


Extraction of Silica from Sugarcane Bagasse, Cassava Periderm and Maize Stalk: Proximate Analysis and Physico-Chemical Properties of Wastes

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Abstract Indiscriminate disposal and burning of agricultural wastes constitute environmental pollution and increase in greenhouse gases emission. Renewable nature and availability of agricultural wastes has stimulated researchers to explore “wastes to wealth creation” policy. Three agricultural wastes were investigated for potential use for silica production. Proximate analysis, thermogravimetric analysis (TGA), compositional analysis, calcination and statistical analysis were carried out to quantify the ash and establish presence of silica. Response surface methodology was used for statistical analysis of CP calcination. The proximate analysis showed that sugarcane bagasse, cassava periderm and maize stalk ash contents are 1.73, 4.93 and 4.80%, respectively. The EDS results showed that their ashes contain 5.22,

6.10 and 7.01% silicon, respectively. XRF results revealed presence of 38% SiO₂ in CP ash. XRD revealed presence of silica and silicates phases. TGA shows that their calcination temperature must be above 500 °C. Numerical optimization of CP calcination gave optimum condition of 700 °C for 270 min to attain 82% weight loss. Calcination regression equation exhibited high coefficient of determination (R²) of 0.8225. The three wastes contain silica and silicates from which silica could be extracted. Calcination temperature and time have been established to be significant in ash content enhancement.

Keywords Proximate analysis · Calcination · Agricultural wastes · Cassava periderm · Maize stalk · Sugarcane bagasse

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Introduction

Several researches have focused on agricultural wastes recycling for extraction of useful products such as cellulose, lignin, carbon, silica, silicon, inhibitors, adsorbents and bio-fuels [1–8]. Most of the research efforts principally focus on a product, neglecting by-products without considering the proportions of other constituents. Narnaware et al. [9] produced solid briquettes and gas from vegetable wastes. Shao et al. [10] and Moreira et al. [11] produced activated carbon, fuel-gas and oil from bio-wastes. These efforts 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 wastes gives approximate information on the elemental composition. Proximate analysis provides information on components such as moisture, volatile matter, fixed carbon and ash contents. Useful products from

the wastes target exploitation of these components except moisture content. Ash contains compounds which enables them to be exploited for extraction of silica [3, 14, 15], pozzolanic material in concrete [16] or reinforcement in composites [14, 17–19]. Generally, more detail information from ultimate analysis are required for fossil fuel applications [20–22].

Procedures required for agricultural wastes recycling usually involve either pyrolysis or calcination as effective thermal treatments. Solid residue as ash or char is obtained after calcination or pyrolysis, respectively but the later could be obtained from the former [10]. Data obtained from thermogravimetric analysis (TGA) indicates temperatures under which the thermal treatment could be carried out [23, 24]. Optimum pyrolysis/calcination temperature is essential for economic, technical feasibility and viability of using the waste in commercial production [25]. Some previous works chose convenient varied temperatures without prior knowledge of thermal behaviour of agricultural wastes been calcined [26, 27]. Temperature of pyrolysis has been shown to vary for most agricultural wastes investigated by Cheng et al. [22] and El-Sayed, Mostafa [28]. The duration of this thermal treatment cannot be obtained from TG/DTG curves. Time is an important factor to consider in material processing.

The main aim of this study is to investigate the physico-chemical characteristics of cassava periderm (CP), maize stalk (MS) and sugarcane bagasse (SB) focusing on their ash contents. The selected agricultural waste residues are substantial in many developing countries like Nigeria [29]. The ash is intended for extraction of silica. Silica has been utilized for several applications across many industries such as pharmaceuticals, archeology, biomedical, electronics and feedstock of silicon (birth of semiconductor revolution). It has been used as intensive blue light emitter, fining agent in food industry, powder flow agent in pharmaceutical industry, extra-terrestrial particles collectors, DNA and RNA extractors, hard abrasives in toothpaste, desiccant, capacitors and silicon production [8, 30–37].

Materials and Methods

Materials

Cassava periderm, maize stalk and sugarcane bagasse used in this study 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 Oluode Arain, Osogbo, Osun State. Analytical electronic balance (HX302T, accuracy is ± 0.01 g) was used for all weight measurement.

Methods

Preparation of Agricultural Wastes

The maize stalks were left to dry in-situ on the farm. Leaves, roots and tassels were removed and then washed with water to remove contaminants (especially soil). The stalks were sliced and chopped into smaller sizes of 3–5 cm. The roots and leaves of sugarcanes were removed before thorough washing to remove contaminants. Bagasse was obtained after manual juice extraction. The brown back of cassava (periderm) was manually removed, washed several times until fine sands were not obvious from the rinsing water. The three wastes were sun dried for maximum of 14 days. Each of the samples was dried inside a 65 cm stainless tray and thereafter stored in separate air-tight polyethylene bags.

Proximate Analysis of Wastes

The stored AWs were pulverized using laboratory blade mill according to ASTM standard as in Table 1. The sieves used are ASTM E 11 Nos 12 (1.70 mm), 18 (1.00 mm), 50 (0.30 mm) and 170 (0.09 mm). Fractions from 18 to 50 mesh sizes were used. Aluminium pans and porcelain crucibles were initially labelled and tared according to ASTM standards [38, 39]. These were kept in a desiccator until use. The tared masses of the containers were used for weight loss calculations.

Table 1 Standard test methods used for proximate analysis

Components	ASTM standards used
Moisture content	E 1756 (2008): Standard test method for determination of total solids in biomass [39]
Ash content	E 1755 (2001): Standard test method for ash in biomass [38]
Volatile matter content	E 872 (1998): Standard test method for volatile matter in the analysis of particulate wood fuels [40]
Fixed carbon content	E 1755 (2001) and E 872 (1998) [38, 40]

The components of different AWs were determined using ASTM standards methods as enumerated in Table 1. Six samples were used and their results averaged for moisture content determination. Minimum of three samples were used and their results averaged, for ash and volatile matter contents. All calculations were based on Sun dried measurements and formula used were obtained from the standards utilized [38–40].

