



Research Article

Preparation and Characterization of Rice Husk Ash and Carbon from Combustion of Raw Rice Husk

Alegbe Monday John^{1,*}, Moronkola Bridget Adekemi¹, Oyesomi Aisha¹, Felix Boluwatife Blessing¹, Agboola Olugbenga Ayodeji², Adekolurejo Ezekiel³, Ejoh Augustine¹

¹Department of Chemistry, Lagos State University, Lagos, Nigeria

²Department of Chemistry Education, Lagos State University of Education, Lagos, Nigeria

³Department of Science Laboratory Technology, Ogun State Institute of Technology, Igbesa, Nigeria

Abstract

Rice production is a significant agricultural activity worldwide which provides food for human consumption and the generation of large amounts of raw rice husk (RRH) as a by-product in which the disposal has become a serious environmental concerns. The aim of this study is to prepare and characterize rice husk ash (RHA) and rice husk carbon (RHC) produced from raw rice husk (RRH). The RHA and RHC are prepared from RRH by burning of RRH in a muffle furnace at varying temperature by combustion process. The RRH, RHA and RHC samples were characterized and quantified using analytical techniques such as X-ray Diffraction (XRD), X-ray Fluorescence (XRF), Scanning- Electron Microscope (SEM), Transmission electron microscopy (TEM), Thermo gravimetric analysis (TGA), Fourier Transform Infrared (FTIR) Spectroscopy and Brunauer-Emmett-Teller (BET). The TGA results revealed that RRH, RHC and RHA samples have thermal stability at temperature which range from 222.40-327.6°C. The X-ray Diffraction (XRD) results identified four mineral phases for the samples RRH, RHC and RHA. The SEM images of the RRH, RHC and RHA samples revealed the spherical irregular particle morphology with different particle sizes and some agglomeration. The TEM further revealed the morphology of RRH, RHC and RHA to be spherical shape with particle sizes of 17.2 nm, 9.96 nm and 5.33 nm respectively. The FTIR adsorption spectra shows the stretching adsorption bands of functional groups in RRH, RHC and RHA with stretching and bending peaks of the samples from one form to another. The XRF results revealed the elemental composition of the samples RRH, RHC, and RHA revealed the elemental composition of SiO₂, Al₂O₃, CaO, MgO, and Fe₂O₃. In conclusion, the RRH, RHC and RHA samples shows that products of the burning process have a higher quality than the raw samples.

Keywords

Raw Rice Husk, Disposal, Combustion, Rice Husk Carbon, Rice Husk Ash, Characterization

*Correspondence: Alegbe Monday John (alegbemj@gmail.com)

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1. Introduction

Rice is an important staple food for approximately half of the world population and the rice husk is one of the most widely available agricultural wastes in many rice producing countries in the world [1, 2]. Raw rice husk (RRH) is a hard protective covering of rice which is removed from rice grain as by-product during the milling process and the rice by-product in milling plant can be recycled into biomass pellets or briquettes for home or commercial use. The chemical composition of rice husk is about 32% cellulose, 20% hemi cellulose, 21% lignin and 20% of other organic matter such as protein and fat [3-5]. Rice husk has been extensively utilized to adsorb phenolic compound [4]. The increasing availability of rice has become part of our everyday food for several households in Nigeria [6]. The disposal of rice husk wastes in countries where large quantities of rice is produced is a big problem but it can be suitable for the purpose of manure [7]. Harmful raw rice husk (RRH) can be eco-friendly to plants, animals, and humans [8]. Large quantity of Rice husk production is important to be stored and reuse as raw material or feedstock rather than disposing it as waste [9, 10]. Presently, RRH wastes are disposed directly into the soil or burnt which result in environmental pollution [11]. RRH can be used as building material, fertilizer, steel production, insulation material and fuel [12, 13]. It possesses various properties that make them suitable for bioethanol production [14, 15]. The rice husk wastes are used as absorbent for removing heavy metals and organic pollutants from wastewater. Because of the increasing growth in population and industrialization, new technologies are developed for waste utilization and to reduce the cost of industrial processing using rice husk (lignocelluloses biomass) as a valuable raw material [16]. Raw rice husk is a free agricultural waste product in many rice producing countries all over the world. RRH is a hard protecting covering of rice grains and it is an abundantly available waste material in all rice producing countries, and it contains about 75 – 90% organic matter such as cellulose, lignin etc and rest mineral components like trace elements, alkalis, and silica. [17]. RRH biomass consists of three polymers which are cellulose, hemicelluloses and lignin [18, 19]. RRH like other lignocellulosic biomass feedstock has been explored as the cheapest feedstock has been explored as the cheapest feedstock for bio-ethanol production [20, 21]. The uses of RRH wastes help to reduce the disposal problem and the cost of waste treatment [21, 22]. RRH is composed of about 75 – 90% organic matter such as cellulose, lignin etc and rest mineral components are trace elements, alkalis, and silica. [17]. It is essentially free as waste product from agricultural sector and forest residue. RRH has high mechanical strength, chemical stability, and possess a granular structure, which makes it a good adsorbent material for removing heavy metals from contaminated wastewater [23]. Air borne RRH particles have been linked to respiratory disease in human [24]. Rice husk is produced after rice harvest and it is a waste product generated on farmlands and rice mills where grains are

