ISSN: 2582-8118 ***Volume 4, Issue 3; May 2024***

INTERNATIONAL JOURNAL OF RESEARCH AND ANALYSIS IN SCIENCE AND ENGINEERING

Web: https://www.iarj.in/index.php/ijrase/index

1. Evaluation of Sokoto Phosphate Rock for Bioactive Glass Production

Isaac Jagusa
Department of Glass and Silicate Technology,
Faculty of Environmental Design,
ABU, Zaria, Nigeria.

Clement M. Gonah
Department of Glass and Silicate Technology,
Faculty of Environmental Design,
ABU, Zaria, Nigeria.

Adele D. Garkida
Department of Glass and Silicate Technology,
Faculty of Environmental Design,
ABU, Zaria, Nigeria.

ABSTRACT

The quest for implant materials to be use in bone tissue engineering, have expanded at a tremendous rate in the previous year's using materials such as metals, plastics, polymers, ceramics, and composites. However, the challenge to obtained materials with excellent biocompatibility, no toxicity, wear and corrosion resistances, convenient mechanical properties, high porosity without implant failure and the risk of infection complication was a concern. Sedimentary phosphate occurs as thin nodular beds and nodular disseminations in the Dange Formation (Paleocene) of Sokoto basin in north western Nigeria. Analysis indicates P2O5 values of 15.8%.and CaO value of 72.72%. The nodules are suitable for the production of bioactive glass on the basis of major elements and P2O5 values.

KEYWORDS:

Sokoto phosphate, bioactive glass, implant materials

1. Introduction:

Sedimentary phosphates are of great economic importance because they constitute most of the raw materials for the manufacture of phosphate fertilizer and some phosphorus-based chemicals.

In Nigeria phosphate rock from Sokoto state are composed of phosphate oxide (33.89%) and calcium oxide (48.35%) average composition along with biologically beneficial trace elements such as Mg, SiO2, F, Na, Mn, Al, K, and Fe. They have wide application in fertilizer, allied chemicals and others industrial used (Okosun & Alkali, 2013; Christopher et al., 2016).

The amount of phosphate oxide contained in phosphate rock from Sokoto state is estimated to have an average weight percentage of 33.89% which is suitable for the production of phosphoric acid that provide phosphate oxide in bioactive glass batch (RMRDC, 2010).

Calcium phosphate products such as bioactive glass are considered better candidates for periodontal regeneration because they act both as a graft material and barrier membrane to promote guided bone regeneration and can also progressively resorb. These are products manufactured mainly from phosphate rock mineral (Kattimani et al. 2016).

2. Materials and Methods:

2.1 Materials:

Phosphate rock deposits from Dan Kilo, Dange-Shuni local government in Sokoto State, Nigeria was chosen out of the four in Nigeria because it is found in commercial quantities (Obaje et al., 2014).

The samples of phosphates rock (21kg) were randomly collected (3 each) from artisanal miners in seven different pits (2m deep) at a distance of 3m within the sample area located.

Figure 1: Phosphate Rocks

2.2 Sample Pretreatment and Beneficiation:

The raw ore is a mixture of phosphate and clays grey rock. Treatment of ores allows the elimination of clays in the material. 6 kilograms of the rock was introduced in demineralized water. The rock was shacked for 30 minutes, and then filtrate containing clays was separated from the solid residue rich in phosphate. For the same sample, operation was repeated 2 times. The solid residue was air dried for 1 week. Then, the rock was crushed using jaw crusher-Retsen type BBI masch.Nr.70992, 40.HCT 89 west German 15L for particles size reduction and ball mill using ball milling machine ATB Kera.b.v soesterbery Holland. The grounded phosphate sample was passed through BSS 60 mesh sieve before the chemical analysis to obtained particle size of 0.25mm methods used by (Muhammed et al, 2014, Umar, 2016).

