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Thermal and Catalytic Conversion of Native Rice Husk into Bio-Oil and Biochar

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ABSTRACT

Pyrolysis process is the most common and favorable thermochemical processes to produce fuels. This research was aimed to assess the potentials of rice husk for the fuel production through pyrolysis. The proximate analysis of husk and biochar sample indicated that it has low moisture of 6.6% and ash 16.16% contents. The low values of both potassium (2033.32ppm) and Sodium (40.83ppm) obtained from this research indicates the suitability of the husk for the pyrolysis process. The percentages of liquid, gaseous as well as solid products at 350°C from both thermal and catalyzed process shows slight increase in liquid products (from 20 to 21%) and significant increase in both solid (from 45 to 62%) and gaseous (from 17.25 to 33.36%) products which can be attributed to the influence of the catalyst used. The major chemical compounds present in both thermal and catalytic pyrolysis processes are fatty acids, carbonyl compounds, alcohols and furans. Higher fatty acids and carbonyl compounds were obtained from the thermal process, while higher furans compound for catalytic process. Therefore, the native rice husk can be suitable biomass for energy conversion.

Keywords: Rice husk; proximate; pyrolysis; pyrolysis oil; trace elements

1.0 INTRODUCTION

The large interest on a bio-based economy has stimulated a fast growing research and development on the second generation lignocellulosic biomass (Gabriele *et al.*, 2011). Lignocellulose biomasses are the most abundant renewable sources of organic carbon on earth and one of cheapest biomass resource materials that are adequately available in large scale for sustainable production of biofuels. Lignocellulose biomass is non-food component of the biomass composed of three major components; cellulose (40-50%), hemicelluloses (25-35%) and lignin (15-20%) (Mamta *et al.*, 2013). The polymeric biomass can be deconstructed into reactive intermediates that can produce biofuels and bio-chemicals through biochemical and thermochemical process.

Agricultural residues are lignocellulose organic materials produced as a byproduct during the

course of harvesting and processing of agricultural crops (Mortensen *et al.*, 2011). They are classified into two categories; crop residues and agricultural industrial by-products (materials produced after crop processing). Crop residues produced during harvesting are called primary or field-based residues, while those that are produced alongside with the products during processing are called secondary or process-based residues. Depending on the mode of handling both field-based and process-based residues have high potential for energy production. These biomass residues may contain high lignin fragments, residual carbohydrates and other organic matter that need to be treated in an environmentally friendly manner so as to leave little or no ecological footprints (Duku *et al.*, 2011).

The present study was designed to determine the potentials of native rice husk sample for the pyrolysis process to produce bio-oil and biochar.

Rice residue (husk) sample was procured from Attajiri rice mill at kware local government area,

2.0 MATERIALS AND METHODS
2.1 Sample Collection and Treatment

Sokoto. The AOAC 1999 methods of analyses with amendment were employed to obtain a required sample for these analyses. The collected sample was washed with distilled water to remove debris, dust and other impurities, and then dried in a hot air circulating oven at 105°C for 24hr to remove moisture. Dried sample was grinded into powdered form and sieve using 40mm mesh then kept for further analysis. The standard procedures were adopted during the preparation of solutions and other required reagents using deionize water.

2.2 Pretreatment of Neat Kaolin

The neat kaolin clay sample was procured from kankara in Katsina State of Nigeria. Then the procured sample was pretreated to remove the impurities present by placing it in 250cm³ of 2M HCl with constant stirring for two hours, washed with distilled water then allowed to cool overnight for the suspension to settle down. Then, the kaolin suspension was washed with distilled water three times and micro sieve was used to filter the clay suspension under suction. The pretreated kaolin clay was dried at 120°C for 2h (Edomwonyi-Otu *et al.*, 2013). The CuO/kaolin catalyst was prepared according to the method adopted from Haber *et al.* (1995) and Sanjay (2013) through incipient wetness impregnation method by dissolving of Cu(NO₃)₂.5H₂O as a precursor in distilled water. After that, the appropriate amount of dissolved salt precursor was impregnated into kaolin clay pores action which serves as support. The mixture will be heated for 2h with the constant stirring of 200 rpm on a heating mantle equipped with stirrer. Then, the

$$\text{Calorific Value} = 4(\text{Crude Protein}) + 9(\text{Crude lipid}) + 4(\text{Carbohydrates}) \dots \dots \dots 2$$

