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Research article

SYNTHESIS AND CHARACTERIZATION OF SILICA PARTICLES FROM YAM AND CASSAVA PERIDERM

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Abstract

In this study, silica particles were synthesized from periderms of yam and cassava. The synthesis process involved the calcination of both periderms to form their respective ashes at 600, 700, 800, and 900°C, followed by purification of the ashes with dilute sulphuric acid (H_2SO_4) and leaching of the ashes in a NaOH solution to form sodium silicate. The sodium silicate was further titrated with HCl to precipitate silica out in gelatinous form. The resulting fine particles were then characterized using X-ray Fluorescence, X-ray diffraction, and Fourier transform infrared spectroscopy. The study showed cassava and yam periderm could be precursors for synthesizing silica nanoparticles with different characteristics and properties. Calcination at 600°C was found to give the highest yield of silica, with the yam and cassava periderms giving 80.89 % and 91.35%, respectively. The findings of this study have potential applications in industries such as pharmaceuticals, construction, and foundry.

Keywords: *Sol-gel; Nanoparticles; Cassava Periderm; Yam Periderm; Silica*

1. Introduction

World municipal solid waste (MSW) output is estimated to be at one billion tonnes per year, with estimates of two billion tonnes per year by 2025 [1]. Out of the MSW output, waste output from the agricultural sector is becoming a significant problem as the waste approaches its near capacity [2]. Many of these agrarian wastes arise from the consumption of farm produce and unwanted harvest materials from crops like maize straw, yam periderm, maize cob, sugarcane bagasse, cassava peels, etc., due to the demand for feeding the increasing human population [3]. Unfortunately, these wastes are not well managed in many developing nations, especially African countries, because they do not know the potential risk of pollution caused by agricultural waste [4]. It is essential to reduce pollution levels and equilibrium of the ecological system by conserving agricultural waste material [5]. To achieve this objective, converting them into valuable materials and shifting from a less cost-effective paradigm of conventional land-occupying substances to transforming waste into something more useful is necessary [6]. Many conservation strategies, such as recycling, dematerializing, and composting, have been explored by many researchers to combat the disposal of hundreds of millions of tonnes of waste substances from agricultural practices [7]. The most potent way of utilizing agricultural waste from tuber crops is to convert them into silica [7]. The extraction of silica from

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naturally occurring crop plants and agricultural waste, such as tuber periderm, cassava periderm, yam periderm, and maize residue, has rekindled attention in recent decades due to the numerous usefulness of silica in areas of medicine, Engineering, chromatography, and catalyst [8].

Agricultural wastes are remnants from processing raw agricultural goods such as fruits and crops. Depending on the farming operation, agricultural waste might be gas, liquid, or solid [4]. Human activities and farming operations have increased the atmospheric concentration of greenhouse gases during the previous two centuries [9]. Agricultural wastes have been used to make various valuable products, which has helped to relieve the problems connected with getting multiple useful products, which has helped to alleviate the issues associated with getting rid of them. Agricultural wastes have been used to make ceramic products, activated carbon, silica, composite reinforcement, and lignin [10]. The two agricultural wastes (cassava and yam peels) used in this project have gone from environmental inconvenience to vital economic resources.

After oxygen, silicon (Si) is the next in line to be the most abundant element on the Earth's crust [11]. Silicon is mainly in polycrystalline, amorphous, or crystalline forms like silica (SiO_2). It may be found in nature, sand, and soil and can be extracted from plant crops via chemical synthesis. Silicon nanoparticles can be synthesized from silicon feedstock using several processes, including combustion synthesis, electron beam evaporation, chemical synthesis, plasma-enhanced chemical vapor deposition, and pulsed laser deposition [12]. The production of silicon particles from bulk materials necessitates chemical methods that are either energy-consuming to produce poisonous compounds or are slow and usually difficult [13].

Silicon dioxide, commonly known as Silica, is a fundamental material widely used in the electronics and ceramics sectors. Due to their small size, ultrafine silica powders offer a broad range of technological applications [14]. Silica-based refractory goods offer superior qualities over agri-based refractory materials, such as solid strength at high temperatures, making them ideal for furnaces [15].

Therefore, this research aims to alleviate the adverse environmental effects of agricultural wastes and enhance the value of cassava and yam periderm by converting them to silica particles.

