



## Exploring the Silicon Composition of Rice Husk Ash and Clay Soil along River Niger-Lokoja, Nigeria

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### Authors' contributions

This work was carried out in collaboration between all authors. Author AAN designed the experiment, carried out the laboratory work, perform the characterization from his research work and wrote the first draft of the manuscripts. Author HDD provided basics on the characterization and author OM supported in carrying out experimental work with the other logistics. All authors read and approved the final manuscripts.

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### ABSTRACT

Rice is a crop that is grown by subsistence farmers in Lokoja-Nigeria at a very small scale. The elemental chemical composition of the soil has great influence on the plant on it, hence, both the soil sample and rice husk from the same site(s) were used to determine silica percentage composition by calcination techniques for possible use as a raw material for silica-based production products. X-ray fluorescence (XRF) determination show that the Silica content in the White Ash from RHA are 45.86%, 45.47% while the silica content from the calcined soil was 45.06%, 44.61% for Site A and Site B respectively. Characterization of Site A White Ash by FTIR gave absorption bands that tally with other researchers. XRD of the same sample shows crystalline characteristics with the formation of crystobalite and traces of tridymite. Rice planted on this river bank soil in Lokoja proved to be affordable raw materials for silica production.

**Keywords:** Rice husk; silica; XRF; FTIR; calcinations; Lokoja.

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## 1. INTRODUCTION

Over the ages, environmental and technological quest towards waste management, utilization coupled with cost reduction in industrial processed waste materials has drawn the use of rice husk/hull ash as a silica-source viable raw material.

In 1938, the existence of Silica was discovered by Martin J.I of Louisiana State University USA. Silica is used by plants for protection and also to improve drought tolerance and disease resistance [1]. Silica plays an important role as ingredients in food, pesticide and personal care products; as fillers in plastics, rubbers and coatings; and as starting materials for semiconductors, silicates and ceramics [2,3].

It was estimated that world paddy production in 2015 was over 746.8 million tonnes which was 0.5% below the 2014 statistics [4]. Rice as a plant absorbs silica in a soluble form called silicic acid,  $\text{Si}(\text{OH})_4$ . These deposited acids in the soil enter the root of the rice crop, transported to the stelai, sheaths and leaves [5]. The soluble silicic acid becomes concentrated into  $\text{SiO}_2 \cdot n\text{H}_2\text{O}$  through evaporation and polymerization process; and this is the formation process of cellulose/Silica composite membrane [6].

Rice husk (RH) is a natural sheath that form and cover the grains of rice. The husk is formed from hard materials including opaline, silica, lignin, cellulose, and hemi celluloses which are referred as lignocelluloses [7]. Rice husk is a by-product of rice production [8,9]; about 20-25% weight of rice is contributed by rice husk [10].

Rice husk is a bio-waste obtained from rice mills, these husk has a tough and abrasive texture with low nutritional importance, low bulk density [11,7,10]; also, disposal process of rice husk has been a major problem over time as the commonest method of disposal available is open burning [12]. This method of burning in open field in all rice mills in Nigeria results in air pollutions, green gas emission and waste of energy [13,14]. Biomass is one of the most promising energy-carrying agents and can play an important role in environmentally friendly energy utilization [15].

Rice husk when burnt in open environment or under controlled temperature produces rice husk ash (RHA) where the ashes produced contain 80-95% Silica [16,17] depending on the calcined temperature. At temperature less or equal to  $700^\circ\text{C}$ , the ash produced has high content of amorphous silica [18]; however, ash produced at

temperature above  $700^\circ\text{C}$  has negligible content of unburned carbon<sup>6</sup>. This leads to crystallization of the ash from amorphous silica into cristoballite or tridymite [19]. The outcome of such ashes could be used for thermal insulation, solar cells, lithium-ion battery anodes, pozzolana, biomedical imaging, and semiconductor electronics etc [20].

The high content of silica in RH presents opportunities for the preparation of value-added silicon based materials. Research on this field has significantly advanced and has also spurred global attention due to quest for sustainable and renewable resources.