separated from Ofada rice. The milling process of Ofada rice is majorly the complete separation of the wanted rice grains from the useful but unwanted husk covering it [25]. Rice husk carbon (RHC) is a derivative of raw rice husk obtained by burning rice husk in the furnace with a regulated temperature of about 400 - 500°C under limited oxygen condition [26, 27]. Charcoal is a carbon material that is produced by the process of carbonization and it is a precursor for making activated carbon. The carbon generated from the rice husk combustion has potential economic applications in different sectors [28]. High energy ball milling technique can used prepare ultrafine powders, and this technique has been shown to effectively produce magnetic materials [29]. Activated carbon is a very good absorbent with a high surface area. RHC is used in the preparation of activated carbon, Pet food fiber, silica and silica compounds, bricks etc. Activated RHC is preferred over several other carbon sources because of its higher surface area and hence better activity in processes where activated carbon is required for different uses [13]. 89-97% silica of rice husk carbon is highly porous and light weight with a very high surface area when compared to the bulk biochar and other constituents are K_2O , Al_2O_3 , CaO , MgO are available in less than 1% [16, 17]. RHC is an excellent raw material for the production of activated carbon whose function varies from being an adsorbent, color remover to being a catalyst support and many other important industrial applications. [30, 31]. Activated carbon is a highly porous adsorbent material with a wide range of industrial uses [17, 32]. Previous studies have shown that biochar is an effective adsorbent for the removal of organic contaminants such as hydrocarbons (PAHs), pharmaceuticals and personal care products (PPCPs), polychlorinated biphenyls (PCBs), polycyclic aromatic and dyes [29, 33].

Rice husk ash (RHA) is a derivative of RRH waste which is obtained by combustion of RRH in a crucible subjected to heating in an electric furnace for 5 hours at a temperature of 400°C to form a black ash substance [26, 34]. There are two types RHA which are white rice husk ash (WRHA) and black rice husk ash (BRHA), depending on if the combustion process is complete or incomplete [35]. RHA is one of the most raw materials that is rich in silica containing about 90-98% silica after complete combustion [36]. RHA is a raw material that is used to produce silica and nano silica formed from the ash can be applied in various fields such as environment, science and industry. Nano silica is widely used as catalyst and in various kinds of organic-inorganic composite materials [37]. Raw rice ash (RHA) is used as a bioadsorbent in vegetable oil refining, removal of heavy metals in the water and purification by removing Arsenic content in drinking water problem in India communities suffering from the health effect [34]. Raw rice husk (RHA) is used in the water purification by removing Arsenic content in drinking water problem in India people in communities suffering from the health effect [38]. RHA acts

as an adsorbent for the adsorption removal of lead and mercury from aqueous water [39]. RHA can remove Methylene blue, humic acid from wastewater [31, 40]. However, the main constituent of ash content is silica, the ion-exchange reaction on the silica surface is achieved by substitution of protons on the surface of silanol groups by the metal ions in the solution [41, 42]. The melting temperature of RHA is estimated as 1440°C that is the temperature at which silica melts [43]. Rice husk ash is used in steel, cement and construction industries. Rice husk acts as an adsorbent for heavy metal removal from waste water. RHA is used to replace silica fume or as an admixture in making low cost concrete block [44]. The RHA has good thermal insulation properties and a high melting point, used in industry to make Cement and Concrete [45]. The benefits of Rice husk ash (RHA) is a derivative of raw

rice husk which is obtained by combusting RRH in a crucible and subjected to heating in an electric furnace for 5 hours at a temperature of 400°C to obtain a black ash substance [46].

2. Materials and Methods

2.1. Sample Collection

Rice husk sample used for the purpose of this research was collected from a rice processing mill at Lafenwa, Abeokuta, Ogun State. About 25 kg of the sample was collected in a plastic bag and sealed immediately to prevent air or moisture content from entering the raw rice husk sample.

