SPECTROSCOPIC CHARACTERIZATION OF A NIGERIAN STANDARD SAND: IGBOKODA SAND

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ABSTRACT: The characterization of the Nigerian Igbokoda Standard Sand was performed by X-ray diffraction, IR and Raman Spectroscopy, and nuclear magnetic resonance techniques. The principal reflections occurring at the d-Spacings of 4.25745, 3.34359, 2.45705, 2.28172, 2.23697, 2.12754, 1.98036, 1.81827, 1.54178 and 1.37190 Å confirm the presence of α-quartz crystalline structure in the cample. The calculated unit cell parameters are: a = 4.9133(2) Å, c = 5.4053 Å, V = 113.00 Å. Presence of Si-O asymmetrical stretching vibrations at 1119.61 – 967cm^{-1}, Si-O asymmetrical stretching vibration at 1180 – 1120cm^{-1} and Si-O Symmetrical Bending Vibration 693cm^{-1} reveal the presence of quartz and that it is in the crystalline form. Bands at around 800cm^{-1} - 822.79cm^{-1} and 786.23cm$^{-1,}$ reveal that the silica is in the form of α-Quartz. The Raman spectrum of the sample has two peaks at around 203cm^{-1} and 463.07cm^{-1} for A$_1$ vibrational mode characteristic of quartz. These peaks reflect that the sample studied belongs to the low temperature quartz. The occurrence of the Raman shift at 463.07cm^{-1} supports the observation from XRD studies which establishes that the quartz observed in this work is α-Quartz. The ^{29}Si NMR spectrum shows a prominent signal at –107.95 ppm correlating to quartz.

Keywords: Sand, X-ray diffraction, infrared and Raman spectra, ^{29}Si nuclear magnetic resonance, Spectroscopic techniques.

1. INTRODUCTION

Silica is made up of one atom of silicon and two atoms of oxygen. It has the chemical formula – SiO_2. Silica is the main component of the mineral quartz. Quartz is the chief constituent of sand [1]. In terms of its crystalline form, silica can be found in nine varying polymorphs. Common among these are quartz, tridymite and cristobalite. Quartz can be further divided into alpha (α) and beta (β) quartz. Low temperature α-quartz transforms to β-quartz upon heating under atmospheric pressures at about 573°C [2]. Quartz is mostly colourless or white but is often coloured by impurities like iron (Ferreira et al, 2015). After feldspar, quartz is the second most prevalent mineral in the earth's crust. It has been reported that about 12% of igneous rocks are quartz [3, 4]. The primary source of quartz is molten magma (lava) from beneath the earth's crust. Heat and pressure effects which occasion ejection of lava from the core make quartz available on the earth's surface [4]. The crystalline conformation of quartz is founded on four oxygen atoms connected together to form a three-dimensional shape referred to as a tetrahedron having one silicon atom at its centre. Several of these tetrahedra are linked together by sharing one another's end oxygen atoms to form a quartz crystal [4].

The unit cell of the crystal make-up involves three SiO_2 groups. The unit cell of quartz is a rhomb (rhombohedral crystal). This rhomb equate to a hexagonal unit cell [5, 6]. X-Ray Diffraction (XRD) helps to answer the question – ''is this sample crystalline or amorphous?''. Infra red (IR) spectroscopy has been backed to be one of the most promising instrumental techniques in the structural and mineral constituent elucidation of silica sand [5]. Raman spectroscopy is a complementary instrumental technique to IR. Together with IR, it furnishes information about bond types. Correct identification of bond types is a precursor to establishing the chemical composition and polymorph of quartz. Other techniques like magnetic resonance also help identify quartz [5]. Silica with at least 95% SiO_2 is favoured for manufacturing and industrial uses. This is owing to some excellent properties which it possesses. Silica is rigid, chemically inert and has elevated melting point. These properties are traced to the strength of the bonds between the atoms [7]. In the face of astronomical increase in world population, without corresponding increase in resources, it becomes necessary to find new sources for raw materials for these industries. Upon finding new sources, it is necessary that they are adequately characterized. In a previous study [8], it was recommended that the Igbokoda sand (IGKS) sample be established as

reference baseline sand for all further engineering work in Nigeria and the West African sub-region.