Thermogravimetric Analysis and Calcination Temperature

Thermogravimetric analysis (TGA) was performed to investigate the thermal behaviour of the selected agricultural wastes from room temperature to 825 °C. This information usually indicates the pyrolysis/calcination temperatures but not the duration of the process. Thermal treatment is usually performed using electric furnaces, at high temperatures. For economic reasons, current (I) and time (t) are directly proportional to quantity of heat (Q) generated. As shown in Eq. 1, resistance (R) is a material property, I and t are very crucial in cost estimation.

$$Q = I^2Rt \quad (1)$$

Due to high thermal stability of CP among the selected wastes, its samples were investigated for optimum calcination temperature and time. Porcelain crucibles were tarred at 450 °C, their masses were measured and recorded as m_1 . 1 g each of 15 CP samples were measured into tarred porcelain crucibles. They were placed in a muffle furnace preheated to 450 °C at 10 °C/min heating rate. Three samples were removed after an hour interval for a maximum of 5 h. The samples were removed and kept in the desiccator to cool to room temperature before weighing. The final masses were measured and recorded as m_2 . This was repeated for 500, 600, 700 and 800 °C.

Design Expert (Version 10) was used for analysis of variance (ANOVA). This was carried out using historical data design of Response Surface Methodology (RSM) with 25 data points. Calcination temperature and time are the numerical factors used to study their synergetic effects on weight loss as the only response. The data used are as obtained from calcination of CP from 450 to 800 °C and 60 to 300 min for temperature and time, respectively. Data point at 450 °C for 60 min was neutralized for regression model improvement.

Characterisation

Agricultural wastes, their proximate analysed (ash and carbonized) samples, and calcined samples were characterized. Schottky Field Emission Scanning Electron Microscopy (SEM JSM 7600F) and Energy Dispersive X-Ray spectroscopy (EDS) were carried out for morphology and chemical compositions. SEM images were obtained at a voltage of

20.0 kV and rendered at $\times 1000$ magnification. EDS scan was obtained at low magnifications. X-Ray Diffraction (XRD; Bruker D8 with generator operated at 40 kV and 30 mA) analysis was conducted to determine phases and crystallinity of the wastes. XRD diffractograms were measured over a range of 2θ from 10° to 90° at step size of 4 degrees per minute. X-Ray Fluorescence (XRF) analysis was carried out using Rigaku ZSX-Primus II. TGA analysis was carried out using Thermal Analyzer (TA Instruments Q600 SDT) with nitrogen gas for purging. Initial mass of 11.5 mg was loaded and heated from room temperature to 825 °C at 10 °C/min.

Results and Discussion

Proximate Analysis

Figure 1 shows that moisture content of MS with 6.30% is the least while 8.37% for SB is the highest. Volatile matter and fixed carbon contents range between 58.50–71.30% and 18.60–29.40%, respectively. Ash contents for all the three wastes have lowest proportions while similar observations have been reported [1, 10, 23]. The trend also conformed to the report of McKendry [41] that the proportions of constituents reduces from volatile matter, fixed carbon to ash content. CP has the highest fixed carbon and ash contents thus gave 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 investigated wastes could be used for production of activated carbon and bio-fuels [10, 25, 42]. Although ash content is considered undesirable for biomass conversion to bio-fuels, it has been found to be useful as pozzolanic materials and extraction of silica [3, 16, 29, 43].

TGA and Calcination

The chars left after thermogravimetric analysis (Fig. 2) correspond to the proportions observed during proximate analysis with values of 34.33, 27.33 and 20.33% for CP, MS and SB, respectively. Thermal characteristics of the three biomass differ particularly during volatilization stage where hemicellulose, cellulose and lignin decomposition occurred [28, 44]. 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 for CP [23]. The first stage of moisture drying occurred up to 200 °C owing to 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 [45].

Fig. 1 Comparison of proximate analysis components of sugarcane bagasse, cassava periderm and maize stalk

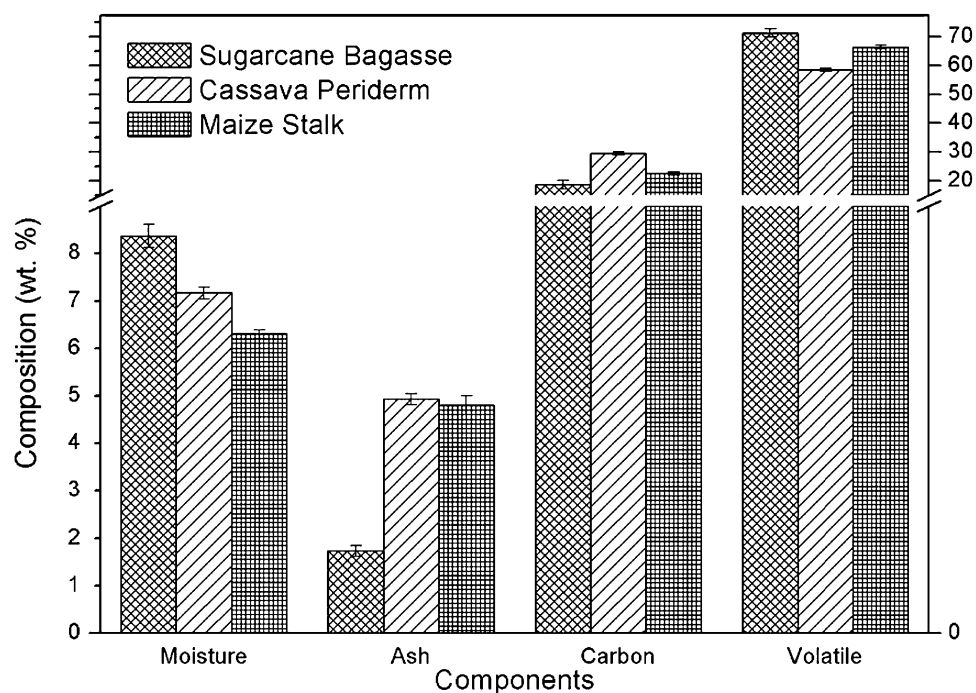
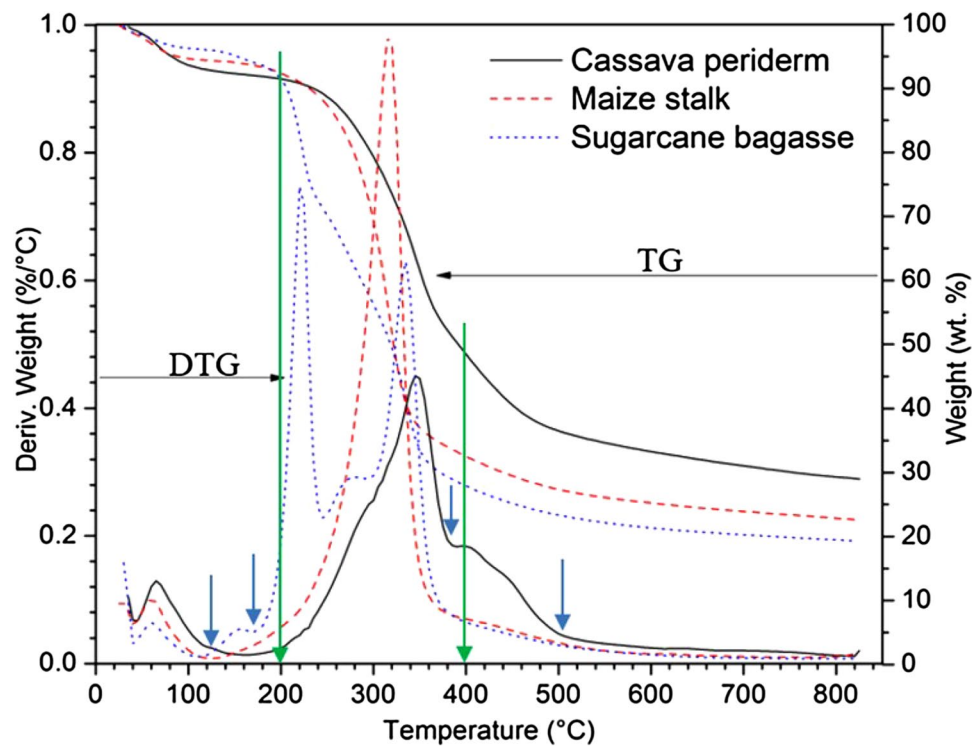