2.2. Sample Location Map

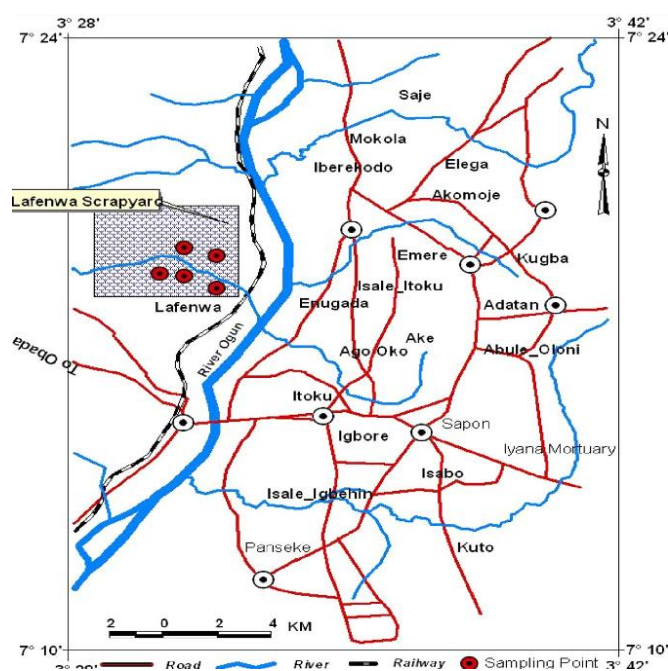


Figure 1. Map of Lafenwa rice husk waste dump, Abeokuta, Ogun state.

2.3. Sample Preparation

The processes carried out in the laboratory to prepare the samples for analysis include drying, burning or combustion at different temperatures to obtain the carbon and ash products. The raw rice husk was sun or air dried to remove moisture for about 48 hours before subjecting it to drying in a regulated oven. It was grinded with mortar and pestle to increase the surface area, and ready for other activities. The raw rice husk was burnt to charcoal and this was carried out in a muffle furnace at 550-600°C for 2 hours to obtain the rice husk carbon. The ash sample was obtained by burning the rice husk sample

at 600-650°C for 3 hours. After obtaining the rice husk carbon and ash, the samples were subjected to grinding into fine powder using mortar and pestle followed by sieving to remove any trace of sand or large particles.

2.4. Preparation of Carbon and Ash Samples

Raw rice husk derivative products were prepared by combustion process to obtain black product and grey ash product which depends on the availability of the oxygen and temperature applied. The preparation processes of making the products are separately explained in the subsections below.

2.4.1. Raw Rice Husk

Raw Rice husk (RRH) was processed by sun or oven drying the samples before pulverizing the dried raw rice husk sample to a powdery form using a mortar and pestle. The powder product was subjected to sieving in order to remove any trace of sand or large particles to obtain a fine powder and the product was kept in a sample bottle or container.

2.4.2. Rice Husk Carbon

Rice husk carbon (RHC) is obtained from raw rice husk by weighing 10 g of dried raw rice husk in a crucible and then placed in an electric muffle furnace and pyrolyzed for 4 hours at a regulated temperature of 550 – 600 °C under limited supply of oxygen conditions to obtain a black product. The black powder product was subjected to sieving in order to remove any trace of sand or large particles present to obtain a fine powder and the product was kept in a sample bottle or container.

2.4.3. Rice Husk Ash Preparation

Rice husk ash (RHA) is obtained raw rice husk by weighing 10 g of raw rice husk in a crucible and placed in an electric muffle furnace and pyrolyzed for 5 hours at a regulated temperature of 600 – 650°C under sufficient supply of air to obtain complete combustion to yield grey ash product. The grey ash powder product was grounded into a fine powder and subjected to sieving in order to remove any trace of sand or large particles in order to obtain a fine powder and the product was kept in a sample bottle or container.

2.4.4. Determination of Moisture Content

Moisture content was determined by weighing 1.0 g of raw rice husk, rice husk carbon and rice husk ash samples were separately weighed into three clean crucibles and subjected in a regulated oven at a temperature of 80 °C for 1 hour. The samples were removed and allowed to cool in a desiccator for 30 minutes before weighing and record the results and repeat the drying process until a constant weight was obtained. The initial weight is the measurement of the beaker and the sample and the final weight is the weight after it was dried in the oven after it was cooled.

Mass of the empty crucible = W

Mass of empty crucible + sample = W1

Mass of crucible + mass of dry sample = W2

Mass of sample = W2 - W

% Moisture Content = $\frac{W - W_2}{W_2} \times 100\%$

2.5. Analytical Techniques

The raw rice husk was subjected to drying using an oven to determine the moisture content and the combustion process of the RRH was carried out using instrument at a regulated temperature of 400°C for carbon and 600°C for ash sample production.

Characterization

XRD of the RRH, RHC and RHA samples was carried out to identify spectrum and mineral composition using a Bruker D8 Advance X-ray diffractometer with Cu K α radiation (45 kV, 40 mA, $\lambda = 1.542 \text{ \AA}$). Scan was conducted on the samples were scanned at 002s from the range of 10–80 °(2 θ). The morphology of the surface of the samples, composition and particle size of the RRH, RHC and RHA were examined with both high resolution scanning electron microscopy-energy-dispersive X-ray spectroscopy (SEM-EDS) using a SEM HITACHI S-4700 electron microscope with spectral imaging system. The internal morphology of the rice husk sample was determined by high resolution transmission electron microscopy (HRTEM) Phillips Tecnai F20 super-twin. XRF elemental composition of rice husk was carried out using a Philips PW 2400 X-ray sequential spectrophotometer to determine the elemental constituents of the rice husk samples. The FTIR was used to determine the functional groups present in the rice husk samples using attenuated total reflectance (ATR). BET surface area of the rice husk samples were measured at a temperature of 77.35K using a quantachrome NOVA 2000 surface analyzer. Thermogravimetric analysis (TGA) was conducted to measure the thermal stability of the prepared rice husk samples determined by a thermal analyzer RB-3000-20, BP Engenharia.

