Suitability of Zircon from Takalafiya for Bio-Ceramic Denture Applications

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Abstract
Dependence on the importation of costly materials is the common complaint by local producers, which contradicts Nigeria's huge endowment of mineral resources. This situation calls for an alternative such as the exploration of local contents such as zircon. This paper analyzed zircon ore from Takalafiya, Taraba State, Nigeria as potential material suitable for bio ceramic denture applications. The petrographic analysis was conducted using a microscope (Olympus CE 0803142), while analysis for elemental composition was carried out using PRO: X: Phenom-World 800-07334 model for the X-ray fluorescence (XRF), then the quantitative and qualitative carried out using X-ray diffraction (XRD), as well as the morphology of sample which was conducted using a scanning electron microscope (SEM). The petrographic results showed the luster of adamantine, the reddish-brown, the yellow-grey color of a streak of white that indicates colors distinction, and the presence of zircon in the mineral matrix. The XRF revealed SiO_2-23.00%, Al_2O_3-0.55%, CaO-0.076%, TiO_2-0.38%, Cr_2O_3-0.009%, MnO-0.027%, Fe_2O_3-1.89%, As_2O_3-0.044%, ZrO_2-72.76%, PbO-0.037%, ThO_2-0.091%, and L.O.I-1.22% respectively. The quantitative and qualitative X-ray diffraction (XRD) analysis showed the presence of zircon as the predominant mineral and followed by silica (quartz). The SEM morphology showed zircon characterized by clusters of massive rhombohedra microstructure, with some slightly curved shape granular crystals. The percentage of zircon being the highest in the proportion revealed the potential requirement for zirconia-based bio ceramic denture applications. However, the presence of 1.89% Fe_2O_3 is white color-deterrent compared to teeth, while 0.09% chrome,0.037% PbO, and 0.091% ThO_2 are toxic been caused by radioactive activity, hence the sample must be purified before its medical applications.

Keywords: Zircon from Takalafiya, Potential, Bioceramic Denture, Application

Introduction

Global economic recession, pricey costs, and recently, the restriction on travel due to the COVID-19 pandemic are among the common complaints by local producers, which calls for alternative measures such as the exploration of local content. These materials include zircon as bio ceramics for denture production. Zirconium silicate (ZrSiO4) occurs as mineral zircon with high refractoriness, tensile strength, and hardness. Its applications keep growing in making glazes, arms, and medical feats (Wahyudi, 2017; Ogundare, 2016; Rajagopalan, 2021). Zircon usually contains sundry elements such as quartz and some impurities ($ThiO_2$, PbO, and Fe_2O_3) due to formation and change factors during their consolidation (Harlow, 2016, Gross, Bergfeldt, Fretwurst, Rothweiler, Nelson and Stricker, 2020). These impurities are usually detrimental to their applications, hence need purification of the unwanted elements to meet the required standard before applications.

Biomaterials or synthetic substances are those put into body tissue as part of an inserted medical device or used to replace an organ, bodily function, for instance, bones and tissue grafts (Boffito & Ciardelli, 2021; Lynch, Kondiah & Choonara, 2021; Arjunan, Baroutaji, Robinson & Praveen, 2021). Also, bio ceramics have a higher tissue response compared to polymers and metals individually (Dorozhkin, 2018; Kumar, Dehiya & Sindhu, 2018; Daculsi, 2016). They are also elective materials used for the regeneration of bone tissues due to their suitable compositional mimicry property of bone's inorganic components (Abbas, Dapporto, Tampieri & Sprio, 2021). Dorozhkin (2018) further averred that a potential biomaterial must also pass all necessary regulatory requirements. Conversely, a denture is an artificial replacement for one or more teeth; or a set of removable *false* *teeth* (Merriam-Webster, 2019 and WebMD, 2019) made from either plastic, nylon, or metal (NHS, 2018).