Fig. 2 TGA and DTG curves for sugarcane bagasse, cassava periderm and maize stalk from room temperature to 825 °C



The DTG curves show kinetics of pyrolysis of the biomass and reveal that dehydration started at initial temperatures but was pronounced from 60 to 120 °C. Volatilization stage involves decomposition of hemicellulose which occurs between 180 and 340 °C, cellulosic breakdown between 250 and 450 °C, and lignin decomposition between 200 and

800 °C [46, 47]. The rapid weight loss (as obvious from the TG curves) can be attributed to hemicellulose and cellulose decomposition [48]. 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

between 200 and 260 °C while cellulosic compounds breakdown occurred between 270 and 300 °C [49]. Due to lignin decomposition temperature range, fractions may be lost concurrently with cellulosic breakdown. The high inflection points at this volatilization stage could also be attributed to the exothermic features of decomposing phases. The volatile matters could be collected as liquid or gaseous biofuels [10, 11, 43]. Carbonization of the remaining chars at this stage could be attributed to the decomposition of lignin (mainly carbon compounds) which started before 400 °C in two main stages. For instance in CP, rapid decomposition occurred up to 500 °C and then at a slow rate above 500 up to 800 °C.

The decomposition profiles at final stage can easily be associated with the fixed carbon contents from the proximate analysis as in Fig. 1 [24]. Similarities also exist when the heights of volatilization stage (from TG curves) and the volatile matter columns in proximate analysis were compared. The weight losses 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.

The final mass of each samples were estimated using *Mass*, $m = m_2 - m_1$. Average mass was calculated and plotted against time. In Fig. 3, mass loss characteristics of CP at different temperatures was investigated to determine optimum duration for calcination. Calcination at temperatures below the main volatilization stage from TG-DTG curves (Fig. 2) results in low mass loss, even for 5 h. Calcination carried out at 200 °C above the end point of volatilization stage gave the same mass loss obtained at 800 °C after 3 h.

For economic reasons, duration is vital to cost of calcination since the amount of energy utilized is directly proportional to time (Eq. 1). Time required for two main stages of calcination will determine the final cost. The first stage requires a constant heating rate which correspond to dehydration stage in Fig. 2. The second stage is characterized with intermittent current usage that could be attributed to volatilization stage. Since calcination takes place in the presence of air/O₂, heat is generated by the combustion of volatile matter from the biomass. The furnace temperature is observed to be above the set temperature during which no current is utilized [50]. Inappropriate calcination temperature (e.g. below 700 °C for CP) requires more time to attain same mass loss.

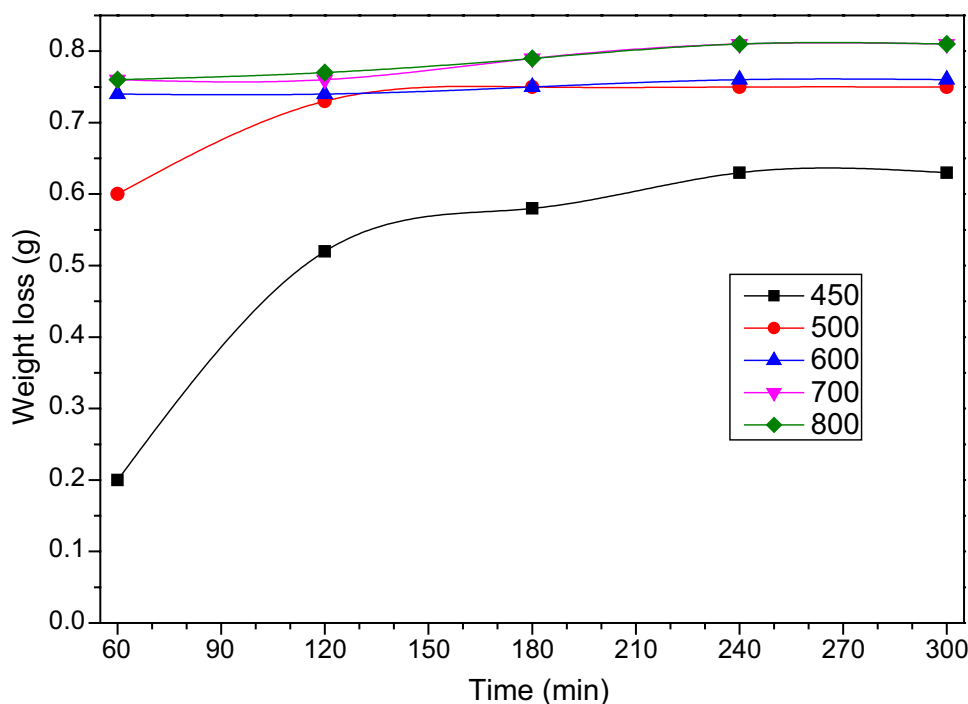
Table 2 shows the analysis of variance for the effect of calcination temperature and time on weight loss during

