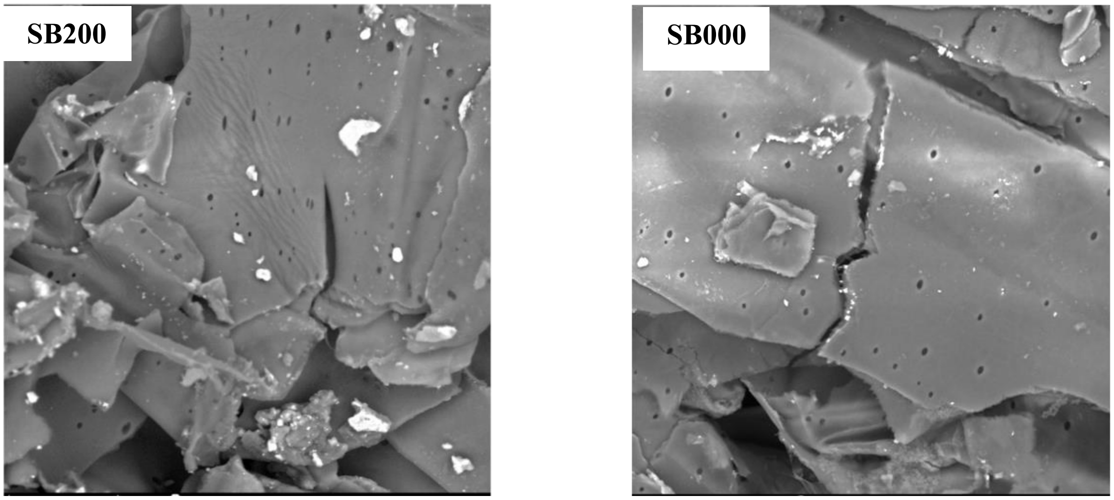

Figure 5: SEM morphology of sample A (SB200) and sample B (SB000); Magnification; ×2000

Brunauer Emmett Teller (BET) Result Analysis

The BET was applied to obtain surface area, pore volume and pore radius of the samples. The Multi-point BET shows that SB000 and SB200 have the surface area of 8.92 and 5.42 m^2g^{-1} respectively (Table 2). The higher surface area of the raw sugarcane bagasse suggests that it has a more porous structure compared to the biochar produced through pyrolysis. Pyrolysis typically leads to the collapse of some of the pores, resulting in a lower surface area in the final biochar product. The pyrolysis temperature at 200°C may not be sufficient to create a highly porous structure in the biochar. Generally, higher temperatures enhance the development of porosity and surface area due to the breakdown of organic materials and the release of volatile compounds (Muhd et al., 2015). The surface area is a critical factor influencing the adsorption capacity of biochar. A lower surface area in the biochar suggests that its ability to adsorb electrolytes for supercapacitor applications is low.

Table 2: Surface Area, Pore Volume and Pore Radius of SB000 and SB200

Samples	Surface area (m^2g^{-1})	Pore volume (cc g^{-1})	Pore radius (nm)
SB000	8.92	5.51	2.09
SB200	5.42	3.33	2.07

In the case of pore volume, the Barrett-Joyner-Halenda (BJH) method was employed due to its suitability for characterizing mesoporous materials. The data in table 2 shows that the pore volume of biomass is greater than that of biochar, this decrease in volume suggests that the pyrolysis process leads to a loss of some of the original pore structures. This is typical as the thermal decomposition of organic materials can result in the collapse of pores and reduction in overall porosity (Muhd et al., 2015).

Furthermore, the pyrolysis at 200°C may not be sufficient to create a highly porous biochar, higher temperatures promote the formation of more stable carbon structures and can enhance pore volume (Muhd et al., 2015). Pore volume is a critical factor influencing the adsorption capacity of biochar. A lower pore volume in the biochar suggests that its ability to adsorb gases, liquid or contaminants may be diminished compared to the raw biomass (Poonam et al., 2015). This can affect its effectiveness in supercapacitor electrodes application.

Lastly, the table 2 data gives the pore radius of SB000 and SB200 as 2.09 and 2.07nm respectively. The close values of pore radius indicate that the overall pore structure of the biomass is largely retained during pyrolysis at 200°C. This suggests that the thermal treatment does not significantly alter the size of the existing pores, which is consistent with findings that lower pyrolysis temperatures may not lead to substantial changes in pore dimension (Talam et al., 2017). At 200°C, the pyrolysis process may not be sufficient to cause significant thermal degradation or restructuring of the biomass. Research indicates that higher temperatures are typically required to induce more extensive changes in pore size and volume due to the breakdown of organic materials and the release of volatile compounds (Liu at al., 2019). The slight decreased in pore radius could indicate that further pyrolysis at higher temperatures might lead to pronounced changes in pore structure, potentially enhancing the porosity and surface area of the biochar. Studies suggest that optimizing pyrolysis conditions can significantly improve the structural properties of biochar (Talam et al., 2017; Noor, 2017; Yaning et al., 2017)

CONCLUSION

Biochar from sugarcane bagasse was produced at 200°C pyrolysis temperature using microwave oven. Although, the intent pyrolysis temperature is a higher temperature but due to constraint from the microwave oven which during the pyrolysis process enter thermal shutdown whenever the temperature of the biomass inside the reactor attained 200°C. This problem is due to fact that the magnetron of the microwave oven is not capable of withstanding the thermal energy generated during the pyrolysis process, therefore, in order for the magnetron not to undergone thermal destruction; the microwave thermal switch trips whenever the magnetron attains the stipulated temperature. This problem can be avoided by using a microwave oven with better features than the model employed in this work. However, despite the limitation of the microwave oven used, the produced char at pyrolysis temperature of 200°C may not found its applications in supercapacitor electrodes due to the low surface area, low Pore volume and low Pore radius. Because the requirements for a biochar to be use as supercapacitor electrodes should have a fairly large surface area (≥1000m^2g^{-1}), pore volume (>2nm for micropores, 2≤5nm for mesopores) and pore radius. Nonetheless, the produced biochar has some qualities which may found its applications in soil fertility improvement, enhanced soil structure, carbon sequestration, pH regulation, pollution mitigation, microbial activity enhancement, etc. This is due to its elemental composition and structure possessed at this pyrolysis temperature. Ultimately, in this work, the investigation revealed and confirms that a biomass that intent to be use as electrodes in supercapacitor application should be pyrolyzed at temperature greater than 200°C for the biochar to be effective in supercapacitor applications. Future research should devise a microwave oven with better features and performance that supersede the type used in this work. This will enable the pyrolysis temperature to attain the desired temperature and obtain a biochar with better quality for use as supercapacitor application.

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