The Healthcare Resource Guide: Nigeria (2019) reported of Nigeria's importation of millions of dollars' worth of orthopedics and prosthetics in the years – 2016 to 2021of 37.6m, and dental products from 2016 to 2021of at 27.4m respectively. This indicates the trajectory of the exploration of the country's huge endowment of solid minerals of different types distributed across its thirty-six (36) states (Alexander, Maina & Barnimas, 2016; Datiri, 2012; Mathias, 2019) with yet slow growth and development of ceramics (Sadiq, Munai & Fai, 2003). Perhaps, the pathetic situation could be due to the overwhelming cost of machines, the lack of trained personnel with high-tech equipment (Yunasa & Ibrahim, 2019 and Umar, 2000), importation of materials (Ogundare, Akinbogun, Kashim & Aramide*,* 2017 and Abraham, 2019) that needs to be tackled.

Although some of these local materials could be processed into what Wang *et al* (2011) considered as bio ceramics that offer physical and chemical properties that sometime exceed their natural equals, however they are prone to some setbacks requiring proper and early analyses before use. Therefore, this study aimed at determining the potential suitability of the Zircon ore from Takalafiya, Bali Local Government Area, Taraba State as a biomaterial for denture applications against the complaints from various producers.

Materials and Methods

Materials

The materials and tools used in the study included slide glass, mounting pin, hot plate, grinding machine, forceps, glass rod, cutting machine, microscope, Olympus CE 0803142, Araldite gum (Epoxy), PRO:X: Phenom-World 800-07334 model, detergents, Canada Balsam Carborundum powder, and methylated spirit. Also, the Reflection-transmission spinner stage with Theta-Theta settings, XR and XRD processing software, and pycnometer was used. The sample material (zircon ore) was sourced from Takalafiya, Bali Local Government on coordinates N 08^0 05^0 58^0 0^0 E 010^0 40^0 19^0 9^0 (115/S, 61 95/S 187), in Taraba State, Northeast Nigeria.

Plate 1: Zircon - Research fieldwork

Methods

The research procedure involved the subjection of the sample to three stages of analysis for characterization thus, petrographic (macroscopic and microscopic), XRF, XRD, and SEM.

Macroscopic and Microscopic Analysis

The sample was first subjected to identification and confirmation as zircon ore. The procedure began with the cutting of the side of interest of the sample using the rock cutting machine. The cut side was ground to powder while observing under a petrology microscope (Olympus CE 0803142). The carborundum powder (an abrasive containing silicon and carbon) and the zircon chip were thinned facing the glass slide. It was marked using a diamond pen and placed on a source of heat (hot plate) for 5 minutes then scrapped to the coverslip. The glass slide was gummed to the coverslip using Canada Balsam Araldite gum (Epoxy) mixed to equilibrium using a glass rod. The bubble air was eliminated by gently rubbing the surface using, and gently heating the slide with forceps. The sample was then dried for about 10 minutes and kept for 2 days, after which the slides were washed using detergents and methylated spirit and then allowed to dry as they were labeled ready for further studies. Also, a pycnometer was used to measure the specific gravity of the zircon using one cube of the sample.

X-ray Fluorescence (XRF) Analysis

The sample was compressed into a compact form and fused with lithium tetraborate flux in a glass bead. An X-Ray beam from a tungsten X-Ray gun was focused on the specimen. The focused X-Rays caused the inherent elements in the sample to excite leading to the emission of secondary X-Rays that are characteristic of the elements. The amount of emission was also directly proportional to the concentration of the elements present in the sample. The X-ray spectrum of the sample was computer-processed to determine the elements present and their proportion by using the PRO: X: Phenom-World 800-07334 model.

X-Ray Diffraction (XRD) Analysis

The sample was analyzed for X-Ray Diffraction after it was first finely ground and homogenized as the average bulk composition was determined. The powdered sample was then prepared using the sample preparation block. It was compressed into the flat sample holder to create a flat, smooth surface that was later mounted on the sample stage in the XRD cabinet. The sample was analyzed using the reflection-transmission spinner stage using the Theta-Theta settings. The two-Theta starting position was 4 degrees and ends at 75 degrees with a two-theta step of 0.026261 at 8.67 seconds per step. The Tube current was 40mA and the tension was 45 VA. A Programmable Divergent Slit was used with a 5mm Width Mask and the Gonio Scan. The intensity of diffracted X-rays was continuously recorded as the sample and detector rotated through their respective angles.

