



## Characterization of Makkoda Silica Sand Deposit in Kano State Northwest Nigeria for Use in Glass Manufacturing

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### ABSTRACT

The suitability of Makkoda silica sand deposits in Kano State Northwest Nigeria for commercial glass production was assessed based on the chemical and physical properties of the silica sand sample collected from the sand deposits. Physical properties such as pH, particle size, grain morphology and the specific gravity examinations were carried out using pH meter, mechanical shaker (sieving method), Scanning electron microscope (SEM) to reveal the shape of the sample and the specific gravity testing machine respectively. The amount of volatile organic matter present in the sample was determined from the results of the Loss on Ignition. Analysis of the percentage chemical compositions of the sand sample was carried out using an X-Ray fluorescence spectrophotometer (XRF) and other metallic elements were determined with the aid of Atomic absorption spectrophotometer (AAS). The results obtained were assessed and compared with the standard requirements for glass making. The pH of the sand sample was found to fall within the neutral range with a value of 7.36 on the scale. Grain size analysis shows that the sand is composed of fine gravel with fine sand having the highest percentage retention fraction of 89.20%. An average specific gravity of 2.58g/cm<sup>3</sup> was obtained, the sample grain morphology was found to be angular. Chemical characterization of the sand sample reveals the sample composed of 94.90% silica (SiO<sub>2</sub>) and 0.16% iron (Fe<sub>2</sub>O<sub>3</sub>). The results of the physical and chemical characterization of the Makkoda silica sand sample reveals that the sand satisfies the minimum requirements for use in glass manufacturing.

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### INTRODUCTION.

Silica sand is one of the abundant solid mineral deposits in Nigeria. According to the 2012 annual report of Raw Materials Research and Development Council (RMRDC), the mineral deposit is found in about 25 states across the country with more than half of the mineral deposit found in the Northern part of the country. Available evidence shows that the silica sand deposits contain a high proportion of quartz (SiO<sub>2</sub>) and can be directly or indirectly used in the manufacture of various industrial products (Edem, *et.al.*, 2014).

Silica sand has been described as an industrial term used for sand or easily disaggregated sandstone that contains a high proportion of silica grains usually more than 95% Silicon Oxide (SiO<sub>2</sub>). According to Platias, Vatisis and Charalampides, (2014) and Bayat, Vapur and Arslan, (2007) silica sand is the most widely used mineral among all the non-metallic commodities for their physical and chemical characteristics and are greatly consumed by the glass industry.

In Nigeria, glass industries are not much and are situated in the Southern part of the country with

only a few available ones in the eastern and northern region, the mineral has not been fully mobilized for extraction and processing, its application has only been limited to building and construction of roads leaving behind the vast high-technological applications (Malu and Bassey, 2003).

Silica sand is valued for its combination of physical and chemical properties, these include high silica content in the form of quartz and low levels of impurities particularly iron oxide ( $\text{Fe}_2\text{O}_3$ ) and chromium oxide ( $\text{Cr}_2\text{O}_3$ ) which are detrimental to the end use of the mineral (British Geological Survey, 2007). The main impurities found in association with the silica sand are iron oxide ( $\text{Fe}_2\text{O}_3$ ), alumina ( $\text{Al}_2\text{O}_3$ ) and Titanium dioxide ( $\text{TiO}_2$ ) others include lime ( $\text{CaO}$ ), Soda ( $\text{Na}_2\text{O}$ ) and potash ( $\text{K}_2\text{O}$ ) (Suratman and Handayani, 2014). Bayat *et.al.* (2007) discuss the grades of silica sand required for specific glass articles. Silica sand suitable for glass manufacturing is however relatively rare because