This is owing to findings which suggested that the sand sample has properties similar to Ottawa standard sand [8]. Ottawa standard sand, sourced from the Middle Ordovician St. Peter Sandstone near Ottawa, Illinois, had been picked by the American Society for Testing and Materials (ASTM) as the reference sand to employ in testing cement and strength of concrete [9]. To the best of our knowledge, no other literature on the spectroscopic characterization of Igbokoda sand exists. The objective of the present investigation is to study the structural composition, mineral constituents, and crystallinity of Igbokoda Standard Sand through vibrational and magnetic resonance spectroscopic techniques due to its importance in cement, geotechnical/geo-environmental research in Nigeria. This should halt importation of standard silica sand for mortar and concrete testing/geotechnical-geoenvironmental engineering research and the indiscriminate /presumptuous use of any sand from the various silica sand deposits in Nigeria.

The Significance and goal of this research is the baseline spectroscopic and molecular characterization of the Igbokoda Standard sand. This would be useful in understanding future reactions/reactive experiments involving this sand as experimental sand. There is the great need to understand the fundamental molecular processes that give rise to the persistence of anthropogenic chemicals in soils which may ultimately inhibit there removal. A complete understanding of the chemistry of complex soil assemblages is prerequisite to accurately assessing environmental and human health risks of contaminants or in designing environmentally sound, cost-effective chemical and biological remediation strategies. Soil has been described as the most complex biomaterial on Earth and extracting key molecular information such as the binding mechanisms of contaminants and agrochemicals currently challenges the limits of modern science. Furthermore, in order to accurately assess chemical contaminant-soil interactions in the environment, it is vital to study pure mineral standard sand such as the Igbokoda silica sand in its natural state without the presence of any contaminant [10,11].

2. EXPERIMENTAL

The characterization of Igbokoda standard sand was carried out using X-ray diffraction (XRD), Fourier transform infrared (FT-IR) spectroscopy, Raman spectroscopy and nuclear magnetic resonance (NMR) techniques. Igbokoda Sand Sample was collected in Igbokoda town (Latitude: 6° 21' 09" N, Longitude: 4° 48' 14" *E)*, along Igokoda-Ayetoro Road in Ilaje Local Government Area. The samples were collected at the proximity of Ofara River. The region is covered by three to eight meter over burden sand with dense clay underneath.

Phase identification of the chemical composition of the sand was obtained by comparing the diffraction signature of the sample with a database of X-ray Diffraction (XRD) mineral patterns. XRD patterns were recorded on a Philips X'Pert X-ray diffractometer equipped with a graphite-monochromated Cu Kα radiation source (40 kV, 30 mA) at the Department of Geology, University of Johannesburg, South Africa (Fig. 1). A diffractogram was collected in the 2θ range between 3° and 90° with a step size of 0.01°, and a scan speed of 1°/min. The XRD analysis was conducted to detect the mineralogical and the crystal structure of the different phases. The XRD pattern was processed using the Joint Committee on Powder Diffraction Standards (JCPDS) card numbers. Sample preparation for XRD analysis entailed the following steps: the sand samples were crushed and then oven-dried at 100 °C, whereafter approximately 10 g was used to make the pellet.

The sand was analyzed for its vibrational spectra with the aid of Fourier Transform IR (FTIR) spectroscopy. IR spectra were recorded using a Bruker Tensor 27 FTIR spectrometer at the Department of Geology, University of Johannesburg, South Africa (Fig. 2) and the samples were analyzed in KBr pellets. Spectra were traced in the range of 4000–400 cm^{-1}, and the band intensities were expressed in terms of transmittance (%). IR spectral analysis permitted the identification of the main molecular groups present in the samples.

Raman spectra were acquired using a PerkinElmer Raman Station 400 bench-top Raman spectrometer at the Analytical Services Division, MINTEK, Randburg, South Africa. The excitation source was a near-infrared 785 nm laser (100 mW at the sample), with a spot size of 100 lm. A spectral range of 100–3200 cm 1 was used. The detector was a temperature-controlled charged coupled device detector (50 °C) including a 1024 256 pixel sensor. Spectra were acquired using Spectrum software and images were acquired using Spectrum IMAGE software, both supplied by PerkinElmer (Bucks, UK).