Table 2 ANOVA for Response surface methodology (RSM) for calcination temperature (T) and time (t)

Source	Sum of squares	Mean square	F value	Prob > F
Model ^a	0.12	0.024	16.68	<0.0001
T	0.090	0.090	63.49	<0.0001
t	0.015	0.015	10.44	0.0046
T ²	0.025	0.025	17.60	0.0005
t ²	9.130E-4	9.130E-4	0.64	0.4333
Tt	1.583E-3	1.583E-3	1.11	0.3052
Residual	0.026	1.421E-3		
Cor total	0.14			

^aAdjusted R-Squared = 0.7732, Predicted R-Squared = 0.7090

Fig. 3 Mass loss of cassava periderm during calcination at different temperatures



thermal treatment of CP within the range of our investigation. Regression model equation in terms of temperature (T) and time (t) is given in Eq. 2. The model's F-value of 16.68 and Prob > F of < 0.0001 imply that the model is highly significant. Values of "Prob > F" less than 0.05 indicate model terms are significant. Hence, calcination temperature, time and second degree of calcination temperature are significant terms. Calcination temperature is the most significant factor for calcination. The relevance of temperature during calcination is also supported by highest F-value of 63.49 and highest actual factor coefficient. Two factors interaction between temperature and time is insignificant since the Prob > F value is greater than 0.1. Consequently, temperature and time have positive effect on weight loss while second degrees and interactions of both factors have negative effects. Ash contents has been reported to increase with temperature which is in tandem with this study [25].

The quadratic model obtained from the data exhibited high coefficient of determination (R^2) of 0.8225. The predicted R-Squared of 0.7090 is in reasonable agreement with the Adjusted R-Squared of 0.7732. Adequate precision value of 12.397 indicates adequate signal for both temperature and time within the design space. The interactive effects of calcination temperature and time on weight loss are shown in Fig. 4. Figure 4a shows effects of calcination temperature and time on weight loss. At low calcination temperature 500 °C for CP (Fig. 2), optimum weight loss cannot be attained. The maximum weight loss attainable for duration of 300 min at this temperature is 71%. Weight loss of 72% was attained at 600 °C within the first 60 min. 82% weight loss at 700 °C after 232 min is predicted by the model.

Figure 4b presents interaction boundaries for all the design space. The optimum temperature could be projected around 700 °C. The difference between the upper and lower limits at any instance of temperature decreases as temperature increases. This minimizes error values for the design space. It is obvious that temperature above 717 °C has no more positive effects on weight loss of CP. Using numerical optimization constraints, calcination at 700 °C for 270 min is the optimum condition for 82% weight loss. This implies that selection of optimum conditions for calcination improves ash yield in ensuring effective volatile matter removal.

$$\text{Weight Loss, mg} = -739 + 3.951T + 1.185t - 2.651 \times 10^{-3}T^2 - 1.051 \times 10^{-3}t^2 - 8.062 \times 10^{-4}Tt \quad (2)$$

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 at $\times 1000$ are shown in Fig. 5. The pores observed in the raw samples of SB (a_1) are attributed to the juice removed from the pulp while that of MS (a_3) represents the skeletal structure of the stalk. The morphology of raw CP (b_1) is unchanged even during volatilization (b_3) because of its bulky flake nature. CP structure was completely crumbled during calcination (b_2) due to volatile matter and fixed carbon removal [51]. This structural deformation was also observed for SB (a_2) and MS (c_2). During volatilization of SB and MS, their structural frameworks were retained because decarbonization did