The organic matter (volatile solid) was determined by subtracting the percentage moisture and Ash content from 100% (Garba, 1999).

$$\text{Volatile Matter} = 100 - (\% \text{Ash} + \% \text{Moisture}) \dots \dots \dots 3$$

The percentage carbon was estimated using equation 5 that is adopted from Abba *et al.*, (2004).

$$\% C = 0.5 \times \text{Organic Matter} \dots \dots \dots 5$$

sample will be suctioned to remove the excess aqueous solution of precursors. The mixture will be calcined at 400°C according to the Ghulam *et al.* (2013).

2.3 Proximate Analysis of the Rice Husk Sample

The moisture content was determined at 105°C in an oven, Ash content was determined at 550°C, crude protein, lipid and fiber were determined in accordance with AOAC (2003), procedures. Crude nitrogen was determined using the Kjeldal procedure and crude protein value was obtained by multiplying nitrogen values by a factor of 6.25, while available carbohydrate was estimated by difference in accordance with equation 1 which is adopted from Hassan *et al.*, (2009).

$$CHO = 100 - (\% \text{ash} + \% \text{crude protein} + \% \text{crude lipid} + \text{crude fibre}) \dots \dots \dots 4$$

The calorific value (in kcal/100g) of both the rice husk sample and bio-char (Solid Product) were estimated according to the method described by Elmuez *et al.* (2014) through multiplying the percentages of crude protein, crude lipid and available carbohydrate by 4, 9 and 4 factors respectively then the product is summed up as shown in equation 3 below. This is done according to the Atwater factor that states, the energy values of 1g each for protein is 17KJ/g (4.0 kcal/g), fat is 37 kJ/g (9.0 kcal/g) and carbohydrates is 17 kJ/g (4.0 kcal/g).

2.4 Mineral Analysis

A sample of 0.5g was put into KJedahl flask to which 24cm³ of the mixture of concentrated nitric acid (HNO₃), Conc. H₂SO₄ and 60% HClO₄ (9:2:1v/v) was added. The flask was allowed to stand overnight to prevent excess foaming (Saharawat *et al.*, 2002). The flask was put on a heating block and digested to a clear solution cooled and the content filtered into 50cm³ volumetric flask. The solution was then diluted. The mineral content (Calcium and Magnesium) were determined using Atomic absorption Spectrometry (AAS). Sodium and potassium content were determined using also using AAS while Phosphorus were determined by colorimetric using vanadium Molybdate (blue) method.

2.5 Pyrolysis Process

Prior to pyrolysis process the dried rice residue sample was dried using hot air again to remove moisture. After that it was further grinded into fine powder. Five grams (5g) of the sample was measured and packed into the Pyrex glass batch reactor. Then the reactor was reassembled and capped with the reactor lid. Nitrogen gas was pumped into the set-up to flush out trapped air in the reactor for 10 minutes prior to the start of the process. The reactor was heated to about 350°C at 10°C/minutes. Then, the temperature was maintained constant for 1hour under nitrogen atmosphere. Similarly, same procedure was repeated using 1g of each catalyst. Both the catalytic and thermal processes were carried out under the atmospheric pressure. At the end of the process, bio-oil and bio-char was collected while

the non-condensable gas was flared to the atmosphere (Nachenius *et al.*, 2013).

The experimental setup used for this work consists of a batch reactor made up of Pyrex glass reactor (length of 145 mm, internal diameter of 37 mm and outer diameter of 41mm) sealed at one end and an outlet tube at other end and Tubular furnace and temperature control system. The assembly of the set-up is shown up in plate 2.1. The Pyrex glass tube is heated externally by the electric furnace, with the temperature being measured by a Cr-Al: K type thermocouple fixed inside the reactor and temperature is controlled by external proportional-integral-derivative (PID) controller. PID controller was used to control temperature of the furnace. The accuracy of the PID controller is $\pm 0.3\%$.