Approximately 2 mg of powdered sample was used to acquire the 29Si spectrum under magic-angle spinning (MAS) conditions. Bruker 500 MHz Avance III NMR spectrometer with solid state capabilities at the Department of Chemistry, University of the Witwatersrand, Johannesburg, South Africa was used (Fig. 3). The NMR spectra was created by Advanced Chemistry Development/ Nuclear Magnetic Resonance (ACD/NMR)

Processor Academic Edition. The benefits of using spectroscopic characterization technique for this Nigerian standard sand in civil engineering practice are numerous. In the field of civil engineering, silica sand is used for several purposes like, in construction, in industries for the production of different construction materials. Sand, which is graded according to a specified standard, is used for testing of Portland cement and concrete, in field density determination using sand replacement method, and in water and sewage treatment plants as a filter media [12]. Due to lack of standard silica sand in Nigeria, the country is losing high foreign currency by importing standard silica sand from abroad. The purpose of this research is the baseline molecular and spectroscopic characterization of the Nigerian standard sand (Igbokoda sand) to facilitate its application Civil Engineering and Geo-environmental Engineering soil matrix/porous media experiments and research.

Fig. 1. Philips X'Pert X-ray diffractometer (Department of Geology, University of Johannesburg, South Africa)

Fig. 2. Bruker Tensor 27 FTIR spectrometer (Department of Geology, University of Johannesburg, South Africa)

Fig. 3. 500 Mhz Nuclear Magnetic Resonance (NMR) Spectrometer (Department of Chemistry, University of the Witwatersrand, Johannesburg, South Africa)

3. RESULTS AND DISCUSSION

3.1 Soil particle Size Distribution and Classification

The particle size distribution curve for the Igbokoda sand is presented in Fig. 4. It reveals that the percentage of Igbokoda sand passing sieve No.200 is 1.58% and the corresponding coefficient of uniformity (C_u) and coefficient of curvature (C_c) are 1.8 and 1.1 respectively. The sand is non-plastic and classified in group A3 (fine sand) according to American Association of State Highway and Transportation Officials (AASHTO) soil classification system. The sand is classified as poorly graded sand SP according to the Unified Soil Classification System (USCS) system [13].
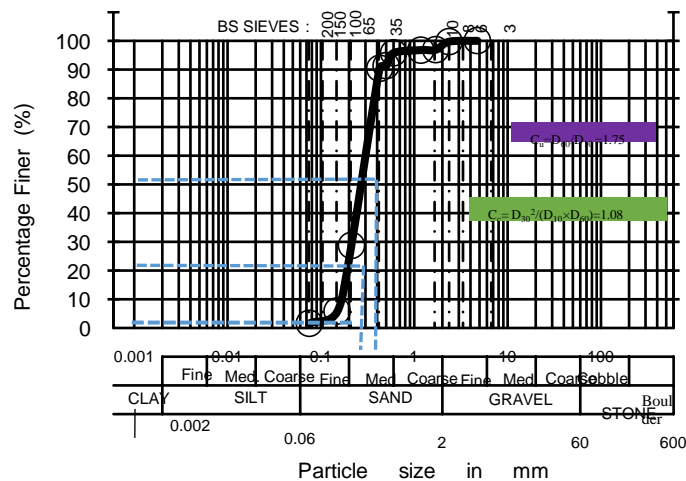

Fig.4 Grain size distribution curve of Igbokoda sand [12].

3.2 X-Ray Diffraction (XRD) Studies

Chemical components of IGKS is presented in Table 1. Petrographic analysis reveals that quartz and kaolinite contents of the sand are 81.4% and 18.6% respectively [8]. The existence of quartz was further investigated by XRD studies. The detected principal reflections at the d-spacings of 4.25745, 3.34359, 2.45705, 2.28172, 2.23697, 2.12754, 1.98036, 1.81827, 1.54178 and 1.37190 Å (Fig. 5) confirm the presence of α-quartz, though low-quartz in the sand sample. The standard unit cell parameters and the crystallographic data of silica sand are the following: Quartz-low (α-SiO2); crystal system hexagonal; space group $P3_121$ (152), $Z = 3$; a = 4.9133(2) Å, c = 5.4053 Å, V = 113.00Å

(Morris et al, 1981). Table 2 provides indexed powder diffraction data on IGKS.

