Mineralogical and Physicochemical Characterization of Enugu Iva-Pottery Silica-Rich Deposit for Ceramics Applications

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ABSTRACT: *The exploration of indigenous raw materials that are pure and simple to process for ceramics is a scalable option for locally-made ceramics products. This study involves the characterization of unexplored silica-rich sand deposits at Iva-pottery, Enugu North local government area in Enugu state Nigeria for their ceramic industrial potential. The physicochemical properties of Iva-pottery silica-rich sand were determined. The crystalline phase components and elemental compositions of the oxide were investigated with an X-ray diffractometer and an X-ray fluorescence spectrophotometer respectively. The silica was used in ceramics composition as a filler and glaze formulation as glass former. The results obtained showed mainly a siliceous crystalline phase with a minor amorphous phase and contains 74.55% SiO_2, 13.02% Al_2O_3, and 0.55% Fe. The grain size in the raw form is in the range of 0.18 mm to 0.075 mm (100mesh to + 200 mesh range) without involving ceramic raw materials processing equipment such as a ball mill as well as a high percentage of alumina (13.02) contents as compared to conventional silica with alumina 0.02% to 0.5% range. The high alumina that forms mullite at sintering temperature brings about a low thermal coefficient and hence, suitable chemical stability and resistance to thermal shock. These rare qualities make the Iva-pottery silica-rich sand an emergent material suitable for glaze formulation and filler in ceramic bodies. Thus, it is recommended that the Iva-pottery silica-rich sand deposits in their raw form can be exploited for the formulation of varied glaze temperature ranges and also for the refractory industry. Iva-pottery silica-rich deposits in their raw form, do not meet the requirements of high tech- ceramics a pplications.*

KEYWORDS: *Ceramic application; Enugu; Iva-pottery silica sand; Minerals; X-ray diffractometer.*

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INTRODUCTION

Background of study

Silica is the most abundant compound on the earth's crust, Silica is the name given to a group of minerals composed solely of silicon and oxygen, the two most abundant elements in the earth's crust, it is composed of one atom of silicon and two atoms of oxygen and it is therefore referred as silicon dioxide with the chemical formula (SiO_2), [1]. Silica as a free mineral which occurs as quartz, silica sand, flint pebbles, agate, onyx, carnelian, jasper, diatomite and opal and can be found in a combined state known as silicate minerals such as clay, feldspar, granite, mica, talc and many other types of rocks [2,3].

The chief and cheapest source of silica is silica sand. Silica has wide applications such as in the manufacture of glass, ceramics, refractory materials, foundry casting, paints, abrasives, and other technical uses depending on the degree of purity [4].

According to Malu and Bassey [5], and recently emphasized and cited by *Alemaka et al.* (2021) [6], the use of silica in Nigeria is constrained in structural constructions such as roads and buildings, however, they attributed this to the lack of research to access the huge deposits of silica unexploited in the country, despite their enormous economical mineral contents which can be used in various industries such as cement, refractory, ceramics, semiconductor, and other high tech application industries. A good number of silica deposits have been reported in different parts of this country [7], some of them are found at Ohaozara, Abakiliki both in Ebonyi state, Okene and Lokoja in Kogi state, Ijero in Ekiti state. Silica deposit with suitable qualities for glaze formulations and other fine ceramics bodies remains the most challenging needs of the local potters and researchers, as high quality, usable and viable natural quartz deposits are generally rare in nature [8].

Geologic setting of the iva-pottery sand deposit

The Iva-pottery silica sand is around the Ekulu River, Milliken Hill, and the Onyeama Coal mine sections located at Iva-valley Ngwo, Enugu North local government area in Enugu state and it is located on coordinates 6.461ºN, 7.458ºE (Fig. 1).