2. Materials and Method

2.1. Materials Preparation

The primary materials used in this work are yam and cassava peels obtained from Babcock University cafeteria in Ilisan-Remo, Ogun State, Nigeria. Other materials include NaOH, HCl, Distilled water, filter paper, and foil paper. The equipment used in carrying out the research consists of a Laboratory Oven, Magnetic stirrer with hot plate, Muffle furnace, Glass beaker, Erlenmeyer flash, Mortar and pestle, Glass funnel, and Centrifuge Pipette.

The sol-gel method started with the purification and acidification of periderms. The two periderm samples were washed in distilled water separately to remove sand and dust particles. The samples were transferred to a tray and kept under the sun to dry for 5 days. After drying the samples, the cassava and yam periderms were divided into four portions and calcined in a muffle furnace at 600°C, 700°C, 800°C, and 900°C, respectively. The resulting silica ash was transferred to a Ziploc bag and labeled.

Acidic purification of the silica ash samples was used to eliminate unwanted metallic and inorganic impurities such as potassium, calcium, and magnesium through dilute H_2SO_4. This process used sulfuric acid (H_2SO_4) of Molar concentration 2M. The silica ash samples from yam and cassava periderm were poured into a beaker and then stirred with a magnetic stirrer on a hot plate at 50°C. The mixture was allowed to settle for 30 mins and then decanted. On settling, two layers were formed: a base layer consisting of the purified periderm ash and the top liquid layer, which contained some impurities that the sulfuric acid reacted with. After decanting, distilled water was used to rinse any trace of sulfuric acid and other contaminants. Then, the eight samples were oven-dried at a temperature of 50°C.

A weighed sample of 80 g was measured from each purified silica ash, added to 100 mL 2M NaOH solution at 60°C for 1 hour, and then filtered off. The eight pure silica ash samples obtained from calcination were dissolved in an alkaline medium to form a sodium silicate solution, following the equation for the reaction:

$$SiO_{2\,(s)} + 2NaOH_{(l)} = Na_2SiO_{3\,(aq)} + H_2O_{(l)} \tag{1}$$

Sodium silicate formation

Fig. 1 Pictures showing (A) Washed and dried Cassava Periderm, (B) Calcination Of Periderm to ash in Furnace, (C) Silica ash from periderm, (D) Preparation of reagent, (E) Washing of silica ash in acid, (F) Labeled purified silica ash, (G) Leaching of Silica Ash in Alkaline Solution, (H) Filtration of Na_2SiO_3 solution, (I) Filtrates of Na_2SiO_3, (J) Titration and precipitation of Na_2SiO_3, (K) Aging of silica gel, and (L) Synthesized aqua silica

After that, the sodium silicate solution was filtered using 150 mm semi-permeable filter paper. The residue was rinsed and discarded. At the same time, all the filtrates were kept for titration to form silica gel through precipitation.
The titration of sodium silicate (Na_2SiO_3) filtrates with hydrochloric acid (HCl) was done at room temperature under vigorous shaking until silica gel was precipitated. While this process was going on, a pH meter was used to monitor the pH level of the reaction.

$$Na_2SiO_{3\,(l)} + 2HCl_{\,(l)} \rightarrow SiO_{2\,(aq)} + 2NaCl_{\,(l)} + H_2O_{\,(l)} \qquad (2)$$

Formation of aqua silica gel

The precipitated aqua silica gel was allowed to settle and aged for 18 hours. After aging, the soft gel was gently broken, and 50 mL of water was added to make a slurry. The supernatant was discarded, and the gels were transferred into a beaker and dried at 80ºC for 24 hours to produce silica xerogels. The Silica xerogel was washed with distilled water to effectively remove minerals and traces of salt from the silica. This is because washing the dried silica (xerogel) with distilled water is more productive than washing the silica gel (aqua gel) before drying and removing minerals.

2.2. Sample Characterization

The sample was characterized using X-ray Fluorescence (XRF), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Fourier Transform Infrared spectroscopy (FT-IR), and Thermogravimetric analyses (TGA) techniques. XRF analytical technique was used to determine the elemental composition of the silica. The samples were measured using an energy-dispersive X-ray fluorescence (EDXRF) spectrometer of model "Epsilon 4" by PanAlytical, Netherlands. 20 g of the prepared sample were weighed each into a sample cup. The cups with the contents were carefully placed in their respective measuring positions on a sample changer. The current used was 14 kV for principal oxides, while kapton filters were used. The periodic table guided the selection of filters while the measurement time for each sample was 100 seconds, and the medium used was air.