Table 1: Chemical composition of IGKS

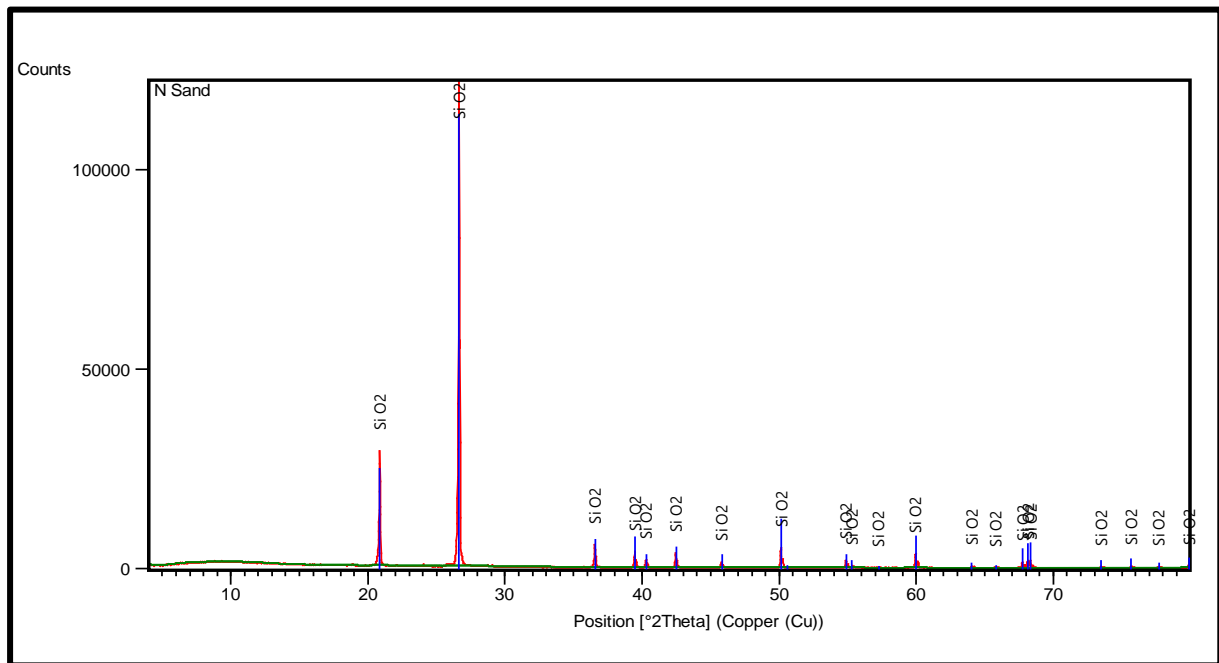| Chemical Component	Percentage (%)
SiO_2	94.24
Fe_2O_3	0.87
Al_2O_3	1.47
TiO_2	0.48
CaO	0.90
K_2O	0.82
Na_2O	0.75
MgO	0.47
Total	**100.00**

Fig 5. Indexed powder XRD pattern of Igbokoda Sand

Table 2. Indexed powder XRD pattern of Igbokoda Sand

Peak No.	D-spacing-sample (Å)	D-spacing-Ref (Å)	Diff D-spacing (Å)	Degrees 2Θ-Sample	Degrees 2Θ-Ref	Diff Degrees 2Θ
1	4.25745	4.2570	0.00045	20.8653	20.85	0.0153
2	3.34359	3.3420	0.00159	26.6390	26.65	-0.011
3	2.45705	2.4570	0.00005	36.5412	36.54	0.0012
4	2.28172	2.2820	-0.00028	39.4608	39.46	0.0008
5	2.23697	2.2370	-0.00003	40.2841	40.29	-0.0059
6	2.12754	2.1270	0.00054	42.5643	42.47	0.0943
7	1.98036	1.9792	0.00116	45.7807	45.81	-0.0293
8	1.81827	1.8179	0.00037	50.1298	50.14	-0.0102
9	1.37190	1.3752	-0.00330	59.9491	59.95	-0.0009
10	1.54178	1.5418	-0.00002	68.3170	68.32	-0.003

2Θ: peak position

D: d-spacing

3.3 FTIR Spectra Analysis

Fig. 6 provides the room temperature FTIR spectrum of IGKS. Table 3 presents the IR absorption band frequencies and their probable vibrational designations. The factor group analysis of quartz forecasts 18 optically active vibrations with four being Raman-active (A_1 symmetry type), five being IR-active (A_2 symmetry) and nine being both Raman and IR-active (E symmetry: doubly degenerate) [14].

