

Experimental Characterization of Physicochemical and Geological Properties of Granite from Olowu, Ibadan, Oyo State, Nigeria

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Abstract -Understanding of the nature and chemistry of valuable minerals in our environment is an asset for maximum exploitation for human need. The physicochemical and geological properties of natural rock samples from Egbeda, Oyo State, Nigeria were studied to categorize the rock samples and to establish their potential applications. The rock samples were characterized using various techniques including XRD, XRF, FTIR, SEM/EDS, TAS plot, BET and water absorption properties. Physicochemical and geological properties confirmed the sample to be granite with low water absorption capacity. The granite is a potential raw material that can be used for exterior walls, interior walls, floor tiles and kitchen walls.

Keywords: Characterization; Construction; Granite; Water absorption

1. Introduction

Granite is a natural stone which has found wide applications mainly in the construction industry as in the construction of bridges, roads, interior and exterior of household walls, floor tiles, etc. (Sousa et al., 2005; Chaki et al., 2012; Barros et al., 2014). Knowledge of the geological and physicochemical properties of granite rock is important to optimize its exploitation, and identify appropriate application, in terms of damage prediction and modifications during application (Chaki et al., 2012). An understanding of parameters such as water adsorption capacity, pore size, chemical, thermal and mechanical properties will be useful information for categorising the application of granite in the construction industry. For instance, road and bridge construction will require high stability (thermal, chemical and mechanical)

while granite with high water adsorption capacity and low concentration of toxic elements/oxides can be used for interior walls in household.

The study area lies within the Western region of Nigeria within Oyo State, Ibadan. Nigeria can broadly be subdivided into three major geological components and these are: the Basement, Granites and Sedimentary Basins (Obaje., 2009). The basement (Precambrian in age) is further subdivided into five regions on the basis of the occurrence of sedimentary basins. These are the Western Nigerian Basement, North Central Nigerian Basement, Adamawa Highland, Eastern Nigerian Basement and the Oban Massif. As discussed by Obiora et al (2009) and references cited within, the basement consists of “migmatitic gneisses, including banded varieties; the schist belts constituted by mica-schists, tremolite-schists, graphite-schists, with occasional marbles and dolomites, calc-silicate rocks, meta-conglomerates and banded iron formation (BIF) and Precambrian granites including porphyritic/porphyroblastic muscovite granites, biotite granites, hornblende-biotite granites, non-porphyritic/non-porphyroblastic granites, aplites, granodiorites, diorites, quartz diorites, syenites, quartz-enstatite granites and enstatite granites (charnockites).” The Migmatite-Gneiss Complex, as described by Rahaman (1988) and Dada (2006), is considered to be the basement *sensu stricto* and makes up the largest component of the Nigerian basement complex. It forms part of the Pan-African mobile belt, the basement complex, which lies to the east of the West African Craton and is sandwiched between Benin to the west and Cameroon in the east (Oyedokun and Igonor., 2013) and includes the Beninian Gneisses of the internal zone of the Pan-African mobile Belt as well as a vast expanse of reactivated high-grade gneisses believed to be Archaean in age and a supracrustal succession thought to be Paleoproterozoic (Schluter., 2006).

Two generations of granites can be identified and these are prominent within the basement rocks. The older granites as termed by Falconer (1911) range widely in composition and age from 750 Ma to 450 Ma (Obaje., 2009). The younger granites (Jurassic in age) are prominently distributed in the North Central Nigerian Basement and occur as ring complexes which form part of a wider province of alkaline anorogenic magmatism (Obaje., 2009). Volcanic sequences such as basaltic lava plateaus, trachyte plugs and domes, large central volcanoes as well as small basalt cinder cones with thin flows are distributed among the more southerly manifestations of Cenozoic volcanism in West Africa (Wright., 1987; Obaje., 2009). Broadly speaking, the sedimentary basins can be stratigraphically divided into formations, the older Cretaceous sedimentary basins of which the Benue Trough, Bida-, and Sokoto Basins are the most prominent. The youngest sequence is the Tertiary Sedimentary Basins of which the Chad Basin is the most prominent as well as the sedimentary sequences of the Niger Delta. The geology of the area of interest in this present study (i.e. Oyo State) falls within the Western Nigerian Basement of which the basement rocks cover almost 100% of the total land surface in this state (Oyedokun and Igonor., 2013); as such the rock sequences will not be mentioned again. A simplified geological map of Nigeria showing the distributions of the three major geological components as well as the location of the present study modified after Obaje (2009) and Schluter (2006) is presented in Fig. 1.

