

PREPARATION OF SILICA FROM CASSAVA PERIDERM

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ABSTRACT

Cassava peel is a substantial by-product of cassava processing and it constitutes about 13% of the crop. Cassava peel is still currently considered a waste often discarded into the environment indiscriminately, and therefore economically underutilized. In the present work, silica was synthesized from cassava periderm via sol gel route. Cassava periderm was burnt at 600 °C to obtain Cassava Periderm Ash (CPA) prior to alkaline leaching of the silica content which was thereafter precipitated with acid. The silica aquagel was then prepared by pH controlled titration and aging, and followed by drying to form silica. Quantitative analysis showed that silica yield from CPA was 61.53%. The morphology, elemental composition and phases of the silica were investigated with SEM, EDX and XRD respectively. SEM revealed that silica particles are small but not uniform. The EDX compositional analysis confirmed the presence of Si silicon among others present. The Sherrer's equation used to evaluate the particle size showed that the average particle size of the silica was 62.69 nm. The silica obtained could be used as desiccant in the laboratory, and industrially, as filler for polymer composites and feedstock for production of silicon used in the manufacture of solar panel.

Keywords: Agricultural wastes, Cassava peel, Cassava periderm ash, Sol-Gel, Silica.

INTRODUCTION

Due to enormous agricultural wastes generated across the globe, researchers have sought ways of utilizing them as sources of raw materials for other applications. The effort has been seen to improve financial status of several people and a means of controlling environmental pollution (Aigbodion *et al.*, 2010). The recycled agricultural wastes have been used for a number of purposes including: energy production (Ki *et al.*, 2013; Liu *et al.*, 2014; Zhao *et al.*, 2014); adsorbents of heavy metals (Kurniawan *et al.*, 2011; Wan Ngah and Hanafiah, 2008); production of activated carbon (Kalderis *et al.*, 2008; Sudaryanto *et al.*, 2006); refractory and ceramic

products (Aigbodion *et al.*, 2010); livestock feed (Aro *et al.*, 2010; Heuzé *et al.*, 2014); construction materials (Agbenyeku and Aneke, 2014; Villamizar *et al.*, 2012), and composite reinforcement (Kaewtatip and Thongmee, 2012; Soykeabkaew *et al.*, 2012).

The rapid increase in the industrial production of cassava products such as cassava flour and garri (cassava flake) has led to more waste that might even be more elusive if something is not done to ensure proper utilization of those waste produced. Tewe and Iyayi (1995) reported that cassava constitutes 20% of total tubers produced annually in Nigeria. Nigeria's cassava production is by far the largest in the world and according to Omongo *et al.*, (2007), the total cassava production in Nigeria was about 46 million tons in 2007.

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Cassava peel consists of two layers namely: the outer layer of cassava tuber which consists of a thicker leathery parenchymatous inner layer, known as cortex is edible and may be consumed as food by humans and animals. The brownish coloured outer layer, known as periderm is widely discarded as waste, it is not edible to animals either except if dried with the former (Oyebimpe *et al.*, 2006). Cassava peels continue to constitute waste in the cassava processing industry despite its usefulness as animal feedstuff (Iyayi and Losel, 2001). Cassava peels have little or no economic value and their disposal is not only costly but also causes environmental problems.

Previously, a large amount of precipitated silica have been produced from various types of agro-wastes such as rice husk ash (Carmona *et al.*, 2013), wheat husk (Javed *et al.*, 2011), corn cob (Mohanraj *et al.*, 2012; Ogunfowokan *et al.*, 2011) and bagasse ash (Affandi *et al.*, 2009a) etc. The economic importance of highly purified precipitated silica include industrial applications as: dryer of compressed air and refrigerants in refrigeration systems, and as filler to improve the mechanical properties of composite materials (Affandi *et al.*, 2009b). Silica powder is a basic raw material that is widely used in industries associated with ceramics, rubber, electronics, catalysis, pharmaceuticals, and dental materials (Prasad and Pandey, 2012). The aim of this work is to consider the production of silica from cassava back peel (*periderm*) via sol gel route, thereby generating wealth from waste and equally solving environmental challenges.