X-ray diffraction (XRD) was used to determine the crystal structure and phases of the silica. The measurements were conducted at the Nigeria Geological Survey Agency`s (NGSA) office in Kaduna. The equipment used is the Rigaku Miniflex XRD (Japanese X-ray scientific and analytical), and the voltage and current used are 40 kV and 15 mA. The measurement was from the range (2θ) of 2° – 70° at a step count of 0.0200º. The X-ray source is a copper anode (λ Cu Kα = 1.5418 Å). The Measurement speed is 0.01°/min.

The SEM was made with Phenom-ProX by Phenom-World Eindhoven, Netherlands. The samples were viewed by placing them on a charge reduction sample holder and introduced into the column of the SEM machine (Phenom ProX), where it was viewed from a Nav-Cam before they were viewed using SEM mode. The image acquisitions were made after adjustments for brightness and contrast. Energy diffractive spectroscopy measurements were carried out on the samples.

The FTIR measurements were made with an Agilent Cary 630 FTIR machine. 15 mg of each prepared silica sample was loaded into the FTIR machine's sample compartment, ensuring proper measurement alignment. Measurement parameters were set, including the wavelength range of 650 – 4000 cm^{-1}, resolution of 2 cm^{-1}, and number of scans (128 scans). Transmission mode was selected for the analysis. A baseline spectrum was obtained by measuring an empty sample compartment to account for any background signals. The spectrum of the silica particles was acquired by taking a sufficient number of scans to improve the signal-to-noise ratio.

The TGA was done only on the cassava and yam periderm to measure the calcination rate under heat. The measurement was made using a PerkinElmer TGA 4000, Netherlands. The samples were dried thoroughly to remove any moisture content. After that, they were separately transferred to the TGA machine crucible. The temperature range for the analysis was set from ambient temperature to 1000°C, with a heating rate of 10°C/min under a nitrogen atmosphere to prevent oxidation.

3.0. Results and Discussion

3.1.X-ray Fluorescence Analysis

Table 1 shows the composition of cassava and yam periderm ash that were calcined at 700ºC and 800ºC, respectively, before the extraction and leaching of silica ash. The result shows that the cassava periderm ash (CPA) calcined at 700ºC has approximately 18.9% silica content, while the CPA calcined at 800ºC has about 21.5% silica content, indicating silica's presence in CPA. The composition of yam periderm ash (YPA) calcined at 700ºC and 800ºC, respectively, before silica ash extraction and leaching shows that the YPA calcined at 700ºC and 800ºC has about 8% silica content, which indicates lower silica presence in YPA compared to CPA. It was observed that Fe_2O_3, CaO, K_2O, and Al_2O_3 are the other significant constituents in the cassava periderm, while Cr_2O_3, MnO, SO_3, TiO_2, and Cl are the minor constituents. K_2O is the considerable constituent in yam periderm (>65 wt. %), while the other constituents are in minor quantities**.**

Table 1 Chemical constituent of Cassava and Yam Periderm Ash from XRF analysis

Constituents (wt.%)	SiO_2	Cr_2O_3	MnO	Fe_2O_3	SO_3	CaO	K_2O	Al_2O_3	TiO_2	Cl
Cassava Periderm @700°C	18.884	0.326	0.295	7.046	4.98	21.961	16.614	10.049	1.086	0.399
Cassava Periderm @800°C	21.532	0.74	0.555	0.9717	3.507	19.892	13.901	10.897	1.075	0.625
Yam Periderm @700°C	8.315	0.122	0.22	2.173	1.703	0.03	74.239	0.56	0.404	0.538
Yam Periderm @800°C	8.318	1.396	0.761	7.653	2.081	0.021	65.346	0.035	0.304	0.556

3.1.1. Chemical Composition of Synthesized Silica

The chemical composition of the synthesized silica from CPA and YPA at 600, 700, 800, and 900°C are shown in Table 2. CPA produced the highest amount of silica, which is similar to the report from previous research by Akhayere et al., 2022 [12]. After it was reported that treating agricultural wastes with HNO_3 or H_2SO_4 after calcination at high temperatures is a very effective technique to eliminate a high percentage of biological and inorganic impurities and produce large volumes of pure, white silica, these results support earlier research, where brownish and blackish ash were observed in all untreated CPA and YPA samples, regardless of temperature. However, treating CPA and YPA with dilute H_2SO_4, followed by leaching with NaOH, resulted in a white-colored silica powder. Based on the XRF analysis results in Table 2, nano-silica synthesized from both CPA and YPA at 600°C have greater SiO_2 particles.