2.3 Physical Properties:

Physical properties determined are colour, textural forms, hardness, particle size, water absorption capacity, specific gravity and density. Textural forms and colour were determined by visual observation. To determine the economic grade size, particle size analysis was determined for the grounded phosphate rock using standard U.S sieves ranging from 4.75mm to 0.15µm in accordance to Tex-907-A. 500g of phosphate powder sample was weigh using weigh balance and carefully poured inside the sieve and vibrated for 160seconds.

The weight retained on each sieve was weigh and recorded and the percentage weight passed was calculated. Water absorption capacity was determined using the method by Fayiga and Obigbesan (2017). This involved weighing rock samples collected from the field and drying in an oven at 100 0C for 30 minutes to eliminate moisture. The oven dried rock samples were then weighed and soaked in water for 2 hours. The capacity of water absorbed was calculated using the equation: Water absorption = Wet sample – Oven dry sample/wet sample x 100. The specific gravity of the rocks was determined using Archimedes principle which states that when a body is totally or partially immersed in a fluid, the upthrust on it is equal to the weight of fluid displaced. This involves weighing of empty container as (Specimen A), container with the material as (Specimen B), container with the material mixed with water as (Specimen C) and container with water as (Specimen D) and the specific gravity was calculated. Hardness was read on MoH's scale while the bulk density of the rock was determined by using square mould 105mm by 105mm and 105mm depth. This involved weighing the empty mould, and then pouring the material for compaction using a square rod to have a uniform distribution. The mould with the material was then weighed and the bulk density was calculated.

2.4 Characterization:

Since XRF technique determines the surface elemental content of particles, samples were finely ground to minimize the possible effect of non-uniform distribution of elements of the material. Grounded samples were loaded into the vacuum sampling chamber of the XRF and analyzed. Elemental content of phosphate rock was determined by X-ray fluorescence (XRF), panalytical mini pal4 ED-XRF made in Netherlands instrument.