MATERIALS AND METHODS

Cassava Periderm Ash

The cassava periderm waste was collected from cassava peels sellers at Better Life Programme for Rural Women market, Isale Osun, Osogbo, Osun State, Nigeria. The brownish part (periderm) which is the outermost layer of the cassava peel was sorted out by hand picking. The periderm was washed thoroughly with distilled water to remove soil and

other dirt. The washed periderm was later leached with hydrochloric acid to remove the soluble inorganic and organic matter present in the cassava peel outer layer. The acid-leached cassava periderm was pyrolysed at 600 °C for 4 hours in a muffle furnace to obtain Cassava Periderm Ash (CPA).

Silica Extraction

450 ml of 1 M NaOH was added to 13 g of CPA and boiled in covered 500 ml Erlenmeyer flasks with magnetic stirring for 1 hour to dissolve the silica and produce a sodium silicate solution. The solution was filtered through Whatman No. 41 ashless filter paper, and the carbon residue was washed with 150 ml of boiling water. The filtrate and the carbon residue were allowed to cool to room temperature and then titrated with 1 M HCl with constant stirring to pH 7. Silica gels started to precipitate just below the pH 10. This was left to age for 18 hours before decanting the supernatant. Distilled water (100 ml) was added to the gels and they were broken to make slurry. The slurries were then centrifuged for 10 to 15 minutes at 4000 rpm, the clear supernatants were discarded. This process was repeated 3 times to ensure that other components were eliminated from the silica obtained. The gels (aquagels) were transferred into beakers and dried at 80 °C for 12 hours to produce xerogels. With this, the water and other impurities present were eliminated. The hardened silica was then reduced to powder by pounding with mortar and pestle. The silica powder was then weighed and stored in airtight plastic bottle. Figure 1 shows the various stages of the silica extraction, and the flow diagram for extraction of silica from cassava periderm ash is as shown in Figure 2.

In Figure 1, plate A shows the treated cassava periderm, plate B the cassava periderm ash, C is the alkaline leaching of the CPA, plate D represents the filtration process, plates E-G refers to the main sol-gel method; titration, gelation and ageing, plate H is the centrifugation process, plate I shows clearly the slurry and supernatant, plate J is the extracted silica acquagel while plate K represents the drying process and plate L is the final silica obtained.



FIGURE 1

Basic steps in the pyro-chemical extraction of silica: (A) cassava periderm, (B) cassava periderm ash, (C) leaching of cassava periderm ash, (D) filtration, (E) solation, (F) gelation, (G) ageing, (H) centrifugation, (I) slurry and supernatant, (J) silica acquagel, (K) drying, and (L) silica.

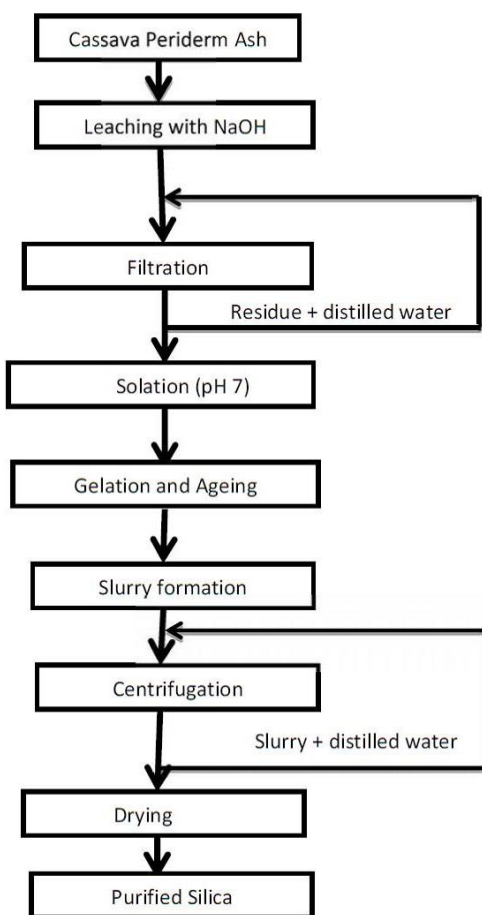


FIGURE 2

Flow diagram for extraction silica from cassava periderm ash.