From the IR spectra (Fig.5) a broad intense absorption from 1119.61 – 967cm^{-1} and two sharp bands at 728.62cm^{-1} and 693.37cm^{-1} appear prominent. The 1119.61 – 967cm^{-1} fall under the set of bands representing Si-O asymmetrical stretching vibrations. Prominent among these are 1119.61cm^{-1}, 1094.63cm^{-1} and 1045.90cm^{-1}. Other less intense bands are observed at 1071.82 and 1022.27cm^{-1} [15].

In addition, slightly broad bands from about 1180cm^{-1} - 1120cm^{-1} with centroid at 1169.89cm^{-1} and at about 1130cm^{-1} were observed. Bands at this region also represent Si-O asymmetrical stretching vibration [15]. This observation is similar to that reported by [16, 5]) and [17, 5] who reported bands at 1150 and 1140cm^{-1} respectively.

Stretching vibration spotted at around 800cm^{-1} helps to tell apart different forms of silica [5]. Consequently, bands at 822.79cm^{-1} and 786.23cm^{-1} show that the silica is in the form of α-quartz.

A sharp band was observed at 728cm^{-1}. A plausible explanation for this observation is that the sand sample may have been contaminated with another mineral – Albite, which absorbs at a very closely to this – 726cm^{-1}. Albite has the chemical formula $NaAlSi_3O_8$. Geochemical analysis from an earlier study on the sample reveals that traces of Na in addition to Si and Al - this result lends credence to the explanation of the band observed at 728cm^{-1} [8].

A sharp peak was observed at 693cm^{-1}. This absorption can be taken to represent Si-O symmetrical bending vibration [15]. This is similar to bands at 694cm^{-1} reported by [5], 693cm^{-1} reported by [18] and 695cm^{-1} [15]. Further, the presence or absence of an absorption band at around 693.37cm^{-1} helps determine if the observed quartz is crystalline or amorphous [5]. Amorphous silica do not absorb at this frequency, while crystalline silica do. Following this, we infer that the quartz in the sand sample is in the crystalline form.

Observation of a slight shift in position of the characteristic peak at 695cm^{-1} may be due to associated minerals or crystal fault of the sample [15]. An example of such associated mineral in this case is kaolinite. Geochemical analysis from a previous study reveals that this mineral is the next most abundant mineral in the sand sample being studied [8]. Kaolinite absorbs at 693cm^{-1} [18, 19]. Without this interference, we may have observed absorption at 695cm^{-1}.

Less intense bands were also observed at 3649.06cm^{-1}, 3025.10cm^{-1}, a doublet at 2860.16cm^{-1}, 1452cm^{-1} and at 1010.32cm^{-1}. The band at 3649.06cm^{-1} can be taken to represent OH stretching of crystalline hydroxyl [18]. OH in crystalline hydroxyl of theoretical kaolin absorbs at 3645cm^{-1} [18]. 1010.32cm^{-1} absorption band can also be assigned to a yet to be named vibration in kaolin (kaolinite is the major constituent of kaolin) [20,18]. 1452.43cm^{-1} can be regarded as the overtone band of a 726.23cm^{-1} which represents the absorption band of a suspected associated mineral – Albite [19].

In summary, the presence of Si-O asymmetrical stretching vibrations at 1119.61 – 967cm^{-1}, Si-O asymmetrical stretching vibration at 1180 – 1120cm^{-1} and Si-O symmetrical bending vibration 693cm^{-1} reveal the presence of quartz. Bands at around 800cm^{-1} - 822.79cm^{-1} and 786.23cm^{-1} reveal that the silica is in the form of α-quartz. Also, presence of a band at 693.37cm^{-1} provides evidence that the quartz is in crystalline form.