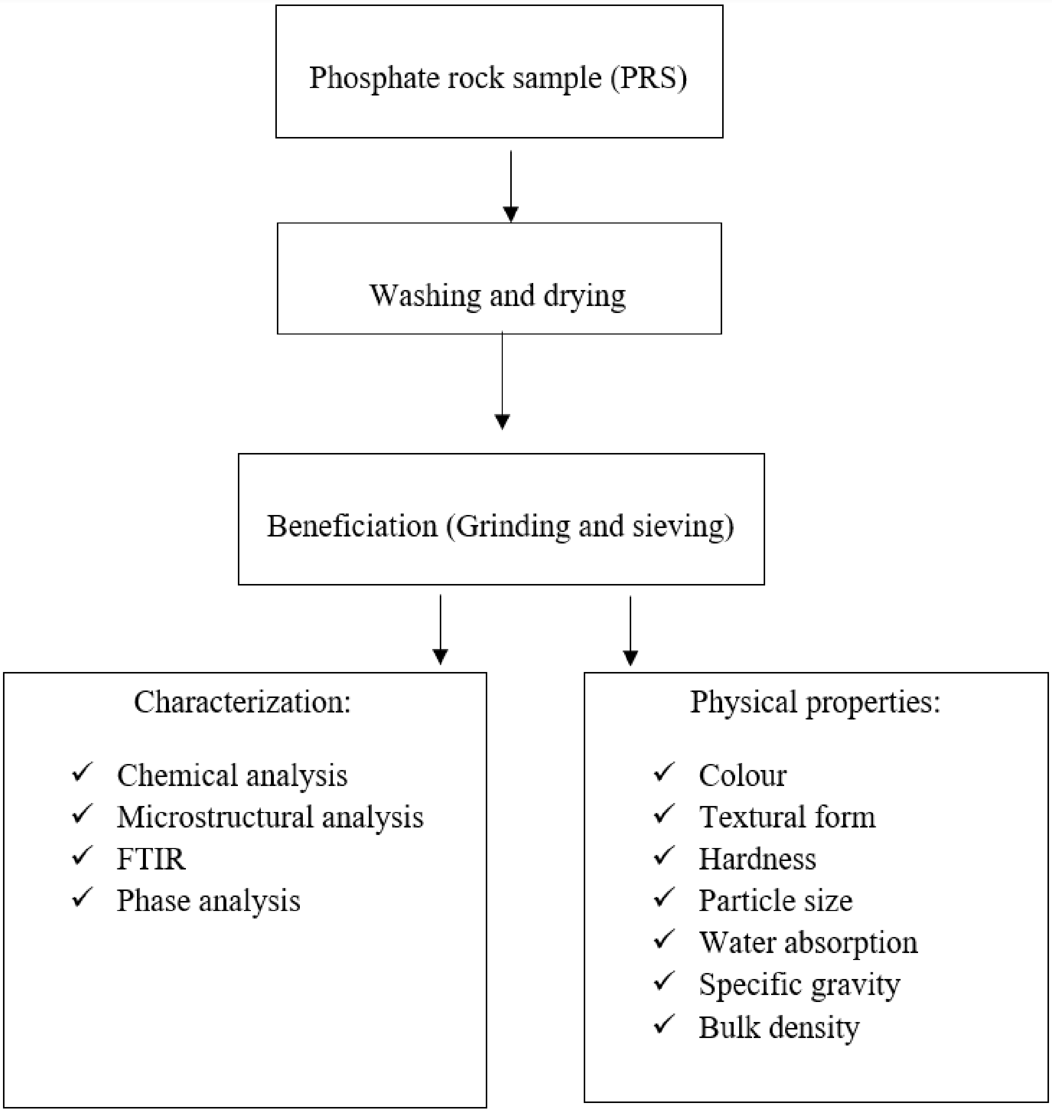

Figure 2: Schematic representation of Preparation and characterization of phosphate rock

The microstructure of the phosphate was characterized in a PRO: X: Phenonm world, model 800-07334, serial number: MVE01570775 scanning electron microscope (SEM). The sample was carbon-coated and observed at an accelerating voltage of 15 kV.

Fourier transform infrared (FTIR, Shimadzu 8400 S), with wave-number range of 4000-400 cm^{-1}, employing KBr pellets operating in a reflectance mode with a 4 cm^{-1} resolution was used to monitor the nature of bonds present in the phosphate powder and the of crystalline phases of phosphate powder sample was investigated.

From this electron density, the mean positions of the atoms in the crystal were examined using Rigaku miniflex300 X-ray diffractometer.

3. Results and Discussion:

3.1 Physical Properties:

3.1.1 Particles Size Distribution:

Sieve analyses are the determination of the gradation or distribution of aggregate particles within a given sample in order to keep to compliance with design and production standards. The particles size distribution of phosphate rock from Dange in Sokoto state in Figure 3 showed 100% passed in 4.75mm sieve size, 98.8% in 2.36mm sieve size, 69.6% in 1.18mm sieve size, 59.4% in 0.6mm sieve size, 30.4% in 0.3mm sieve size, 12% in 0.15mm sieve size respectively.

From the results, the particle size fractions of the material show a normal distribution, indicating that the particle size fractions respond to the theories of comminution. That is, the energy consumed in size reduction is proportional to the area of new surface produced (Umar et al, 2016).

