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EXTRACTION OF SILICON FROM AFIKPO BEACH SAND AND RICE HUSK USING MAGNESIOTHERMIC REDUCTION REACTION

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ABSTRACT

The extraction of silicon from Afikpo beach sand and rice husk using magnesiothermic reduction reaction was studied. The raw materials (beach sand and rice husk) were initially pre-treated by drying in sunlight and later in an oven for 3 hours at 150 ^oC. The rick husk was thermally treated in a gas kiln at 500 ^oC for 4 hours to obtain rice husk ash (RHA), while the Afikpo beach sand was ground and sieved using a 100 (150 µm) and 240 (63 µm)-mesh sieves. Chemical leaching was conducted using hydrochloric acid (HCl) for 2 hours for both Afikpo beach sand and RHA, while only the RHA was alkali treated using NaOH, after which the precipitates from both samples were dried and calcined in an air-gas atmosphere at 700 ^oC for 2 hours. The calcined products underwent magnesiothermic reduction in a nitrogen gas atmosphere at 750 ^oC for 2 hours, followed by a final acid leaching using HCl to remove unreacted magnesium and residual impurities. Characterization was performed using x-ray fluorescence (XRF) and scanning electron microscope (SEM). XRF results indicated high levels of retained impurities, with silicon content measured at 51.45 % for RHA and 70.53 % for beach sand extract. SEM micrographs revealed heterogeneous and irregular shaped microstructures, attributed to incomplete impurity removal and non-uniform reduction. These findings underscore the need for optimization of purification and processing steps to improve the quality and yield of silicon from these locally sourced materials for potential use in semiconductor devices, batteries, photovoltaic cells and electronic applications.

KEYWORDS: Rice hush ash, chemical leaching, magnesiothermic, x-ray fluorescence, Microstructures.

INTRODUCTION

Silicon (Si) is a fundamental element in modern technology, with applications ranging from semiconductor devices to photovoltaic cells (solar panels). Over 90 % of the earth's crust is composed of silicate minerals, making silicon one of the most abundant elements in nature. Beach sand is one of the richest sources of silica (SiO_2), while agricultural residues such as rice husk are emerging as sustainable and cost-effective alternatives for silica production (Park & Lee, 2018).

Globally, there is an increasing demand for high-purity silicon, driven by the proliferation of electronics, solar energy technologies, and advanced materials (Zhao et al., 2022). Extracting silicon locally from natural resources such as Afikpo beach sand and rice husk offers a sustainable, low-cost, and environmentally friendly solution that can help reduce import dependency, boost local industries, and support the transition towards renewable energy technologies. Si is an essential element widely used in various industries, including electronics, solar energy, glass manufacturing, and metallurgy. Its primary source is silica (SiO_2), which is abundant in beach sand and agricultural waste like rice husk ash (Ramesh et al., 2020).

Nigeria, with its vast natural resources, possesses significant silica sand deposits, including Afikpo Beach Sand in Afikpo North LGA of Ebonyi State. Nigeria is one of the largest rice producers in Africa, generating tons of rice husks annually as agricultural waste. Rice husk contains up to 90–98% amorphous silica after controlled combustion, making it an excellent supplementary raw material for silicon production (Muthadhi & Kothandaraman, 2010).

Despite these resources, Nigeria remains dependent on imported silicon for industrial use, limiting its technological capacity and increasing production costs (Adebayo & Olaleye, 2019). There, Nigeria's reliance on imported silicon-based raw materials results in high production costs, reduced competitiveness in manufacturing, and limited growth in sectors such as electronics, glass, and renewable energy (Okonkwo & Uchenna, 2020).

Aim and Objectives of the Study

Although deposits of silica have been identified in various parts of Nigeria, including Afikpo, and rice husk is widely available as agricultural waste, little research has been conducted on developing scalable, cost-effective methods to extract and process high-purity silicon from these sources (Eze & Nwosu, 2018).

Based on the above given background, this study aims to investigate the feasibility of extracting silicon from Afikpo beach sand and rice husk. The objectives of the study include:

1. To analyze the chemical composition of Afikpo beach sand and rice husk ash to confirm silica content.
2. To develop cost-effective and environmentally friendly methods for extracting silicon from both materials.
3. To evaluate the purity and quality of the silicon obtained from both sources.
4. By achieving these objectives, this study will contribute to Nigeria's industrial growth, reduce import dependence, and promote sustainable utilization of natural and agricultural resources (Rahman et al., 2019).