SEM analysis

The sample was subjected to analysis following Sinnott-Armstrong (2010) to retrieve the main information presented by SEM on the surface detail of an object, which essentially produced a magnification image of the object/sample for the topographical, compositional, and morphological characterization based on grain size.

Results and Discussions

The results of the characterization of zircon ore from *Takalafiya* for possible use in Bioceramic denture applications are reported in the order in which the experiments were conducted as thus:

Macroscopic and Microscopy: Petrographic Interpretation

The macroscopic and *microscopy* akin to petrographic interpretation results of the sample provided pertinent information about the visual inspection data of a clue to the chemical composition, physical properties: structural issues and distribution, occurrence and class of species as well as the color. Crystal habit and system, hardness and specific gravity, luster, color, streak, cleavage, fracture, and occurrence; which form the physical aspect are shown in Table 1.

Table 1: Physical Properties of Petrographic Analysis of Zircon from Takalafiya, Taraba, Nigeria

Name	Chemical Composition	Crystal habit and system	Gravity and specific gravity	Luster	Colour	Streak	Cleavage	Fracture
Zircon	$ZrSiO_4$	Tetrahedral prismatic also in dendritic grains	7.5 4.3	Adamantine	Reddish-brown, yellow-grey	Colorless White	Imperfect	Conchoidal fracture

The geochemical features *showed a* structural appearance of imperfect cleavage, and fracture of conchoidal due to weathering, which suggest crystal habit and a system of tetrahedral prismatic zircons. Also, the mineralogical composition of dendritic grains is of very high relief and makes textural measurements like the orientation of grains, in a polycrystalline nature that concurred with Dutrow and Clark's (2021) description of zircon. Equally, the detrital nature of the sample fits the depiction of zircon based on the preexisting data of the types of formation sources explained by Sawaki, Suzuki, Asanuma, Okabayashi, and Hattori, Saito, and Hirata (2017). Likewise, the results showed infinity with tetragonal polycrystalline zirconia, which can partially stabilize with 3 mol% yttria as accounted for by Kleverlaan *et al* (2015).

Similarly, the occurrence and crystal class in Table 1 revealed that the accessory mineral of more acid igneous rocks, with the luster of adamantine; detrital deposits minor mineralogical distinctions and of the tetragonal crystal class with densest of the major gemstones, which is in line with Alden (2019) clue on zircon. Also, the sample showed fire suitability of 1170 - 2370^{0}C, which is relatively similar to the result of Saridag, Tak & Alniacik's (2013) study. The high strength revealed by the material shows its potential to be a beneficial factor for use being consistent with the literature data on *hyacinth* or *jacinth* species (Arem, Clark & Smigel, 2019). The results of tetrahedral prismatic with dendritic grain characterize the property of teeth, hence having the potential of being suitable for the development of dentures.

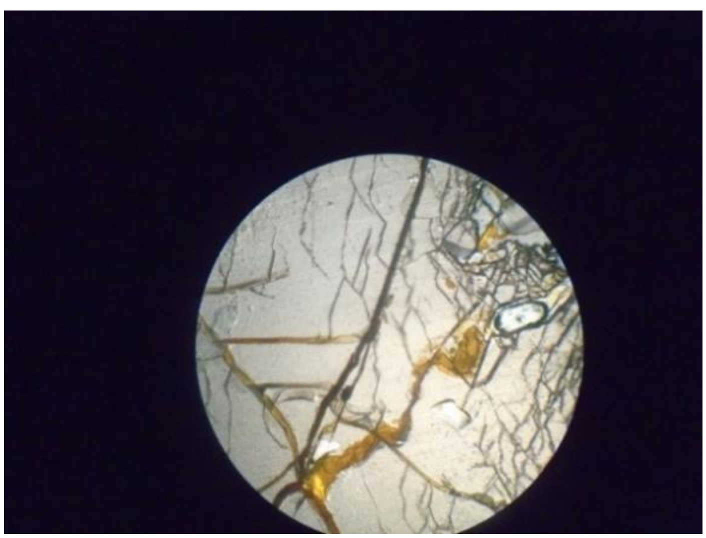

Plate 2: Petrographic Microscopically

The traces of reddish-brown, yellow-grey colors and a streak of colorless white, revealed the white-colored minerals like quartz, while the black is biotite Plate 2 is likely to be due to the inclusions of

ThiO$_2$, PbO, and Fe_2O_3 caused by radioactivity. The fractures showed the weathering effect that correlates with the study by Pidgeon, Nemchin, Roberts, Whitehouse, and Bellucci (2019) consistent with the typical characteristics of the zircon ore study by Gross *et al* (2020). Remarkably, despite these inclusions sample exceeds the premium standard of zircon as pointed out by Snyders, Potgieter, and Nel (2005) and Breiter *et al* (2014). Conversely, the radioactive materials however require removal and synthesis of material before use as recommended by Saridag, Tak, and Alniacik (2013).