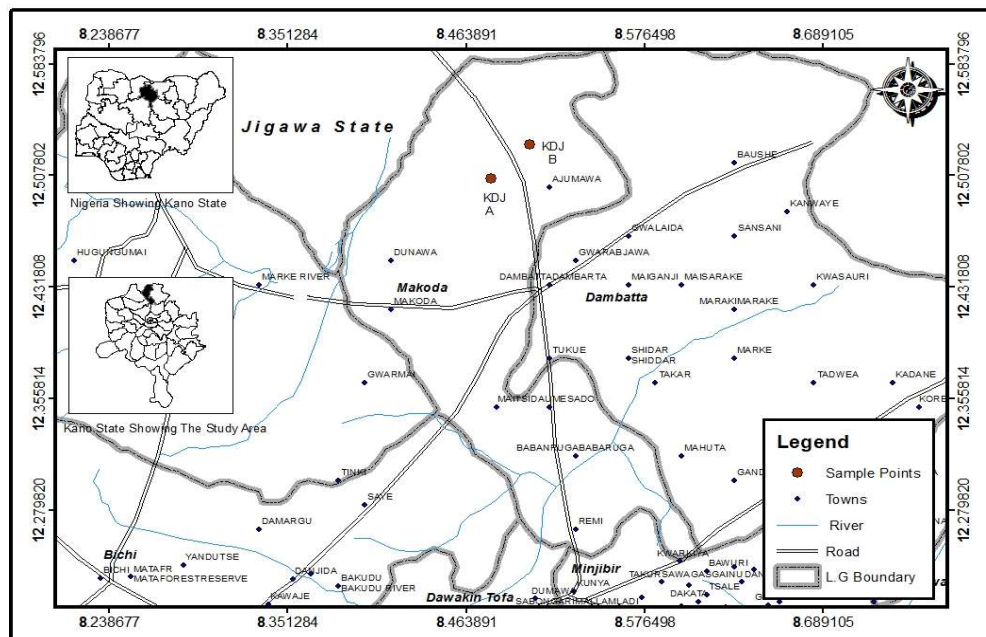
of the need for high purity. The essential requirements for silica sand used in glass manufacture are that it must be even in grain size more than 90% of the grains must lie in the range of 125-500 $\mu\text{m}$  and its chemical composition must meet the standard requirement.

The Makkoda Silica sand deposits lie within the basement complex of northern Nigeria and Chad formation that covers Dambatta and Makkoda Local Government Areas of the state ( $\text{N}12^\circ 32' 20''$  and  $8^\circ 28' 47''$ ). The area falls within a transition zone between the schist belt of Kazaure and the Chad formation to the East. The silica sand in the site is white and fine-grained friable and well sorted (NIMG, 2016). According to the preliminary geological report on the silica sand deposit by NIMG (2016), it is estimated at six million tonnes covering an area of about 135 hectares. Therefore the investigation of the physical and chemical characteristics of Makkoda Silica sand deposit for glass making is of paramount importance.



Plate 1: Makkoda Silica Sand Deposit Site

**Fig. 1:** The Map of Makkoda Silica Sand Deposit showing the study site



## MATERIALS AND METHODS.

The methods involved in the research are sample collection, sample pre-treatment and sample characterization. The materials used in the research work are the sand sample obtained from Makkoda silica sand deposits of Makkoda Local Government, Kano State. Alumina crucibles, Laboratory glassware for laboratory bench work, chemical reagents of analytical grade, instruments and equipment for the characterization and chemical analysis. The silica sand samples obtained was first poured on a flat surface coned and quartered to obtain the representative fraction which was divided into four equal parts by using a straight edge. The two alternate quarters were mixed again for further quartering. After which the representative fraction of the sample obtained was sieved through 200 and 100 sieves, washed with distilled water and dried in an open space for seven days and finally in an oven for seven hours at 110°C (Edem, *et.al.*, 2014). The pre-treated sample was subjected to characterization to determine its physical properties and elemental composition. Atomic Absorption Spectrophotometer (Model) and X-ray

Fluorescence spectrophotometer (S2 RANGER BRUKER) were used to determine the various elements and the per cent composition of oxides present in the sample. Other parameters studied in the sample were the sample pH, Grain size distribution, sample-specific gravity, acid demand value (ADV) and loss on ignition (LOI)

### pH Test

The pH of the sand sample was analyzed according to the AFS 51B-00-5 Standards. The pH was determined by weighing 25g of the silica sand and transferred it into 100ml distilled water. A magnetic stirrer was used to mix the sand and water for 5minute, and the pH was measured using a digital pH meter "Mettler Toledo S20K Model" at the Instrumentation Lab of the department of pure and industrial chemistry, Bayero University, Kano.

