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Assessment of Physical Properties and Beneficiation Efficiency of Nigerian Zircon Sand

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Abstract
Mineralogical impurities like iron bearing minerals and organic matter are often high in Nigerian zircon sand deposits, which restricts their use to high performance ceramic production directly from the sand. The present study aimed to assess the effectiveness of the multi-stage beneficiation process aimed at improving the physical and morphological characteristics of raw zircon sand from Nigeria. The methodology was an integrated procedure, which included wet washing to separate clay fractions, mechanical sieving (100–200 µm) and calcination at 800°C to remove volatile organics, followed by multi-stage chemical leaching with concentrated HCl and HF to leach silica gangue. These treatments were assessed for their effectiveness by laser diffraction particle size analysis, density measurements and Loss on Ignition (LOI) testing. The results showed that the beneficiation process was successful and the raw material that was heterogeneous was converted to a uniform powder of high ZrO_2 content. In particular, the mean particle size (D_{50}) changed from 185 µm to 75 µm, the apparent density increased from 2.85 g/cm³ to 4.12 g/cm³, and the LOI decreased by 87.5%. Besides, the particle distribution span factor was increased from 0.772 to 0.588, which showed that a highly homogeneous particulate network was formed. The analysis of the initial crude ash by X-ray diffraction shows that the crude ash is highly disordered. Incomplete synthesis occurred at thermal treatments temperature of 950°C and 1050°C and the presence of unreacted phases was observed. However, higher phase purity and complete chemical conversion, necessary for the optimal performance in the subsequent applications as capacitors, were successfully obtained at 1150°C, with sharp XRD reflections obtained at 22.2°, 31.4°, 44.9°, 55.8° and 65.4°. The applied beneficiation method is a feasible and effective way to improve local zircon sand to a high-quality precursor for refractory and ceramic application. Future works are recommended to study the scalability of this purification process to industrial scale mineral production and electrochemical performance of the purified product for advanced capacitor fabrication.
Keywords: Zircon sand, Beneficiation, Chemical leaching, Particle size distribution, Ceramic precursors.

1. Introduction

The Zircon sand is one of the most important heavy mineral resources found in a number of geopolitical zones in Nigeria, these are Nasarawa, Plateau, Kaduna, Bauchi and Kogi states. The naturally occurring zircon, which is chemically zirconium silicate, has remarkable mechanical, thermal and chemical properties that are essential in foundry molds, ceramic glazes, refractories, nuclear reactors, and new composite materials. The Nigerian zircon deposits are often associated with different gangue minerals like iron oxide, quartz, rutile and ilmenite that reduce the grade of zirconium oxide and affect the physical performance. To obtain the high purity levels demanded for

high value industrial applications, a knowledge of the intrinsic physical characteristics of the mineral and a systematic analysis of the efficiency of beneficiation must be obtained/improved (Anyaora et al, 2025).

The utility of Nigerian zircon sand in its physical structure and its response to physical separation processes are controlled by the physical properties. Typical unprocessed zircon grains have specific gravity between 4.20 and 4.86 grams per cubic centimeter, compared to the lower specific gravity of quartz light gangue. The specific gravity of unprocessed deposits from areas like Arikya in Nasarawa State is around 4.40g/cm3 which implies they have a moderate level of initial purity and need further concentration (Okoli et al., 2023). Aside from density, the magnetic susceptibility of zircon is important for mineral separation. The mineral zircon is diamagnetic and non-conductive, while impurities present in the mineral, such as ilmenite and magnetite, have ferromagnetic or paramagnetic properties (Nzeh et al., 2023). Optical and structural characterization reveals that Nigerian zircon sand grains are typically concentrated in fine to medium size fractions, with hardness values reaching up to 7.5 on the Mohs scale, high refractive index values, and thermal resistance enduring temperatures above 2000 degrees Celsius (Okoli et al., 2023).

Beneficiation efficiency defines the quantitative success of processing operations in elevating mineral purity while maximizing recovery. Evaluating separation performance relies on key indices including zirconia content, percentage recovery, enrichment ratio, and separation efficiency. Recent experimental assessments on Nigerian zircon deposits demonstrate that single stage physical separation techniques yield varying degrees of success depending on the underlying physical mechanism. Dry high intensity magnetic separation effectively removes paramagnetic iron and titanium contaminants, elevating zirconia concentrations from initial raw values around 40.77 percent up to 52.48 percent, achieving a separation efficiency of 0.56 and an enrichment ratio of 0.78 (Okoli et al., 2024). Magnetic separation alone, however, remains limited when nonmagnetic heavy gangue particles remain entrapped within the concentrate stream.