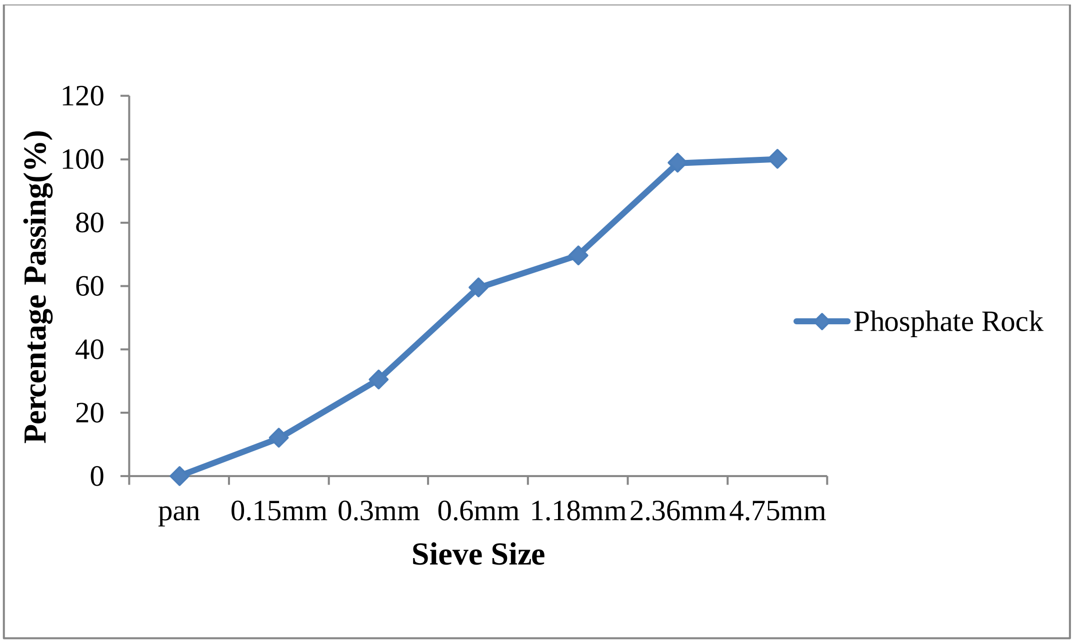

Figure 3: Showing the particle size distribution of phosphate rock from Dange in Sokoto

3.1.2 Textural Form, Colour, and Hardness:

As shown in Table 1a, the phosphate sample exits in nodular textural form with hardness of 5.0 on MoH's scale. This is an indication of high rock strength and more so phosphate rocks are silvery grey and dark grey in colour as observed by (Adesanwo et al., 2010, Fayiga et al, 2017).

Table 1a: Showing the textural, colour and hardness of phosphate rock

Property	Phosphate Rock
Textural form	Nodular
Colour	Silvery grey and dark grey
Hardness	5.0

3.1.3 Specific Gravity, Water Absorption and Density:

As shown in Table 1b, the water absorption capacity of 10.1, on the average was obtained from the phosphate rock. The value obtained is low than 11.1 for phosphate minerals ores sighted in literature, indicating higher rock strength and the higher rock strength may contribute to technical problems in the processing. The bulk density of the material was 2539kgm-3 (Fayiga et al., 2017). the specific gravity of 2.75, on the average, was obtained for the phosphate rock. The value obtained is slightly higher than 2.65 for phosphate minerals ores sighted in literature but agrees with a range of 2.9–3.2 reported for apatite group minerals. The slightly high value could be attributed to the presence of other impurity minerals associated with the phosphate rock in accordance with the earlier work done by (Umar et al, 2016).

Table 1b: Showing the specific gravity, water absorption and density of phosphate rock

Property	Phosphate Rock
Specific Gravity	2.75
Water Absorption	10.1
Bulk Density	2539kgm^{-3}

3.2 Chemical Composition Analysis (ASTM F1185):

X–ray fluorescence analysis data of the phosphate rock nodules from Dange in Sokoto state as present in Table 2. Result showed that the elements are relatively abundance in nature as major and trace constituents. The CaO and P2O5 values are (72.72 and 15.8 wt%) respectively; these are indicative of high apatite content, although P2O5 content suggest that is more of phosphate occurrence in grey shale as it falls within the range of 10-32% P2O5 while the enhanced CaO content suggests that calcite was present as observed by (Onuigbo et al, 2017).

The P2O5, CaO and Fe2O3 values of constitutes are over 90% by weight of the rock material; this can be related to lower quantities of other minerals in the rock, particularly the detrital components. The iron content is (8.96 wt%) in the phosphorite, however this value has been very significantly affected by secondary iron mineral deposition related to the weathering and cannot be ascribed to the sedimentary environment. The trace element values of Cl, TiO2, V2O5, MnO, CoO4, CuO, GeO2, SrO, Y2O3, ZrO, CeO and HgO, are all less than 3.0 wt%.