3.4 Raman Spectra Analysis

Raman spectrum can be segmented into three portions (Table 4) [5]. The Raman spectrum of Igbodoka sand sample is presented in Fig. 7. The Raman spectrum of the sample has two peaks at around 203cm^{-1} and 463.07cm^{-1} for A_1 vibrational mode characteristic of quartz [21, 5, 22]. These peaks reflect that the sample studied belongs to the low temperature quartz [5].

Asell and Nicol (1968) [23] undertook a study to understand the pressure dependence of Raman frequencies of α-quartz. They reported that the Raman shift at 464cm^{-1} under atmospheric pressure moved to about 495cm^{-1} under a pressure of 40kbar. In contrast, the Raman shift was observed at 463.07cm^{-1} implying that the impact pressure witnessed by the quartz in this work is lower than 40kbar.

Islam and Datchi (2015) [22] have demonstrated that observed Raman shifts/line and associated intensities depend on the polarization of the incoming and outgoing beams and the orientation of the sample. In their work, the 464cm^{-1} line shifted 464.644cm^{-1} with an intensity of 2792units when the polarization of the incoming and beam outgoing light was set to horizontal and the c-axis of the α-quartz crystal was placed at the horizontal polarization direction. This arrangement id termed 'x(zz)x' in the Raman configuration. Other Raman

configurations namely; x(yy)x, z(yy)z and z(xx)z gave Raman shifts higher than 464cm⁻¹ with the least intensity being 233units. It could be inferred that the 463.07cm⁻¹ shift observed in this work with an intensity of 3319 units belongs to other Raman configurations outside those studied by [22], for example, the y(zz)y.

The occurrence of the Raman shift at 463.07cm⁻¹ establishes that the quartz observed in this work is α-quartz [5]. Unlike in the work of [5], crystallinity of the quartz identified in the sample could be established from the Raman spectrum obtained.
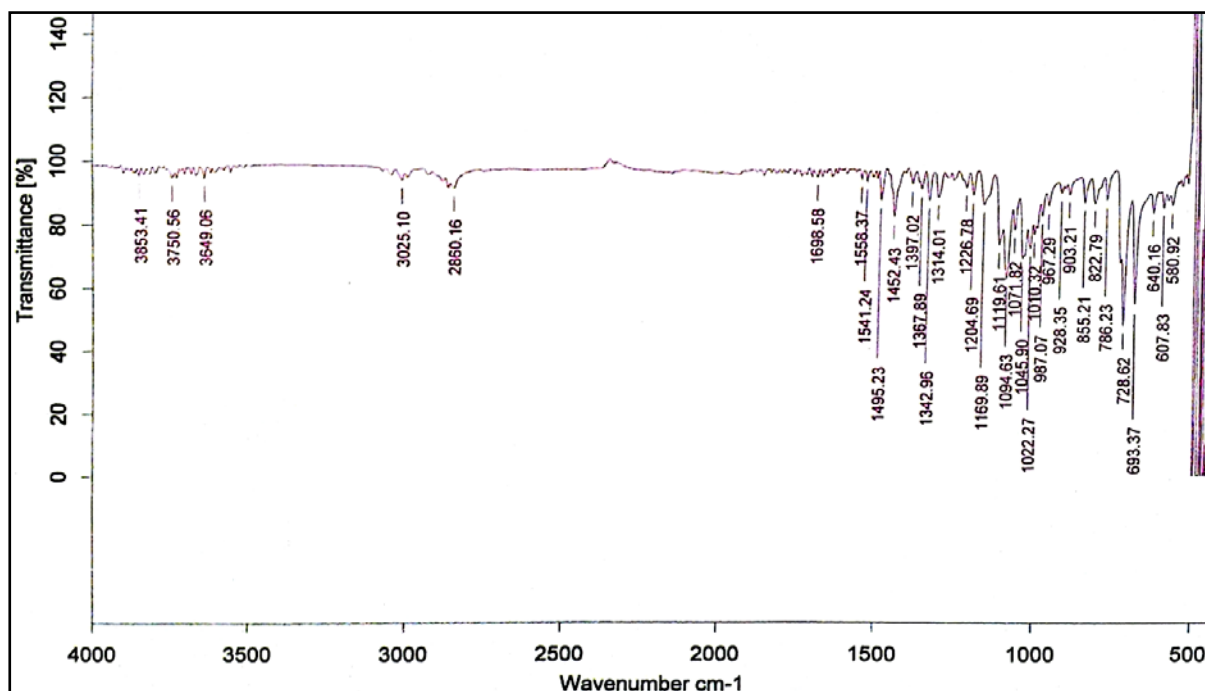
Fig. 6. Room temperature IR spectrum of Igbokoda Sand