Plate 1: Reactor Set-up for Pyrolysis Process

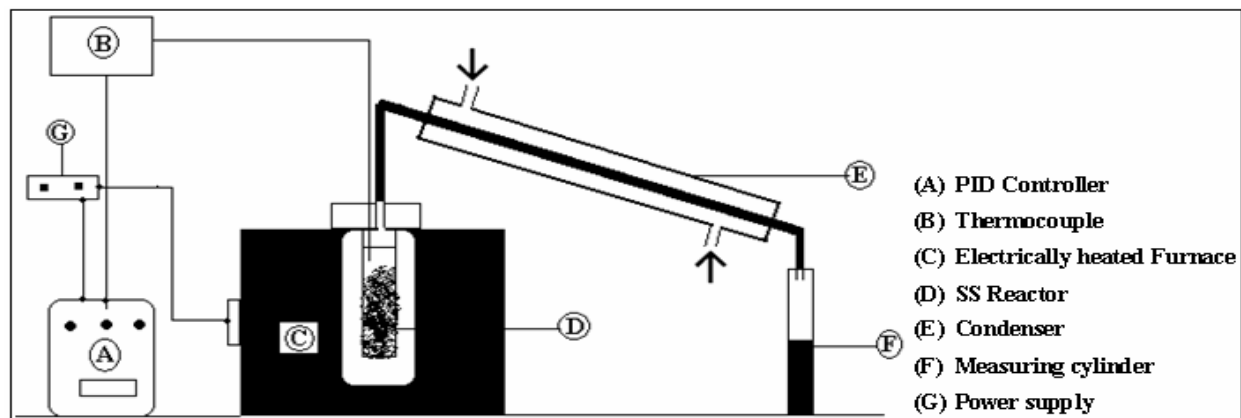


Plate 2: Schematic Diagram for Pyrolysis Set-up

At the end the pyrolysis process, the liquid product obtained was taken for GC-MS analysis at Yobe State University, Damaturu. The analysis was carried out using Agilent GC-MS (GC 7890B, MSD 5977A) that was equipped with a capillary column Petrocol™ DH 24160-U, (100 m length, 0.25 mm diameter, 0.5 μ m stationary phase) using a 1:0 split ratio operating at 80°C for 1 min at the heating rate of 10°C/min until 200°C was reached and kept at this temperature for 4 min, then heated at 10°C/min until 280°C and kept at this temperature for 5 min. A quadrupole mass Spectrophotometer (MS) detector was used with a 70 eV electron impact ions generator, operating in the range 40-450 m/z. Total ion chromatography (TIC) was obtained with a signal/noise ratio of five, and composition was reported as percent peak areas without any response factors correction. Compounds were identified using the NIST mass spectral library. The analysis was carried out in accordance with Jinang *et al.* (2013).

3.0 RESULTS AND DISCUSSION

3.1 FTIR Analysis on the Kaolin and CuO/kaolin Catalyst

The doped CuO on Kaolin sample used as catalyst was characterized using FTIR analysis determined its bands and vibrational frequencies shown in Appendix 3. The results obtained indicated that the band at 1050 cm^{-1} was attributed to the cluster of Al^{3+} and O^{2-} ions for the Al-O vibration, this is quiet similar to Nurudeen *et al.* (2015) report. The bands obtain are also similar to that of Aroke *et al.* (2013) which are bands 3701 and 3542 cm^{-1} region that corresponds to OH stretching (hydroxyl sheet), 3626 cm^{-1} frequency band that corresponds to the inner layer OH (Al-O-H) stretching which falls close to 3623 cm^{-1} and 3622 cm^{-1} . The peak at 50 cm^{-1} corresponds to Si-O deformation. The characteristic bands observed at 432.3 cm^{-1} and 497 cm^{-1} can be also attributed to the Cu-O stretching which corresponded with what Ethiraj and Kang (2012) reported for CuO.

3.2 Proximate Analysis and Ultimate Analysis of Rice Husk and its Biochar

The proximate analysis which comprises moisture and ash contents, Volatile Organic Matter, fixed carbon and Calorific value of both Rice Husk substrate and its Char are presented are below in Table 1.