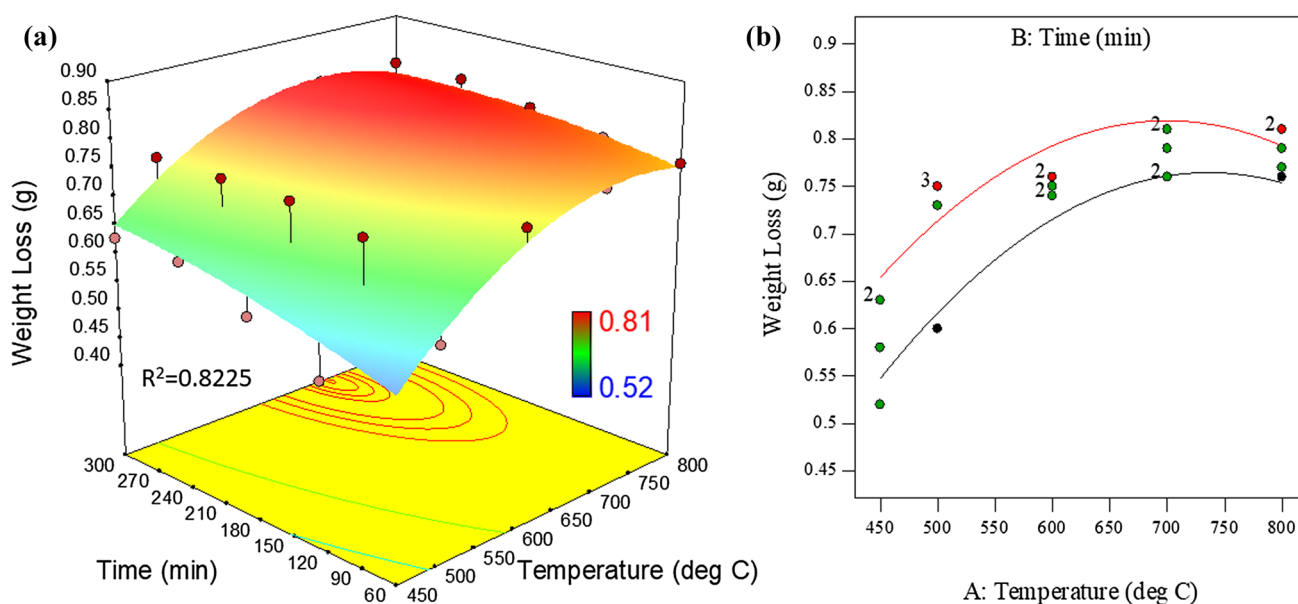
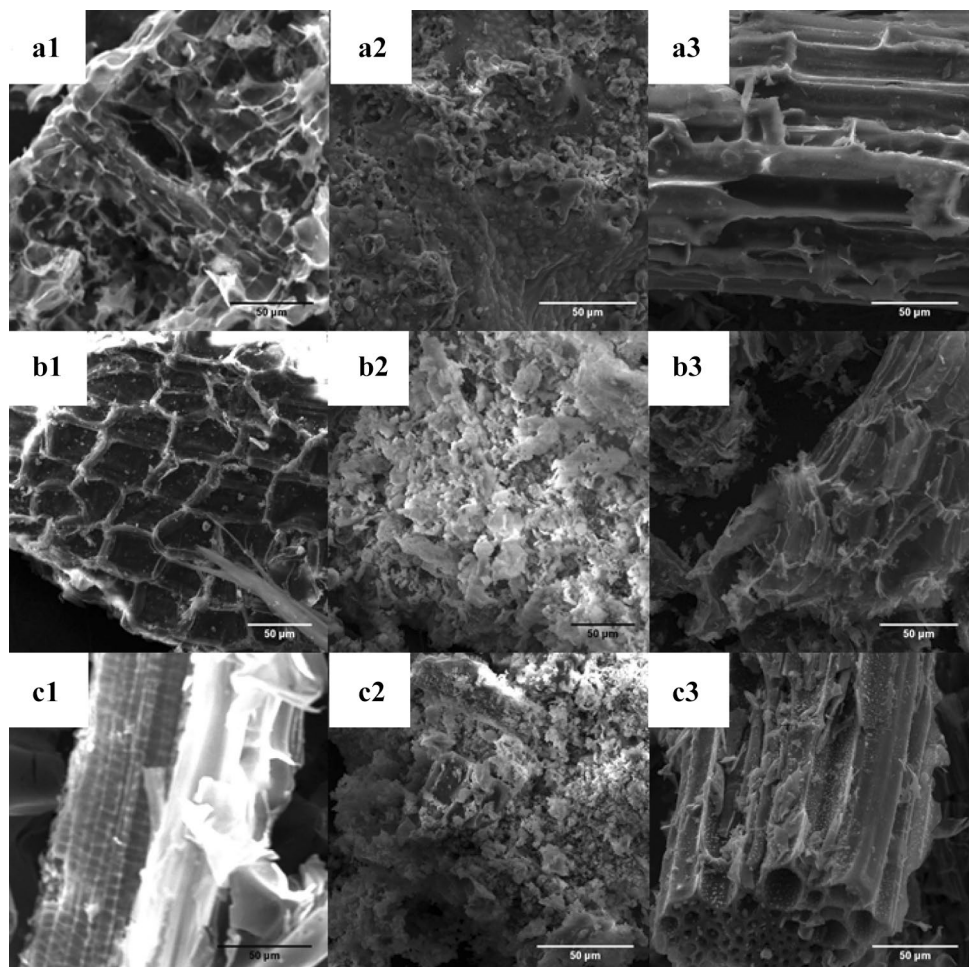


Fig. 4 Graphical effects of temperature and time on weight loss during calcination by **a** 3D surface model plot, and **b** interaction model plot

Fig. 5 SEM micrographs for sugarcane bagasse, cassava periderm and maize stalk. [a sugarcane bagasse, b cassava periderm, c maize stalk, 1 raw, 2 calcined at 575 °C, and 3 volatilized at 950 °C]



not occur according to the procedure [40]. Lignin, which forms the main structural supporting tissue of plants, was decomposed but its high carbon percent left was strong enough to support the structure for both biomasses [52–55]. Through pores were formed by both SB (a_3) and MS (c_3) thus increasing the rate of their decompositions during volatilization stage (Fig. 2). 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 residues [44].

To establish how much siliceous products could be obtained from the wastes, Energy Dispersive X-ray spectroscopy (EDS) was carried out. The compositions of each biomass at different stages are shown in Table 3. Silicon is more abundant in MS than in CP with least amount observed in SB. Extraction of silica from ashes obtained after calcination has been established from previous researches [3, 14]. 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. The residues obtained at 575 °C have higher silicon contents because both volatilization and combustibles are removed. The products of this procedure

could be associated with calcination products. The calcination temperature is also above the volatilization temperatures from thermogravimetric analysis (Fig. 2).

Figures 6 and 7 show the progress of calcination process for CP where oxides of metals were increasing. The percentage of carbon decreased as temperature and time increased because of oxidative environment in the furnace chamber. Combustion of volatile matters and carbon takes place in air. The heat generated at this stage is responsible for increased furnace temperature and intermittent no usage of electricity during the procedure. Carbon, which was above 80% (Fig. 6a), dropped to proportions below 40 (Fig. 6b), 16 (Fig. 6c), 11 (Fig. 6d) and 10% (Fig. 7) at 500, 600, 700 and 800 °C, respectively. The different gradients observed for each species could be attributed to temperature and time effects on volatile matters. This also corresponds with the observed features in Fig. 2 as regards stages of volatile matter decomposition. There is no much difference between the proportions of various chemical species at 700 and 800 °C. This shows that calcination of CP at 700 °C for a duration of 300 min is adequate. This condition corresponds with optimum condition from numerical optimization of the quadratic

Table 3 Chemical composition of sugarcane bagasse, cassava periderm and maize stalk

Element	Composition (at.%)								
	Sugarcane bagasse			Cassava periderm			Maize stalk		
	Raw	575 °C	Volatilized	Raw	575 °C	Volatilized	Raw	575 °C	Volatilized
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
Na	0.13	2.13	<<	<<	<<	0.12	<<	<<	<<
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	<<	<<	<<	<<	<<	<<	<<