In the development of technical or fine ceramics, milling of silica to its finest particle sizes is very important but the cost of milling has limited its use. Most of our local potteries and tertiary institutions are limited to terracotta wares (unglazed red pottery), neither because of the lack of knowledge of how to formulate the glazed body nor because of the availability of the silica deposit, but because of the lack

Fig. 1: Location map of the study area.

of access to silica of high purity and very fine particle sizes and also lack of equipment to have this enormously available silica milled to the required standard qualities [9]. Therefore, the need to explore, exploit, and characterize nature-formed silica deposits of high purity and required fine particle sizes would be a promising option for sufficient and sustainable development of traditional ceramics, porcelain, and all whitewares ceramics. To identify, and obtain some raw materials suitable for ceramics body and glaze development in Iva-pottery Enugu-North in the Enugu state of Nigeria, the physical properties of clay, felspar, and Iva-pottery silica-rich sand mixtures and determination of the maturing temperature suitable for the glazes and the ceramics bodies form the basis of this work.

EXPERIMENTAL SECTION

Materials

The silica sand samples were collected from Iva-pottery after field studies were done to evaluate the location and the size of the deposit. The physical, crystalline phase components (minerals) and elemental compositions were investigated with an X-ray diffractometer and X-Ray Fluorescence Spectrophotometer respectively. The grain size percentages were determined following the standard method of sieve analysis. The color of the sample before and after firing at 1200ºC was observed. The X-Ray Diffraction (XRD) was done under a wide-angle range using XRD GBC EMMA powdered diffractometer with monochromatic CuKα radiation (λ=1.541Å) voltage of 40kV and 40mA. The diffraction angle was scanned from 10^0 to 701^0, 2θ at a step size of 0.05^0 and a rate of 3.57^0/min. The degree of crystallinity was measured using MATCH crystal impact identification from a powdered diffraction software package (version 3.4.2 Build 96) utilizing the FullProof Suit program (2.05) for Rietveld Refinement.

Table 1: The percentage composition of ceramics body and code of different formulation.

Clay%	Iva-pottery silica-rich sand%	Feldspar (%)	Code
50	10	40	A
50	20	30	B
50	30	20	C
50	40	10	D
50	50	0	E

Methods

Sample preparation

The lumps of the silica were crushed and pulverized with wooden mortar and pestle and the powdered silica was sieved with 100 mesh sieve. This would give powder particles of diameter less than or equal to 150 µm. The silica was soaked with water in a big container and was well stirred, left for 24 hours, and excess water was decanted to remove impurities of organic materials that passed the sieve. This process was repeated two times morning and evening for two days to wash properly. The washed silica was air-dried in an open place for two days.

The physical analysis of the compounded bodies with Iva-pottery silica-rich sand

The formulation of the ceramic bodies was done with Iva-pottery silica-rich sand, clay, and feldspar to ascertain the feasibility of using the Iva-pottery silica-rich sand in the production of ceramic whitewares.

The percentage composition of ceramics bodies developed with 10% - 50% Iva-pottery silica-rich sand is shown in Table 1, and the physical properties of the developed bodies were determined and evaluated.

The moistened formulated body was then molded into a rectangular shape using a rectangular metallic mold. This was performed for each sample and the test pieces were air-dried and subsequently fired to 1200ºC in a locally fabricated kiln.

Determination of water absorption, porosity, and density

The fired test pieces obtained after firing were then weighed and the weight was recorded as dry weight, *M1* (g). Thereafter, the test pieces were soaked in water for one hour, then removed, cleaned weighed immediately, and recorded as soaked weight, *M2* (g). The suspended weight of the test pieces was then determined by the use of a lever balance and recorded as *M3* (g). The apparent porosity and bulk density were then calculated [10].

$$\text{Water Absorption}(\%) = \frac{100[\text{M2} - \text{M1}]}{\text{M1}}$$

$$\text{Bulk Density} = M_1 / [M_2 - M_3]$$

$$\text{Apparent Porosity}\ (\%) = \frac{100\,[M_2 - M_1]}{M_2 - M_3}$$

Glaze formulations

The Iva-pottery silica-rich sand and borax were mixed using a line blending method of body formulation [11] as seen in Table 2 and were applied on test tile to find out the fusion and glassification potentials of Iva-pottery silica-rich sand with a flux (borax).