Characterization

Microstructural characterization as well as elemental composition was carried out to examine the structure and composition of the silica produced from cassava periderm ash. This was accomplished by ASPEX Scanning Electron Microscope (SEM) equipped with an Energy Dispersive X-ray spectroscopy (EDX) at an accelerating Voltage of 15.00 kV. The X-Ray diffraction intensity of the silica produced was determined using Panalytical X'pert powder diffractometer, Cu-K α radiation ($\lambda=1.54 \text{ \AA}$) using an acceleration voltage of 30 kV and current of 20 mA. The X-Ray diffraction pattern of the silica sample was recorded at a range of 20° - 120° 2θ Bragg's angle, at a rate of $3.25^\circ/\text{min}$. The particle size of the silica was determined from the X-ray diffraction data using Debye – Sherrer's formula as given in equation (1) (Mathew and Narayanankutty, 2010).

$$C_s = \frac{0.89\lambda}{\beta \cos \theta} \quad (1)$$

Where C_s is the particle size, λ is the wavelength of the inci-

dent X-ray beam, β is the full width at half maximum (FWHM) of the X-ray diffraction peaks and θ is half of the angle 2θ corresponding to the peak.

RESULTS AND DISCUSSION

Scanning Electron Microscopy (SEM)

About 2 g of silica powder extracted from cassava periderm ash was analyzed with Scanning Electron Microscope (SEM) to study the microstructure of the sample. The SEM micrographs of the silica prepared are shown in Figures 3 and 4. The black region on the micrographs is the adhesive black paste coated on sample holder to enhance conductivity and to reduce charging the silica powder to be analyzed. The detailed imaging information regarding the morphology of the sample shows that the sample has a non-uniform mix of lumpy and smaller grains.

Energy Dispersive Spectroscopy

Figure 5 shows the EDX spectrum obtained from the extracted silica which shows the presence of silicon, oxygen, sodium and chlorine. The prominent peaks originated from the element of silicon and oxygen. The same observation was also reported by Villarreal et al., (2013). The presence of Na and Cl was an indication of residual sodium chloride after the titration. Both silicon and oxygen peaks correspond to the silica produced (Kamath and Proctor, 1998). Table 1 shows the particle size of the extracted silica. Using the Debye – Sherrer's formula, the average particle size for the silica is calculated to be 62.69 nm which is in the range of nanoparticles. This is possible because the sol-gel method employed has been verified by various researchers (Henry, 2004;

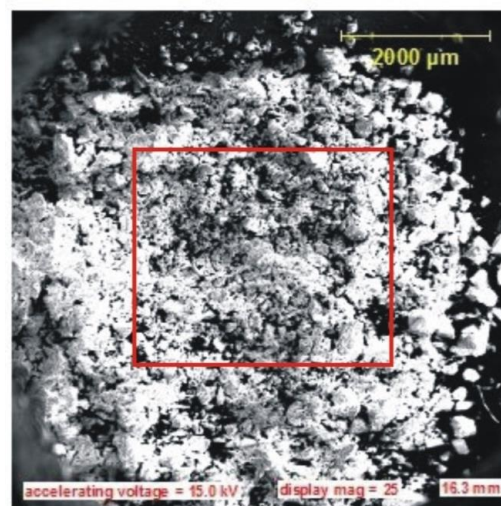


FIGURE 3

SEM micrograph of Silica from CPA at 25x magnification

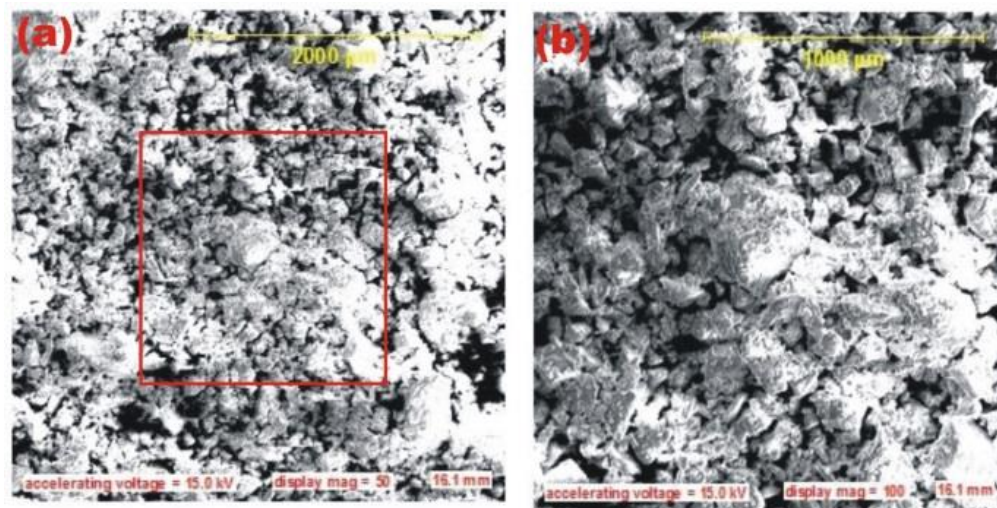


FIGURE 4
SEM micrographs of Silica from CPA at (a) 50x and (b) 100x magnification