### Grain Size Distribution Analysis.

The grain-size analysis was carried out using the standard set of sieve according to BS 1377:PART 2:1990 standard method to determine the percentage ideal fraction of the



silica sand sample The grain-size analysis was carried out to determine the percentage ideal fraction of the silica sand samples using a standard set of sieves. 100g of the dried sample was poured into a sieving machine. The machine was electrically agitated for 30 minutes and the percentage retentions of grain size of each sample on each sieve calculated. (Freestone, 2005; Ushie *et al.*, 2005; Sundararajan *et al.*, 2009).

#### Specific Gravity Analysis

Specific gravities of the silica sand samples were determined as prescribed by ASTM-962-15 Using a graduated cylinder (Pycnometer method) and the result was calculated from the relation given below.

$$\text{Specific Gravity} = \frac{WT_1}{\{WT_2 - (WT_3 - WT_1)\}} \dots\dots\dots (i)$$

Where  $WT_1$  = Weight of the silica sand sample  
 $WT_2$  = Weight of pycnometer filled with water  
 $WT_3$  = Weight of pycnometer, silica sand sample and water.

#### Acid Demand Value Test

The ADV test was carried out according to the method adopted by Sundeen (1978). 50g of the sand sample was placed in a 400ml beaker containing 50ml distilled water. A 50ml solution of 0.1M HCl acid was added into the mixture and stirred for 15min. The solution was decanted and filtered with the sand being washed with distilled water severally, the wash was added to the filtrates which were titrated against 0.1M NaOH solution to a phenolphthalein endpoint the test procedure was repeated five times. The ADV number was calculated using the equation (ii) given below (Foundrymag.com, 2017).

$$\text{ADV} = X - Y \dots\dots\dots (ii)$$

Where X= volume of 0.1M HCl and Y= volume of 0.1M NaOH at the endpoint.

#### Loss On Ignition (LOI)

10g of the sample was transferred into a clean crucible and placed in a muffle furnace and heated to 950°C for 1hour to determine the loss on ignition Oliver, *et.al.*,(2001).

#### CHEMICAL ANALYSIS

Atomic Absorption Spectroscopy and X-ray Fluorescence Spectroscopy were used to study the mineral content of the silica sand samples. Scanning Electron Microscopy (SEM) was applied to further characterize the morphology and orientation of the grain particles in the silica sand Samples. The sample was subjected to acid digestion to determine the concentration of Manganese (Mn), Magnesium (Mg) and Potassium (K) present in the sample. A mixture of Nitric acid and Per-chloric acid ( $\text{HNO}_3/\text{HOCl}_3$ ) were used for the digestion. The metals were released into the solution and analyzed using atomic absorption spectrophotometer (AAS) at the Department of Soil Science Bayero University, Kano. X-Ray fluorescence spectrophotometer (S2 RANGER BRUKER) at the Centre for Dryland Agriculture Bayero University, Kano, was used to determine the per cent composition of Silicon oxide ( $\text{SiO}_2$ ), Iron oxide ( $\text{FeO}_3$ ), Calcium oxide ( $\text{CaO}$ ), Aluminium oxide ( $\text{Al}_2\text{O}_3$ ), Potassium oxide ( $\text{K}_2\text{O}$ ), Titania ( $\text{Ti}_2\text{O}$ ), Sulphide oxide ( $\text{SO}_3$ ), Zirconium oxide ( $\text{ZrO}_2$ ) and Phosphorous pentoxide ( $\text{P}_2\text{O}_5$ ) in the sample according to the method adopted by (Rentalla and Lorring, 1992, Ademoroti, 1996 and Anaekwe and Hassan, 2017).

#### RESULTS AND DISCUSSIONS

Tables I, II and III present the physical properties, result of grain size distribution analysis and the chemical composition of the silica sand sample. The pH of the representative sand sample was analyzed in triplicate according to AFS SIB-00-5 Standards with a pH meter model "Mettler Toledo S20K" at the department of pure and industrial chemistry, Bayero University, Kano. The result shows that the sample is neutral with a mean pH of  $7.48 \pm 0.01$  as shown in table I. The specific gravity of the silica sand samples was determined as described by ASTM-962-15 the test procedure was repeated five times and the mean is shown in Table I. Acid demand value test was carried out five times, for each test three sets of values were obtained and the mean ADV number calculated is shown in Table I. LOI analysis was conducted at the Kaduna, National Geo-science



Laboratory. The result obtained after igniting three sets of an equal weight of the sample five times is shown in Table I.