Production of ceramics whitewares using iva-pottery silica-rich sand in body and glaze formulation

The Recipe A (clay 50%, iva-pottery silica-rich sand 10%, and feldspar 40%) in Table 1 above was used in the body production of pin electric porcelain insulators (low voltage) for electricity distribution while the glaze with the formulation; Feldspar 50%, iva-pottery silica-rich 25% Nafuta clay 10 % and borax 15% was made and applied on bisque (900ºC) of pin electric porcelain insulators and fired to 1200ºC the gloss temperature as determined is shown in Fig. 5.

RESULTS AND DISCUSSION

Physical Appearance of the Iva-pottery silica-rich Sand after firing

The pure sample (Iva-pottery silica-rich sand without flux) was fired at 1200ºC, as seen in plate 1. At this temperature, the sample became brighter without any sign of fusing or transforming to other polymorphs of silica. The observation is in complete agreement with *Stevens et al*. [2] and *Wembo et al*. [22] The color difference was a result of the vaporization of organic matters and trace elements such as Fe and Ti in the silica as these and some other transition elements may color silica and are temperature dependent [3].

Table 2: Line test blending of Iva-pottery silica sand and borax.

	Parts by weight									
Test No;	A	B	C	D	E	F	G	H	I	J
Silica	10	9	8	7	6	5	4	3	2	1
Borax	0	1	2	3	4	5	6	7	8	9

Table.3. Result of sieve analysis of Iva-pottery silica-rich sand

ASTM Sieve	Sieve opening	% passing
80	180µm	100
100	150 µm	90
120	125 µm	50
200	75 µm	15

The grain size of the silica sand is in the range of 0.18mm to 0.08mm with 100% of the silica passing through 0.18mm, 90% passing through 0.15 mm, while 50% passed through 0.125mm, and 15% passing through 0.075 mm size range as shown in table 3. This showed clearly that Iva-pottery silica-rich sand is fine in nature and the grain size is within the required standard for the production of glass and whiteware ceramics of 36 to +120 mesh range. The industrial application of silica is dependent on the purity and the grain size distribution of silica, with large size is deleterious in most of the high-tech silica-based structural and white wares ceramics industry, this brings about incomplete homogenization of the body, hence poor densification and glassification of the bodies. It is also observed that very fine grain may also have a crazy effect on glass production. Also in ceramic bodies as fillers, the very fine grain size does not effectively check the shrinkage properties of the body and hence low strength. The workable grain size distribution of silica is reported within 50 to 140 mesh size [10]. It is observed from above that the grain size distribution of Iva pottery silica-rich sand on their raw states falls within the recommended screen mesh size.

Physical Characterizations of formulated batches of ceramics with Iva- pottery silica-rich

The Physical characterization of ceramics bodies formulated with Iva-pottery silica-rich deposits such as apparent porosity, water absorption, and bulk density was used to study the physical behavior of the body formulated with Iva-pottery silica-rich, kaolin clay and feldspar. The results are shown in Figs. 1-5.

The result of the apparent porosity of ceramic bodies developed with Iva-pottery silica-rich sand is shown in above Fig. 1. The apparent porosity indicates the level of densification and vitrification of ceramic bodies. At the temperature of 1200ºC, the apparent porosity values of 11% to 20% for samples A to D, are within the standard required for insulating bricks, earthen, and stoneware productions [11]. However, the apparent porosity of the sample made from a formulation of Iva-Pottery Silica-rich (IPS) sand and clay at an equal proportion of 50% was found to have very high porosity hence, was attributed to the lack of feldspar in the mixture, as feldspar being a fluxing agent contains some fluxing oxide, such as potassium, sodium oxide. This, at high temperature, brings about the liquid phase in the body of ceramics called mullite, this mullite brings about lacing and sealing of the bodies. Also, the high apparent porosity observed at recipe E (Clay 50%: IPS sand 50%) is a result of the high content of the quartz as quartz content increases the porosity increases and the shrinkage decreases consequently the bulk density is reduced. This is in agreement with other authors *Kimambo et al.* [11]. Apparent porosity as observed in the recipe, E is within the 15% to 20% required standard for the production of earthenwares products such as tableware, tiles, kitchen wares, and refractory insulating fire bricks [12, 13].