The quantity of SiO_2 in the CPA samples was lowest at 900°C, yielding 81%. At lower temperatures of 600°C, 700°C, and 800°C, the yield of SiO_2 is 91.34%, 87.4% and 91.2% respectively. Some of the principal contaminants in the extracted silica are chlorine and alumina. The use of HCl in the extraction process and soil nutrients in agricultural activities appears to have resulted in high levels of these chemicals, according to Akhayere et al., 2019 [1]. The quantity of SiO_2 in the YPA samples was lowest at 900°C, yielding 37%. At lower temperatures of 600°C, 700°C, and 800°C, the yield of SiO_2 is 61.92%, 79.06% and 80.89% respectively. Significant quantities of K_2O and Cl were observed in the samples, especially at higher temperatures.

The chlorine content is more than 2% across all temperature ranges investigated, indicating that not all chlorine was neutralized in $NaSiO_3$ with the addition of HCl. The XRF results are consistent with those reported by Athinarayanan et al., 2015 [16]. Various other oxides are found to be present depending on the impurity level. The estimated average weight percentage of silica in CPA and YPA before extraction is approximately 11.16%, indicating that silica was effectively extracted from all the ash samples.

Table 2 Chemical constituents of the extracted silica at different temperatures from the XRF analysis

	Compound amount from CPA (wt.%)				**Compound amount from YPA (wt.%)**			
Constituent	**600°C**	**700°C**	**800°C**	**900°C**	**600°C**	**700°C**	**800°C**	**900°C**
SiO_2	91.349	87.404	91.21	81.369	80.889	79.058	61.924	37.184
Cr_2O_3	0.025	0.021	0.011	0.02	0.021	0.013	0.051	0.023
MnO	0.026	0.033	0.021	0.032	0.035	0.026	0.02	0.087
Fe_2O_3	0.219	0.354	0.146	0.414	0.182	0.175	0.167	0.858
SO_3	0.511	3.912	0.764	2.922	0	0.459	0.945	0.324
CaO	0.284	1.081	2.224	0.446	2.409	0.667	4.66	0.563
K_2O	0.82	0.62	0.552	2.383	1.204	8.046	13.909	25.108
Al_2O_3	2.259	2.709	2.081	2.878	1.675	1.528	1.766	2.606
TiO_2	0.314	0.328	0.247	0.368	0.091	0.086	0.167	0.939
Cl	4.042	2.096	2.668	8.968	13.236	8.859	13.973	26.286

3.2. X-ray Diffraction (XRD Analysis

3.2.1. X-ray Diffraction Analysis of Ash Periderm

The crystalline nature of the synthesized silica from YPA and CPA was investigated using X-ray diffraction. Fig. 2. depicts the XRD pattern of CPA and YPA at 800°C. The YPA samples produced showed a peak at 27°, indicating the semi-crystalline nature of the ash. At the same time, the X-ray diffraction graph of CPA did not show any visible peaks, confirming the amorphous nature of the ash.

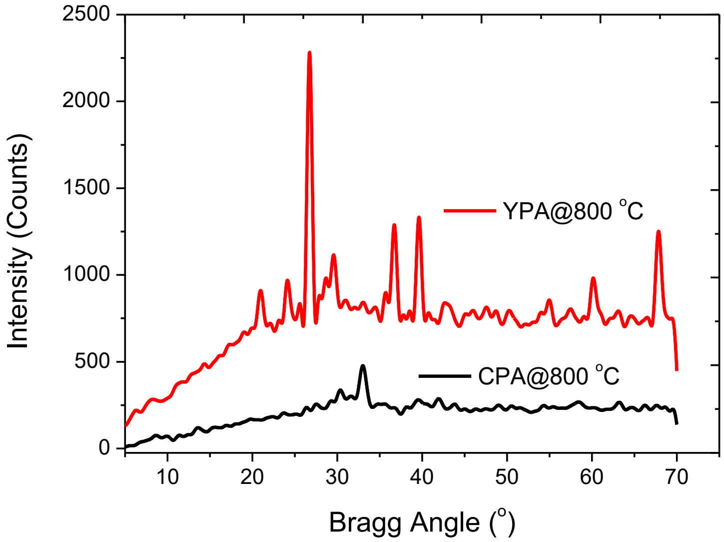
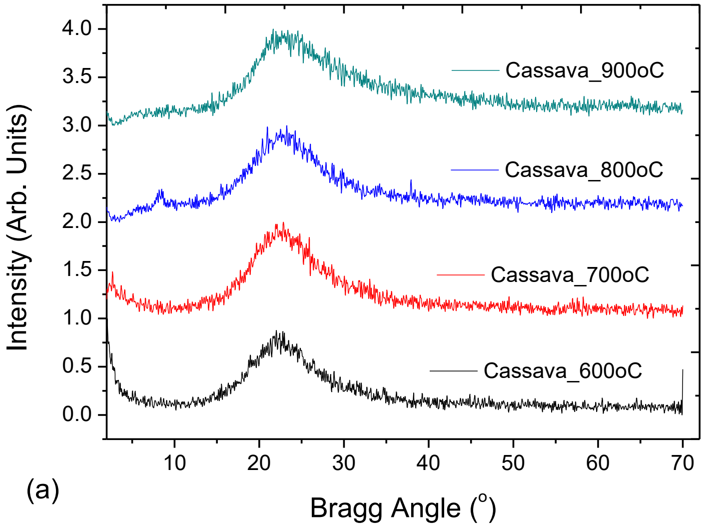