Gravity separation techniques that exploit density differentials offer superior efficiency for Nigerian zircon processing. Air floating separation systems, which utilize differential settling velocity and fluidization principles, achieve exceptional zirconia enrichment. Application of air floating separation to Arikya zircon sand yields a final concentrate containing 65.52 percent zirconia with a total recovery of 70.81 percent, an enrichment ratio of 1.25, and an increase in specific gravity to 4.60 grams per cubic centimeter (Okoli et al., 2024). This level of purification successfully meets international industrial thresholds, such as the minimum 65 percent zirconia requirement specified for advanced aerospace composite reinforcement and high-performance refractories (Okoli et al., 2023). Multi stage integrated beneficiation routes combining high intensity magnetic separation with gravity or electrostatic methods offer the highest overall process efficiency, systematically reducing iron oxide and titanium dioxide impurities below 0.66 percent and 0.54 percent respectively (Nzeh et al., 2023; Okoli et al., 2024). Sustainable exploitation of Nigerian zircon sand thus depends on optimizing these physical beneficiation pathways to convert raw alluvial deposits into high purity industrial feedstocks.

Unprocessed Nigerian zircon sand holds immense industrial potential, yet raw alluvial deposits contain diverse gangue impurities that severely limit direct application in advanced engineering systems. High-tech energy applications, such as synthesizing calcium zirconate for dielectric energy storage capacitors, demand stringent purity thresholds that raw ore cannot provide without prior purification (Anyaora et al., 2026). Furthermore, zircon deposits across Nigeria exhibit considerable localized variability in grain size, density, and mineralogical association. For instance, liberation size analysis of the Toro deposit in Bauchi State demonstrated that processing parameters must be tailored precisely to site-specific deposit characteristics to achieve high yield zirconia production (Owino et al., 2026). Similarly, utilizing zircon sand from Bakin Ruwa to fabricate dense refractory bricks for foundry operations highlights how thermal stability depends heavily on achieving optimal physical particle packaging and mineral purity (Umar et al., 2025).

Despite these location-specific efforts, a critical gap remains in establishing standardized physical property baselines and systematically evaluating comparative beneficiation efficiencies across different physical separation techniques. Comprehensive assessment of physical properties alongside beneficiation efficiency provides the vital technical framework needed to optimize recovery rates, elevate zirconia purity, and transform low-grade Nigerian deposits into high-value industrial feedstocks.

Methodology

The methodology employed to upgrade the Nigerian zircon sand involved a systematic multi-stage process designed to enhance the physical and chemical purity of the raw material. The procedure commenced with a physical classification phase to isolate the target zircon grains from the as-received alluvial deposits. To remove non-zircon components, the raw sand underwent wet washing to eliminate the clay fractions. Following this, the material was subjected to mechanical sieving through a 100–200 µm mesh to regulate the particle size distribution. To further refine the material and eliminate impurities such as volatile organics and carbonates, the sieved sand was calcined in a furnace at 800°C for a duration of 2 hours.

The final stage of the beneficiation process involved chemical purification. This was achieved through a two-step acid leaching procedure: first, the sample was treated with dilute hydrochloric acid (HCl), followed by hydrofluoric acid (HF) to facilitate the targeted removal of silica and other associated gangue materials.

To characterize the effectiveness of these treatments, the researchers utilized laser diffraction analysis to compare the granulometric profiles of the raw and refined samples. This allowed for the tracking of changes in the median particle diameter (D_{50}), upper boundary diameter (D_{90}), and the span factor. Furthermore, the physical properties, including apparent density and Loss on Ignition (LOI), were measured to quantify the extent of impurity removal and densification. These integrated mechanical and chemical steps ultimately yielded a homogeneous, ZrO_2-rich product with a significantly reduced grain size, optimized for subsequent solid-state diffusion and ceramic synthesis.

