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Effect of Thermal Stagnation on the Pore Evolution and Fuel Properties of Microwave-Pyrolyzed Rice Husk

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Abstract- This study evaluates the potentiality of rice-husk biochar produced by microwave pyrolysis for its suitability use as fuel-energy. The microwave pyrolysis temperature set to 350oC with maximum operating power of 700W and residence time of 30 minutes, the sample was thermally stagnated at pyrolysis temperature of 200oC, Thermogravimetric analysis (TGA) and structural analysis using Brunauer-Emmett-Teller (BET) was conducted on the sample to explore its weight loss at stagnated temperature, surface area and pore structures respectively. The surface area was found to be 2.76×102 m2g-1, pore volume of 3.191419 × 10-1cc/g and pore size of 1.8916 nm. The TGA result in the temperature range 30oC to 200oC shows the mass loss of 2.19%. It is evaluated that rice husk treated at this pyrolysis temperature (200oC) has faster ignition and more stable burning in direct-heating appliances, reduce problems linked to wet fuel (steam quenching, smoky start-ups). This sample can be blended with coal in existing power plants or direct cooking; this reduces carbon emissions and utilizes the high volatile content of the RH to improve the ignition characteristics of the coal.

Keywords: Biomass, Pyrolysis, Microwaves. Thermal-stagnation Fuel-energy

I. INTRODUCTION

Energy produced and used in ways that support human development in all its social, economic and environmental dimensions is what is meant by sustainable energy [1]. In 2017, the International Energy Agency estimated that 1.1 billion people do not have access to electricity and more than 3 billion people rely on the traditional fuelwood for cooking, mostly using inefficient stoves in poorly ventilated spaces [2]. In Nigeria, the National Bureau of Statistics estimates that 55% of Nigerian lack access to electricity and those who have access suffer erratic supply, Furthermore, the World Energy Outlook 2012 reported that of 80 countries, Nigeria ranks 66th with an energy development index (EDI) of 0.11, such lack of access to modern energy limits income generation and creates a vicious cycle of deprivation that trap people in poverty and any projected economic growth could be hindered [2].

In Nigeria, there is an energy source that is almost never discussed and is not contained in any government energy policy document–energy generation from waste. Any waste treatment process that generates energy in the form of electricity, heat, or transport fuels is considered as energy recovery [2]. Nigeria has substantial biomass potential of about 144 million tons per year [3]. The Nigerian environment contains enormous amounts of waste: municipal solid waste (MSW), food waste, industrial waste, and animal waste, and these are major problem in the country if they are not utilized as energy recovery.

Biomass is an abundant organic material that, in addition to its use as a fuel, can be upgraded to generate biochar. Biochar is a porous carbonaceous solid produced when organic biomass is heated in a closed container with no or limited oxygen supply [4]. Chars can be shaped into different forms (pellets, briquettes …) to be easily transported [5].

Chars can be produced by various thermal processes with restricted oxygen supply; the most common production techniques are pyrolysis that could be carried out in pyrolizers. Microwave pyrolysis is another technique that has been recently applied at laboratory scale, this technique is characterized by the presence of hot spots in the biomass, and brings to chars with higher heating value (HHV) and specific surface area than those obtained by conventional pyrolysis [5]. Pyrolysis is a thermo-chemical process in which organic material is converted into a carbon-rich solid (char) and volatile matter (liquids and gases) by heating organic solid waste in the absence of oxygen [6]. The main operating parameters in pyrolysis are heating rate, pyrolysis temperature, and residence time [7].

Various researches have been conducted in which biochar from biomass was produced at different pyrolysis temperature. The high heating values (HHV) of biochar produced at 250, 350 and 450°C were 24, 23.64 and 23.08 MJ kg-1 [4], while the HHV for coal vary between 14 and 35 MJ/kg [8]. This characteristic of biochar along with the high tendency of slagging indicates that biochar could be used as a source of energy [4]. This study aims to evaluate the potentiality of rice-husk biochar produced by microwave pyrolysis method for a sustainable source of fuel-energy.

II. MATERIALS AND METHODS

Sample collection

The rice-husk is collected from local rice millers in Kura local government town, Kano state. The selection of this feedstock for biochar production is based on their availability as waste materials.