Preparation of silica aerogel from rice husk is a good alternative to the conventional production materials from tetraethylorthosilicate (TEOS), tetramethylorthosilicate (TMOS), sodium silicate solution (water glass) which are more expensive and carcinogenic [21]. The technique of preparation of aerogels includes a sol-gel step-to-wet gels which are carefully dried to preserve the pore structure and the surface area. Silica aerogel are also produced in a form of monolith, powder, beads, blanket, flexible sheets etc, however, monolith preparation form is much easier compared to aerogel particle [22].

The chemical composition of rice husk ash depends on the type of soil for growing the rice plants, fertilizing practices, temperature/duration of calcination/burning and environment of plantation. All these factors influences the percentage of silica found in the rice husk ash.

The objective of this work was to explore the level of Silicon available in clay soil and Rice husk around River Lokoja ( $7.50^\circ\text{N}$ ,  $6.44^\circ\text{E}$ ) bank which is one of the major Rivers in Nigeria. Lokoja is a city in Nigeria; it lies at the confluence of the Niger and Benue rivers and is the capital of Nigeria's Kogi State. Selected rice was from Ganaja village and Nataco Village along Lokoja river axis. Metallurgical grade silicon was produced from the clay deposit while white silica was produced from one of the sampled rice husk having the highest silicon oxide composition.

## 2. EXPERIMENTAL

### 2.1 Materials

Paddy Rice was collected from small rice farm at Ganaja-Lokoja (Site A) and Nataco-Lokoja (Site B) all by the river bank. From the same sample sites, clay soil upon which the rice was planted was also collected for analysis. The rice was

processed and rice husk ash was extracted for this work. All chemicals used are analytical grade; therefore, no further purification was done. De-ionized water is applied for all synthesis and treatment processes.

## 2.2 Sample Collection/ Preparation

The production of silica from clay soil and rice husk involved thermal treatment method using two major simple processes; pre-treatment using acids, calcination for rice husk; drying, calcination process for the clay soil.

The rice husk (15 g of ash was mixed with 20 cm<sup>3</sup> of 30% w/w H<sub>2</sub>SO<sub>4</sub> aqueous solution) from Site A and B were washed in de-ionized water properly, followed by drying in oven at 90°C for 24 hrs. Samples were refluxed with H<sub>2</sub>SO<sub>4</sub> to reduce the presence of metallic impurities for 2 hrs and further washed, dried for 24 hrs at 90°C. Dried samples were calcined at 900°C [11,14] in a muffle furnace for 2 hrs, allowed to cool and the white silica was obtained. The silica samples obtained using H<sub>2</sub>SO<sub>4</sub> acids and degree of agglomeration of silica particles were analysed using Scanning Electron Microscopy (SEM), XRF, and FTIR characterization was carried on a sample with the highest percentage composition of Silicon oxide (Site A).

Wet soil samples from the various sites were sun dried for a whole day and visible particle removed. Samples were heated in an electric furnace for 1 hr at 100°C to attain stable temperature and allowed in the furnace for 6 hrs to cool; after which it was calcined at 900°C for 1 hr. XRF analysis was carried out to ascertain the chemical percentage composition.

## 3. RESULTS AND DISCUSSION

The conditions for SEM, XRF, FTIR, XRD (software) test for the analysis is; the SEM was operated at an accelerating voltage of 5 to 20 kV;