XRF Analysis

The results of the elemental composition are presented in Table 2 and Figure 1.

Table 2: Chemical Composition of Zircon of Takalafiya in Percentage

Oxide	Concentration (in %)
SiO_2	23.00
Al_2O_3	0.55
CaO	0.076
TiO_2	0.38
Cr_2O_3	0.009
MnO	0.027
Fe_2O_{3s}	1.89
As_2O_3	0.044
ZrO_2	72.76
PbO	0.037
ThO_2	0.091
L.O.I	1.22

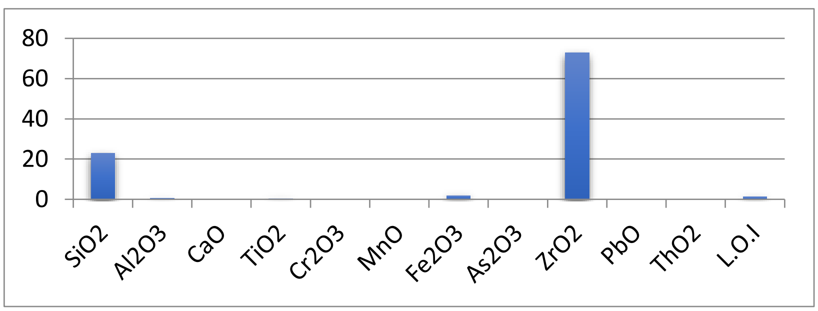

Figure 1: Values of the chemical elements in Zircon in chart

Although the results of the chemical composition *values* or elemental mineral content of the zircon sample in Table 1 and Figure 1 were established, hence it was further confirmed with XRF as opined by Ogundare (2016), Nanoscience Instruments (2018) and Manappallil, (2016) whose studies suggest that material must be tested before and after upon use (Sikalidis, 2011) revealed in Table 2, as well as the XRD result in Table 3 established the material to be zircon*. However, t*he presence of SiO_2, Al2O$_3$, Cao, TiO_2, Cr_2O_3, MnO, Fe_2O_3, As_2O_3, ZrO_2, PbO, and ThO_2 (see Table 2) is consistence with the typical zircon ore as opined by Harlow (2016) and Gross *et al* (2020), who attributed such to the geological fact that materials such as zircon that they contain natural radioactivity.

For instance, Table 2 revealed 72.76% of (ZrO2), which possess significant potential in comparison with the premium-grade quality given the standard in comparison to the zircons obtained in other geographical locations. Similarly, the implication of the high content of zircon in the sample indicated that its recovery into zirconia will yield substantial quantity, and with less effort. This makes the sample potentially suitable for the proposed enterprise, which is the development of dentures. Despite the significant potential of the sample, the presence of ThO_2 and PbO, AsO_3 (are toxic), and

Fe_2O_3 (susceptible to exhibiting shades of brown contrary to the ideal natural color of teeth: white, hence tend to jeopardize the aesthetic quality) are setbacks. However, the investigated properties revealed results that are acceptable for standard denture development as earlier mentioned, yet require purification for medical compatibility.