3. Results

3.1. Moisture Content

Figure 2 presents the moisture content of the rice husk and its derivatives: RRH (1.28%), RHC (1.68%), and RHA (1.72%) that was determine which revealed moisture content of ash the highest and RRH to be the least. The results shows that RHA > RHC > RRH and the moisture content absorbed can be attributed to the nature of the particle, season, and their particle sizes.

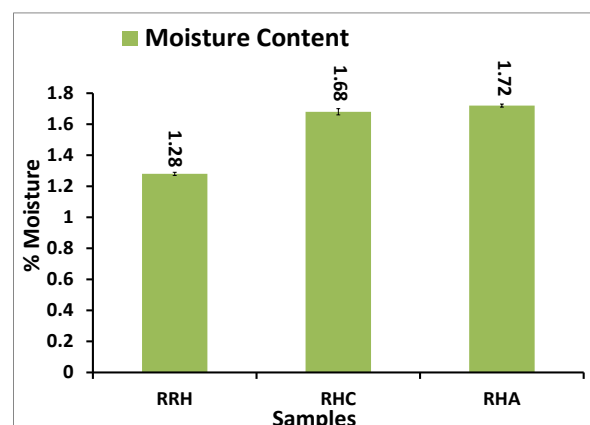


Figure 2. Moisture Content of Rice Husk samples.

3.2. Characterization

The raw rice husk (RRH), rice husk carbon (RHC) and rice husk ash (RHA) were characterized to assess the transformation process that has taken place using characterization using the following techniques: XRD, SEM, TEM, XRF, TGA, FTIR and BET. The characterization results RHC and RHA prepared from RRH is presented in Figures 3-11.

3.2.1. XRD

Figure 3 presents the XRD spectral pattern and mineral quantification analysis of RRH, RHC and RHA. The particle size was calculated using Scherer's equation for RRH (8 nm), RHC (2 nm), and RHA (2 nm). The rice husk and its derivatives all possessed four mineral phases each with different composition. The XRD spectrum result for RRH revealed a broad band sharp spectral peak indicating crystallinity which consists of four mineral phases with percent composition of Urea (58%), Graphite

(12%), Cristoballite (17%), Mellite (13.4%). RRH revealed that Urea has the highest composition while graphite has the lowest composition. The RHC spectrum revealed a broad spectral peak which indicates crystallinity with four different mineral phases with composition of Graphite (66%), Osumilite (5%), Silicon Dioxide (24%), Hanksite (4%). RHC shows that Graphite has the highest composition while Hanksite has the lowest composition. The XRD spectrum of RHA revealed four mineral phases with their mineral composition are: Graphite (2.63%), Osumilite (10.42%), Silicon Dioxide (86.9%), Hanksite (0.09%). The RHA result shows that Silicon Dioxide has the highest mineral composition while Hanksite has the lowest composition. The transformation of the RRH to RHC and RHA was very clear, only a single mineral phase of graphite is common to the three (RRH, RHC and RHA) samples. RHC and RHA had the same type of mineral phases except that their compositions are different.