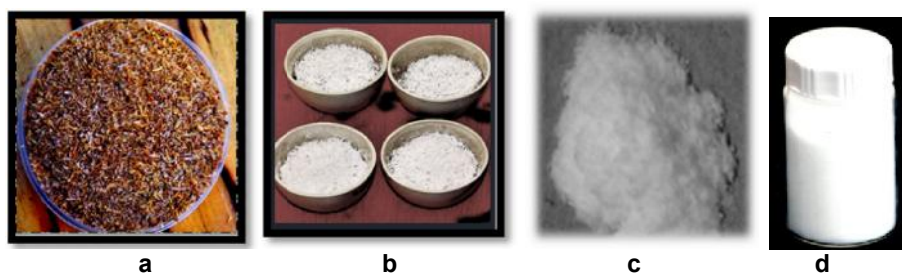
X-ray diffractograms was taken using Cu K $\alpha$  radiation at scan speed of 3°/ min. The samples were rotated at precisely one-half of the angular speed of the receiving slit, so that a constant angle between the incident and beams is maintained; FTIR system using 1 cm<sup>-1</sup> resolution, 22 meter path length, and a broad band MCT detector. The XRF system was controlled by a PC running the dedicated Mini Pal analytical software.

## 3.1 Compositional Analysis of Rice Husk

Fig. 1 shows the images of rice husk before and after combustion.

Once the organic compounds were decomposed, many empty spaces were left between the veins. It can be seen in Fig. 1c that ashes are distributed on the burnt rice husk. These ashes mainly consisted of silica. Because very little ash remained after combustion, silica content in RH relatively low at the first burning. As the incineration temperature increases, there appears to be some accompanying phase changes.

In determining the element content of RHA (white silica), the X-ray Fluorescence (XRF) and X-ray Diffraction (XRD) were conducted, its XRD from site A shown in Fig. 2 indicates that the structure of silica present is of amorphous material having a diffused peak of 2778 counts at about  $\theta = 22^\circ$ . A typical chemical composition of RHA obtained after calcination and grinding is shown in Table 1. From Table 1, it can be seen that the SiO<sub>2</sub> for RHA are 98.17% and 97.35% with 0.38% and 0.36% of potassium (K<sub>2</sub>O) for Site A and B respectively. There are some minor oxides such as sulfate, and calcium oxide. The obtained RHA from these two sites can be also classified as pozzolan provided the SiO<sub>2</sub> present was > 70% with loss of ignition (LOI) < 6% of which LOI for RHA is about 4.81% (ASTM C618:2003).



**Fig. 1. Visual stage of the transformation of rice husk to white silica**  
**(a) Rice Husk, (b) Rice Husk Ash, (c) White silica, (d) Silica glow at night after exposure to visible light during the day**

The X-ray diffractogram shows four distinct crystalline peaks at 22.0204°, 26.6996°, 35.8573°, 60.0719° indicating that the sample is crystalline in nature with the formation of cristobalite and traces of tridymite. This is in agreement with the work of Kapur [23] who

studied the structural behaviour of silica over a temperature range of 400-1500°C and reported that at combustion temperature above 900°C [11,14], the silica in rice husk ash consisted of cristobalite and a small amount of tridymite.