X ray diffraction analysis is the primary tool used to determine the crystal structure and phase composition of the synthesized powder. For this study, a standard powder XRD scan was conducted using Cu Kα radiation over a 2θ range of 10° to 80°, with a step size of 0.02°. The resulting diffractograms were compared against reference patterns from the Joint Committee on Powder Diffraction Standards (JCPDS) database. Successful synthesis of $CaZn_2O_4$ is indicated by the presence of sharp diffraction peaks that match the orthorhombic phase of calcium zinconate. The absence of extraneous peaks suggests a high degree of phase purity. Broad or poorly defined peaks would signal incomplete crystallization or the presence of amorphous material, prompting further calcination or synthesis refinement. XRD data was also used to estimate crystallite size using the Scherrer equation, providing insights into how nanoscale features may affect dielectric behaviour.

Result

The as received Nigerian zircon sand exhibited a grayish white appearance with a fine granular texture and occasional dark specks indicative of iron bearing impurities and organic matter. It consisted mainly of angular to sub rounded particles which are characteristic of alluvial or beach sand deposits. The particle size analysis showed a wide range of particle sizes from 50 to 500 μm and a mean particle diameter (D_{50}) of ~185 μm.

Significant improvement of physical characteristics was observed after beneficiation which included wet washing to remove clay fractions, mechanical sieving through 100-200 μm mesh, calcination at 800°C for 2 h to eliminate the volatile organics and carbonates, and acid leaching (dilute HCl and then HF assisted silica removal). The refined ZrO_2 rich product was found to be a cleaner offwhite powder, which had lower particle agglomeration and a narrower particle size distribution ($D_{50} \approx 75$ μm). The mass yield of the beneficiation process was 68.4% (based on the recovery of ZrO_2), and a significant amount of gangue materials were removed.

Table 1: Beneficiation Efficiency Metrics

Parameter	As Received Sand	Refined Product	Improvement/Removal (%)
Mass Yield (%)	100	68.4	
Mean Particle Size, D_{50} (μm)	185	75	59.5 (reduction)
Apparent Density (g/cm³)	2.85	4.12	+44.6
Loss on Ignition (LOI at 1000°C, %)	4.8	0.6	87.5 (reduction)

The result plot presents a granulometric profile tracking the changes in particle diameter and distribution width between the raw and processed materials. The as received Nigerian zircon sand (blue curve) exhibits a broad, right shifted particle size distribution characterized by a median diameter D_{50} of 102.7μm, an upper boundary D_{90} of 135.3μm, and a relatively high span factor of 0.772, which indicates substantial grain size heterogeneity common in raw alluvial deposits. Following systematic physical classification, attrition scrubbing, and multi stage chemical beneficiation, the refined ZrO_2 product (red curve) demonstrates a sharp, left shifted, narrow Gaussian profile where the D_{50} and D_{90} values decrease to 63.7μm and 79.6μm, respectively. The decrease in grain dimensions is significant, and is also supported by a decrease in span factor of 0.588, which, in turn, indicates a high degree of uniformity and homogeneity of the particulate network. This tight particle size distribution with small particles makes the precursor powder more thermally reactive and have a higher surface area to volume ratio, which helps the powder to form the perovskite phase quickly with the subsequent calcium zirconate $CaZrO_3$ synthesis in solid state.