The relatively chemical composition observed in the Phosphate rock indicates typical of sedimentary phosphorites as these rocks are renowned for their lateral and vertical variability. The low values of alkalis, radionics elements and undesirable heavy metals indicates that direct use of phosphate rock nodules from Dange in sokoto state as source of phosphorus for industrial production can be considered environmentally safe as observed by (Obeja et al, 2014). Therefore, the result of these analysis as shown in Table 2 revealed that with the values of 72.72% CaO, 15.8% P2O5, 8.96 Fe2O3 and 2.477% of other oxides, presence in the phosphate rock can used for bioactive glass and bio-ceramic production. In addition, the presence of 8.96% Fe2O3 content is useful and high enough to act as catalyst in the crystalline growth of hydroxyl apatite, which can enhance the bioactivity of bioactive glass and as nucleating agent for bio-ceramic production. More so, it has been reported that iron doped sample acts as quick drug delivery agent as showed high hemocompatibility compared to pure HAp sample as observed by many authors (Guta et al, 2005., Wu et al, 2008., Lee et al, 2012., Li et al, 2013., Wu et al, 2015).

Table 2. Composition of the phosphate rock

Compound (oxides)	Conc. Unit (Wt %)
P_2O_5	15.8
Cl	0.654
CaO	72.72
TiO_2	0.28
V_2O_5	0.006
MnO	0.919
Fe_2O_3	8.96
Co_3O_4	0.054
CuO	0.018
GeO_2	0.035
SrO	0.299
Y_2O_3	0.15
ZrO_2	0.047
CeO_2	0.01
HgO	0.005

3.3 Microstructural Analysis:

Figure 4 is the SEM micrograph of phosphate powder showing a heterogeneous microstructure as analyzed. Moreover, the observation achieved by SEM shows that the powder sample is agglomerated and clustered. This is an indication for substantially major phases with great difference between percentages as shown by XRD. also the SEM micrograph reveals the presence of three essential colors, a black, a white and the gray colour that indicates the presence of more than two mineralogical phases in the sample. Phosphate rock in Sokoto is said to be cellophane in nature; this affirmation is in good agreement with the result of X-ray diffraction pattern and literature by (Umar et al, 2016).

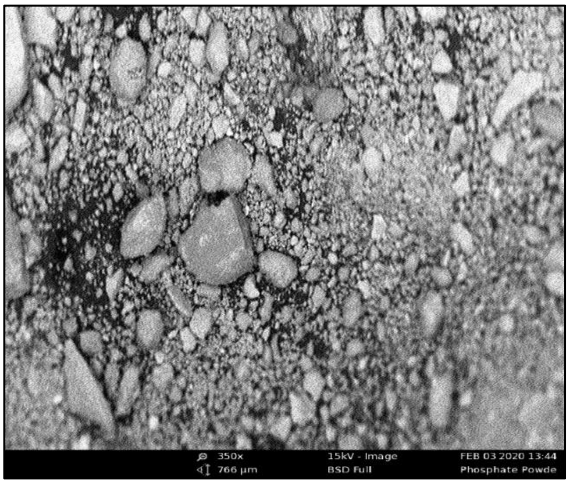

Figure 4: SEM of phosphate powder. Magnified at 350×, 766µm

3.4 Phase Analysis:

The diffraction pattern of phosphate rock from Dange in Sokoto state when subjected to x-ray diffraction analysis revealed a sharp peak zone centered at 29.890 2ϴ signifies the high crystalline nature of the material. The XRD study shows the presence of flourapatite, hydroxylapatite, and calcite. Calcite as the most dominant mineral as confirmed by the XRF result (equivalent of 72.72 % CaO), followed by the phosphate group minerals (equivalent of 15.8 % P2O5) and Iron group mineral (equivalent of 8.96 % Fe2O3). The hydroxylapatite, fluorapatite and carbonate-fluorapatite belong to the apatite family of phosphate minerals which are mostly characterized by high percentage of lime with fluorapatite containing high iron content and this explains the reason for the high iron content in the phosphate rock. The presence of these minerals could be attributed to the marine sedimentation of the organic-rich diatomaceous material such as bones, teeth and other animal shells. The peaks in the diffraction patterns are relatively sharp and well resolved in confirmation of earlier work done by (Ibrahim et al. 2010, Umar et al, 2016).

