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Preparation and characterization of nanosilica from sorghum husk ash by chemical method

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Preparation and characterization of nanosilica from sorghum husk ash by chemical method

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Abstract

Extraction of nanosilica from Sorghum Husk Ash (SHA) was performed in this study by acid leaching method. Two methods of calcination was adopted (open and controlled method) in the production of SHA. Amorphous silica was produced by dissolving a quantity of SHA in hydrochloric acid solution which made the removal of heavy metals possible. The extracted/pure silica was rinsed with deionized water which lowers the pH to 7.0. The SHA silica was characterized using XRF, XRD and BET techniques. Silica and mineral contents of SHA were determined by XRF, while the X-ray diffraction patterns revealed the amorphous nature of extracted silica. Pure silica yield from SHA was 89.30%, while moisture content was 1.09%. The average particle size, BET surface area, pore volume, and pore diameter of SHA samples are 77.05 nm, 723.020 m^2/g, 4.448e +01 cc/g, and 2.072e+00 nm respectively. The outcome of the research study reveals that the properties of the extracted silica satisfied all the requirements as established by relevant literature. Consequently, the high volume amorphous nanosilica with minimal mineral contaminants can be produced from SHA by the acid leaching method.

Keywords: Sorghum husk ash; amorphous silica; nanosilica, BET surface area, X-ray diffraction

1   Introduction

Nanotechnology was defined as the science and technology of modifying, examining, and monitoring the performance of material's behaviour at the nanoscale [1, 2]. It was further defined nanoscale materials as a set of substances where at least one measurement is less than approximately 100 nanometers [1]. A nanometer is a million smaller than a millimeter, that is

smaller than the diameter of a cobweb/human hair. Nanotechnology can also be described as the process of creating a device or material with building blocks at the molecular scale [3].

Silica has wide applications in dental material, ceramics, pharmaceutical products, chromatograph column packing, detergents, adhesives, and electronics [4-6]. Silica has also been adopted as a forerunner for a variety of inorganic and organometallic materials which have wide applications as catalysts and in thin films or coatings for electronic and optical materials [7]. A low energy method to produce pure silica from rice husk ash with 91% extraction yield, has been developed [8, 9], acid leaching [10] and gasification [11].

Guinea corn is a cereal crop locally known as grain sorghum which belongs to the general name of *Sorghum*, and reported to be the most popular cereal grain produced in Africa [12-14]. Guinea corn/sorghum grain is mostly cultivated in northern Nigeria in states like Kaduna, Bauchi, Plateau, and other northern states. When harvested, it is normally processed mechanically by the use of combined harvesters or manually by threshing with sticks leaving a large quantity of residue (husk) constituting environmental waste annually.

Hence, objective of this research work is to extract amorphous silica (using the open and controlled burning) and nanosilica from sorghum husk ash, using the acid (HCl) as leaching agents to remove the heavy metal impurities. The prepared amorphous silica and nanosilica were characterized using XRF, XRD, and BET techniques.

2   Materials and Methods

Sorghum husk was obtained from Bida local government area of Niger state Nigeria, after the harvest seasons. The sorghum husk was washed and sun dried. Samples of sorghum husk were calcinated by open burning and controlled burning at 600 ^{o}C for 3 h at a heating rate of 10 ^{o}C/min in a muffle furnace at National Cereal Research Institute (NCRI) Baddegi, and left over night to cool in the furnace.

2.1 Silica Extraction

After the calcinations, the ashes obtained (from both open and controlled) were collected and subjected to grinding for 10 min in a ball mill. The ground ashes were kept separately in a desiccator for future material characterization tests. Nanosilica was extracted from SHA sample that gives the highest silica by adopting the acid pretreatment method, where 10 g of SHA was weighed and subjected to immersion in the acid solution (hydrochloric acid,) at a concentration of 1N acid solution for 2.5 h with constant stirring at ambient temperature. After the acidic solution was drained off, SHA was rinsed with deionized water until the pH

Nanotechnology Applications in Africa: Opportunities and Constraints

IOP Conf. Series: Materials Science and Engineering **805** (2020) 012023

IOP Publishing

doi:10.1088/1757-899X/805/1/012023

rose to 7.0, then it was filtered and oven dried at 50 C for 24 h. In addition, further milling was performed using the Los Angeles abrasion machine.

2.2 Moisture content of silica

Water content of the air dried silica was determined using an air oven method [15]. About 1 g of the sample was heated in aluminum moisture pan at 130 ^{o}C for 1 h. The sample was cooled in a desiccator and weighed thereafter. The weight loss (%) was recorded as the water content of sample.