A brief overview of Silicon

Silicon is a chemical element with symbol Si and atomic number 14 with a diamond cubic structure, giving it a high mechanical strength (Ashcroft & Mermin, 1976).

It is classified as a metalloid, and exhibits properties of both metals and non-metals. Si is the second most abundant element in the Earth's crust, accounting for ~28% of its composition (Brown, 2009). It is mostly found as silicon dioxide (SiO_2) in sand, quartz, and silicate minerals. Pure silicon is usually obtained by reducing silica with carbon in an electric arc furnace (Klein & Dutrow, 2007). The table below shows a summary properties of silicon element.

Table 1. Selected Properties of Silicon.

Property	Type	Value	Reference
Density	Physical	2.33 g/cm³ at 25 °C	Callister & Rethwisch, 2020
Melting Point	Physical	1,414 °C	Lide, 2004
Hardness	Physical	~7 on Mohs scale	Kittel, 2018
Bandgap	Electrical	1.12 eV	Sze & Ng, 2007
Resistivity	Electrical	~52 Ω·cm	Streetman & Banerjee, 2016
Thermal conductivity	Heat	149 W/m·K	Callister & Rethwisch, 2020

Applications of Silicon

Silicon is utilized in the following areas:

1. Electronics & Semiconductors as microchips, transistors, integrated circuits as well as in solar energy for photovoltaic cells (Green et al., 2022).
2. Construction: Glass, cement, ceramics (Mehta & Monteiro, 2019).

3. Automotive & Aerospace: Silicon carbide for high-performance components (Zhang et al., 2020).
4. Biomedical: Porous silicon for drug delivery (Tasciotti et al., 2008), and
5. Energy Storage: Silicon anodes in next-generation lithium-ion batteries (Liu et al., 2012).

2. MATERIALS AND METHOD

2.1. Materials

The materials, chemicals, laboratory tools and equipment used in the study were:

- Raw Materials: Rice husk and Afikpo beach sand.
- Chemicals: Hydrochloric acid (HCl), sodium hydroxide (NaOH), magnesium powder and distilled water.
- Laboratory Tools: Crucibles, beakers, mortar (and pestle), stirrers (both glass rods and magnetic stirrers), filter paper and sieves [100 (150 µm) and 240 (63 µm)-mesh sieves].
- Laboratory Equipment: Ball mill, magnetic separator, pH meter, kiln, scanning electron microscope and XRF analyzer.

2.2. Method

2.2.1. Sample Collection and Preparation

The Afikpo beach sand was sourced from Afikpo beach in Afikpo North LGA of Ebonyi State, Nigeria. It was washed with distilled water, oven-dried at 150 °C, milled with a ball mill, and sieved through a 100-mesh and 240-mesh sieve. Magnetic separation was performed to remove iron impurities. The rice husk was also sourced from a local rice mill in Afikpo, Afikpo North LGA, Ebonyi State, Nigeria. It was washed thoroughly to remove dirt, dried in sunlight, oven dried for 3 hours and finally thermal treated in a gas kiln at 500 °C for 4 hours to obtain the rice husk ash (RHA). The resulting RHA was collected after cooling.

2.2.2. Extraction of Silicon from Afikpo Beach Sand

The following steps were undergone for the extraction process:

- Acid Leaching

About 100 g of the prepared Afikpo beach sand was mixed with 500 ml of 2 M HCl. The mixture was stirred continuously for 2 hours to dissolve any metallic impurities. Thereafter, it was filtered, washed with distilled water to remove residual acid and allowed to dry.

- Calcination

The acid-leached beach sand was then calcined in an electric kiln at 700 °C for 2 hours to eliminate residual organics and moisture.

- Magnesiothermic Reduction

The calcined beach sand was mixed with magnesium powder in a molar ratio of 1:1. The mixture was ground and stirred thoroughly in a mortar using pestle to ensure homogeneity before being placed in refractory crucibles, and sintered for 2 hours at 750 ^{0}C in nitrogen gas atmosphere.

The reduction followed the reaction: $\mathbf{SiO_2 + 2Mg \rightarrow 2MgO + Si}$

- Post-Reduction Treatment

The reduced product was treated with HCl and stirred for about 1 hour to dissolve MgO and any unreacted Mg. The suspension was then filtered, washed with distilled water until neutral pH was achieved, and dried at 150 °C to obtain silicon powder.