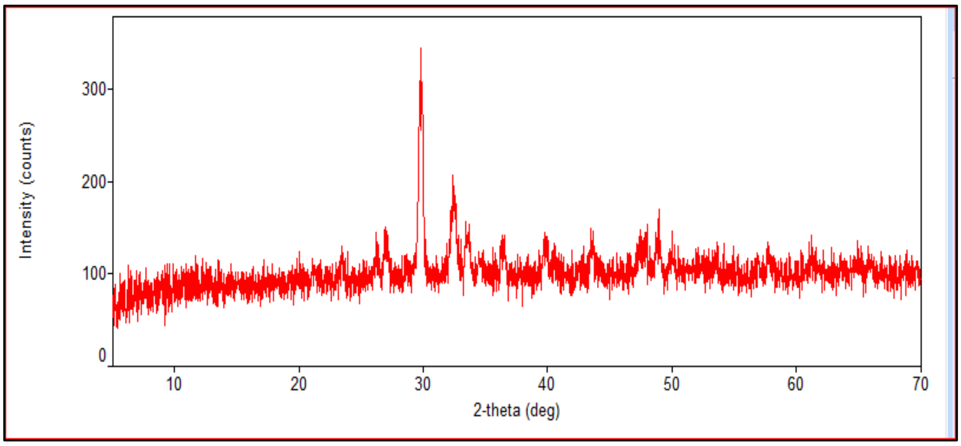

Figure 5: XRD of phosphate powder, Position (2theta copper Cu)

3.5 Fourier Transformed Infrared (FTIR):

FTIR was used to characterize the chemical functional groups of the phosphate rock sample. The FTIR spectra of the phosphate rock sample showed in Figure 6 As observed, the spectrum of the phosphate powder reveals bands at 424.35 cm-1, 470.65 cm-1, 569.02 cm-1, 605.67 cm-1, 673.18 cm-1, 709.83 cm-1, 808.20 cm-1, 873.78 cm-1, 1039.67 cm-1, 1101.39 cm-1, 1429.30 cm-1, 1639.55 cm-1, 1797.72 cm-1, 2281.87 cm-1, 2511.40 cm-1, 2854.74 cm-1, 2924.18 cm-1, 2976.26 cm-1, 3204.47 cm-1, and 3693.81 cm-1.

The IR spectra of the characterized powder showed vibration bands of PO43- group of the apatite structure which are detected: (424.35 cm-1, 470.65 cm-1, 569.02 cm-1, 605.67 cm-1, 673.18 cm-1, 709.83 cm-1, 1039.67 cm-1, 1101.39 cm-1). The spectra show the presence of stretching and liberation bands characteristic of the hydroxyl ion in the apatite structure such as (2281.87 cm-1, 2511.40 cm-1, 2854.74 cm-1, 2924.18 cm-1, 2976.26 cm-1). This gave a convince evidence that the present phases were fluoroapatite and hydroxylapatite as the XRD analysis confirmed. The band at 1429.30 cm-1 is attributed to the antisymmetric vibration of the CO32- group, while the band observed around 808.20 cm-1 and 873.78 cm-1 are assigned to the symmetrical vibration of same group.

These bands are characteristic of calcite as confirmed by phase identification. The band at 1639.55 cm-1 and 1797.72 cm-1 are attributable to the trapped water molecules in the pores by capillarity. A large peak observed around 3204 cm-1 and 3693 cm-1, is attributed to the molecules of water adsorbed on the surface of phosphate ore, OH group in confirmation with earlier work done by (Abdallah et al, 2014).