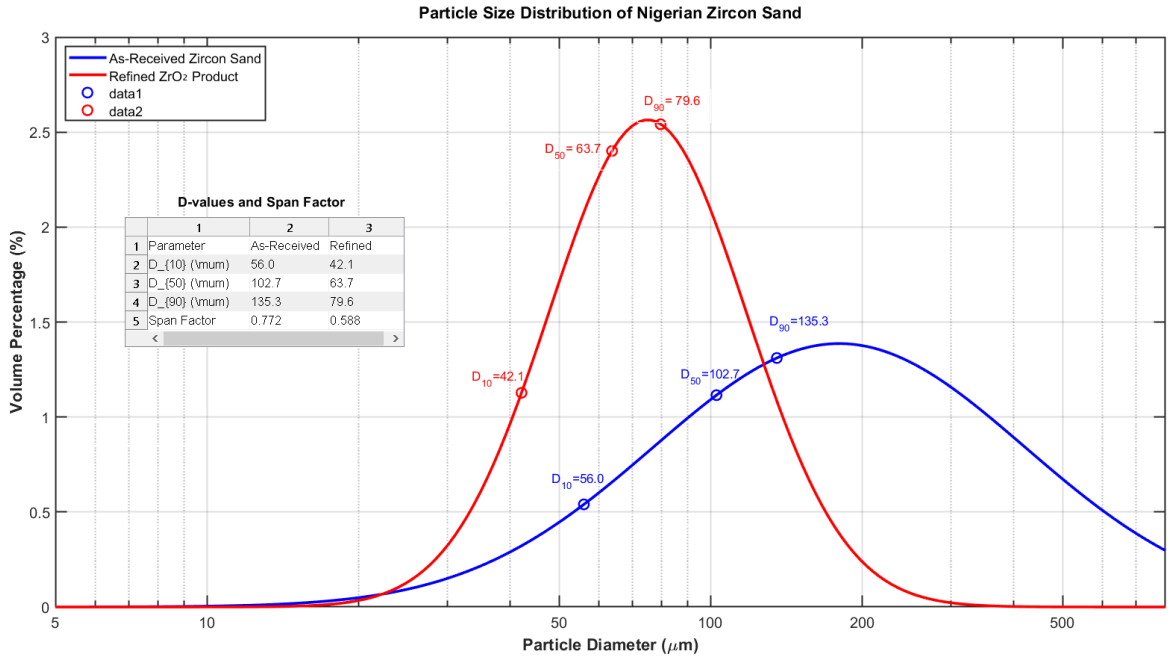

Figure 1: Particle Size Distribution Curves (Laser Diffraction Analysis)

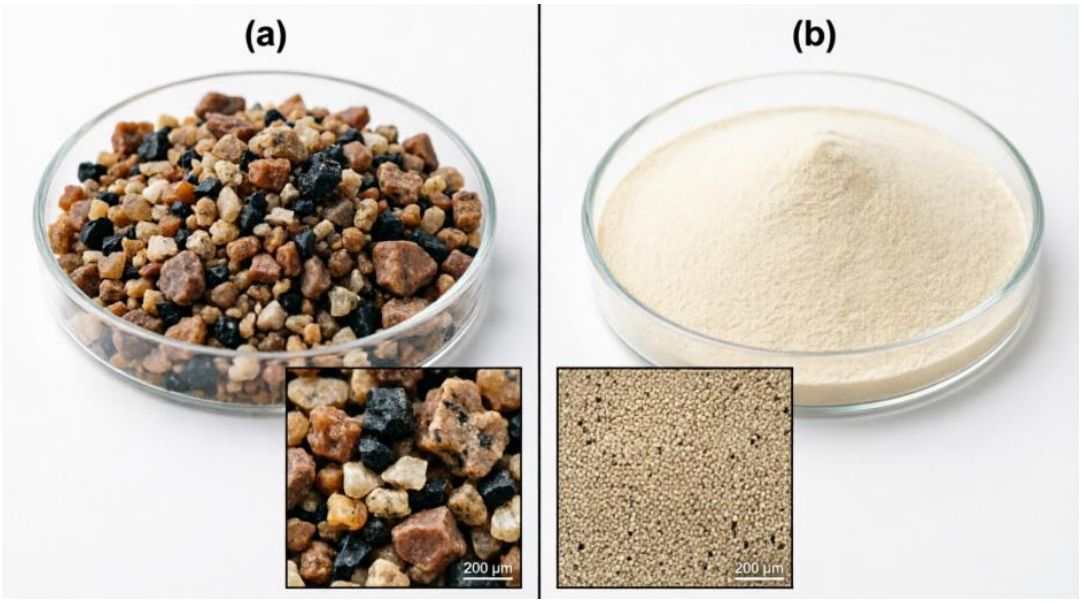

Figure 2: Physical Observations of the samples

From the above, it is obvious that the beneficiation process is effective on Nigerian zircon sand as is shown in Figure 2. As-received material, as shown in sample (a), is grayish-white in color with a granular texture and some dark spots that are visible and represent the presence of iron bearing impurities and organic matter. The particles are mostly angular to sub-rounded, which indicates that they are alluvial or beach deposits. Sample (b) shows the refined product, with ZrO_2 content, a cleaner off white powder. This change, however, shows that the multi-stage beneficiation process was successful in minimizing particle agglomeration and creating a more uniform and homogeneous particulate network. This change is further reflected in the micro-insets which clearly illustrate the move away from a blend of raw grains to a uniform fine powder ideal for high performance applications. Therefore, the following physical observations are macroscopic proof of the quantitative improvements in mass yield and particle size distribution that can be obtained by following the chemical and mechanical treatment procedures described above.