The high porosity of the body is not accepted in stone white wares production hence, can be improved by

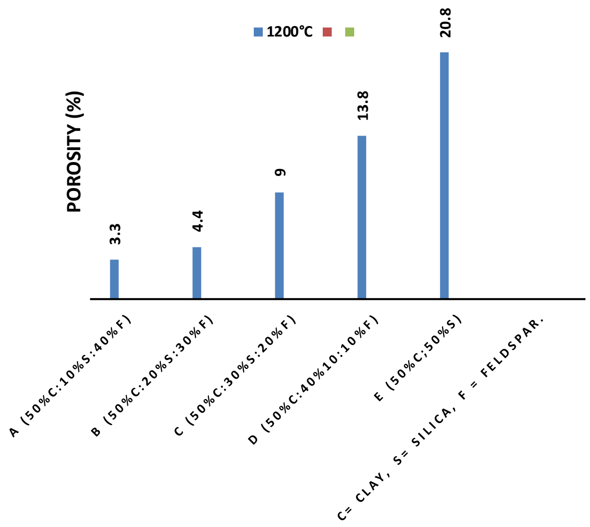


Fig. 2: The apparent porosity of ceramics bodies formulated with Iva-pottery silica-rich sand.

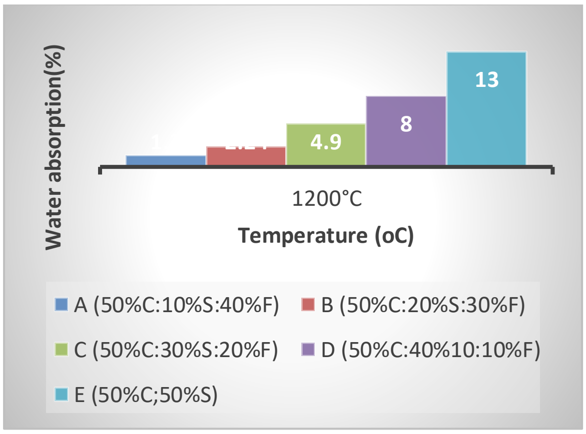


Fig . 3: Effect of Iva-pottery silica-rich(IPS) sand on the water absorption of Ceramics body

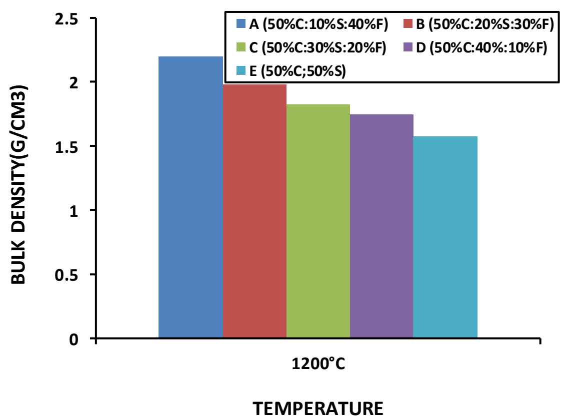


Fig. 4: The effect of (IPS) sand on the bulk density of ceramics body.

reducing to 2% and below by increasing the feldspar contents, the fluxing agents and subjecting to 1200ºC [11].

The bulk density as shown in Fig. 4 ranges from 1.58 to 2.2g/cm^3. The bulk density has its highest value with recipe A at the temperature of 1200^{0}C and decreases down. It is observed, that the bulk density decreases with an increase in the Iva-Pottery Silica-rich (IPS) sand ratio at the same vein, it increases with a feldspar ratio increase and thus it is expected to have a successive improvement in the modulus of ruptures. In other words, bulk density describes how dense and compact the body is, it is however directly related to the strength of a body. It is also observed that apparent porosity and water absorption have a reverse progression with bulk density and modulus of rupture.