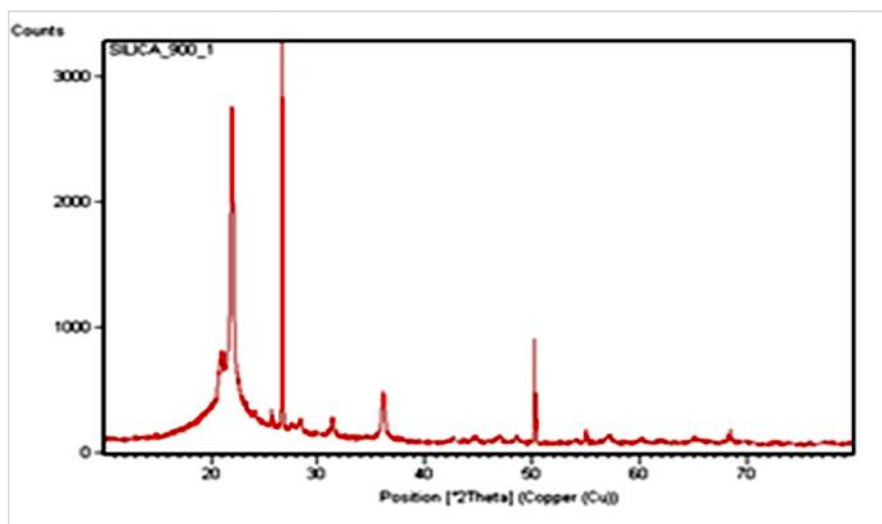
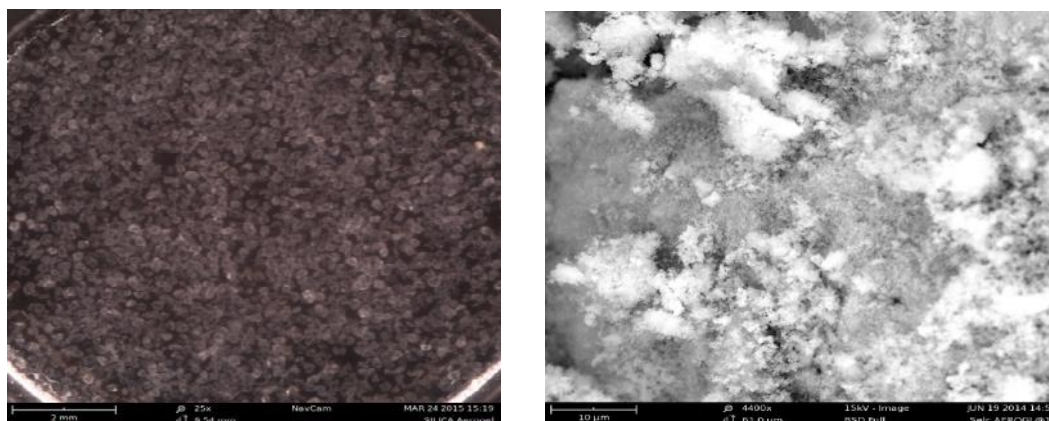


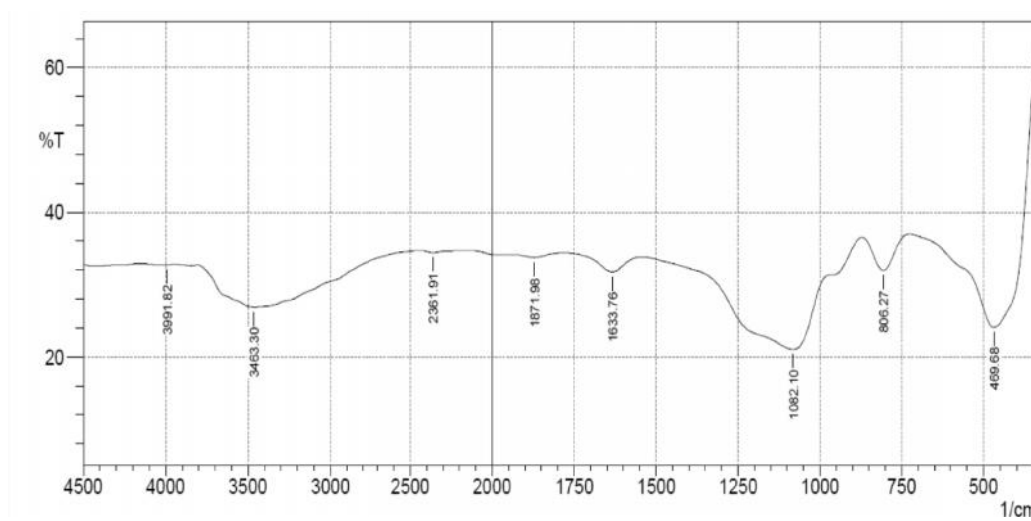
Fig. 2. XRD patterns of RHA of site a sample

