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Editors

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Proximate Analysis and Physicochemical Properties of Sugarcane Bagasse, Cassava Periderm and Maize Stalk

J. A. Adebisi^{a,b,d,*}, J. O. Agunsoye^a, S. A. Bello^c, M Haris^d, M. M. Ramakokovhu^e, M. O. Daramola^f, and S. B. Hassan^a

^a*Department of Metallurgical and Materials Engineering, University of Lagos, Nigeria*

^b*Department of Materials and Metallurgical Engineering, University of Ilorin, Nigeria*

^c*Department Materials Science and Engineering, Kwara State University, Malete, Nigeria*

^d*Department of Physics, Karunya University, Karunya Nagar, Coimbatore 641 114, India*

^e*Department of Chemical, Metallurgical and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa*

^f*School of Chemical and Metallurgical Engineering, University of Witwatersrand, Johannesburg, South Africa*

Abstract

Recycling of agricultural wastes has attracted researchers to reduce their indiscriminate and unguided disposal that pose environmental challenges. The amount of several products from such recycling are proportional to the amount of volatile matter, carbon and ash contents. In order to investigate the potential of obtaining quantifiable amount of silica from three agricultural wastes (cassava periderm, maize stalk and sugarcane bagasse), their proximate and physicochemical characteristics were investigated. SEM, EDS, XRD and TGA analysis were carried out to comprehend their characteristics. Cassava periderm and maize stalk have higher ash content relative to sugarcane bagasse. The amount of free silica obtained from their XRD also revealed greater potential of yielding more silica than sugarcane. Sugarcane bagasse gave more volatile matter while cassava periderm has more carbon. It is therefore feasible to extract silica from all the wastes but with low yield from sugarcane.

Keywords: Proximate analysis; agricultural wastes; cassava periderm; maize stalk; sugarcane bagasse; physicochemical properties.

1. Introduction

Several researches have focused on agricultural wastes recycling for extraction of useful products such as cellulose, lignin, carbon, silica, silicon, inhibitors, adsorbents and biofuels [1-9]. Principal focus of most of them is on by-products without considering the proportions of other constituents. Narnaware, Srivastava [10] produced solid briquettes and gas from vegetable market wastes while Shao, Jiang [11] produced activated carbon, fuel-gas and oil from corn cob which are better waste management approaches [12, 13]. Proportion of constituent elements and components will give salient information on how to best manage wastes from these sources. Chemical analysis of the wastes gives approximate information on the elements present. Proximate analysis will provide information on components such as moisture, volatile matter, fixed carbon and ash contents but with sulphur content in cases of

*Corresponding author. Tel.: +234-803-042-233-0.

E-mail address: adebisijeel@gmail.com, adebisi.ja@unilorin.edu.ng

utilization as fossil fuels. Generally, ultimate analysis is required for fossil fuel applications according to some American Society for Testing and Materials (ASTM) standards [14, 15].

The aim of this study is to determine the amount of ash that can be obtained from three agricultural wastes as feedstock for silica extraction. The microstructural details of their raw, carbonized and ashes will enhance their applications in surface and environmental sciences, most especially in adsorption processes[16, 17]. A good micrograph of a sample will give detailed morphological structure of the sample, its pore sizes and distribution. The current work also investigates the physicochemical characteristics of cassava periderm, maize stalk and sugarcane bagasse which are substantial agricultural wastes and residues in many developing countries like Nigeria [18].

Nomenclature

CP	Cassava Periderm
MS	Maize Stalk
SB	Sugarcane Bagasse
AWs	Agricultural Wastes

2. Materials and Methods

2.1. Materials

The agricultural wastes CP, MS and SB used in this work were obtained from cassava (*Manihot esculenta*), maize (*Zea mays*) and sugarcane (*Saccharum officinarum*) respectively. All the wastes were sourced locally in Nigeria: cassava from Betterlife market, Isale-Osun, Osogbo, Osun State, South-West; MS from University of Ilorin farm settlement, Ilorin, Kwara State, North Central; and sugarcane from OluodeArain, Osogbo, Osun State.

2.2. Methods

2.2.1. Preparation of the Agricultural Wastes

The maize stalks were left dried in situ on the farm. The leaves, roots and tassels were removed before the stalks were chopped into smaller sizes of 3 – 5 cm. They were then washed with water to remove any contaminants (especially soil) before sun drying for 10 days. The roots and leaves of sugarcane were removed before washing and rinsing carefully to remove contaminants. Bagasse obtained after juice extraction was sun dried for 14 days. The brown back peels (periderm) of cassava were manually removed, washed several times until fine sands were not obvious from the rinsing water. CP was then sun dried for 7 days. All the drying was carried out using 65 cm stainless tray and thereafter stored in separate air-tight polyethylene bags.