Experimental Set-up

A modified microwave-assisted pyrolysis system was used for the experiments. The microwave oven product is LG with model MS2044DMB having microwave frequency and maximum output power of 2.45 GHz and 700W, respectively. The microwave oven was equipped with a process controller which enables timing control of the process. The process controller has a temperature detection range 0-400°C to allow easier programming and data processing. Temperature was measured by inserting the tip of an ungrounded K-type thermocouple 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%) purges into the reactor at 0.2 mL/min [9].

Pyrolysis Procedure

A rice-husk biomass sample weighted 50.0g of was placed in crucible. The thermocouple was inserted to the center of the biomass sample; the pyrolysis temperature was set to 350°C with maximum operating power of 700W. The temperature was gradually attained the temperature of 200°C within 60 minutes with residence time of 30 minutes. No increased in temperature was observed, the temperature remained within 200°C. After the pyrolysis, the crucible and its contents was allowed to cool. Thereon, the sample was measured to be 46.0g.

Product Yield

The product yield after pyrolysis was calculated through percentage ratio of biochar mass (pyrolyzed sample) to biomass [10].

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

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

Structural and Thermogravimetric Analysis (TGA)

The biomass product (biochar) was subjected to Brunauer-Emmett-Teller (BET) and Thermogravimetric Analysis (TGA). 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) to examine the surface area, pore volume and pore diameter of the samples while TGA is meant to determine the derivative mass loss of the sample.

III. RESULTS AND DISCUSSION

Pyrolysis Result

The specific experimental observation is the complete thermal stagnation of rice husk at 200°C for a 30-minute residence time, despite continuous application of 700 W microwave power. This phenomenon is dictated entirely by the dielectric properties of the material, the heat generated internally within the material (P_G) often referred to as volumetric power dissipation, is governed by the relationship:

$$P_G = 2\pi f \varepsilon_0 \varepsilon'' E^2 \qquad (2)$$

where f is the microwave frequency (typically 2.45 GHz), ϵ_0 is the permittivity of free space, ϵ'' is the dielectric loss factor, and E is the electric field strength. The heating efficiency relies critically on ϵ'', which quantifies the material's ability to dissipate electromagnetic energy into heat [11].

In the raw or wet state, the rice husk's initial heating up to 100°C and beyond is driven by the high dielectric loss of free and bound water. The high ϵ'' provided by the moisture ensures efficient coupling of the 700 W microwave power, leading to a relatively rapid temperature rise toward 200°C. As the temperature approaches 200°C, the majority of the free and easily removable bound water has been vaporized. The remaining material is largely dry lignocellulose (cellulose, hemicellulose, and lignin), which is inherently a low-loss dielectric material. The instantaneous heat generation rate (P_G) plunges sharply because the primary heating mechanism (dipolar polarization of water) has been depleted, and the secondary heating mechanism (conductive losses from char) has not yet been established. The rice husk transitions from an efficient absorber to a material that is largely "electromagnetic-wave transparent" in the relevant frequency range. This transition creates a dielectric minimum trap where the rate of heat generation collapses, making it impossible for the continuous 700 W input to raise the material temperature further.

To overcome the 200°C plateau and resume heating, the system requires a fundamental change in the material's dielectric properties, specifically a dramatic increase in ϵ'', which necessitates sufficient internal heat generation to accelerate pyrolysis. The most established method to circumvent the low ϵ'' trap is the introduction of a material with high intrinsic dielectric loss (a susceptor or absorber). Since biochar is known to be a good microwave absorber, mixing a small percentage of pre-made biochar (often referred to as 'char seeding') with the raw rice husk feedstock provides an immediate conductive heating pathway.

The susceptor absorbs the 700 W efficiently, generating intense localized heating ("hot spots"). This concentrated heat is then transferred to the surrounding low-loss dry rice husk by conventional conduction and convection, forcing the local temperature of the rice husk past the critical 250°C threshold required for its own char nucleation. This bypasses the inherent dielectric limitation of the raw biomass, enabling the 700 W input to become highly effective almost immediately.

Biochar Yield

Out of 50g of biomass used for the conversion, the final product obtained was 46g. This gives the yield of 92% of the product according to equation (1), and 8% of initial mass loss. The mass loss at the first stage (50–200°C) was mainly related to removal of water, CO_2, CO, and other small molecule volatile compounds [18]. As the product did not attain the second phase of pyrolysis (i.e. thermal decomposition of the product in order to produce the char) due to the problem mentioned in section (4.01) above, the product sample was further characterized and analyzed for possible useful applications.