From the above Fig. 5, it is observed that there is a general decrease in bulk density (2.2 to 1.01g/cm^3) from composition A to E as also explained in Fig. 4. The effects of sintering temperature on the bulk density show that there is a significant increase in the bulk density of the compositions with an increase in the sintering temperatures up to 1200ºC for recipes A to D after which, there is an observed decrease in bulk density. This is mainly attributed to the formation of bloating and blisters from the gaseous elements which is progressively released as the temperature increases. From the Fig. 5 it is also noted that the bulk density of E with 50% silica and 50% clay increases beyond 1200ºC, this shows that it needs a farther temperature range before its vitrification points. This is due to the absence of feldspar in the composition which acts as a fluxing agent, it brings down the vitrification temperature of the body. The observation is the same with other researchers [11].

Chemical and mineralogical analysis

The result of the chemical analysis of Iva-pottery silica-rich is shown in Table 4. It is observed that silica (SiO_2) formed the major component of the Iva-pottery silica-rich sand followed by alumina (Al_2O_3) while other metal oxides are present in smaller amounts. It is observed that the silica content of IPS from the XRF result is moderate and below the British Standard BS2975 for quartz composition but the alumina (Al_2O_3) is high above the British Standard BS2975 for silica sand composition and relatively low with clay composition, hence the name " Iva-pottery silica-rich", (IPS) in short form.

Table 4: Result of chemical analysis of the Iva-pottery silica-rich Sand.

Component	% oxides composition
SiO_2	74.56
Al_2O_3	13.02
Fe_2O_3	0.55
Na_2O	1.59
MoO	0.031
MgO	0.031
K_2O	0.75
CaO	0.28
TiO_2	5.28
SnO_2	0.032

Table 5: Result of Chemical Analysis of the Feldspar.

Component	% oxides composition
SiO_2	64.65
Al_2O_3	16.1
Fe_2O_3	1.86
Na_2O	2.99
MgO	0.031
K_2O	10.22
CaO	0.098
MnO	0.100
TiO_2	1.080
L. O. I	1.60

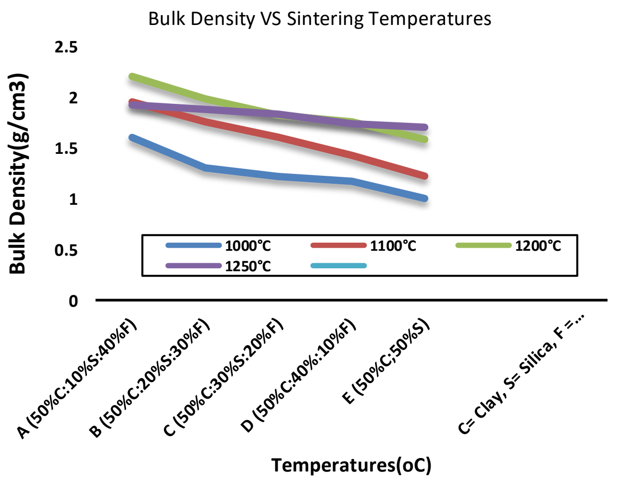


Fig. 5: Bulk density vs sintering temperatures of five ceramics body composition with IPS sand.

The silica content of Iva-pottery silica-rich sand was found to be moderate and this satisfies the requirement for the manufacture of ceramics (>60.5%), refractory bricks (>51.7%) and high melting clays (53-73%) [15]. However, from the studies of *Salah et al.* [16] and also Duvuna and Ayuba, [17] in their intensive investigations of silica sand deposits for general ceramics applications and glass productions respectively from their results it is observed that transition metal oxide (fe_2O_3, TiO_2 Cr_2O_3) > 1% has deleterious effects on the whiteware ceramics and glass body, this is because of their coloration and fluxing ability at relatively high temperatures. Thus, from the result in Table 4, alumina (13.02%) is not within the accepted standard in glass production because of its viscous effects of alumina and while Titanium oxide (5%) is also more than the required accepted limit for the production of hi-tech ceramics. The percentage of iron oxide in the silica sand sample falls within the standard requirement for ceramics 0.5-1.2% and refractory bricks 0.5-2.4% [17], while it does not meet the limit standard for optical and colorless glass production. The Na, K, and Ca indicate the occurrence of feldspar and probably mica and calcite exist in less concentration.