2.2.2. Proximate Analysis of the Wastes

The stored AWs were pulverized using laboratory blade mill according to ASTM standard [19]. The sieves used are ASTM 11 Nos 12 (1.70 mm), 18 (1.00 mm), 50 (0.30 mm) and 170 (0.09 mm) while fractions from 18 and 50 were used. The aluminium pans and porcelain crucibles were initially labelled and tared according to ASTM standards [20, 21]

which were thereafter kept in desiccator until used. The tared masses of the containers were used for calculations in this work.

The components of the different AWs were determined using ASTM standard methods as enumerated in Table 1. Six samples were averaged for moisture content determination while at least three samples were averaged for ash and volatile matter contents. All calculations are based on Sun dried measurements and formula used can be obtained from the ASTM standards employed [20-22].

Table 1: ASTM standards used for proximate analysis

Components	ASTM standards used
Moisture content	E 1756 (2008): Standard Test Method for Determination of Total Solids in Biomass [21].
Ash content	E 1755 (2001): Standard Test Method for Ash in Biomass [20].
Volatile matter content	E 872 (1998): Standard Test Method for Volatile Matter in the Analysis of Particulate Wood Fuels [22].
Fixed carbon content	E 1755 (2001) and E 872 (1998) [20, 22].

2.2.3. Characterization

In order to obtain information about morphology and chemical compositions of each AWs and their proximate analysed (ash and carbonized) samples, Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray spectroscopy (EDS) were carried out. X-Ray Diffractometric (XRD) analysis was conducted to determine the phases and structures present. Thermogravimetric analysis (TGA) was also performed to investigate the thermal behaviour of the various AWs from room temperature to 825°C.

3. Results and Discussion

3.1. Proximate analysis

Fig.1 shows that moisture content of MS with 6.30 % is the least while 8.37 % for SB is the highest. Volatile matter and carbon contents range between 58.50 – 71.30 % and 18.60 – 29.40 %, respectively. Ash contents for all the three wastes selected have lowest values. Close observations have been made in literature for similar biomass [9, 11, 23-26] based on dry mass. The trend also conformed with the review of McKendry [27] that the proportions of constituents reduces from volatile matter, fixed carbon to ash content. CP has the highest carbon and ash contents thus given the highest char value. The percentage of volatile matter (71.30 %) in SB is the highest which is 5 % above that of MS. Due to quite low ash content in SB, it is desirable to explore this agro-waste for carbon and volatile matter utilization. All the wastes could be used for production of activated carbon and bio-fuels [9, 11]. Although ash content is considered undesirable for biomass conversion to bio-fuels [28], it has been found to be useful as pozzolanic materials [29, 30] and extraction of silica [3, 18].