Brunauer Emmett Teller (BET)

Different BET methods were applied to determine the structure which includes; surface area, pore volume and pore radius of the biochar.

Surface area

The average surface area of the sample was found to be 2.76×10^{2} m^2g^{-1}, this value is significantly higher than what is typically reported in scientific literature for rice husk treated at only 200°C without any chemical treatment using microwave pyrolysis. In some previous studies, BET surface area of rice husk was reported to be 172.04 m^2/g above the microwave pyrolysis temperature of 450°C and residence times (5–15 min) [12], while 190 m^2/g was obtained at microwave pyrolysis temperature of 600°C in combination with Predetermined quantity of granular activated carbon, GAC (rice husk: GAC as 5:1) which was added as microwave absorber to enhance the pyrolysis process temperature [13], in another study revealed by [14], a BET surface area 217.03 m^2/g rice husk was obtained with the sample undergone water washing–torrefaction pretreatment prior to the microwave pyrolysis process, the pyrolysis temperature was controlled at 550°C with 20 minutes of residence time [14]. At 200°C, rice husk undergoes a process called torrefaction (mild pyrolysis). Even though it has not yet reached the high-carbon "charcoal" state, it is a superior fuel compared to raw rice husk for several reasons [15]: (1) Increased

Energy Density: At 200°C, the material loses moisture and oxygen-rich volatiles. This concentrates the carbon, increasing the Higher Heating Value (HHV). (2) Hydrophobicity: Torrefied rice husk does not absorb water from the air, making it more stable for storage and transport. (3) Grindability: The heat breaks down the fibrous structure, making it easier to pulverize for use in industrial furnaces or power plants. The surface area of 2.76×10^{2} m^2g^{-1} at 200°C is exceptionally high for that temperature, which suggests the material might be even more valuable as a specialized adsorbent than a simple fuel.

Pore volume

The average pore volume of the sample as determined by BET is found to be 3.191419×10^{-1}cc/g. When compared with reported values of rice husk treated at 200°C in literature, such as 0.195726 cm^3g^{-1} obtained by microwave pyrolysis of rice husk at 500ºC and the operating power of 700W [14], 0.1229 cm^3/g was also reported at microwave pyrolysis of rice husk at temperature of 450ºC, power of 1000W and holding time of 5 minutes. This lower pyrolysis temperature likely preserved the delicate silica scaffold of the rice husk, preventing the "melting" or "fusing" that happens at higher heat achieving more "holes" with less heat energy. At this temperature, moisture and some light gases (volatiles) have been removed, which opens up the pores without destroying the structural "scaffold." A biomass material with pore volume of 0.3191 cm^3g^{-1} can be effectively employ for soil amendment in terms of soil aeration, water retention, regulating of soil pH and promoting microbial activity [14, 16 & 17].

In the fuel industry, a "smokeless" fuel usually has a Volatile Matter (VM) content below 15–20%, the samples pyrolyzed at 500°C are true "Biochars." They have very low VM, so they burn with almost no visible smoke, similar to charcoal. The 200°C sample is "torrefied." It still contains a significant amount of volatile matter. Under normal conditions, it would produce some smoke. However, high pore volume (0.3191 cm^3g^{-1}) changes this. Smoke is usually caused by incomplete combustion when the fuel doesn't get enough oxygen to burn its gases, however, with high pore volume oxygen can penetrate the husk much more deeply and quickly allows those gases to mix with air and burn completely. Rice husk ash is roughly 90% silica (natural glass) [18]. At very high temperatures (typically above 700°C–900°C), this silica can melt and form a hard, glassy crust called slag or clinkers, which can ruin a stove [19]. By processing at 200°C (lower temperature), the biogenic silica in the sample remains in its amorphous (non-glassy) state [20]. During use as a fuel, this silica scaffold stays rigid and prevents the fuel from collapsing into a dense, air-blocking lump. This keeps the fire "breathing" until the very end [21].

A rice husk pyrolyzed at 200°C under microwave power of 700W with residence time of 30 minutes can be efficient for a domestic cooking fuel. It is easier to ignite and produces less total ash residue than biochar, while its superior porosity ensures that it burns much cleaner than raw, untreated rice husk.