The result of the chemical analysis of feldspar used as a flux in the development of ceramics bodies with the Iva-pottery silica-rich sand is shown in Table 5. It is observed, that it is potassium feldspar with the major constituents of silica (SiO_2).

X-Ray Diffraction (XRD) studies

The XRD of the Iva-pottery silica-rich sand sample is shown in Fig. 6, below.

The graph shows that the samples are crystalline as evidenced by the sharp and distinct peaks. The diffractogram shows that the minerals are identified as quartz and kaolinite. The match analysis report also revealed other insignificant minerals such as muscovite, ilmenite, magnetite, Rutile, and calcite, the result agrees with the XRF result.

SEM-EDX analysis of Iva-pottery silica sand

The Energy dispersive X-ray spectroscopy was employed to identify the elements and the atomic composition of the silica sand.

The EDX spectrum as shown in Fig.7 and Table 7 above indicates the high concentration of Si in the Iva-pottery

Table 7: The atomic composition of Iva – pottery silica-rich sand.

Element	Weight %	Atomic %	Error %
O K	4.75	8.86	16.04
Al K	2.22	2.46	6.99
Si K	79.47	84.53	2.41
Y L	2.08	0.7	19.23
Nb L	7.96	2.56	13.63
Sn L	3.52	0.89	22.17

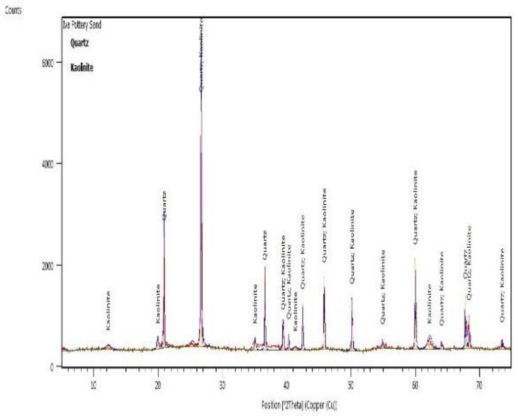


Fig. 6: X-Ray Diffractogram of Iva- pottery silica-rich sand sample.

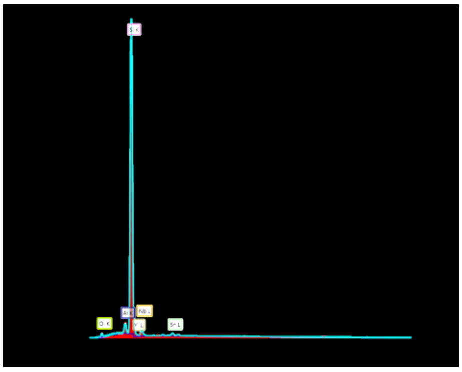


Fig. 7: The EDX spectrum of Iva-pottery silica-rich sand.

silica-rich sand while O, Al, Y Nb, and Sn are in minor concentration, this agrees with the XRF and XRD chemical analysis results. However, EDX did not indicate the presence of Fe and Ti, while chemical analysis indicates their presence as shown in (Table 4).

The Scanning Electron Microscope (SEM) was used for morphological elucidation of the Iva-pottery silica-rich sand. The (Fig. 8) showed the microstructure of the Iva-pottery silica-rich sand to have irregular grain shapes and sizes. The microstructure or the pore size distribution as calculated using Image J software showed a range from 4 to 35 μm. The average grain size of the silica was found to be 10.5 μm. This also agrees with *Haniza* and *Azimah* [18] report in their study of the grain size of silica.

The FT-IR Spectroscopy Analysis

FT-IR spectroscopy was employed to study the mineralogical composition of the Iva-pottery silica-rich sand. The sample showed prominent absorption bands at 1082.01cm^{-1}, 1032.46cm^{-1}, 796.50cm^{-1}, 778.57cm^{-1}, 694.16cm^{-1} and 465.33cm^{-1} as shown in Fig. 9 below. The bands at 778.57cm^{-1} and 694.16 cm^{-1}are due to the Si-O symmetrical stretching vibrations and Si-O symmetrical bending vibrations respectively [18] while the presence of quartz bands at 1082.01cm^{-1}, and 1032.46 cm^{-1}, are indicators of asymmetric stretching vibrations of the silicate tetrahedron [20] and [21].