2.2.3. Extraction of Silicon from the Rice Husk Ash

The following procedures were undergone for the extraction process:

- Acid Leaching

About 100 g of RHA was mixed with 500 ml of 2 M HCl in a beaker. The mixture was stirred continuously for 2 hours to enhance dissolution of metal impurities. It was then filtered, and the residue was washed with distilled water until it became an acid-free.

- Alkali Treatment

The acid-leached RHA sample was mixed with 500 ml of 2 M NaOH in a beaker. The mixture was stirred continuously for 2 hours to extract silica in the form of sodium silicate. The slurry was later filtered, and the filtrate containing sodium silicate was collected.

- Precipitation

Acid (HCl) was added slowly to the filtrate with constant stirring until the pH was adjusted to 7. Stirring was continued for 2 hours to allow silica to precipitate completely.

The precipitate was filtered, washed with distilled water, and collected.

- Calcination

The precipitated silica was placed in a crucible and calcined in an electric kiln at 700 °C for 2 hours to obtain purified SiO_2.

- Magnesiothermic Reduction

The purified SiO_2 from RHA was mixed with magnesium powder in a 1:1 molar ratio. The mixture was stirred thoroughly and placed in crucibles and thermally treated in nitrogen atmosphere at 750 ^{0}C for 2 hours.

- Post-Reduction Treatment

The reduced product was treated with HCl and stirred for 1 hour to dissolve MgO and unreacted magnesium. The suspension was filtered, washed with distilled water until neutral pH, and dried at 150 °C to obtain a purified silicon powder.

3. Characterization

Characterization was performed using x-ray fluorescence (XRF) and scanning electron microscope (SEM). XRF analysis was used to determine the chemical composition of the raw materials and the final products, while SEM analysis was used to observe the surface morphology or the microstructure of the extracted silicon.

4. RESULTS AND DISCUSSION

4.1 Results

Figures 1 to 3 show the raw and processed rice husk and Afikpo Beach sand, respectively.

Fig. 1. Prepared and sieved raw (a) Rice husk ash (AH_1) and (b) Afikpo Beach sand (BH_1)

Fig. 2. Leached and calcined (a) Rice husk ash (AH_2) (b) Afikpo beach sand (BH_2)

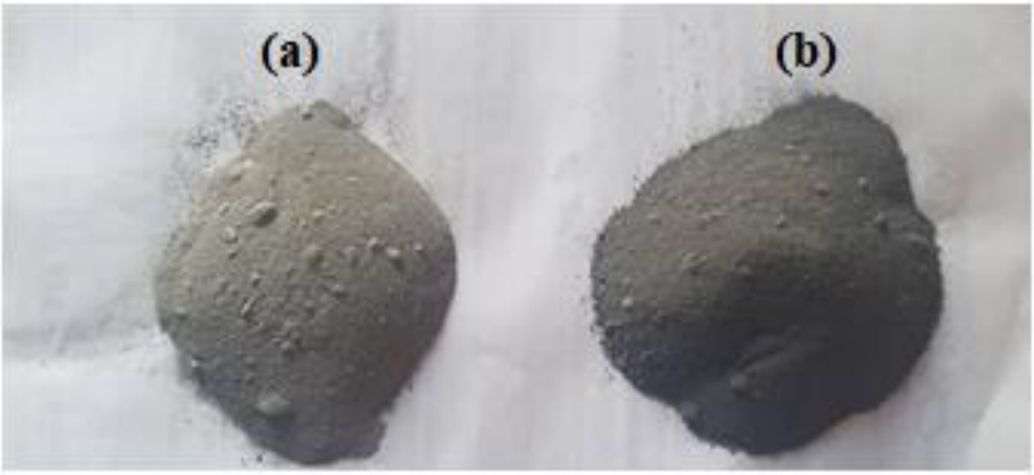

Fig. 3. Magnesiothermic Reduced (a) Rice husk ash (AH_3) (b) Afikpo beach sand (BH_3).

Elemental compositions of the samples (AH_1, AH_2, AH_3, BS_1, BS_2, and BS_3) generated from the XRF analysis are shown in Tables 1 and 2, respectively.

Table 1. Elemental Composition of the Rice Husk Ash

Element	Mass/Mass Percentage (m/m %)		
	AH_1	AH_2	AH_3
Si	34.71	33.27	51.45
K	20.37	1.01	0.299
Px	14.62	0.323	0.232
Fe	10.74	0.184	0.0740
Ca	8.50	0.444	0.245
Sx	4.51	0.359	0.587
Mn	1.61	0.0056	0.0695
Ti	1.27	0.0412	0.0111
Al	1.18	0.61	3.47
Cl	0.890	63.72	2.97
Zn	0.868	0.0114	0.0160
Rb	0.208	-	-
Zr	0.177	-	-
Cu	0.162	-	-
TTE	0.183	0.0073	-
Mg	-	-	40.54

TTE = Total Trace Elements

Table 2. Elemental Composition of the Afikpo Beach Sand.