<< Below detection level

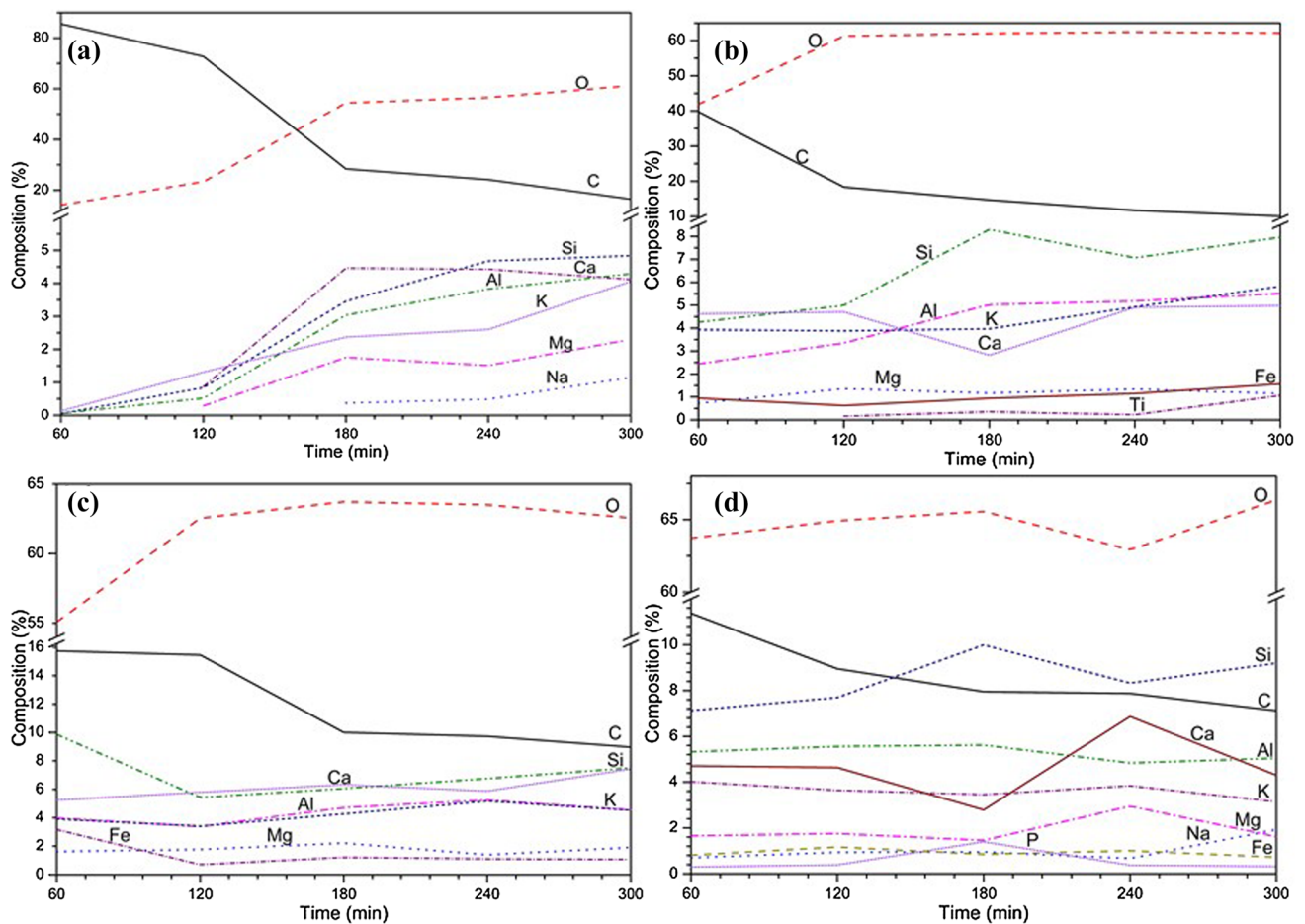
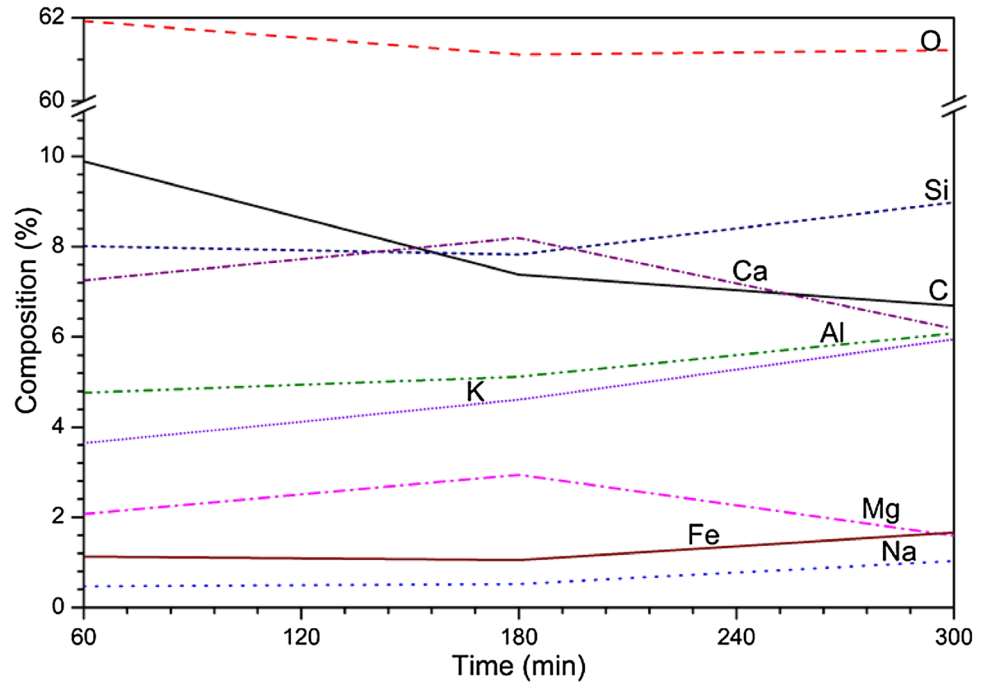
**Fig. 6** Chemical composition of cassava periderm calcined at different temperatures **a** 450 °C, **b** 500 °C, **c** 600 °C and **d** 700 °C

Fig. 7 Chemical composition of cassava periderm calcined at 800 °C



model in Eq. 2. Progressive increase in the percentage of oxygen validates formation of the oxides whose values correspond with the XRF results in Fig. 8 at 800 °C. Figure 7 shows the quantitative elemental composition (EDS) of solid residue after calcination of CP at 800 °C. The proportion in descending magnitude is Si, Ca, Al, K, Fe, Mg and Na. This order is verified by the XRF result in Fig. 8 except for detection of more oxides due to higher efficiency of the instruments used.