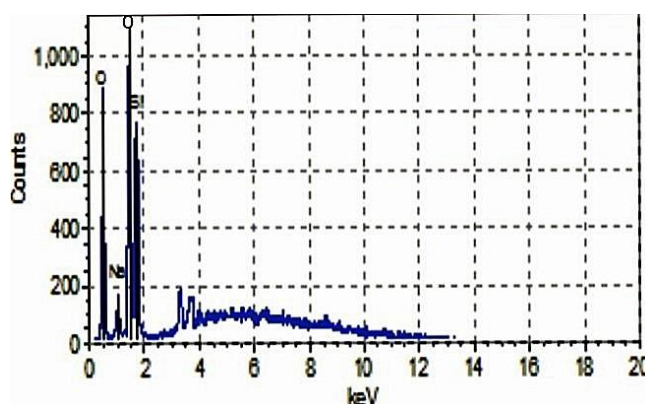


FIGURE 5
EDX Spectrum of the synthesized silica

Ogunfowokan *et al.*, 2011; Pokropivny *et al.*, 2007; Sakka, 2005; Simonenko *et al.*, 2013) to be suitable for nano-structuring of materials from bottom-up (Overney, 2010).

XRD Analysis

The XRD pattern of the synthesized silica from cassava periderm ash is as shown in Figure 6.

The XRD peaks of the silica are sharp and also revealed

TABLE 1
Particle size of Synthesized Silica using Scherrer's equation

Bragg's angle [2θ (degree)]	FWHM [Δ2θ (degree)]	Scherrer's particle size (nm)	Average particle Size (nm)
23.1780	0.2480	64.65	62.69
47.6887	0.2480	69.24	
57.4322	0.3306	54.17	

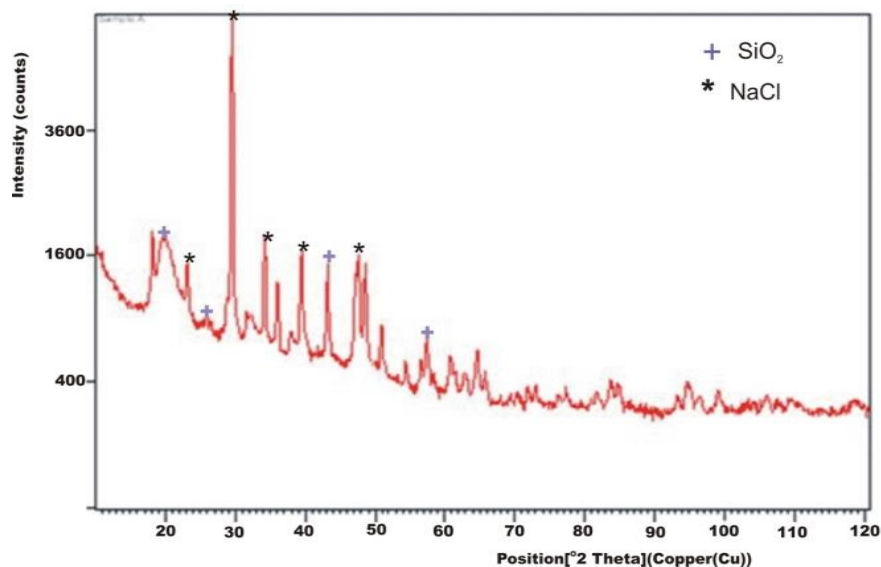


FIGURE 6
XRD pattern of silica obtained from CPA

the presence of an impurity which is sodium chloride (NaCl) that emanated during titration and gelation. The impurity was suggested and explained in the previous research by Pijarn et al., (2010) with direct evidence from XRD analysis.

CONCLUSION

An agricultural waste, cassava periderm, was converted into valuable product by sol-gel method yielding 61.53% silica from CPA. The SEM micrograph revealed that the grains are small but not uniform. XRD result confirmed the presence of silica and NaCl as an impurity. The average particle size of the silica was 62.69 nm.

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