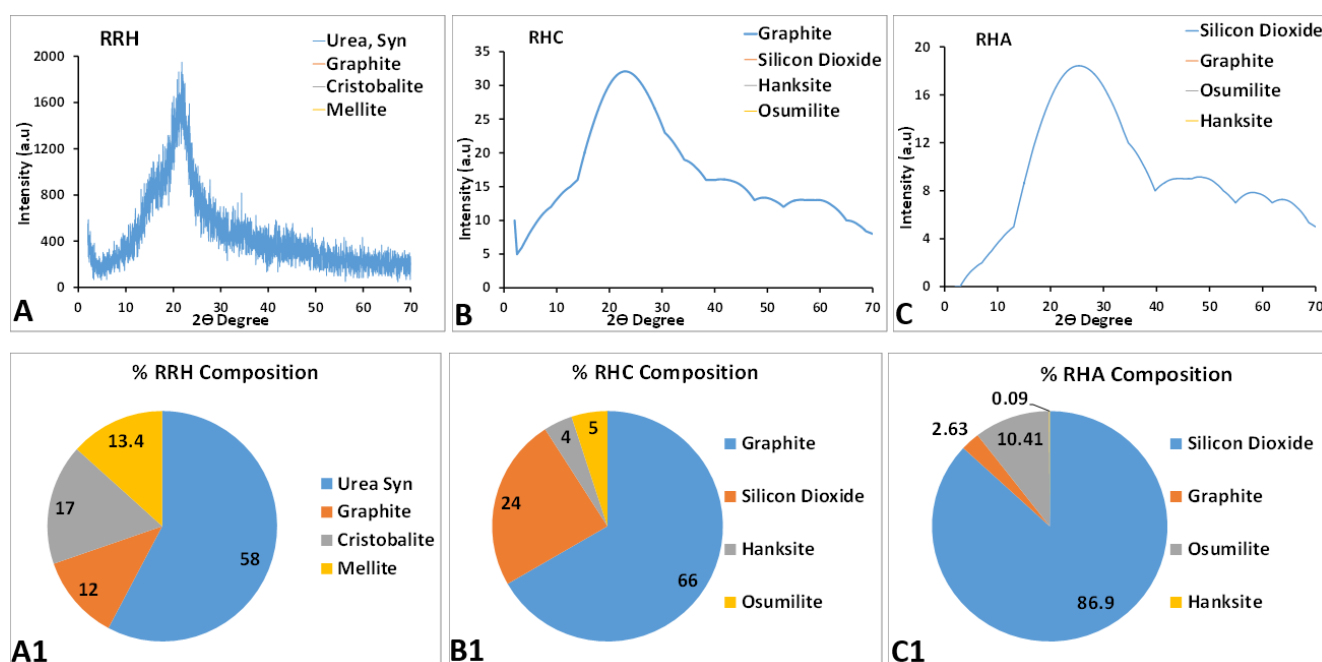


Figure 3. RD spectrum (A) and mineral composition (B) of RRH, RHC and RHA Samples.

3.2.2. SEM

SEM images of RRH, RHA and RHC in Figure 4 revealed irregular spherical particle shape with rough surface indicating some agglomeration which was attributed to the presence of moisture in the samples. A close observation of the images of the samples showed solid, loosely packed structures that are

porous and crystalline. The structure shows the trend of changes that took place before and after the combustion of the RRH with respect to RHC and RHA prepared samples. The high agglomeration in the RHC and RHA could be as a result of their high moisture content. The morphology of the samples are spherical in shape but with irregular particle sizes indicating absorption of moisture that caused agglomeration presented in Figure 4.

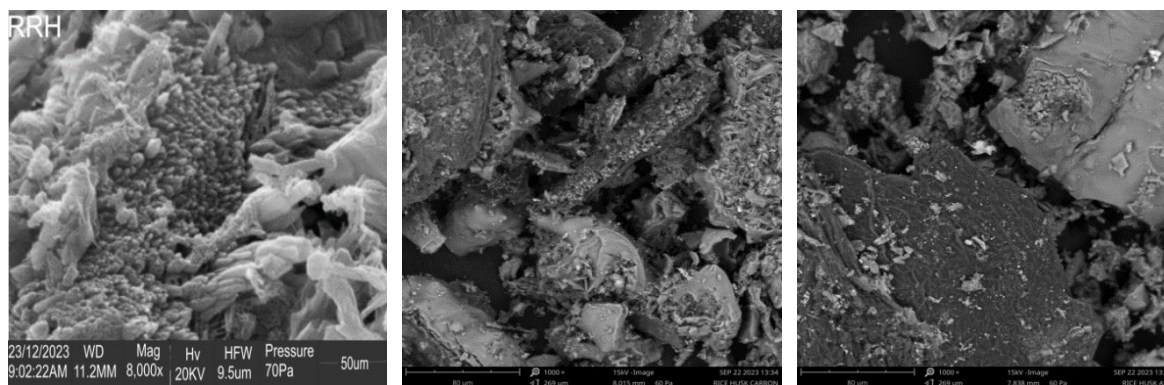


Figure 4. SEM images of RRH, RHC and RHA Samples.

3.2.3. HRTEM

The HRTEM images of RRH, RHA and RHC sample results in [Figure 5](#) revealed the internal morphology of the rice

husk and its derivative samples are crystalline, spherical in shape with irregular particle sizes and their average particle sizes are 17.24 nm, 9.61 nm and 5.33 nm respectively.

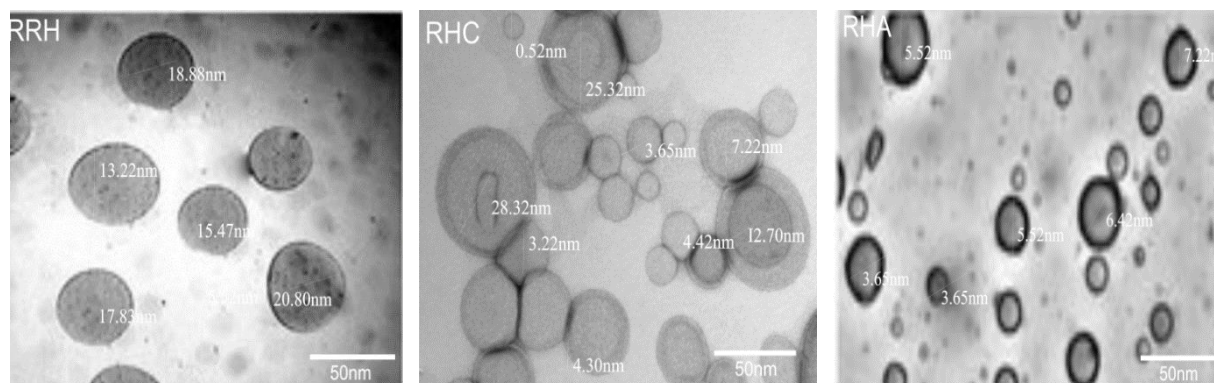


Figure 5. HRTEM images of RRH, RHC and RHA Samples.