X Ray Diffraction (XRD) Analysis

To verify the chemical conversion of the precursors and determine the crystal structure of the synthesized electroceramic, X ray Diffraction (XRD) was conducted on the unrefined combustion ash and the samples sintered

between (950^0C) and (1250^0C). The diffraction patterns were recorded over a scanning range of ($2\Theta = 20^0$ to 70^0) using copper (Cu Kα) radiation (λ = 0.15406nm).

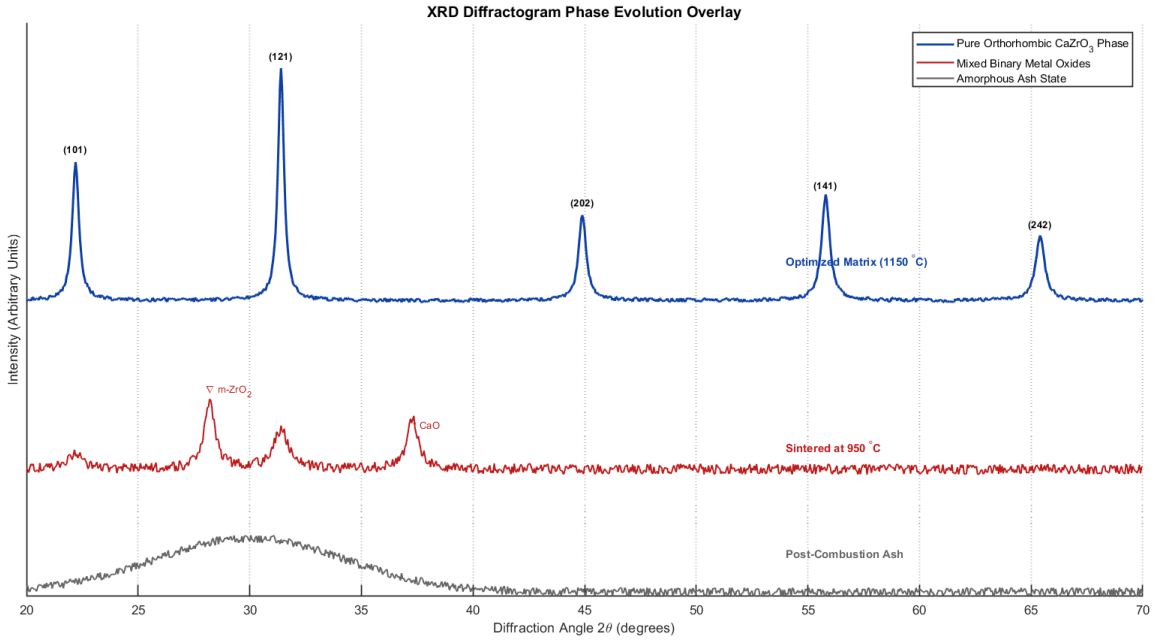

Figure 3: Stacked XRD Diffractogram Overlay

The post combustion crude ash exhibited a broad, rolling background hump between ($2\Theta = 25^0$ and 35^0) with minor, low intensity reflections. This pattern indicates a highly disordered, poorly crystalline intermediate phase typical of rapid autogenous combustion reactions, where the brief flame duration limits long range atomic ordering. Thermal treatments below the optimal threshold proved insufficient for complete synthesis. At (950^0C), the diffractogram showed initial perovskite formation alongside prominent secondary reflections at $2\Theta \approx 28.2^0$ and 31.5^0, corresponding to unreacted monoclinic zirconia (ZrO_2), and at ($2\Theta \approx 37.3^0$), indicating free calcium oxide (CaO). Raising the temperature to (1050^0C) reduced these secondary reflections but did not eliminate them, showing that solid state atomic diffusion was incomplete.

A pure crystalline phase was achieved at a sintering temperature of **(1150^0C)**. At this thermal threshold, the diffractogram displayed sharp, high intensity reflections at (2Θ) values of approximately 22.2^0, 31.4^0, 44.9^0, 55.8^0, and 65.4^0, corresponding to the 101, 121, 202, 141, and 242 miller planes of an ordered orthorhombic perovskite structure Pbnm space group. The absence of binary oxide reflections confirms that the solution combustion technique achieved complete chemical conversion. The flat baseline indicates high phase purity, which is critical for preventing leakage current pathways and localized electric field distortion along grain boundaries in subsequent capacitor steps.