Fig. 2 X-ray diffraction patterns of cassava and yam periderm ash at 800°C

3.2.2.X-ray Diffraction Analysis of Silica

Figs. 3a and 3b show the X-ray diffraction profiles generated for silica at various calcination temperatures using cassava and yam periderm, respectively. The temperature, duration, and extraction method contributed directly to the production of amorphous silica. For CPA, all samples display a single peak at an intensity of about 780 cps and a Braggs angle of 22°, indicating the presence of amorphous silica. This result correlates with research by Athinarayanan *et al.,* 2015 [16]. We also noticed that the XRD graph of silica obtained from YPA has more peaks than silica from CPA, which means that silica extracted from YPA has more crystal structure than silica from CPA. As the calcination temperature increased, the crystallinity of silica from YPA increased.



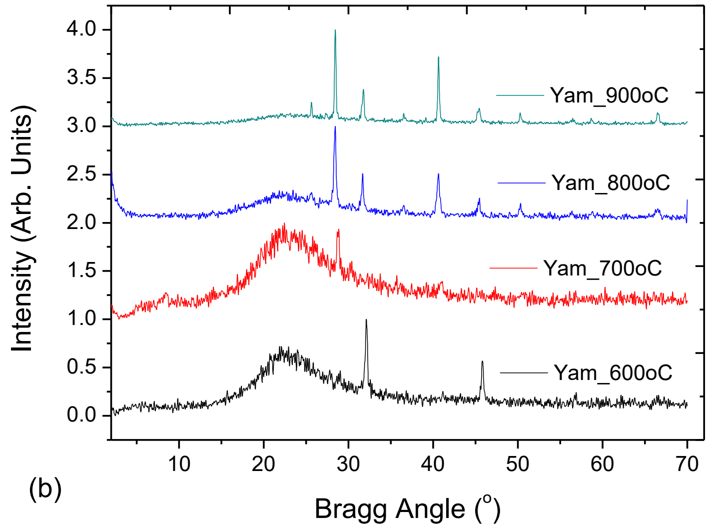


Fig. 3 X-ray Diffraction Patterns of silica extracted from (a) cassava periderm ash at various temperatures and (b) yam periderm ash at various temperatures

3.3. FTIR Analysis

Analyzing the functional groups and secondary structures in the calcined periderm ash and the extracted silica was done. Typically, silica contains functional groups found in quartz, such as silanol (Si-O), hydroxy(O=H), and siloxane groups (Si-O-Si) [17]. FTIR peaks in the spectra are usually narrow and can be attributed to the vibration of a particular chemical bond or functional group in the molecule [18]. The peak noticed at 1000 cm^{-1} in Fig. 4, and an FTIR spectra graph of CPA and YPA at 800°C reveals the presence of silicate ions in the respective ash.