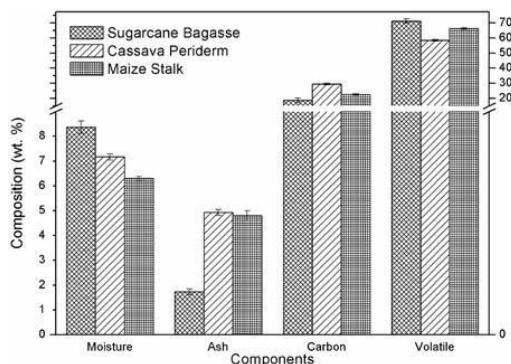


Fig. 1: Proximate analysis of SB, CP and MS

3.2. Thermogravimetric Analysis (TGA)

The chars left after thermogravimetric analysis (Fig.1) corresponding to the proportions observed during proximate analysis obtained as 34.33, 27.33 and 20.33 % for CP, MS and SB, respectively. Thermal characteristics of the three biomass differ completely during volatilization stage where hemicellulose, cellulose and lignin decomposed [31]. Their weight loss (TG) curves showed three main stages of dehydration, volatilization and carbonization with inflection points occurring before 200, 400 and 500°C respectively [25, 32, 33]. The first stage of moisture drying occurs up to 200°C owing to the presence of water of crystallization in the structures of biomass but with evolution of light volatiles around 150°C for SB as can be observed from its derivative of weight loss (DTG) curve [34]. The DTG curves that showed kinetics of pyrolysis of the biomass revealed that the main dehydration started at initial temperatures but well pronounced from 60 to 120°C. Volatilization stage involves decomposition of hemicellulose which occurs between 180 and 340°C [35], cellulosic breakdown between 250 and 450°C, and lignin decomposition between 200 and 800°C [33]. The rapid weight loss (as obvious from the TG curves) can be attributed to hemicellulose and cellulose decomposition [32]. MS has simplest decomposition profile with a single peak spanning from 200 to 360°C while that of CP is also similar with moderate profile but up to 380°C. SB has a distinctive decomposition profile for hemicellulose decomposition prior to 200°C and 260°C with cellulosic compounds breakdown occurring between 270 and 300°C. Because of the low temperature decomposition of lignin, fractions may be lost concurrently with cellulosic breakdown. Carbonization of the remaining chars at this stage is established to be decomposition of lignin (mainly carbon compounds) which started before 400°C in two main stages; rapid decomposition up to 500°C and then at slow rate above 500 to 800°C.

The decomposition profiles at this stage can easily be associated with the carbon contents from the proximate analysis as in Fig 1. Similarities also exist when the heights of volatilization stage (from TG curves) and the volatile matter columns in proximate analysis are compared. The weight loss within this range (200 – 400°C) are 63.59, 59.85 and 43.08% for SB, MS and CP, respectively. This variation could be associated with carbonization stage loss which is more pronounced for CP.

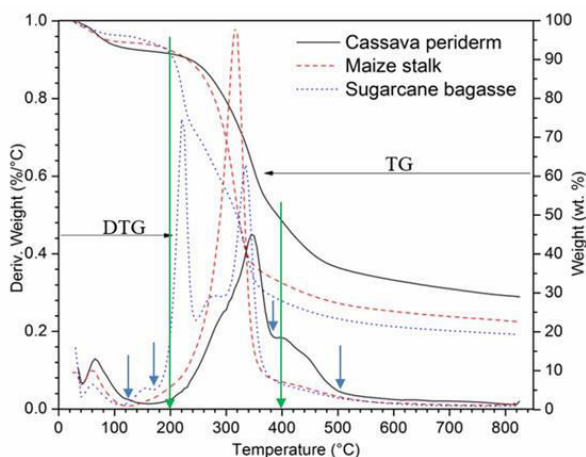


Fig. 1: Thermogravimetric curves for TG and DTG of CP, MS and SB

3.3. Microscopy and Chemical Analysis

Scanning electron microscopy was performed on the biomass in order to study their morphology as they are heated to get varied ashes during calcination at 575°C and char during volatilization at 950°C. The micrographs obtained are shown in Fig.2. The pores observed in the raw samples of SB (a_1) is due to the removal of juice from the pulp while that of MS (a_3) represent the skeletal structure of the stalk. The morphology of raw CP (b_1) is unchanged even during volatilization (b_3) since it is a bulky flake with very tough physical appearance. CP structure was completely damaged during calcination (b_2) since all the volatile matter and carbon were removed in the processing [36]. This structural deformation was also observed during calcination of SB (a_2) and MS (c_2). During volatilization of SB and MS, their structural framework were retained because carbonization did not occur according to the procedure [22]. Lignin which forms the main structural supporting tissue of plants was removed but its high carbon percent left was still enough to support the structure for both biomasses. The fibrous pores formed by both SB (a_3) and MS (c_3) increased their surface areas thus increasing the rate of their decompositions during volatilization stage (Fig.1). This justifies the observed sharp and short range peaks at this stage. The microspheres found around the pores are the chars of the volatilized cellulose, hemicellulose and lignin that occupied the pores initially [31]. Nevertheless, this observation established that surface area is crucial to the application, the chars of SB and MS could be employed especially in adsorption phenomenon.