3.2.4. FTIR

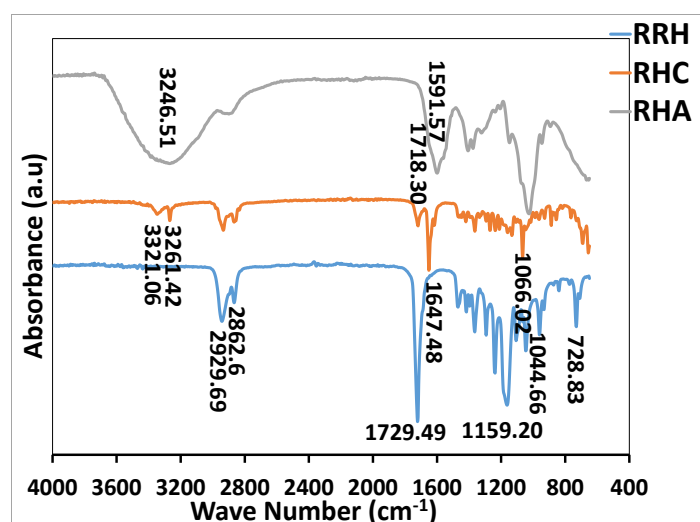


Figure 6. FTIR absorption bands of RRH, RHC and RHA Samples.

This section presents the FTIR absorption bands of the RRH, RHC and RHA which is presented in Figure 6. The result revealed the transformation that occurred in the conversion of the raw rice husk to RHC and RHA that RRH has high amount of moisture with a broad band at 3246.51 cm^{-1} for RHA while that of RHC had small peaks and RRH had no visible peak. The RRH, RHC and RHA FTIR spectra bands at 2929.69 cm^{-1} , 2862.6 cm^{-1} , $1729.49\text{--}1647.48\text{ cm}^{-1}$, $1159.20\text{--}1044.66\text{ cm}^{-1}$ and 728.83 cm^{-1} are for C-H, C=O, C-O, Si-O-Si and Si-OH stretching respectively [4, 47].

3.2.5. TGA

Figure 7 revealed the TGA thermal stability of RRH, RHC and RHA as the temperature increases. The thermal degradation of rice husk and its derivatives weight loss can be attributed to the removal of moisture content and the maximum weight loss was observed for RHA from $216.96\text{--}482.59^\circ\text{C}$, RHC $307.76\text{--}482.59^\circ\text{C}$ and RRH $333.72\text{--}545.72^\circ\text{C}$. The materials were stable at certain temperature before declining and the stability of the raw rice husk and its derivatives are $\text{RRH} > \text{RHC} > \text{RHA}$. Study has shown that weight loss occurs from $400\text{ to }500^\circ\text{C}$ as it can be attributed to the degradation of hemicellulose and cellulose [48].

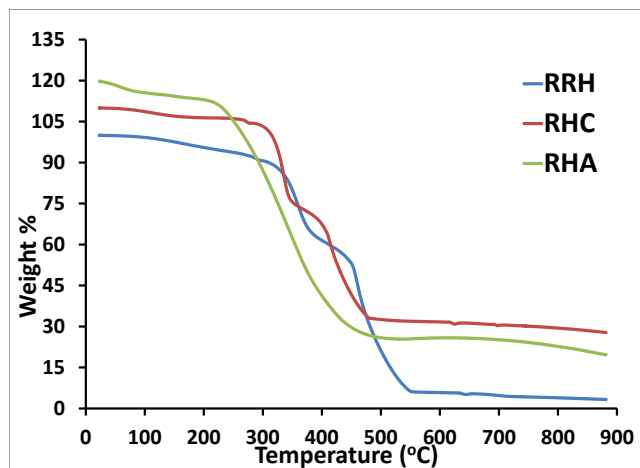


Figure 7. TGA analysis of RRH, RHC and RHA Samples.

3.2.6. XRF

Figure 8 present the XRF results revealed the elemental composition of the rice husk and its derivatives and RRH, revealed the elemental composition of SiO_2 (0.62%), Al_2O_3 (0.01%), CaO (0.36%), MgO (0.05%) and Fe_2O_3 (0.52%). RHC revealed the elemental composition of SiO_2 (44.63%), Al_2O_3 (0.99%), CaO (0.45%), MgO (5.52%) and Fe_2O_3 (0.17%) while RHA revealed the elemental composition of SiO_2 (64.77%), Al_2O_3 (1.27%), CaO (0.57%), MgO (3.47%), and Fe_2O_3 (0.42%). The SiO_2 composition in RRH was 0.62% while that of RHC and RHA were 44.63% and 64.77% respectively indicating that the combustion had converted bulk of the

shaft to silica. The elemental composition of the RRH, RHC and RHA revealed that RHA had the highest composition of SiO_2 , Al_2O_3 , and CaO while RRH and RHC had the highest metal oxides in Fe_2O_3 and MgO respectively. SiO_2 is the backbone, major component and most important ingredient used in the production of ceramics such as toilet wares, tiles, electrical appliances, glass wares, etc [46]. The high silica content of the RHA shows that it can be categorized as pozzolanic material which is used in concrete and mortar which eventually cut cost of construction [48-50].