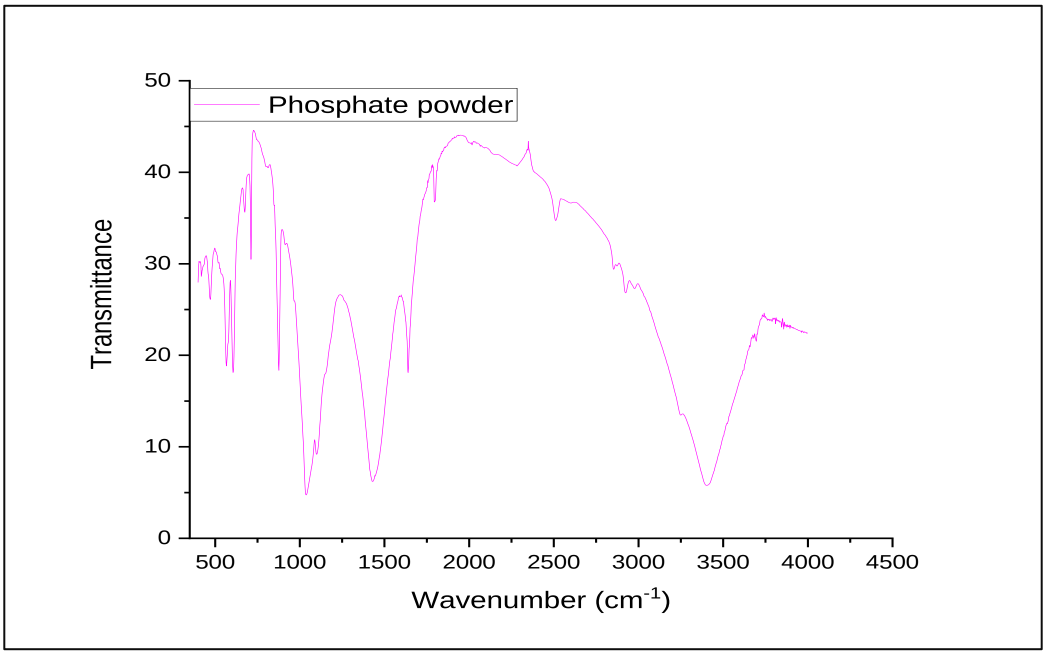

Figure 6: FTIR of phosphate powder

4. Conclusion:

The phosphate rock obtained from Dange in Sokoto State, North-West Nigeria could serve as a useful and viable low-cost material for preparing large scale calcium and phosphate source for the preparation of bioactive glasses with potentials for commercialization.

References:

1. Abdallah A., Abdalazeem A., Mustafa A. (2014). Qualitative and quantitative analysis of phosphate rock from Hazm Al-jalamid area, northern Saudi Arabia. *International Journal of Basic and Applied Sciences, 3(3),* doi:10.14419/ijbas.v3i3.2755, www.sciencepubco.com/index.php/IJBAS
2. Adesanwo O. O., Dunlevey J. N., Adetunji M. T., Adesanwo J. K., Diatta S. & Osiname O. A. (2010). Geochemistry and mineralogy of Ogun phosphate rock, *African Journal of Environmental Science and Technology* Vol. 4(10), pp. 698-708, http://www.academicjournals.org/AJEST, ISSN 1991-637X ©2010 Academic Journals.
3. Christopher O. A., Alaga T. A., Ogbole O. J., (2016). Geological Site Suitability in Sokoto State, Northwestern Nigeria, for Phosphates Prospecting. *Greener Journal of Geology and Earth Sciences,* 4(2): 040-050, ISSN:2354-2268 http://doi.org/10.15580/GJGES.2016.2.062716108
4. Fayiga A. O. and Obigbesan G. O. (2017). Physico-Chemical Characterization of Ogun and Sokoto Phosphate Rocks, *Global Journal of Pure and Applied Sciences* vol. 23, 27-34, Doi: https://dx.doi.org/10.4314/gjpas.v23i1.4
5. Guta A. K., & Gupta M., (2005). Synthesis and surface engineering of iron oxide nanoparticles for biomedical application. *Journal of biomaterial,* 26, 3995. Doi: 10.1016/j.biomaterials.2004.10.012
6. Ibrahim, S. S., El-Midany, A. A & Boulos, T. R. (2010). Economic Preferences of Mechanical Activation Over Mineral Beneficiation for Phosphate Rock Direct Applications. *Physicochem. Probl. Miner. Process.* 44: 63–78.
7. Kattimani V.S., Krishna P. L., Chakravarthi P., Managutti A., & Katwala H., (2016). Egg shell derived hydroxyapatite as bone graft material in maxillofacial skeleton – A randomized blind multi-center clinical trial. Front. *Bioeng Biotechnol*. Conference Abstract: 10th World Biomaterials Congress.
8. Lee N., & Hyeon T. (2012). Designed synthesis of uniformly sized iron oxide nanoparticles for efficient magnetic resonance imaging contrast agents. *Journal of chemistry society Rev.* 41, 2575. Doi: 10.1039/CICS15248C
9. Li L., Jiang W., Ino K., Song H., Lan F., Wu Y., & Gu Z. (2013). Superparamagnetic iron oxide nanoparticles as MRI Constrast agents for non-invasive stem cell labeling and tracking. *theranostic.* 3, 595. Doi: 10.7150/thno.5366
10. Muhammad A. Q., Mahmood A., Saghir A. & Iftikhar U.H., (2014). Synthesis of Dicalcium Phosphate Used as Feed Additive For Animals From Phosphate Rock as Potential Cost-Effective Raw Material *International Journal of Chemistry Science: 12(1), 2014, 111-120 ISSN 0972-768X, www.sadgurupublications.com,*Institute of Chemistry, University of Punjab, LAHORE, PAKISTAN
11. Obaje, S. O., Ojutalayo, A., Ogedengbe, O & Okosun, E. A., (2014)a. Nigeria's Phosphate and Uranium Mineral Occurrences: Implication for Mineral Investment.

Journal of Environmental Earth Science. 4(1): 83-89, ISSN 2224-3216 (Paper) ISSN 2225-0948 (Online)

12. Obaje S.O., Okosun E.A., Amoka I.S. & Kwaha J.Z. (2014)b. Geochemical Characterization and Ore Dressing Potential of Sokoto Phosphate Rock, Northwestern Nigeria *Universal Journal of Geoscience* 2(7): 187-194, http://www.hrpub.org, DOI: 10.13189/ujg.2014.020701
13. Okosun E. A. & Alkali Y. B. (2013). The Geochemistry, Origin And Reserve Evaluation Of Sokoto Phosphate Deposit, North Western, *Nigeria Earth Science Research*; Vol. 2, No. 2; ISSN 1927-0542 E-ISSN 1927-0550
14. Onuigbo E.N., Okoro A.U. & Nwokeabia, C.N. (2017). Clay Minerals as Indicator of Phosphatogenesis: A Case Study of Sokoto Basin, Northwestern Nigeria *Journal of Natural Sciences Research* www.iiste.org, ISSN 2224-3186 (Paper) ISSN 2225-0921 (Online), Vol.7, No.14.
15. RMRDC (2010). National Strategy for Competitiveness in Raw Materials and Products Development in Nigeria, 5-43. https:// www.rmrdc.gov.ng/..../raw materials-update/60-raw-materials-research and development -council.html.
16. Umar A.H., Yaro S.A., Abdulwahab M., & Dodo M.R. (2016). Characterization of Sokoto Phosphate Rock for Beneficiation, *Journal of Raw Materials Research*, Pg 74-93, jormar 10 (2), ISSN: 1597-3204, https://www.researchgate.net/publication/316089778
17. Wu W., He Q. G. & Jiang C. Z. (2008). Magnetic iron oxide nanoparticles: Synthesis and surface functionalization strategies nanoscale. *Resc. Lett.* 3397. Doi: 10.1007/S1167-008-9174-9
18. Wu W., Wu Z., Yu T., Jiang C., & Kim W. (2015). Recent progress on magnetic Iron oxide nanoparticles and biomedical application. *Science and technology of advanced materials* 16, 2. https://doi.org/10.1088/1468-6996/16/2/023501