Pore size

Statistical analyses show that, the pore size of the sample as determined by BET is found to be 1.8916 nm within the confidence interval. This reveals that micropores are developed in the husk [22]. Generally, at temperatures below 550 °C the macropore and mesopore increased with increasing temperature, while the micropore decreased. This could be due to the elimination of volatiles at higher temperatures leading to the formation of larger pores as well as the merging of micropores to form mesopores [23,24]. The pyrolysis of rice husk at this temperature (200ºC) reduces its oxygen contents but increases the carbon contents, thus increased the HHV of the husk [15].

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) stands as one of the most prevalent methodologies for investigating pyrolysis. It is frequently employed to analyze the kinetic and thermodynamic characteristics of pyrolysis reactions. TGA is a technique utilized to examine thermal decomposition behavior and conduct kinetic analysis, typically at specific temperatures and lower heating rates. By plotting temperature against mass loss under a controlled heating rate and within an inert atmosphere, TGA generates thermogravimetric (TG) and derivative thermogravimetric (DTG) curves figure 1 to 2. These curves illustrate the mass loss resulting from the chemical and physical changes that occur during the thermal decomposition of biomass [25].

The Thermogravimetric Analysis (TGA) of the sample conducted at air environment shows different distinct thermal degradation stages as it is heated from 30°C to 950°C at a rate of 10°C/min, Figure 1. The initial sample weight was 13.209 mg, and the final residue is 19.63% (2.593 mg). This results in a total weight Loss of 80.37%.

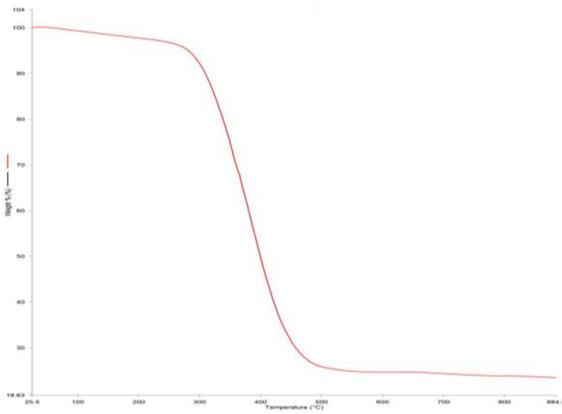

Figure 1: TGA of rice husk pyrolyzed at 200ºC

Fig. 2 illustrated that from 30 °C to 200 °C, the first mass loss of about 2.19 % (12.912mg), this weight loss is relatively low when compared to other rice husk TGA result conducted in air environment with mass loss of 9.6% in this temperature range [26]. This suggest that the sample in used was either well-dried before the analysis or has a low hygroscopic nature. But this is attributed to the mild pyrolysis (torrefaction) undergone by the sample.

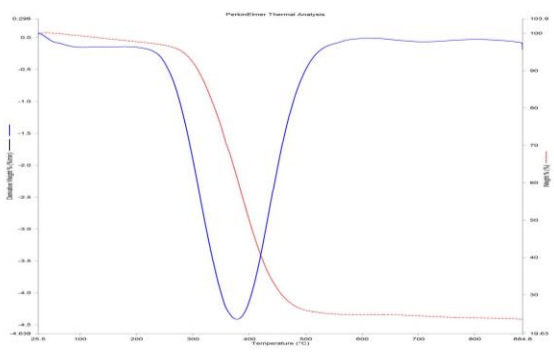

Figure 2: TGA, DTG curve of the rice husk pyrolyzed at 200ºC

IV. CONCLUSION

Rice husk pyrolyzed at 200°C under 700 W of microwave power with a residence time of 30 minutes demonstrates significant potential as an efficient domestic cooking fuel. This material is easier to ignite and produces a lower total ash residue compared to biochar, whilst its superior porosity ensures a much cleaner combustion than raw, untreated rice husk. The minimal mass loss of 2.19% up to 200°C indicates that the fuel is essentially dry; due to the mild pyrolysis at stagnated temperature of 200°C Consequently, it offers a higher net heat output, faster ignition, and more stable combustion in direct-heating appliances, thereby mitigating issues associated with wet fuel, such as steam quenching and smoky start-ups. Furthermore, the total weight loss of 80.37% suggests that the majority of the sample is combustible. This fuel is suitable for blending with coal (typically comprising 5–10% biomass) in existing power plants or for direct cooking applications. Such blending not only reduces carbon emissions but also utilizes the high volatile content of the rice husk to enhance the ignition characteristics of the coal.

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