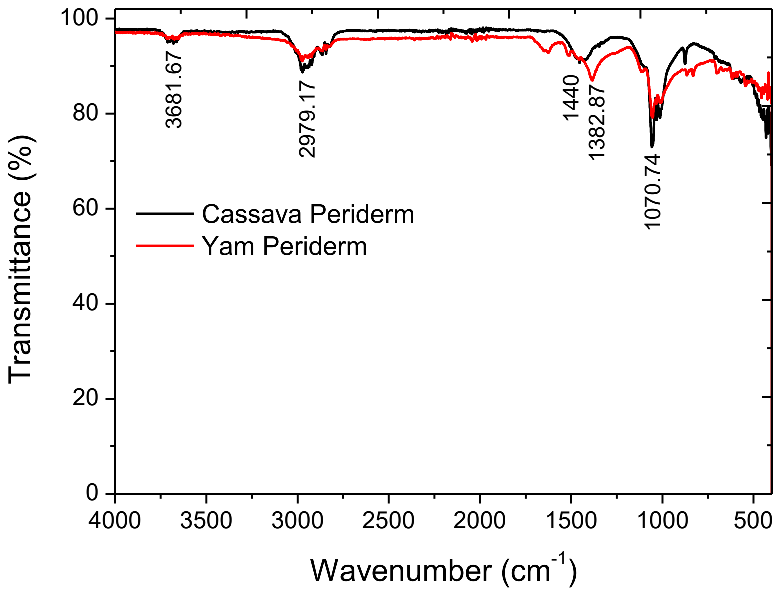


Fig. 4 Fourier Transform Infrared spectra of Cassava periderm Ash and Yam Periderm Ash at 800°C

Figs. 5a and 5b show the FTIR spectra for all the extracted silica samples by critically identifying the key functional groups in the extracted silica. All the spectra are similar and have been removed, indicating homogeneity in the functional groups of the extracted silica samples. The wide band at about 3410 cm^{-1} to 3470 cm^{-1} in all the graphs is caused by the stretching vibration of the O-H bond between silanol groups (Si-OH) and the adsorbed water molecules on the silica surface. The Si-O-Si asymmetric stretching vibration causes the band to be at 1070 cm^{-1} to 1090 cm^{-1}, whereas the network Si-O-Si symmetric bond stretching vibration causes the band to be 791 cm^{-1} to 806 cm^{-1}.

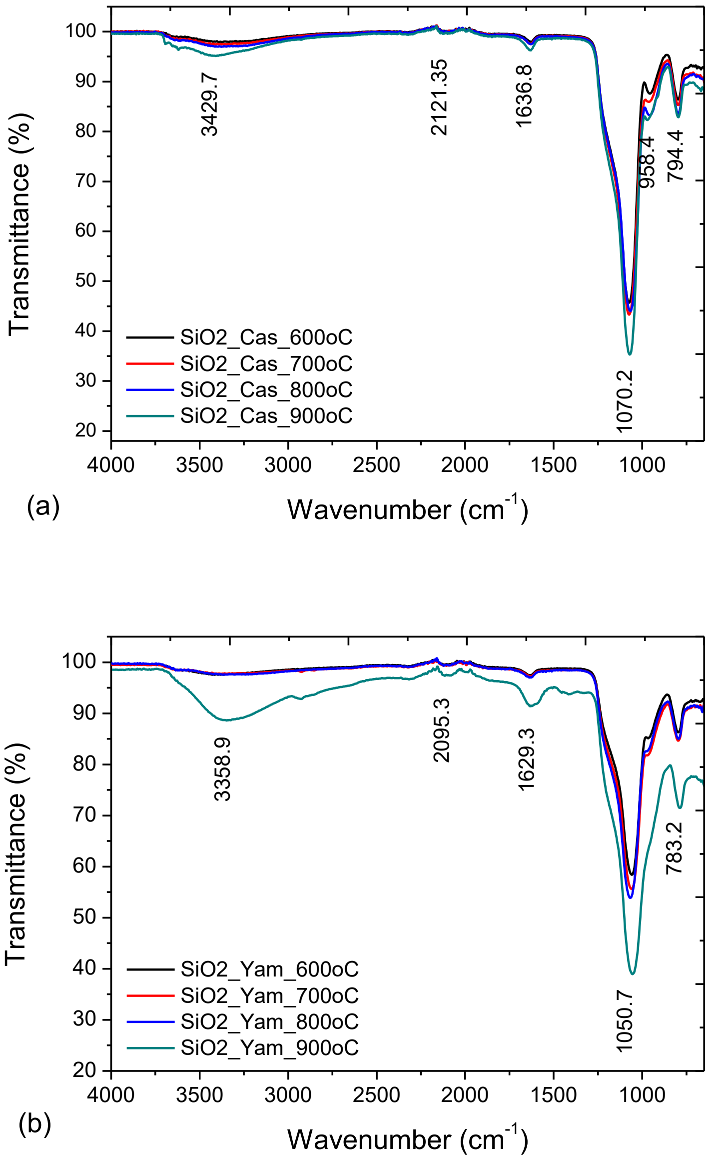


Fig. 5 FTIR spectra of SiO$_2$ extracted from (a) cassava periderm ash (CPA) at 600, 700, 800, and 900^{o}C and (b) yam periderm ash (YPA) at 600, 700, 800, and 900^{o}C