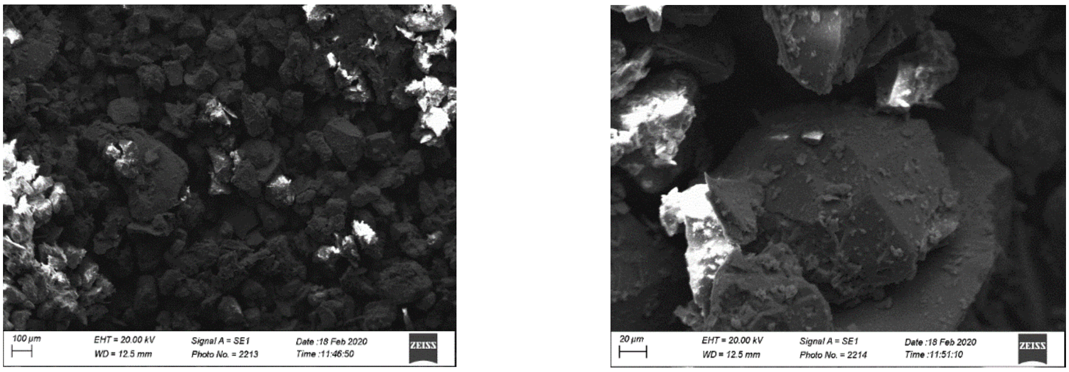
Fig. 9. Also gives two less intense absorption bands at 3691.75 cm^{-1} and 3619.53 cm^{-1} indicating the presence of hydrogen bond (-OH) associated with the sample.

The insignificant absorption bands at 2800cm^{-1}-2900cm^{-1} show that the sample contains organic matter impurities in a small amount. Hence, the Loss on Ignition (LOI) of the sample is expected to be below.

The results of FT-IR Spectroscopy as obtained in the study of the sample conformed well with the reports of *Salah et al.* [16].

Test melting results of line test blending of samples fired at 1200ºC

The samples were fired to 1200ºC after it was formulated as shown in Table 2. **A** did not fuse, there was



(a) (b)

Fig. 8: (a) and (b): SEM microgams of the Iva-pottery silica sand sample at different magnifications.

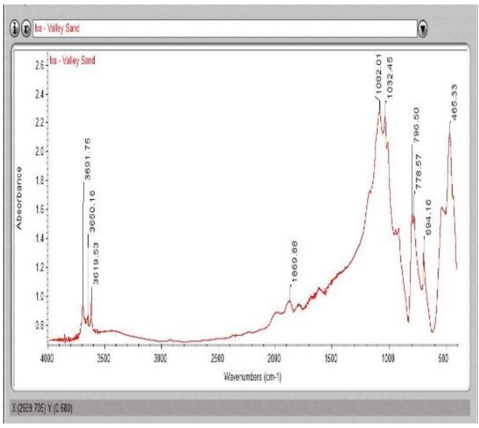


Fig. 9: The FT-IR Spectrum of Iva-pottery silica sand.

no change in phase, it only appeared brighter as shown in plate1, this shows the high purity level of the sample, sample A does not have any alkali oxide or flux added, this could be the reason why there was no transformation of polymorph at 1200ºC. The similar thermal behavior of pure silica with and without flux was reported by *Wembo et al*. (2021) [22] in the Microstructure Study of Phase Transformation of Quartz in Potassium Silicate Glass at 900 ◦C and 1000 ◦C. **B**, **C,** and **D** fused on the test tiles but did not melt at all (plate 2). This could be due to the low amount of flux or alkali oxides,10%, 20% and 30% as shown in Table 2. According to *Wembo et al.* [22] the rate of phase transformation of quartz particles is mainly affected by the number of alkali oxides (K+ ions) in silica samples. The samples **E**, **F,** and **G** with 60%, 50%, and 40% Iva-pottery silica-rich as seen in plate3**,** fused and melted matt while **H, I** and **J** with 30%, 20%, and 10% Iva-pottery silica-rich respectively melted glossy while sample J ran off the surface of the body, this is shown in plate 4. It is important to point out that these samples are likely to run when applied on a ceramics body hence, to check the streaking effects, the right proportion of alumina should be introduced through kaolin to increase the viscosity and correct the glaze.