Element	Mass/Mass Percentage (m/m %)		
	BS_1	BS_2	BS_3
Si	71.22	72.50	70.53
K	12.35	11.81	11.34
Fe	6.08	5.16	7.93
Al	5.47	4.90	4.76
Ca	1.44	1.07	1.48
Cl	1.08	2.52	0.265
Ti	0.786	0.734	0.729
Ba	0.644	0.584	1.28
Px	0.475	0.355	0.856
Sr	0.145	0.133	0.242
Zr	0.112	0.121	0.249
Mn	0.103	0.0289	0.178
Rb	0.0518	0.0482	0.0678
Zn	0.0140	0.0148	0.0311

The SEM morphology of the extracted silicon elements from the rice husk and the beach sand is presented in Figures 4 and 5, respectively.

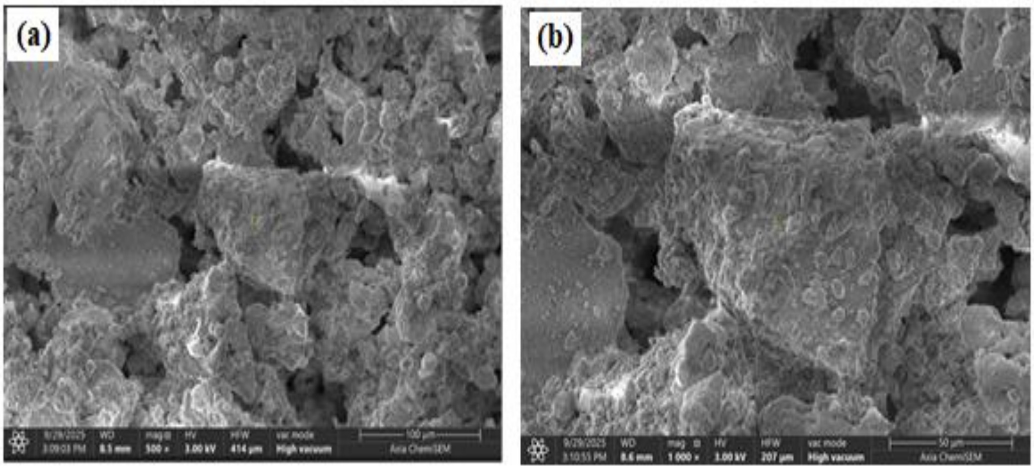

Fig 4. SEM morphology of magnesiothermic reduced rice husk ash, AH_3 at magnification (a) 500x (b) 1000x

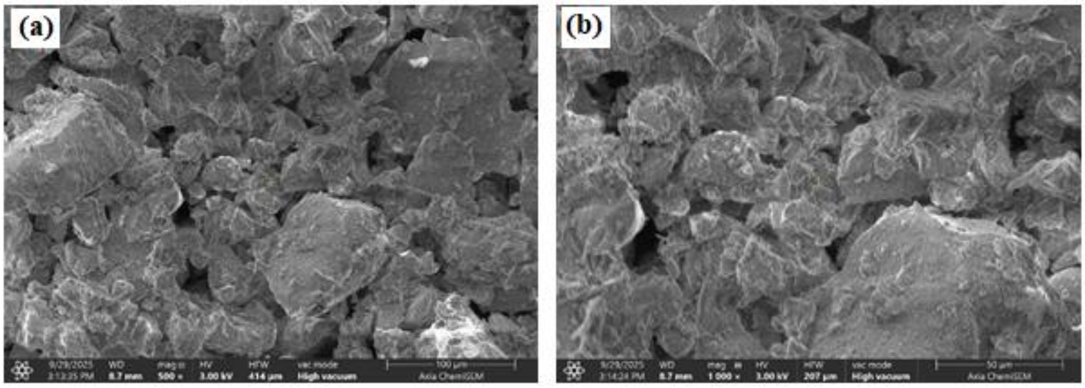

Fig 5. SEM morphology of magnesiothermic reduced Afikpo beach sand, BH_3 at magnification (a) 500 x (b) 1000 x

DISCUSSION

From Tables 4.1 and 4.2, high impurity contents are observed even after leaching, calcination and magnesiothermic reduction. Several causes have been deduced to this effect. The causes include:

1. Incomplete Leaching

Leaching with HCl primarily target metallic impurities such as Fe, Al and Ca. However, a single-stage or low concentration acid treatment may not completely eliminate these. According to Park et al., (2001) multiple-step acid leaching for example, using HCl and followed by HF is often required for higher purity.