3.4. Scanning Electron Microscopy

The scanning electron micrographs of silica samples synthesized from YPA and CPA at 700 and 900°C were measured. Fig. 6 shows their SEM and EDX spectra, respectively. The picture from the SEM result was analyzed using Image J software, and it was discovered that particle size ranges from 100 µm to 150 µm in diameter, with an average diameter of 125 µm were obtained. In Figs. 6a to 6d, the EDX spectra showed a high intensity of Si alone in the spectra, confirming the presence of silica in the sample and some traces of contaminants**.**

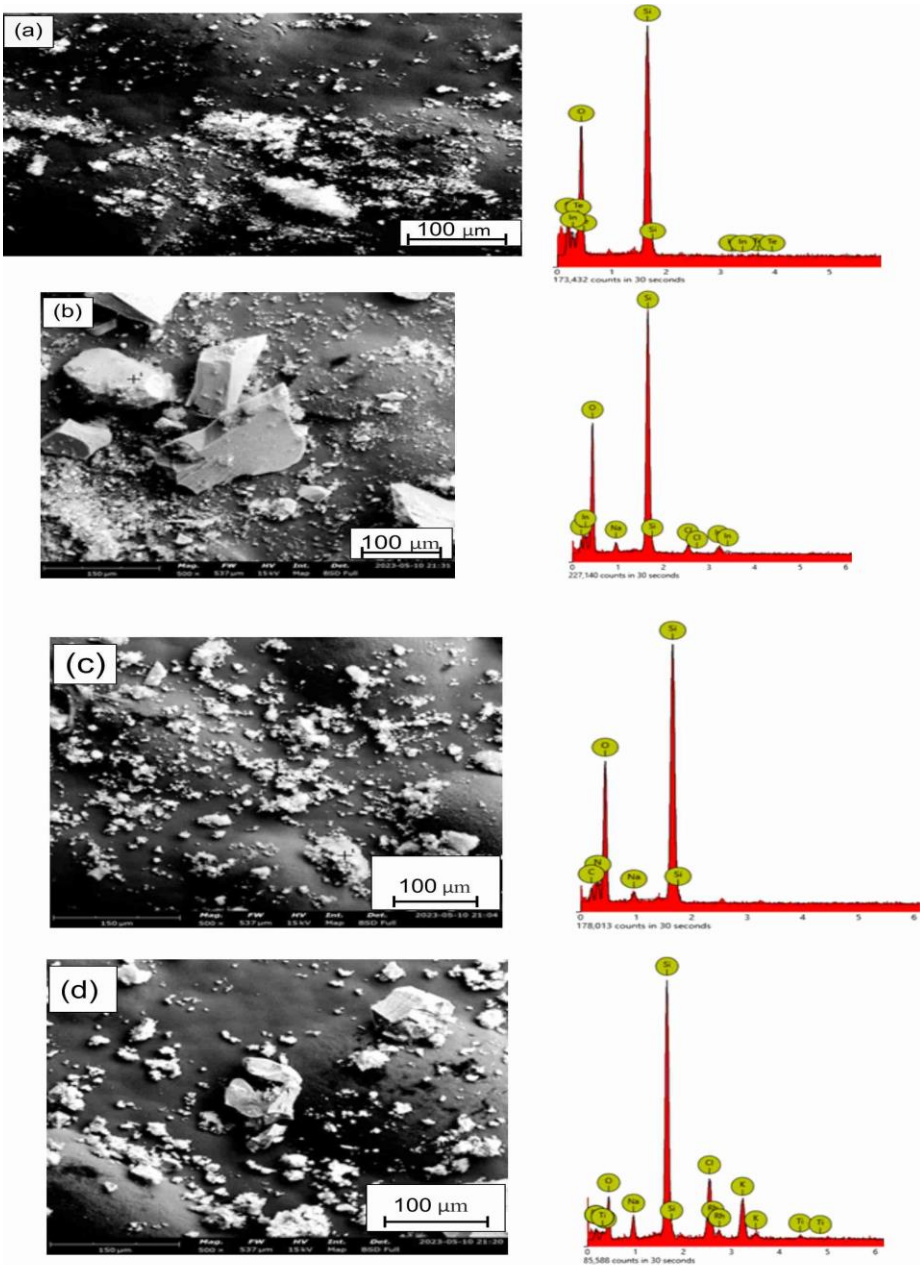


Fig. 6 Scanning electron microscope images and energy dispersive spectroscopy graphs of (a) SiO_2 from CPA at 700°C, (b) SiO_2 from CPA at 900°C, (c) SiO_2 from YPA at 700°C, and (d) SiO_2 from YPA at 900°C