The application of the Iva-pottery silica-rich sand in the production of low electrical porcelain insulators (whiteware ceramics)

The Iva-pottery silica-rich sand was used in the production of low voltage porcelain insulators to determine the potentials in ceramics whitewares productions and refractories. The Iva-pottery silica-rich was used both in body and glaze formulation at 10% and 25% respectively. The samples shown in plate 1 and plate 2 were fired at 900ºC for bisque and subsequently glazed and gloss fired to 1200 ºC which was determined to be the best densification temperature of the formulation as seen in Fig. 5. It is observed that the bisques do not have cracks on bisque and gloss fired came out without dunt or craze on the porcelains, this is attributed to the uncommon constituent of the Iva-pottery silica-rich as identified with XRF and XRD in Table 4 and Fig. 5 respectively, it is shown that the silica has high alumina as compared with conventional silica. This alumina in the Iva-pottery silica-rich and also in the clay with quartz form mullite at high temperature. According to *Rashed et al*. [23], this mullite has a low thermal expansion coefficient, hence high stability and thermal shock resistance. A porcelain and glaze composition with Iva-pottery Silica-rich sand has

Plate 1 Test result of sample A (silica without flux) fired at 1200°C

Plate2 Test result of samples B, C and D fired at 1200°C

Plate 3 Test results of samples E, F and G fired at 1200°C

Plate 4 Test results of samples H, I and J fired at 1200°C

a high amount of alumina and are expected to be stable at high temperatures, therefore, preventing cracks due to sudden changes in the volume of particles during quartz inversion. Thus, veritable in refractories and porcelain industries [24]. Currently, the natural Silica-rich from Iva-pottery Enugu is being used at the Projects Development Institute (PRODA***)*** ceramics workshop for ceramics body and as glass former in glaze development.

CONCLUSIONS

The physical, chemical, and mineralogical analysis of natural silica-rich from Iva-pottery Enugu Nigeria revealed that the deposit has a fine size range of 0.15 mm to 0.08 mm (100mesh to + 200 mesh range) without involving ceramic raw material processing equipment such as ball mill or any other form of beneficiation just wash, dry and use. The XRD and XRF revealed that Iva-pottery silica-rich has a significant percentage of silica and alumina at 74% and 13% respectively. It is observed that the silica content of (IPS) result is moderate and below the British Standard BS2975 for quartz composition but the alumina (Al_2O_3) is high above the British Standard BS2975 for silica sand composition and relatively low with clay composition [4] hence the name " Iva-pottery silica-rich", (IPS) in short form.

This alumina in the Iva-pottery silica-rich and also in the clay with quartz form mullite at high temperature. This mullite has a low thermal expansion coefficient, hence high stability and thermal shock resistance, therefore, preventing cracks due to sudden changes in the volume of particles during quartz inversion [24]. The optimum sintering temperature was 1200ºC with the highest bulk density of 2.2 (g/cm^3). The sand was found suitable for the production of both low and high-temperature glaze, ceramics, refractory bodies, tiles, and porcelain. Based on the specification for glass and silicon productions, IPS does not meet the requirement considering the low SiO_2 (75%) and high Al_2O_3 (13%) content compared with 99.2% SiO_2 and 0.5% Al_2O_3 required. Thus, the Iva-pottery silica-rich deposits have the qualities required for the production of various whiteware products and glazes in their raw state but do not meet the requirements of the high-tech- application industry in its raw form. We recommend that Iva-pottery silica sand should be subjected to beneficiations and further characterization for possible applications in white glass, optical glass, and silicon productions.

Plate 1 low voltage pin porcelain insulators bisque fired at 900°C

Plate 2 Low voltage pin porcelain insulators fired gloss at 1200° C

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