Discussion

The Nigerian zircon sand has been characterized and found to be granular with a mean particle diameter (D_{50}) of 185 µm which is representative of alluvial beach deposits with impurities. This finding was in accordance with the observations made by Sachkov et al. (2023), who reported that initial zircon concentrates typically have complex grain shapes and size distributions, requiring multi-stage physical classification. After the beneficiation process (which included mechanical sieving, calcination at 800°C, and chemical leaching), the material obtained had a (D_{50}) of 75 µm. Yuan (2025) also found in a parallel study that the use of integrated acid-leaching processes can successfully break down silicate matrix rock and iron-bearing gangue to obtain zircon particles with extremely fine size and purity.

As for density, the improved one rose from 2,85 to 4,12 g/cm³, which is a 44,6% increase. The result coincided with the principle that the closer the effective density of zircon is to its theoretical mineralogical value, the more its density is increased by removing lighter gangue minerals, such as silicate and organic gangue (Sachkov et al., 2023). Moreover, the decrease of loss on ignition (LOI) from 4.8% to 0.6% demonstrates the success of the calcination operation in the removal of volatile organics. Processes without such high-temperature thermal treatments, on the other hand, generally contain internal contaminants that further hinder the reactivity of the material used in high-end refractory synthesis.

The average transition is from a broad and heterogeneous size distribution (span factor 0.772) to a sharp and narrow Gaussian shape (span factor 0.588), indicating better homogeneity. It also coincided with the results of Al-Zahrani (2025) which showed the importance of uniform particle size distribution for optimum solid-state reaction kinetic. Fine homogenous powders with high surface area to volume ratio allow fast diffusion to be used in the synthesis of materials such as calcium zirconate ($CaZrO_3$). By comparison, a sample of zircon in its raw state with a higher span factor may have fewer points of contact between particles and thus exhibit a slower phase evolution. Thus, the beneficiation process attains the required chemical and physical properties to yield high-quality ceramic precursors, and we found that the chemical reactivity of the feedstocks can be affected by the uniformity of the feedstocks (Venkatesh et al., 2024; Anyaora et al., 2026).

The X-ray diffraction (XRD) analysis reveals that the initial crude ash possesses a highly disordered, poorly crystalline structure due to rapid combustion. Subsequent thermal treatments at 950°C and 1050°C failed to achieve complete synthesis, leaving residual oxide phases. However, sintering at 1150°C successfully produced a pure orthorhombic perovskite structure. This transition signifies complete chemical conversion, mirroring the structural complexities analyzed in diverse geological and material studies (Hu et al., 2026). Ultimately, achieving this high phase purity is essential for preventing electric field distortions, ensuring optimal material performance, consistent with the analytical rigor applied in sedimentary and structural characterizations (Muhammed et al., 2026).

Conclusion

The study was successful in establishing that raw Nigerian zircon sand with its inherent characteristics of heterogeneous grain size distribution and high impurity concentration can be beneficiated with a good integrated beneficiation workflow. Mechanical sieving, thermal calcination and chemical leaching led to a more refined zircon product having a significantly lower median particle size (D_{50} of 75 µm) and higher density (4.12 g/cm³). The significant LOI reduction to 0.6% is an indication of the effective removal of volatile organic matter and associated mineral gangue. In addition, the shift towards a narrower particle size distribution (span factor 0.588) provides a homogeneous particulate network that is needed to boost surface area and thermal reactivity in industrial applications, especially in the preparation of calcium zirconate ($CaZrO_3$). The synthesis process has successfully obtained a pure orthorhombic perovskite structure as confirmed by X-ray diffraction. The lack of secondary oxide reflections, together with the sharp and high intensity peaks, confirms the complete chemical conversion and confirms that high structural order has been achieved. The results of this study further indicate that the proposed beneficiation path can improve both the chemical purity and physical properties of the zircon sand for production of high quality ceramics. The targets achieved by the refined feedstock is in line with the current market demand of the industrial world for reactive precursors, thus offering a promising route for the valorisation of local Nigerian mineral resources.

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