Table 1. XRF results obtained from RHA and soil along river Niger-Lokoja

Compound	Unit	River Niger Lokoja soil site A	RHA Lokoja site A	River Niger Lokoja soil site B	RHA Lokoja site B
Na <sub>2</sub> O	%	0.07	ND	0.05	0.11
MgO	%	1.14	0.12	1.24	0.33
Al <sub>2</sub> O <sub>3</sub>	%	1.2	0.18	1.75	0.18
<b>SiO<sub>2</sub></b>	<b>%</b>	<b>96.46</b>	<b>98.17*</b>	<b>95.51</b>	<b>97.35</b>
P <sub>2</sub> O <sub>5</sub>	%	ND	0.3	ND	0.48
SO <sub>3</sub>	%	0.16	0.2	0.24	0.4
K <sub>2</sub> O	%	ND	0.38	ND	0.36
CaO	%	0.02	0.35	0.08	0.37
TiO <sub>2</sub>	%	0.11	0.02	0.13	0.02
V <sub>2</sub> O <sub>5</sub>	%	0.03	ND	0.04	ND
Cr <sub>2</sub> O <sub>3</sub>	%	0.08	ND	0.08	ND
Mn <sub>3</sub> O <sub>4</sub>	%	0.02	0.05	0.03	0.1
Fe <sub>2</sub> O <sub>3</sub>	%	0.66	0.16	0.55	0.23
ZnO	%	0.04	0.01	0.3	0.01
SrO	%	0.01	0.01	ND	0.01
Y <sub>2</sub> O <sub>3</sub>	%	ND	0.03	ND	0.02
ZrO <sub>2</sub>	%	ND	0.01	ND	0.02
BaO	%	ND	0.01	ND	0.01
Total	%	100	100	100	100
Impurities	%	3.54	1.83	4.49	2.65
<b>Silica</b>	<b>%</b>	<b>45.06</b>	<b>45.86</b>	<b>44.61</b>	<b>45.47</b>



**Fig. 3.** SEM images of silica nanoparticles from RH. Left: silica nanoparticles from calcination at 900°C (25x magnification); Right: silica nanoparticles from the calcination at 900°C (4400x magnification) of the H<sub>2</sub>SO<sub>4</sub> treated RHs



**Fig. 4.** FTIR spectra of RHA site a sample at prepared calcination of 900°C temperatures

FTIR was conducted in order to investigate the chemical bonding state of surface modifying agent with aerogels calcined at 900°C temperatures (Fig. 4 above). There are characteristic bands of silica aerogel at ~1082.10, ~806.27 and ~469.68 cm<sup>-1</sup>. The ~1082.10 cm<sup>-1</sup> band is associated with Si-O-Si asymmetric bond stretching vibration [24]; while the band at ~806.27 cm<sup>-1</sup> is assigned to a network Si-O-Si symmetric bond stretching vibration. The bands at ~469.68 cm<sup>-1</sup> are associated with a network Si-O-Si bond bending vibration. The bands at ~3463.30 and ~1633.76 cm<sup>-1</sup> are associated with the -OH absorption band which is caused by physically adsorbed water [25]. The small absorption peaks at ~2361.91 and ~1871.96 cm<sup>-1</sup>

may be associated with -OC<sub>2</sub>H<sub>5</sub> terminal group [21].

#### 4. CONCLUSIONS

A study on exploring the silicon percentage composition link between the soil and planted rice husk properties of the silica product obtained has been presented. It is clear from the processing methods that, the higher the soil silicon oxide, the higher the percentage composition of silicon oxide found in the RHA. The obtained RHA and river sand from Lokoja river bank are rich in high silicon oxide above 70%, hence, are recommended for industrial application; the obtained silica from RHA can also be used as pozzolan in various applications

in high concrete as substitute to silica fume; advanced materials like SiC, Si<sub>3</sub>N<sub>4</sub>, elemental Si and Mg<sub>2</sub>Si can be sourced from this site as agreed by Chandrasekhar et al. [26]. X-ray fluorescence (XRF) determination show that the Silica content in the White Silica from RHA are 45.86%, 45.47% while the silica content from the calcined soil was 45.06%, 44.61% for Site A and Site B respectively. Characterization of Site A White silica by FTIR gave absorption bands that tally with other researchers. XRD of the same sample is crystalline in nature with the formation of cristobalite and traces of tridymite. Rice planted on this river bank soil in Lokoja proved to be affordable raw materials for silica production.

### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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