XRD Analysis

The results are demonstrated in Figure 2 for Zircon ore from Takalafiya

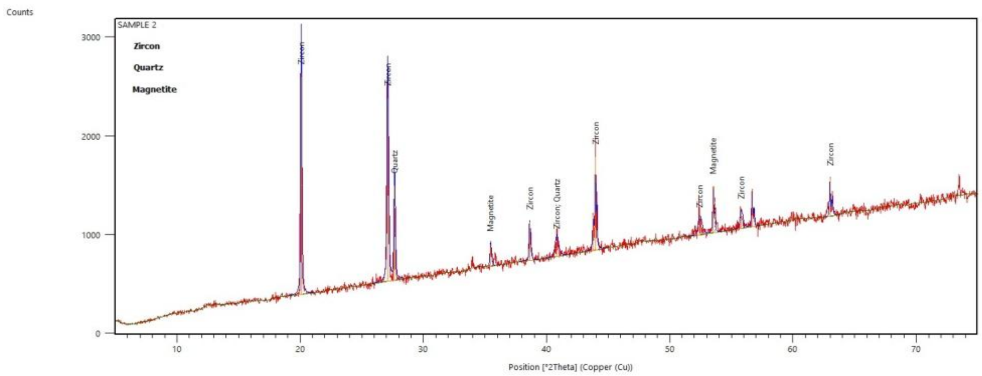

Figure 2: Major minerals identified by X-Ray Diffraction Pattern

The results of the XRD determine the phases/microstructure and their crystal structure or material composition information provided the images of the surface of the sample at extremely high magnifications revealed. The peak in intensity that occurred when the mineral contains lattice planes combined with d-spacings appropriate to diffract X-rays at that value of θ. Although each peak consists of two separate reflections ($K\alpha_1$ and $K\alpha_2$), at small values of 2θ the peak locations overlapped with $K\alpha_2$ appearing as a hump on the side of $K\alpha_1$, the greater separation occurred at higher values of θ. Typically these combined peaks are treated as one.

The 2λ position of the diffraction peak was typically measured as the center of the peak at 80% peak height as seen in Figure 2. Also, the intense peaks apparent at 20^0, 27.5^0, 28^0, 44^0 2θ correspond to what crystal plane of zirconia by the ICDD database. Also, smaller features of the XRD spectrum were assigned to the crystal phases of zirconia. Besides weak peaks, they were assigned to associate with oxides impurities. Results are commonly presented as peak positions at 2θ and X-ray counts (intensity) in the form of a table or an x-y plot (Figure 2). Intensity (I) is either reported as peak height intensity, that intensity above background, or as integrated intensity, the area under the peak. The relative intensity is recorded as the ratio of the peak intensity to that of the most intense peak (relative intensity = I/I_1 x 100).

The d-spacing of each peak was then obtained by solution of the Bragg equation for the appropriate value of λ. Once all d-spacings have been determined, automated search/match routines compare the ds of the unknown to those of known materials. Because each mineral has a unique set of d-spacings, matching these d-spacings provides an identification of the unknown sample. After a systematic procedure is used by ordering the d-spacings in terms of their intensity beginning with the most intense peak. Files of d-spacings for hundreds of thousands of inorganic compounds are available from the International Centre for Diffraction Data as the Powder Diffraction File (PDF). The peaks obtained from these analyses were matched with the minerals phases from the PDF 2 ICDD database attached to the XRD processing software, XPert High score Plus also from Panalytical. The XRD graph peaks for zircon ore perfectly agree with Bragg's peaks appearing in the XRD pattern.

Table 3: Mineralogical Composition of the Material

Mineral name	Compound name	Empirical formula	Chemical formula
Zircon	Zirconium silicate	Zr0.9Hf0.05REE0.05SiO4	Zr4Si4O16 (ZrSiO4)
Magnetite	Ferrous-ferric Oxide	Fe3O4	Fe2+Fe3+2O4
Quartz	Silica mineral	Si_3O_6	SiO_2

The results obtained also agreed with other works reported by Gross, *et al,* (2020), Boch and Niepce (2017), Suastika, Karelius, and Sudyana (2018), which stated that dark color commonly associated with most zircon minerals is caused by the presence of impurities particles in compounds of the magnetic element (Harlow, 2016). The common mineral phases usually associated with most zircon minerals, raw Talalafiya gemstone crystallographic parameter revealed tetragonal crystal for Zr, while that of Si is hexagonal and that of Fe_3O_4 is cubic. The X-ray diffraction results are shown in Table 4 and Figure 2 revealed the presence of three mineral phases; zircon, the mineral of interest, with quartz and magnetite as non-valuable associated minerals.