Table 1: Proximate Analysis of Rice Husk and its Biochar

Proximate Analysis	Rice Husk	Biochar
Moisture content (%)	6.667 \pm 0.289	3.01 \pm 0.01
Ash content (%)	16.167 \pm 0.577	37.43 \pm 0.81
Crude Protein (%)	1.080 \pm 0.008	2.726 \pm 0.020
Crude Lipid (%)	0.510 \pm 0.010	0.533 \pm 0.058
Carbohydrates (%)	67.920 \pm 1.34	46.87 \pm 0.327
Volatile Matter (%)	77.166 \pm 0.26	59.56 \pm 1.35
Fixed Carbon (%)	38.58 \pm 0.506	29.78 \pm 0.370
Calorific Value (MJ/Kg)	280.59 \pm 0.022	203.181 \pm 0.022

The proximate composition of a biomass provides a simple way of investigating the fuel quality of a solid biomass sample. The moisture content of both rice husk and biochar sample presented in Table 1 are 6.607 \pm 0.289% and 3.001 \pm 0.01% respectively which is slightly lower than Awoyale *et al.* (2019). However, the obtain values are lower than 8.80% for Tanzanian and 9.33% for Abakaliki waste rice husk as reported by Mhilu (2014) and Ekwe (2013) but compared to 6.70% for coffee husk as reported by Mhilu (2014). The lower

moisture content of both husk and its biochar show that the sample can be stored for over a long period of time without any spoilage. The ash content from the rice husk sample is 16.167 \pm 0.577% while its biochar sample is 37.43 \pm 0.81% both have lower content compared to Ekwe (2013) work. Therefore, the rice husk from this research can be a good potential for thermochemical conversion such as pyrolysis as it contains lower incombustible proximate parameters such as moisture and ash content. The high ash content according to

Abdullahi and Sulaiman (2013) indicates the presence of high alkali metal, especially potassium (K) and sodium (Na) which are known to have an actively catalytic nature in promoting secondary reactions during the pyrolysis process and fuel production.

Crude lipid content of husk and biochar was $0.510\pm 0.010\%$ and $0.533\pm 0.058\%$ respectively. The values obtained are compared to 0.49% for waste husk from Abakaliki as reported by Ekwe (2013). The Lipid content of the given biomass show its quality for biofuel production. While the crude protein content for both sample and biochar are $1.080\pm 0.008\%$ and $2.726\pm 0.020\%$ respectively. The value obtained is lower than 2.51% for husk but compared to biochar as reported by Ekwe (2013). Similarly hydrocarbon content of both rice husk and biochar were found to be 38.58% and 20.22% respectively. Higher hydrocarbon content influences the combustion process of the biomass sample. The values obtained are in conformity with Filomena *et al.* (2015).

The carbohydrate contents of the samples was found to be $67.290\pm 1.34\%$ and $46.87\pm 0.327\%$. This indicate that both the husk and biochar may find

application in bioenergy generation. Volatile matter composition of husk and biochar is $77.166\pm 0.26\%$ and $59.56\pm 1.35\%$ respectively which is higher than 67.60% and $61.53\pm 0.23\%$ as reported by Filomena *et al* (2015), Ghodke and Naidu (2017) respectively. The fixed carbon content was found to be $38.58\pm 0.506\%$ and $29.78\pm 0.370\%$ for husk and biochar. The obtained value is higher than $34.87\pm 1.25\%$ for Indian rice husk as reported by Ghodke and Naidu (2017) and 6.3% for Portugal rice husk (Filomena *et al.*, 2015). Carbon influences the combustion process and also it is known to have undergone oxidation with hydrogen (H) and exothermically releases thermal energy which is more intense with high carbon contents in the sample (Abdullahi and Sulaiman, 2013). Therefore, the rice husk and its biochar produces in Nigeria has shown a good potential for energy production which is supported by high calorific energy value as indicated in Table 1 above.

3.3 Trace Elements Analysis

The trace elements from both Rice Husk sample and its Char are shown in Table 2 which indicates that the Rice Husk sample contains higher percentage of Potassium and Sodium than its char.