Table 3. Important IR bands of silica sand along with their designations

Band (cm⁻¹)	Assignment
1119.61 – 967	Si-O asymmetrical stretching vibrations
1180 – 1120	Si-O asymmetrical stretching vibration
693	Si-O symmetrical bending vibration
3649.06	OH stretching of crystalline hydroxyl

3.5 ²⁹Si NMR Spectrum

High-field shifts are brought about by escalating the degree of condensation of SiO_4 from single to double tetrahedra, to chains and cyclic layered structures, and eventually to three-dimensional frameworks. This is easily noticed with silicates with same cations [24, 5]. The identified signal at –107.95 ppm in IGKS is due to quartz and should correlate to unsubstituted Si(OAl) in SiO_2 (low quartz) (Fig. 8) [24, 25, 5]. This supports the XRD analysis result. Smith et al [25] reported that both the Si-O bond length-chemical shift and bond strength-chemical shift relationships are helpful aids for studying the structures of crystalline silicates. However, the agreement between Si-O bond length and isotropic chemical shifts is bad, particularly for the chain silicates, having chemical shifts between –80ppm and –90 ppm.

Table 4: Important Raman bands of silica sand along with their designations

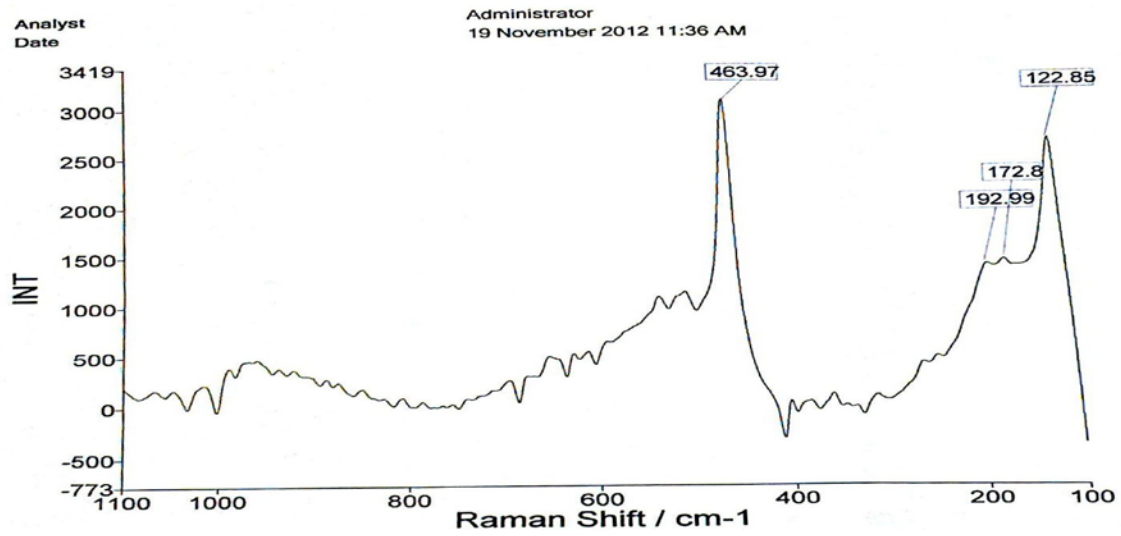| Band (cm⁻¹)	Assignment
>1050 and 700 – 800	Si-O stretching modes
350 – 500	O-Si-O bending modes
<300	Si-O-Si bending and torsional modes