The presence of these mineral phases can be attributed to the differential mineralization of the deposit that may have resulted from the biogenic and geochemical tectonic activities of the earth's mantle. There are differences also in the peaks of each of the revealed or dominant elements. For instance, Zr (Zircon) shows the highest peak, followed by Si (Quartz), then Fe_3O_4 (Magnetite) respectively, which agrees with the visual observation made of the SEM in Plate 2 of the thin analysis. However, it indicated that they can be separated from each other easily after comminution based on their physical and chemical nature while double or complex phases will be comminuted to a point that will enhance their liberation for possible physical separation or leaching.

SEM Analysis
The results are presented in Table 4 and Plates 3-9.

Table 4: SEM of Zircon of Takalafiya Showing Different Magnifications

S/N	Plate	Magnification (x)	Grain Size (mm & um)
a.	3	250	107mm
b.	4	300	894 µm
c.	5	500	536 µm
d.	6	600	447 µm
e.	7	800	335 µm
f.	8	1000	268 µm
g.	9	1500	179 µm
h.	10	2000	134 µm

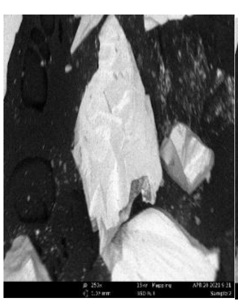
Plate 3

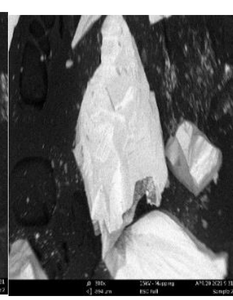
Plate 4


Plate 5

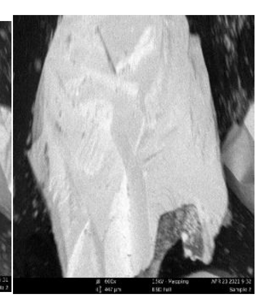
Plate 6

Plate 7

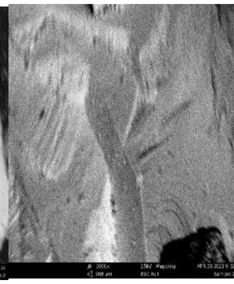

Plate 8

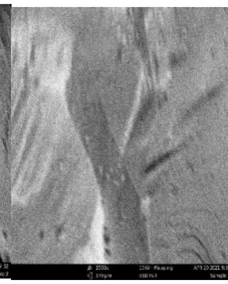

Plate 9

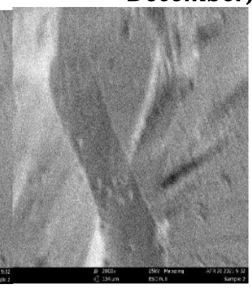

Plate 10

Plates 3 - 10 SEM images of zircon ore at different magnifications.

The SEM analysis conducted provided the results of images based on the various magnification (see Table 4), which revealed the topography/morphology structure or features of the sample (Plates 3-10). This is in agreement with Wang *et al* (2011), which opined that SEM is essential for applications where resolutions greater than those provided by optical microscopy are required. More so, given that SEM is one of the most popular analytical tools due to its ability to provide high-resolution images with excellent depth of field.

Conclusions

The zircon ore from *Takalafiya* for bio ceramic denture applications was successfully characterized for petrography, XRF, XRD, and SEM. The results of experimentations of the sample revealed significant potential as local (indigenous) content suitable for bio ceramic application. The chemical results obtained are comparable with the worldwide average based on the premium standard. The study contributes to the exploration of indigenous natural resources as potential material for medical grade dentures thus limiting the overreliance of Nigeria on costly given the rate of the dollar. This jeopardizes the GDP, hence detrimental national economy.

Although there are inclusions (impurities) $ThiO_2$ and PbO that are toxic, as well as Fe_2O_3 and Cr_2O_3, that counter the white color which is the ideal color of natural teeth. these data are based on the required properties for bio ceramic products, which pose some challenges. However, the sample showed good properties with a zircon content of 72.76%. Also, the silica content of 23% could play a crucial role as a glass-forming agent, which makes the material a versatile candidate for the desired application.

Further study should be conducted on the sample given the inclusions of impurities aforementioned that should undergo treatments for an upgrade to meet the required standard of making dentures.

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