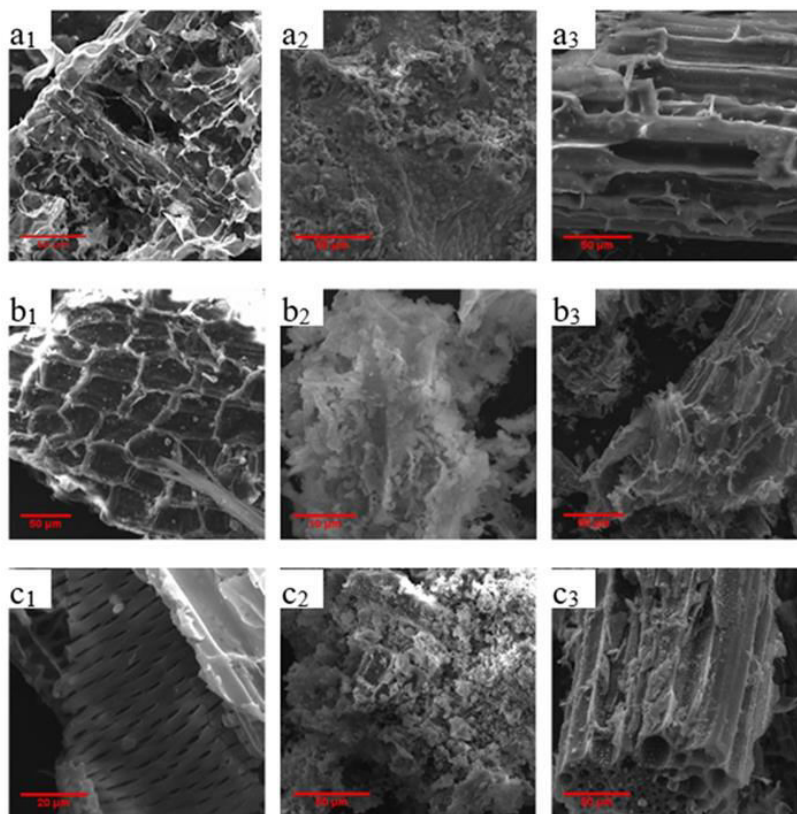


Fig.2: SEM micrographs for SB, CP and MS. [a=SB, b=CP, c=MS, 1=raw, 2=calcined samples at 575°C, and 3=volatilized samples at 950°C]

In order to establish how much siliceous products could be obtained, Energy Dispersive X-ray spectroscopy (EDX) was carried out and the compositions of each biomass at different stages are shown in Table 2. The silicon is more present in MS than CP with least amount of SB. Extraction of silica from ashes obtained after calcination has been established from past literature [3, 18]. The presence of metals (e.g. Al) that could form amphoteric oxides should be taken into consideration as this may affect the final purity of the silica.

Table 2: Chemical composition of SB, CP and MS using EDX

Composition (at. %)									
Element	Sugarcane Bagasse			Cassava Periderm			Maize Stalk		
	Raw	575	Volatile	Raw	575	Volatile	Raw	575	Volatile
C	62.70	<<	91.01	55.74	14.17	70.77	63.23	18.25	82.68
O	30.66	64.22	8.26	35.01	58.44	23.26	33.96	53.39	11.73
Mg	2.33	2.50	<<	<<	2.22	0.19	<<	1.74	<<
Si	0.15	5.22	<<	3.68	6.10	2.58	2.46	7.01	1.44
Al	0.22	<<	<<	2.66	3.64	1.81	<<	<<	<<
K	0.89	18.45	0.73	<<	5.11	0.35	0.35	14.90	3.78

Ca	2.09	<<	<<	1.86	8.86	0.41	<<	<<	<<
Fe	<<	<<	<<	1.04	1.45	0.39	<<	<<	<<
P	0.65	2.66	<<	<<	<<	0.04	<<	<<	<<
S	<<	2.25	<<	<<	<<	<<	<<	2.76	<<
Cl	0.19	<<	<<	<<	<<	<<	<<	1.96	0.36
Ti	<<	<<	<<	<<	<<	0.08	<<	<<	<<
Mo	<<	2.57	<<	<<	<<	<<	<<	<<	<<
Na	0.13	2.13	<<	<<	<<	0.12	<<	<<	<<

<< Below detection level

3.4. Phase Identification

Figs 4-6 show the XRD spectra of SB, CP and MS of sun-dried (raw), calcined and volatilized samples with focus on silica/silicate phases. Fig 4 revealed that free silica is only feasible during calcination of the bagasse at relatively high temperature. The free silica recombined at volatilization temperature of 950 °C to form complex silicates (Magnesium Aluminum Silicate and Sodium Tetramethyl Ammonium Silicate Hydrate) even though the process is temporary. Silicon is present in the raw SB as chantalite (Calcium Aluminum Silicate Hydroxide) which is an inorganic mineral with a tetragonal structure and magnesiochrochite (Magnesium Iron Aluminum Silicate Hydroxide) with similar mineral source but orthorhombic crystallographic structure [37]. From Fig 5 and Fig 6, free silica is present in their raw samples with some silicates and complex hydroxides. Some of the free silica transformed to complex silicates during calcination and volatilization as can be observed in both CP and MS. The free silica can be extracted by either calcination and acid leaching [38] or via hydrometallurgical process, sol-gel [39].