The grain-size analysis was conducted according to BS 1377: PART 2:1990 Standard. The test was repeated five times and the result obtained is given in Table II. The silica sample was chemically analyzed to determine the elemental and percentage composition of metal oxide. Two types of techniques were applied in the studies; the atomic absorption

spectrophotometer (AAS) which was used to determine the concentration of Magnesium (Mg), Lead (Pb) and Manganese (Mn), the sample was run five times and the result of the analysis is shown in Table III (a). The per cent metal oxide present in the sample was determined using the X-ray Fluorescence spectrophotometer S2 Ranger Model Xflas Bruker, and the result obtained after running the sample five times is presented in table III (b).

## RESULTS

**Table I:** Physical Properties of the silica sand sample

S/No	Parameter	Value
1	Sample pH	7.48 ±0.01
2	Sample Specific Gravity	2.57±0.04
3	Acid Demand Value	27.58±0.02
4	Loss on ignition	0.71±0.01

**Table II. Result of Grain-size Distribution Analysis**

Mesh No.	Mesh Aperture (mm)	Particle Description	Weight retained (g)	% Composition
50	0.300	Medium Sand	1.06	0.87
60	0.212		3.27	
100	0.150	Fine Sand	306.14	89.42
200	0.075		140.96	
	Pan	Silt or Clay	48.1	9.71
<b>Total</b>			<b>499.49g</b>	<b>100%</b>

**Table III (a):** The Result of AAS analysis

Elements	Concentration in mg/kg				
	Readings	I	II	III	IV
Magnesium (Mg)	2.30	6.60	2.56	4.21	5.26
Lead (Pb)	0.02	ND	ND	ND	ND
Manganese (Mn)	0.005	ND	ND	ND	ND

- ND = Not detected



**Table III (b):** The Result of the per cent composition of Oxides in the Samples (XRF Analysis)

Oxides	I	II	III	IV	V	Percent Mean Composition
SiO <sub>2</sub>	94.95	94.92	95.07	94.99	94.62	94.91±0.15
Al <sub>2</sub> O <sub>3</sub>	1.54	1.22	1.23	1.68	1.37	1.41±0.27
P <sub>2</sub> O <sub>5</sub>	0.74	0.77	0.73	0.69	0.73	0.73±0.02
SO <sub>3</sub>	0.55	0.54	0.53	0.55	0.55	0.54±0.01
CaO	0.14	0.14	0.16	0.12	0.12	0.14±0.02
TiO <sub>2</sub>	0.12	0.13	0.12	0.12	0.12	0.12±0.01
Fe <sub>2</sub> O <sub>3</sub>	0.16	0.16	0.17	0.16	0.16	0.16±0.01
K <sub>2</sub> O	0.09	0.09	0.08	0.09	0.09	0.09±0.01
ZrO <sub>2</sub>	0.04	0.02	0.02	0.02	0.02	0.02±0.02

## DISCUSSION

The average mean pH value of the silica sand was found to fall within the neutral values of 7.36. The observed values were in line with the published values in earlier research work which exist in the range of pH 7-9 (Murthy and Rao, 2016). The pH of silica sand gives the water-soluble level of acidity or alkalinity of the sand (Doremus, 1973). Higher or lower pH values signify the presence of acidic or basic oxide in a given silica sample. The pH of close to neutral (7.0) is, therefore, the best for glass making (Paul, 1982).

The specific gravity of silica sand calculates how much of the gravel is required to attain a specific depth in a cylindrical shaped container at a given temperature. A lower specific gravity indicates the presence of light impurities or grain porosity while a higher value indicates the presence of heavier minerals like carbonates and iron oxide. Makkoda Silica sand was found to have an average specific gravity of 2.57±0.04 which is in line with the established standard for glass making. The mean ADV value of 27.73 obtained shows that the silica sand samples have a soluble carbonate which can be reduced by beneficiation and or upgrading process. ADV is a measure of soluble carbonates present in a

given sand sample; the value determines a low or higher concentration of alkali or carbonates reacting materials in the sample. Their presence is an indicator of whether a given silica sand sample can be used for glass making (Edem, *et. al.* 2014). Very pure silica sand with no impurities will have an ADV number of zero. However, if the sand contains impurities that can react with the hydrochloric acid, the ADV number will increase. The more impurities in the sand that react with the hydrochloric acid and neutralize it, the higher will be the ADV number. Some impurities in sand will not react with the dilute hydrochloric acid and may be acidic. In this case, the ADV number can be less than zero ([www.foundrymag.com/pH-ADV](http://www.foundrymag.com/pH-ADV)).