Phase Identification

Figures 9, 10 and 11 show the XRD spectra of SB, CP and MS of sun-dried (raw) and proximate analysis products. The samples contain silica and silicate phases depending on the treatments. Figure 9 indicates that free silica is only formed during calcination of SB at relatively high temperature. The free silica recombined at volatilization temperature of 950 °C to form complex silicates (magnesium aluminium

Fig. 8 Chemical composition of cassava periderm using XRF

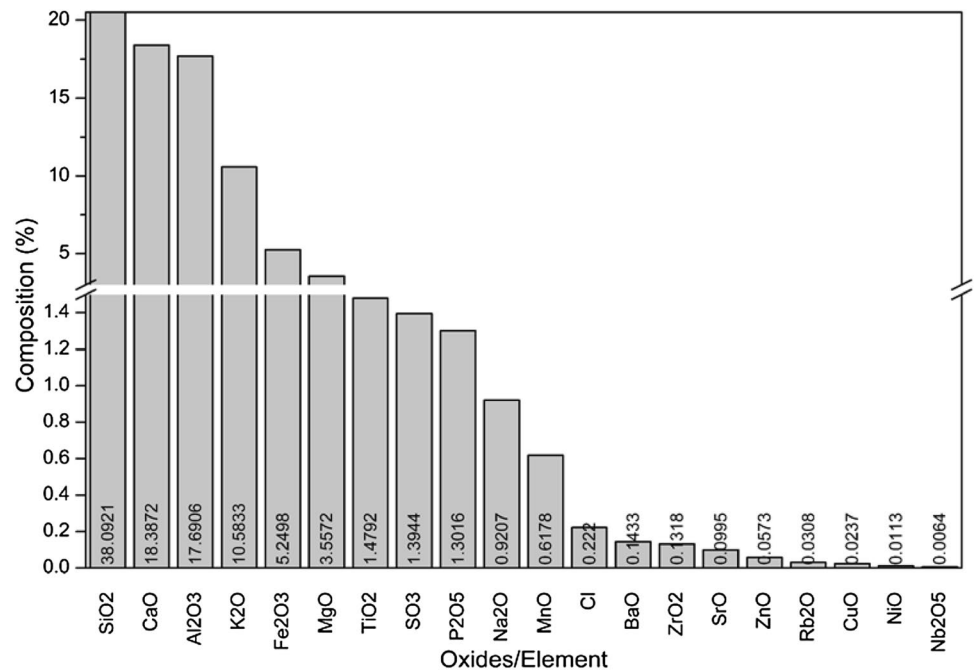


Fig. 9 XRD of sugarcane bagasse of proximate analysis products for as sun dried, calcined at 575 °C and volatilized at 950 °C

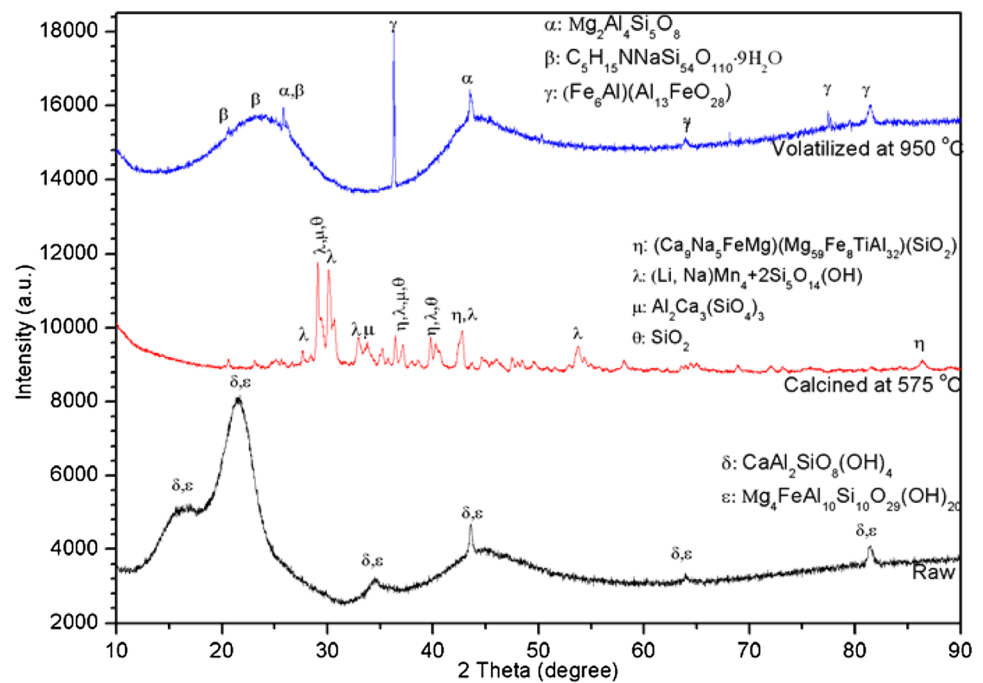
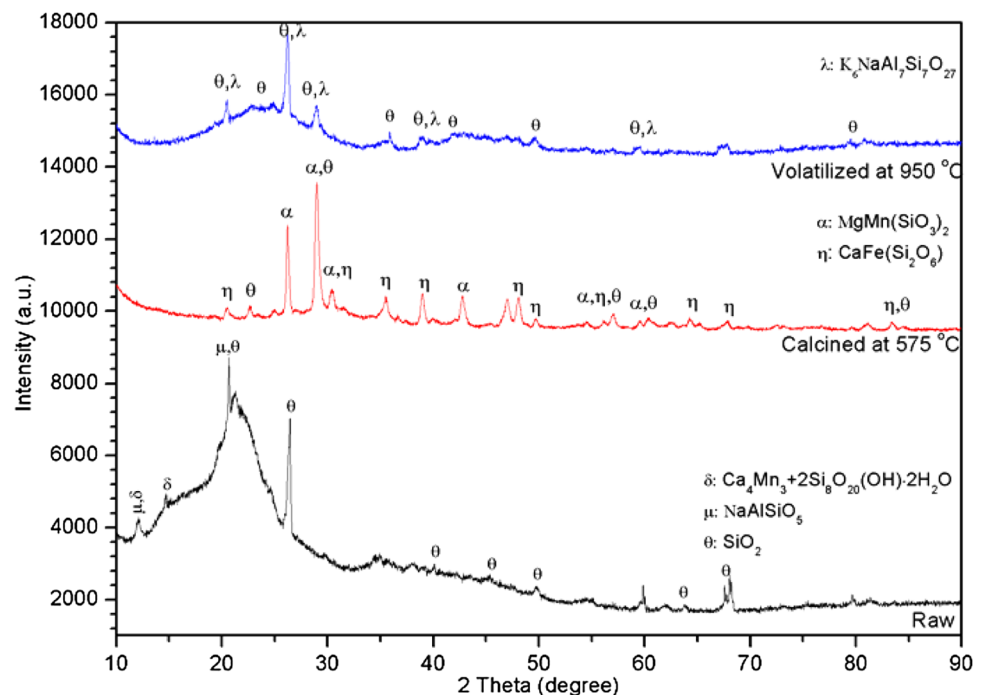