Extraction of silica from the various silicates is of great importance because their abundance is quite overwhelming. They have properties similar to most minerals and gemstones but usually reactive in alkaline medium [40]. The use of sol-gel also dissolves unwanted silicates which impair the purity of the final silica [41].

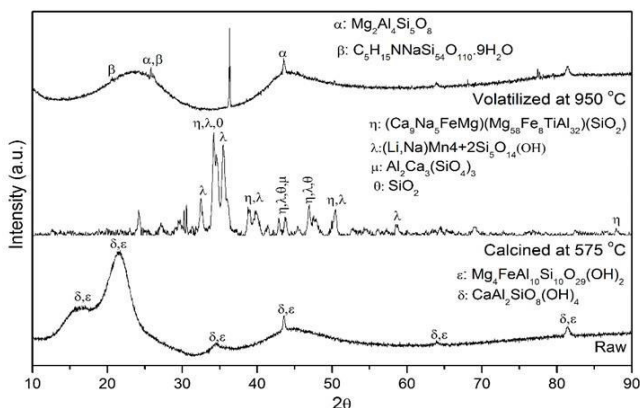


Fig.4: XRD of sugarcane bagasse; as sun dried, calcined at 575°C and volatilized at 950°C

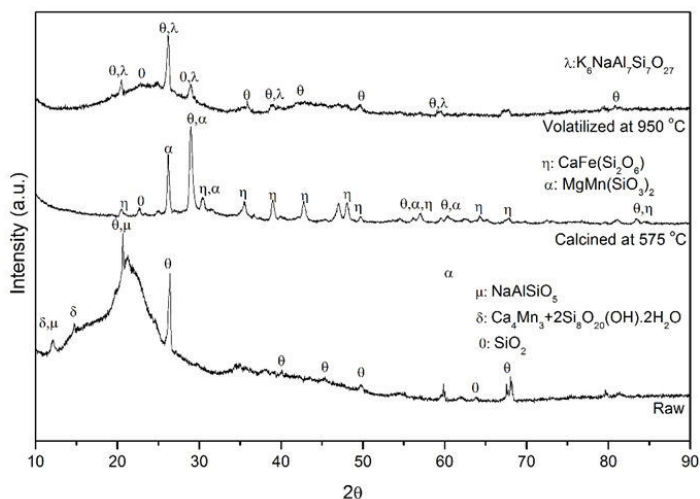


Fig.5: XRD of cassava periderm; as sun dried, calced at 575 oC and volatilized at 950 oC

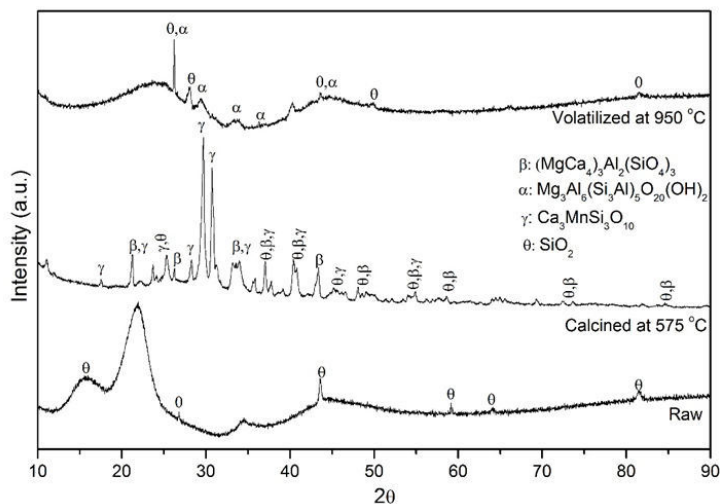


Fig.6: XRD of maize stalk; as sun dried, calced at 575°C and volatilized at 950 °C

4. Conclusion

All the agricultural wastes investigated have silica present in their ashes but the amounts of free silica differ. More silicates evolved during calcination while CP and MS have free silica present in their raw samples. Ash, volatile matter and carbon contents for SB, CP and MS are 1.73, 4.93 and 4.80%; 71.30, 58.50 and 66.37%; and 18.60, 29.40 and 22.53% respectively. SB and MS could be employed in adsorption application due to high surface areas. There is a need to establish relationships between the various stages of TGA and the proximate products as this work observe a trend between them. More detailed investigation is required to establish these correlations.

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