Water, organic matter, carbonate mineral and siliciclastic diatomic content are estimated sequentially by measuring the weight loss in silica sand samples after heating at selected temperatures. It is used as a quality parameter commonly carried out for solid mineral deposits to ascertain the level of loss of volatile matter when a sample is subjected to a temperature of 950°C – 1000°C (Dean, 1974; Velda, 1992; Ademoroti, 1996; Oliver, 2001). The result of the LOI test in this research reveals that the Makkoda silica sand has a low level of



soluble and dissolve organic matters that appear as liquid inclusion in glass materials which is detrimental to the end-user.

Grain size distribution plays an important role in silica sand requirement. Large grains do not mix properly with other grains in the batch while too fine grains create air bubbles in the glass final products (Robert, 2002).

The result of this research shows that the grain size distribution fraction from Makkoda Silica sand was found to have an average of 89.42% fine sand and therefore falls within the recommended size range of 40-100 mesh (Sundeen, 1978 and Robert, 2002).

The result obtained from the sample revealed that the highest percentage mean composition of SiO<sub>2</sub> in the silica samples was 94.91% and significant amounts of Iron oxide (Fe<sub>2</sub>O<sub>3</sub>) 0.16%, Aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) 1.41%, Phosphorous oxide (P<sub>2</sub>O<sub>5</sub>) 0.73%, Sulphide (SO<sub>3</sub>) 0.54%, lime (CaO) 0.14%, Titania (TiO<sub>2</sub>) 0.12%, Potassium oxide (K<sub>2</sub>O) 0.09% and Zirconium oxide (ZrO<sub>2</sub>) 0.02%. The metallic elements identified are Magnesium with a mean concentration of 4.17mg/kg followed by Lead with 0.02mg/kg and Manganese with 0.005mg/kg respectively (Table 4.9). This result is following the work of (Waziri, Audu and Shuaibu, 2013) in the Assessment of the Levels of Some Heavy Metals Content in the Glass Sand Deposit in Kazaure, Nigeria.

The result obtained in this research was found to be very similar to previous studies on the silica minerals in various places as reported by (Shakila, Allandin and Quaisar, 1999) and Sundararajan, *et.al.*, (2009).

It has been therefore proven that the Makkoda silica sand deposit contains 94.91% silica (SiO<sub>2</sub>) and 0.16% iron oxide (Fe<sub>2</sub>O<sub>3</sub>) which necessitated the development of a beneficiation method to upgrade the Silica content and reduce the level of the iron oxide (Fe<sub>2</sub>O<sub>3</sub>) content to achieve the minimum standard requirement of 95-99% and 0.005% respectively as established by the American Ceramic Society, National Industrial Sand Association, National Bureau of Standards, Indian Standard and the Standard Specification for Raw Materials Testing in Foundries and many others.

## CONCLUSION

The physical characterization of the sand deposit revealed that the average pH value of the sand was found to be 7.36. The result of grain size analysis shows that the sand sample composed of fine gravels in which the fine sand have the highest percentage retention fraction of 89.20%, silt 10.00% and the lowest percentage goes to the medium sand with 0.81%. The sand was found to have an average specific gravity of 2.58g/cm<sup>3</sup>. The chemical characterization of the sample indicates that an average of 94.90% of the sample contains silicon oxide (SiO<sub>2</sub>), iron oxide (Fe<sub>2</sub>O<sub>3</sub>) which is detrimental in glass manufacturing was found to have a concentration of only 0.16% in the sample. The physical characterizations of the sand deposit yield a result that is following the established standard for glass sand by ASTM, ISI and BSI. The global recommended standard for grain size distribution agreed with the result of the grain size analysis obtained in this research. The chemical characterization analysis of the silica sand, therefore, proves that Makkoda silica sand is suitable for the manufacture of different varieties of glass articles

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