2. Organic and Carbon Residues in RHA

If calcination is not optimized, residual carbon and unburned organics may remain in the ash, hence affecting the purity and reduction efficiency (Krishnarao *et al*., 2001).

3. Magnesiothmic Reduction Limitation

Mg reduction can be incomplete or result in secondary phases, e.g. MgO, Mg_2Si and unreacted silica especially when reaction conditions such as temperature, duration Mg: SiO_2 ratio, etc. are not optimized (Nguyen *et al*., 2014).

4. Silica phase in Beach Sand

According to Yuvakkumar *et al* (2013), beach sand contains crystalline silica (quartz) which is more resistant to chemical leaching compared to amorphous silica in RHA. Therefore, the crystalline structure can trap impurities or prevent full reduction

5. Contamination During Processing

The use of non-inert containers or contaminated crucibles during high temperature treatment can introduce trace melts into the product (Yadav *et al*., 2010).

Consequently, the relatively low silicon content, 51.45 % for rice husk and 70.53 % for beach sand after the extraction steps can be attributed to several technical, chemical and procedural limitations as supported by various studies.

These include incomplete combustion, inadequate leaching, limited reduction conversion and contamination. Achieving > 90 % silicon typically requires high-purity amorphous silica, multi-step leaching and controlled reduction processes. Amorphous silica is preferred due to its higher reactivity while quartz (beach sand) requires, more energy (high temperature) and often results in incomplete reduction (Yukkumar et al., 2013).

The SEM images (Figures 4.10 and 4.11) show irregular, non-uniform surfaces with foreign particle inclusions (seen as dark spots or agglomerates) and undefined grain boundaries. The description observed is as a result of the low purity and incomplete processing of the RHA and beach sand.

The low silicon purity from RHA and beach sand directly affects the microstructural clarity, morphology and quality observed in SEM. This therefore, compromises the materials suitability for high performance applications such as in photovoltaic or semiconductors. The resultant effects of the low purity (high presence of impurities) on the SEM images are summarized as follows:

1. Presence of residual impurities

The SEM images appear irregular, non-uniform with foreign particle inclusions which appear as dark spots. This is as a result of the poor leaching and incomplete reduction, leaving behind MgO, Mg_2Si or unreacted SiO_2 (Yuvakkumar *et al*., 2013).

2. Poor Crystallinity and Morphology

Instead of sharp-edged silicon crystals, the SEM reveals amorphous or fragmented structures, indicating incomplete or low temperature reduction reaction.

RHA contains amorphous silica that was not fully converted to silicon, while the beach sand (crystalline SiO_2) resisted reduction under mild conditions (Nguyen *et al*., 2014).

3. Agglomeration and Particle Clustering

Instead of discrete silicon particle as expected, the SEM images showed agglomerated clusters, making grain boundary analysis difficult. The high impurity content caused the magnesiothermic by-products to bind particles together thereby reducing the clarity of the microstructure.

4. Lack of clear silicon grain boundaries, hence grain edges appeared blurred with undefined grain boundaries.

CONCLUSION

This study investigated the extraction of silicon from rice husk and Afikpo Beach sand using a combination of drying, sieving, acid leaching, thermal treatment and magnesiothermic reduction methods. The primary aim was to explore sustainable, low-cost local sources of silicon for potential applications in electronics and renewable energy technologies.

The processing techniques showed promising results, with silicon content of 51.45 % and 70.53 % for rice husk ash and Afikpo beach sand, respectively. However, the relatively low silicon content (< 90 %) in the final product can be attributed to various factors, including incomplete purification or leaching, inefficient calcination, magnesiothermic reduction limit and contamination during processing as evidenced by the XRF analysis and the SEM images. Giving the growing global demand for sustainable and low-cost materials in photovoltaic, batteries, and semiconductors, the utilization of abundant resources such as rice husk and beach sand holds significant promise.

However, further research is recommended to enhance the extraction efficiency, silicon purity and percentage; and crystallinity suitable for advanced renewable energy and electronic applications.

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