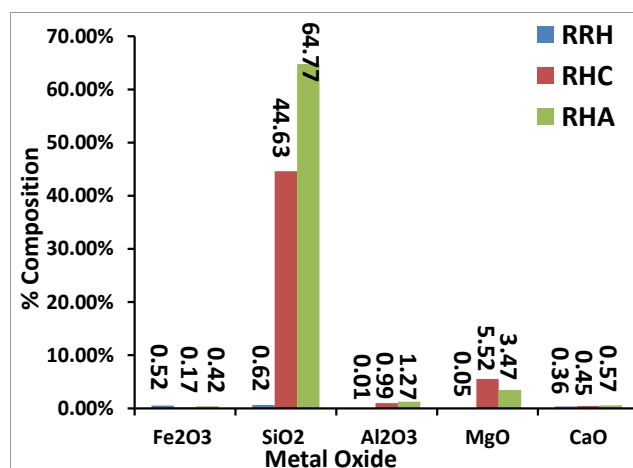


Figure 8. XRF elemental composition (A) and BET Micro pore analysis (B) of RRH, RHC, and RHA Samples.

3.2.7. BET

The BET method analysis assumes that gas molecules form a uniform layer on the surface of the material which allows adsorption to occur in a monolayer form. The micropore (A) and Langmuir (B) of the samples is presented in Figure 9. The BET revealed the micropore (A) diameters of RRH, RHA and RHC to be 2.74 nm, 1.73 nm, and 2.66 nm respectively indicating $\text{RRH} > \text{RHA} > \text{RHC}$ while the Langmuir (B) revealed this trend of $\text{RHC} > \text{RHA} > \text{RRH}$. The Langmuir isotherms of RRH, RHC and RHA revealed that the gas molecules formed a uniform layer on the surface and the adsorption occurs in a monolayer form and the Langmuir isotherms plot gives a linear graph [51]. The pore size distribution in Figure 10 revealed the cumulative surface area and cumulative pore volumes of the samples which indicate the surface area of these samples are $\text{RHC} > \text{RRH} > \text{RHA}$. The multipoint BET in Figure 11 revealed that as the relative pressure increase, volume and the multipoint also increase in this order $\text{RHC} > \text{RHA} > \text{RRH}$. The surface area of the rice husk and its derivatives are RRH ($314.0\text{ m}^2/\text{g}$), RHA ($194.3\text{ m}^2/\text{g}$) and RHC ($57.32\text{ m}^2/\text{g}$). The type of RHA surface area depends on what it is to be used for such as catalyst, adsorbent, synthesis of zeolites, or pozzolanic material in construction work should have larger surface area [52].

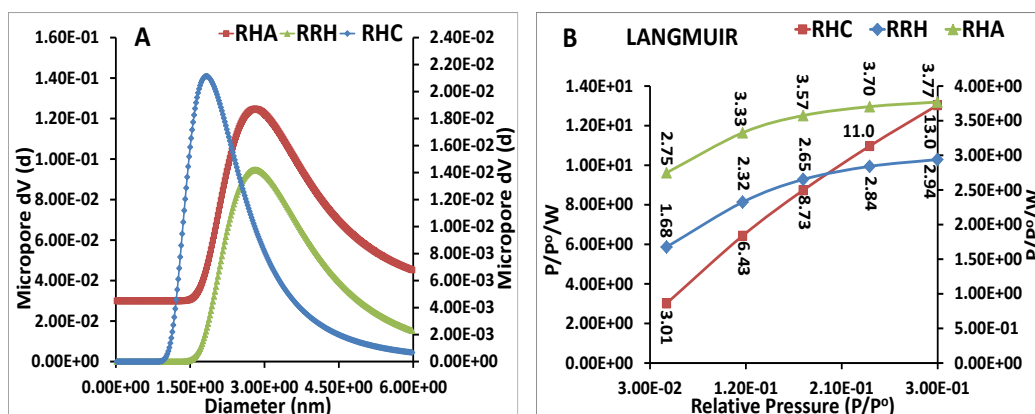


Figure 9. BET Micropore and Langmuir of rice husk and derivatives (RRH, RHC and RHA).