2.3 Characterization

Quantitative chemical analysis of the SHA was executed by X-ray fluorescence. The particle size was assessed by the laser diffraction analyzer Easysize 2θ (OMEC, Zhuhai, China). The surface area and pore volume of silica was measured by Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) methods, respectively, according to ASTM D3663-03 using Micromeritics Tristar 3000 Surface Area (Micromeritics, Norcross, GA, USA) and Porosity Analyzer (Micromeritics, Norcross, GA, USA).

X-ray diffraction (XRD) of the sorghum husk ash and silica extract were monitored using X-ray minidiffractometer MD-10, at an acceleration voltage of 25 kV and current of 400 μA. The diffraction angle was scanned from 10° to 75° 2θ, at a rate of 3.25^o /min.

3.0 Results and Discussion

3.1 Chemical Compositions and Physical Properties of SHA and Silica

Chemical compositions and physical properties of SHA before and after pretreatment are summarized in Tables 1 and 2, respectively. It can be seen that silica oxide forms the main component (76% and 89%) of SHAs with trace elements in the form of composite oxides K_2O, Na_2O, CaO, MgO, Fe_2O_3, and Al_2O_3. In the case of controlled burning SHA, 76.105% of silica was produced by calcining SH at 600 ^{o}C for 2 h. Potassium (K) was detected to account for the highest concentration (3.91%) among the metallic trace elements. The SHA after leaching yielded silica content of 89.30% having surface area of 723.020 m^2/g.

The trace elements were reduced to a very low level, especially in the case of potassium which was reduced to 2.3% and aluminum not detected. HCl as leaching agent also have excellent performance in washing off alkali metals and improving the surface area, as well as reducing the carbon residue content of RHA as earlier reported [16]. The average particle size, BET surface area, pore volume, and pore diameter of SHA samples after extraction are given in Table 2.

**Table 1**. Chemical Composition of SHA and silica extracted (%)

SHA Samples	Si O$_2$	Al$_2$O$_3$	Fe$_2$O$_3$	CaO	MgO	K$_2$O	Na$_2$O	SO$_3$	LOI
Open Burning	76.057	2.66	1.48	2.04	1.23	3.90	0.06	0.12	3.1
Controlled Burning	76.105	2.71	1.23	3.32	1.27	3.91	0.07	0.13	3.0
After extraction	89.03	ND	0.36	1.82	0.05	2.30	ND	0.03	2.85

ND, not detected

Table 2. Physical properties of SHA silica

SNPs Tests	Avg part. size	BET surf area	pore Volume	Avg pore dia
After extraction	77.05 nm	723.020 m^2/g	4.448e +01cc/g	2.072e+00 nm

The concentration and water content of the silica was 89.30% and 1.09% respectively. The X-ray diffractogram of amorphous silica concentration in SHA is shown in Figure 1. The broad X-ray diffraction pattern which signifies typical amorphous solids [9] shows that the extracted silica is principally amorphous. Diffraction peak at theta = 22 degree affirm the formation of amorphous silica, however, it has been reported that diffraction broad peak at theta = 22 degree indicates amorphous silica along with some crystalline silica [17].
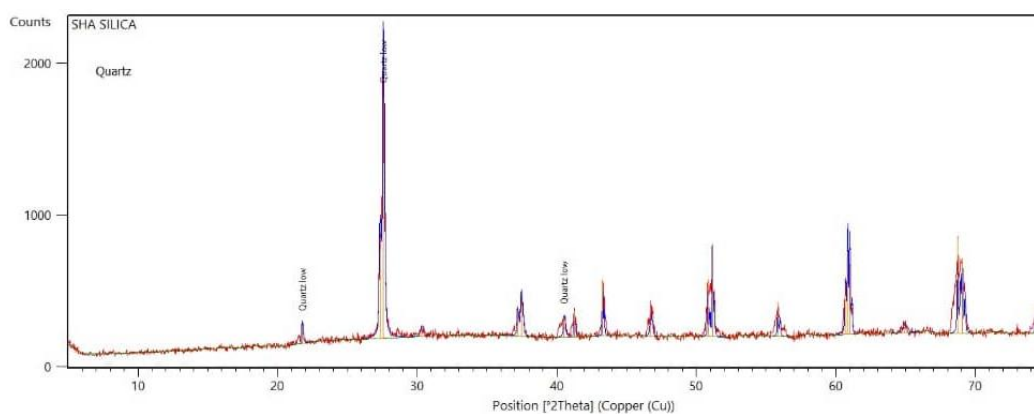
Figure 1. XRD of extracted amorphous silica

4.0   Conclusion

This study affirmed that after treatment, pure silica extracted from SHA was 89.30% with moisture of 1.09%. The average particle size of 77.05 nm falls within the standard range for nanoparticles. In addition, the nanosilica is highly amorphous as revealed by the XRD

analysis. The results of the research reveals the properties of the extraction silica satisfied all the requirements as established by relevant literature. Consequently, the high volume amorphous nanosilica with minimal mineral contaminants can be produced from SHA by the acid leaching method.

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