3.5. Thermal Analysis of Periderm

Fig. 7 shows the differential thermal analyzer and thermogravimetric plot of cassava and yam periderm under thermal decomposition. It is evident from the curve that the cassava and yam periderms had a mass loss at temperatures ranging from about 30-100°C, which is attributable to the removal of moisture content and organic solvents present in both periderms. Another decomposition process was recorded in the temperature range of 180°C to 500°C, which is attributable to periderm disintegration to ash under the calcination process. Yam periderm takes a slightly higher temperature to start decomposing compared to cassava. The differential thermal analyzer graph shows that the yam periderm takes a higher temperature to start decomposition. Overall, the reactions are mainly exothermic in nature from room temperature up to about 400 °C. Above this temperature, up to 550°C, the reactions become endothermic.

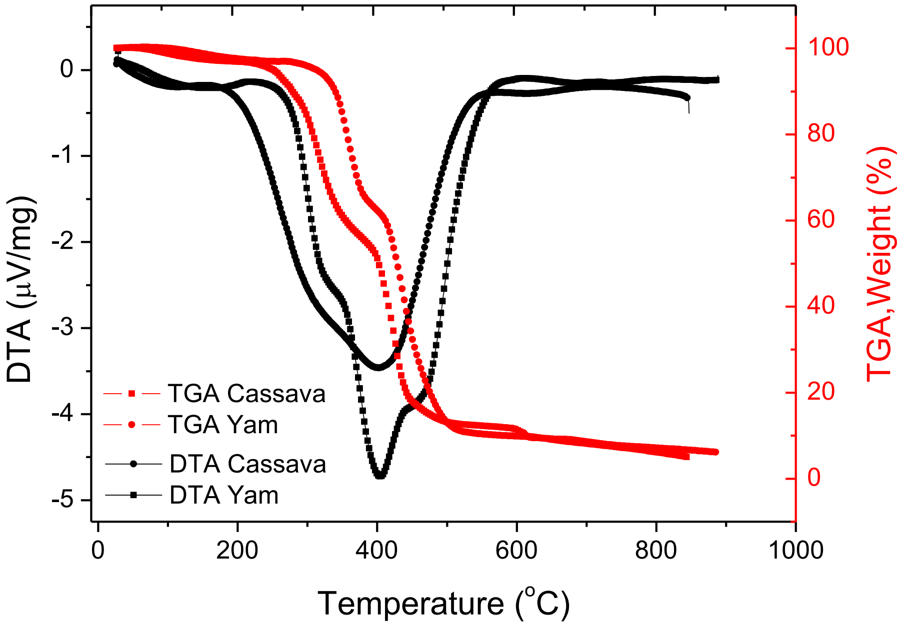


Fig. 7 Plot of Thermogravimetric analysis for cassava and yam periderm from room temperature to 1000°C.

4.0 Conclusion

The synthesis and characterization of silica particles from cassava and yam periderm using the sol-gel method is an efficient and cost-effective technique for producing high-quality micro-particles. The results show that the synthesized micro-particles exhibited a narrow-size distribution, high purity, and structural stability. The XRF and FT-IR spectroscopy results revealed the successful formation of silica particles with minimal contaminants and impurities. The XRD spectroscopy showed the crystalline nature of the silica produced with characteristic peaks at 2θ = 22.5° and 795 cm^{-1}, respectively, for yam periderm. Scanning electron microscopy images showed that the micro-particles have irregular shapes, with diameters ranging from 50-150 μm. Although crystalline silica particles were produced using yam periderm, the yield of silica from cassava periderm was higher. The synthesized micro-particles of silica show remarkable potential industrial usefulness. They offer a promising alternative to conventional drug delivery systems because of their low toxicity, easy surface adjustment, and biocompatibility.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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Author Contributions

Ayomide Famodu conducted the experiment and wrote the first draft. Henry Mgbemere conceptualized the topic, plotted the graphs, and interpreted the results. Eugenia Obidiegwu revised and edited the manuscript and also analyzed the results. All the authors read and approved the final manuscript.

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