Fig. 10 XRD of cassava periderm of proximate analysis products for as sun dried, calcined at 575 °C and volatilized at 950 °C

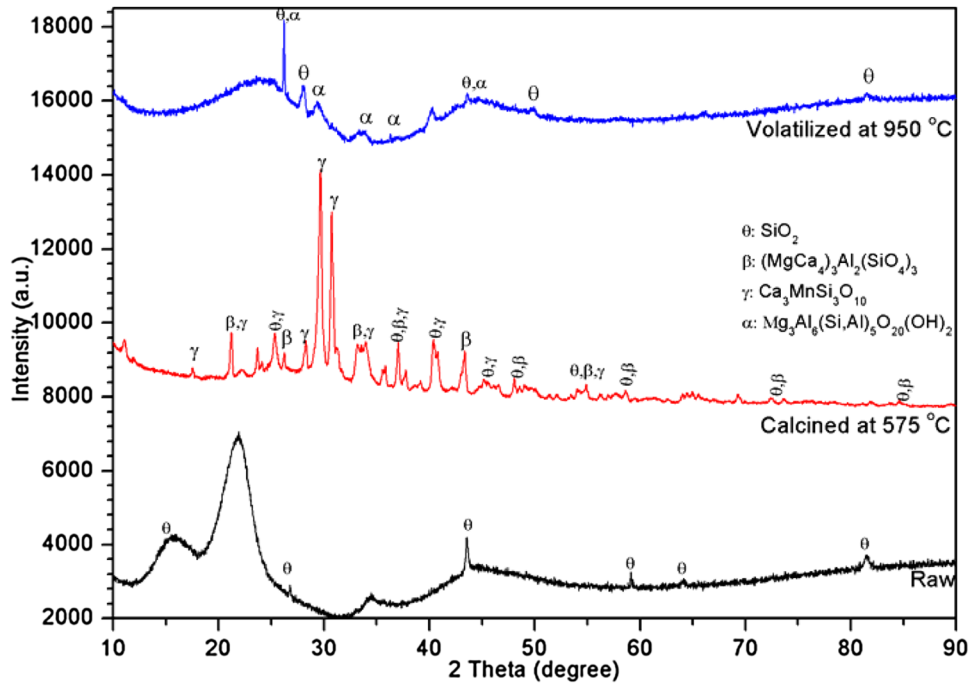


silicate and sodium tetramethyl ammonium silicate hydrate). Silicon is present in the raw SB as chantalite (calcium aluminium silicate hydroxide) which is an inorganic mineral with a tetragonal structure. It is also detected as magnesiocarpholite (magnesium iron aluminium silicate hydroxide) with similar mineral source but orthorhombic structure [56]. From Figs. 10 and 11, 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 both in CP and MS. The free silica can be enhanced and/or extracted by calcination and acid leaching or via hydrometallurgical process, sol-gel [14, 26].

Extraction of silica from siliceous compounds like agricultural waste residues is important. Availability of abundant agricultural wastes for silica extraction has been reported

Fig. 11 XRD of sugarcane bagasse of proximate analysis products for as sun dried, calcined at 575 °C and volatilized at 950 °C



[29]. They have properties similar to most siliceous minerals and gemstones but usually reactive in alkaline medium at low temperatures [15]. The use of chemical extractive process like sol-gel dissolves unwanted silicates. This could impair the purity of the final silica product [57]. This reactivity features of the ash is also employed leaching unwanted soluble phases [58].

Conclusions

- All the agricultural wastes investigated have silica present in their ashes but the amount of free silica differ. 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.
- All the selected agricultural wastes have different thermal decomposition profiles. Thermogravimetric analysis gave a calcination temperature of above 500 °C for SB, CP and MS.
- EDS results showed that samples calcined at 575 °C contain highest silicon contents of 5.22, 6.10 and 7.01% for SB, CP and MS, respectively. Acid leaching the ash could be effective for removal of unwanted oxides. Presence of amphoteric oxide like aluminium oxide should be avoided as may impair final silica purity.
- XRF result shows that CP ash contains more than 38% SiO_2 with oxides of Ca, Al, K, Fe, Mg, Ti, S, P, Na and Mn. It also contains traces of Cl, BaO, ZrO_2 , SrO, ZnO, Rb_2O , CuO, NiO and Nb_2O_5 .

- Calcination of CP at 700 °C for 240 min in a muffle furnace gave 81% weight loss. Numerical optimization using RSM gave an optimum calcination condition of 700 °C for 270 min for 82% weight loss.
- XRD spectra confirm the presence of silica and siliceous compounds in the selected agricultural wastes prominently after calcination of their raw samples.

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