Table 2: Trace Elements present in Rice Husk and its Biochar

Element	Rice Husk (ppm)	Biochar (ppm)
Sodium (Na)	40.833 ± 1.44	17.37 ± 0.31
Potassium (K)	2033.300 ± 57.07	350.40 ± 0.05
Calcium (Ca)	0.917 ± 0.0291	2.83 ± 0.066
Magnesium (Mg)	0.150 ± 0.500	ND
Phosphorus (P)	3.35 ± 0.015	ND

ND= Not detected

Biomass is inherently composed of inorganic constituent that undergoes some chemical transformation leading to fouling and slagging effects during chemical reaction or conversion process such as pyrolysis. The high ash and potassium content are significant, as it is very clear that high ash content particularly high potassium contents lead to the low liquid yields in pyrolysis process. Also these elements were found out to cause detrimental reactions. Although the presence of these metals in biomass might be inevitable, their effect is considered to be minimal at lower acceptable limit (that is less than 2%).

concentrations. The results from this research shown in Table 3.2 indicates that the sodium and potassium were low (40.42 and 2033.30ppm respectively) for rice husk sample. Thus, demonstrating that the secondary reactions or cracking will be minimized during the pyrolysis process. The results are lower than that of Janewit *et al.* (2008) studies that obtained concentration of 210ppm (0.21mg/g) for sodium and 5370ppm (5.37mg/g). Present research indicates that the trace elements are within the

3.5 Mass Balance of Rice Husk for both thermal and Catalytic Pyrolysis process

The result of the mass balance of the Rice Husk pyrolysis process shown in Table 3 indicated that

CuO/Kaolin catalysts gave higher char than thermal process while but yielded lower gaseous product than thermal process.

Table 3: Mass Balance of Rice Husk Pyrolysis for both thermal and Catalytic Process

Product (%)	Thermal	CuO/Kaolin
Gaseous	17.25±0.76	33.36±0.34
Liquid	20.42±0.76	21.62±0.83
Char	62.33±0.49	45.02±0.16

The percentages of liquid, gaseous as well as solid products at 350°C for both thermal catalyzed processes were presented on Table 3. From the results it can be observed that 17.25% (gaseous), 20.42% (liquid) and 62.33% (solid char) were obtained from thermal process. A slight increase in liquid products (from 20 to 21%) and significant increase was also observed from both gaseous and solid products after catalytic process at same temperature. Thus, this clearly indicates that the

catalyst has strong influence on the yields of the pyrolysis products on the substrate. According to the Siraam *et al.* (2015), catalysts enhance secondary cracking process for a substrate during the pyrolysis process. This trend was also enlightened by Meesuk *et al.* (2011), whom observed that, catalytic pyrolysis influences secondary cracking of primary organic vapors into lighter hydro-compounds.

Table 4: Relative Percentage of Chemical Compounds Presents in the Pyrolysis Oil from Rice Husk

Product	Thermal (%)	CuO/Kaolin (%)
Fatty Acid	51.64	14.43
Carbonyl (Ketone & Aldehyde)	14.06	7.95
Alcohol	5.79	13.04
Furans	12.94	20.05
Other	15.57	44.53

The major chemical compounds present in both thermal and catalytic pyrolysis processes were presented in Table 3.4 above. The percentage peak of the chemical compound from thermal process are fatty acids (51.64%), carbonyl compounds (14.06%), alcohols (5.29) and furans (12.94) while fatty acids (14.43%), carbonyl compounds (7.93%), alcohols (13.04) and furans (20.05) were

obtained from catalytic process. It is clearly observed that higher percentage of fatty acids and carbonyl compounds were obtained from the thermal process while higher furan compounds were obtained from catalytic process. Thus, this prevails that the catalyst used has significant effect on the pyrolysis end products.

The present study was designed to determine the potentials of native rice husk sample for the pyrolysis process to produce bio-oil and biochar. The moisture content of the rice husk sample analyzed shown that the moisture of rice husk sample and its biochar have low moisture content compared to the literature. Similarly, low concentration of trace elements was observed from the sample such as sodium and potassium compared with the literature. This demonstrated

4.0 CONCLUSION

minimal secondary reactions is expected to occur when pyrolysing this sample. The percentages of liquid, gaseous as well as solid products at 350°C for both thermal catalyzed process were process show significant increase difference between the catalyzed and uncatalyzed process. The percentage peak area from GC-MS analysis indicates that major chemical compounds present in both thermal and catalytic pyrolysis processes are fatty acids, carbonyl compounds, alcohols and furans. Higher

fatty acids and carbonyl compounds are observed from the thermal process while higher furans compound from catalytic process.

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