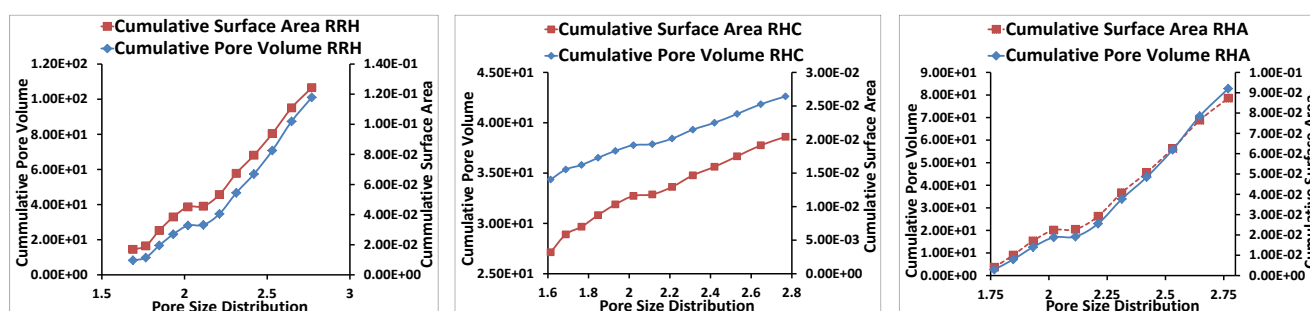


Figure 10. Pore Size Distribution of rice husk and derivatives (RRH, RHC and RHA).

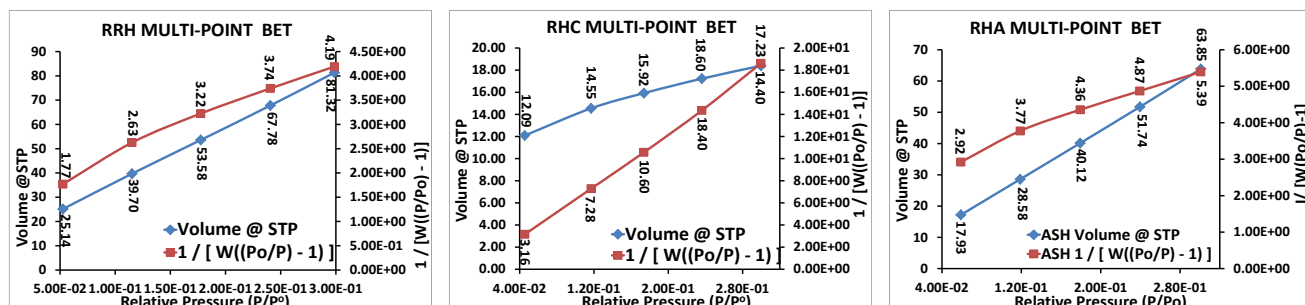


Figure 11. Multi-point BET of rice husk and derivatives (RRH, RHC and RHA).

4. Conclusion

- 1) The XRD results revealed that the rice husk and its derivatives (RRH, RHA and RHC) different spectra and mineral phases with their elemental% composition such as Graphite, Silicon Dioxide, Hanksite and Osumilite. Graphite had the highest% composition of 66% while Hanksite had the least% composition of 4%. RRH Urea, Graphite, Cristobalite, Mellite. Urea had the highest composition of 58%, Cristobalite (17%), Mellite (13%), while Graphite had the lowest composition of (12%)
- 2) The SEM of RRH, RHA and RHC revealed spherical

shape with rough surface which indicate some agglomeration

- 3) The TEM of RRH, RHA and RHC revealed spherical shape with smooth surface
- 4) The BET revealed RRH, RHC and RHA surface area, multi-point BET, pore size distribution, micropore, and Langmuir
- 5) The TGA revealed the RRH, RHC and RHA thermal stability
- 6) The XRF results revealed the elemental composition of the RRH, RHC, and RHA revealed the elemental composition of SiO₂, Al₂O₃, CaO, MgO, and Fe₂O₃
- 7) The FTIR adsorption spectra for RRH, RHC, RHA shows the stretching adsorption bands of functional

groups in raw rice husk, with stretching and bending peaks of transformation from one form to another

- 8) The rice husk and its derivatives serve as a low cost adsorbent for treatment and purification of wastewater

Abbreviations

RRH	Raw Rice Husk
RHC	Rice Husk Carbon
RHA	Rice Husk Ash
XRD	X-Ray Diffraction
WRHA	White Rice Husk Ash
BRHA	Black Rice Husk Ash
XRF	X-Ray Florescence
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
TGA	Thermogravimetric Analysis
BET	Brunauer Emmette Teller
IC	Ion Chromatography
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometry

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Author Contributions

Alegbe Monday John: Conceptualization, Supervision

Moronkola Bridget Adekemi: Formal Analysis, Writing – review & editing

Oyesomi Aisha: Data curation

Felix Boluwatife Blessing: Writing – review & editing

Agboola Olugbenga Ayodeji: Data curation

Adekolurejo Ezekiel: Writing – original draft, Writing – review & editing

Ejoh Augustine: Writing – original draft, Writing – review & editing

Conflicts of Interest

There is no conflict of interest among the authors or with any other party.

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