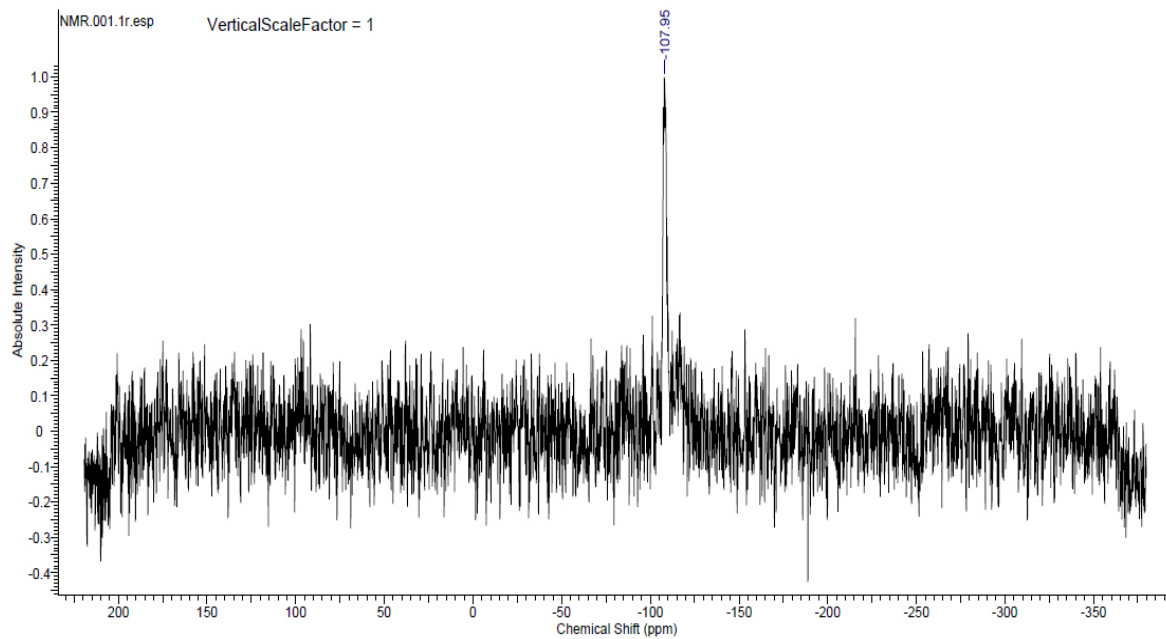Fig. 7. Raman spectrum of Igbokoda Sand at room temperature

Fig. 8. Solid state ^{29}Si spectrum of Igbokoda Sand

4. CONCLUSION

Characterization of IGKS was undertaken using X-ray diffraction, IR, Raman and NMR techniques. The detected principal reflections at the d-spacings of 4.25745, 3.34359, 2.45705, 2.28172, 2.23697, 2.12754, 1.98036, 1.81827, 1.54178 and 1.37190 Å confirm the presence of α-quartz, though low-quartz in the sand sample. Presence of Si-O asymmetrical stretching vibrations at 1119.61 – 967cm^{-1}, Si-O asymmetrical stretching vibration at 1180 – 1120cm^{-1} and Si-O symmetrical bending vibration 693cm^{-1} reveal the presence of quartz and that it is in the crystalline form. Bands at around 800cm^{-1} - 822.79cm^{-1} and 786.23cm^{-1}, reveal that the silica is in the form of α-quartz. The Raman

spectrum of the sample has two peaks at around 203cm^{-1} and 463.07cm^{-1} for A_1 vibrational mode characteristic of quartz. These peaks reflect that the sample studied belongs to the low temperature quartz. The occurrence of the Raman shift at 463.07cm^{-1} supports the observation from XRD studies which establishes that the quartz observed in this work is α-quartz. The ^{29}Si NMR spectrum shows a prominent signal at –107.95 ppm correlating to quartz. Through this research, there has been improvement in understanding of the relationships between physical properties and spectral response of Igbokoda standard sand in the visible and near-infrared regions. Raman spectroscopy has furnished information about bond types and established the chemical composition and polymorph of quartz mineral in the Igbokoda standard sand. Nuclear magnetic resonance (NMR) technique has revealed the porous media morphology and geometry. These relationships would be very useful in fine aggregate/porous media experimental applications of the Nigerian standard sand (Igbokoda sand) in Building Technology, Construction Engineering, Civil Engineering and Geo-Environmental Engineering Research.

5. ACKNOWLEDGEMENT

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