This paper presents the results of the characterisation of granite samples obtained from a deposit site in Olowu in Egbeda Local Govt. Area, Ibadan, Oyo State, Nigeria to evaluate its potential as a raw material for building and categorise its application in the construction industry. The study focused on the following properties of the granite: geological (type of granite), chemical (oxide composition, water absorption capacity, surface area, pore volume and size, functional groups, chemical symmetry, pH of rock sample after soaking rock in water, and density), physical (colour), and microstructure analysis (surface morphology, grain and pore sizes).

2. Experimental

2. 1. Materials

The Granite samples were collected from Olowu, Egbeda Local Government Area, Ibadan, Oyo State at coordinates $7^{\circ}22'01.1''N$ $4^{\circ}01'22.9''E$.

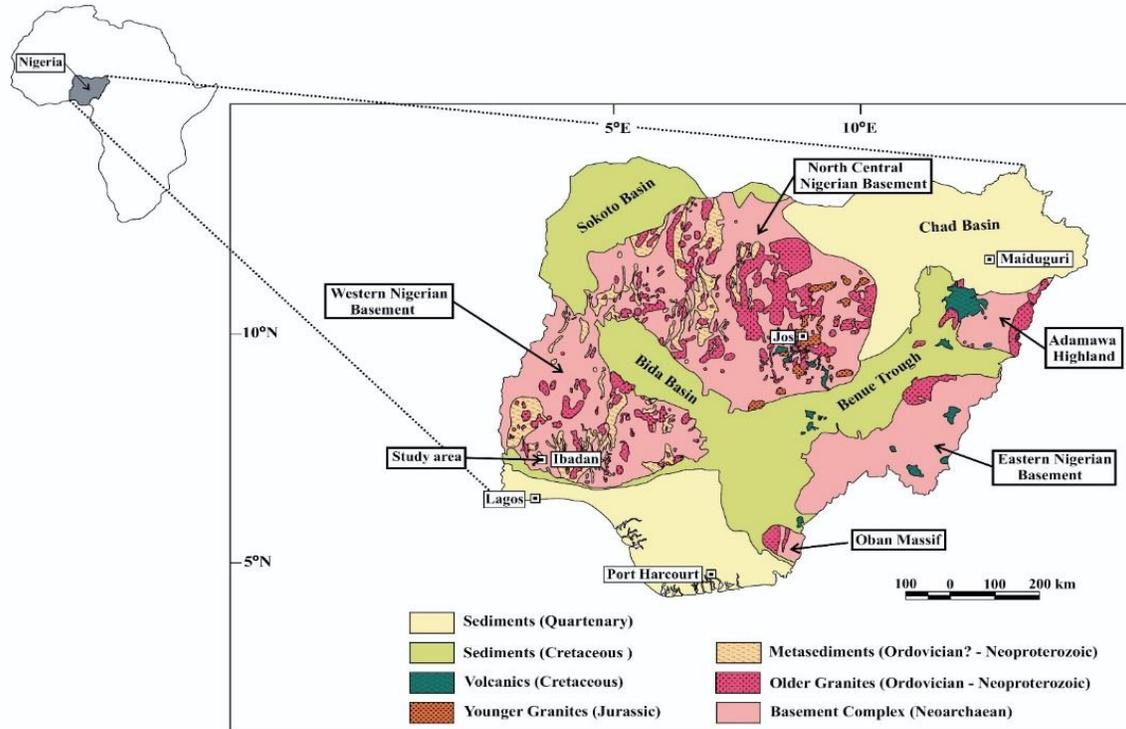


Fig. 1. Simplified geological map of Nigeria showing the distributions of the three major geological components as well as the location of the present study (Modified after Obaje 2009 and Schluter 2006).

2. 2. Characterization

2. 2. 1. Physicochemical Properties

The chemical composition of the rock samples was analysed using a Rigaku, ZSX Primus II X-ray Fluorescence (XRF) spectrometer. Phase identification of the chemical composition of the rock samples 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 Rigaku Ultima IV X-ray diffractometer equipped with a graphite-monochromated Cu K α radiation source (40 kV, 30 mA). A diffractogram was collected in the 2 θ 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 JCPDS card numbers. Sample preparation for XRD analysis entailed the following steps: the granite samples were crushed and then oven-dried at 100 °C, whereafter approximately 10 g was used to make the pellet. The microstructure and the chemical analysis of the rock sample was analysed using a scanning electron microscope (SEM) model TESCAN equipped with Oxford instrument X-Max (EDS). A thin section of the rock sample was prepared along with crushed samples of the rock; the samples were then mounted on a stud and carbon coated and irradiated with a beam of electrons at 20 kV. Secondary electron (SE) and backscattered electron (BSE) micrographs were used for optimum imaging of samples. A gas pycnometer (Micromeritics AccuPyc 1340 Pycnometer) was used to measure the density of the rock. Water absorption analysis was done using a total immersion method. This method comprises the measurement of the water absorption rate and maximum water absorption capacity of a material. The total water absorbed can be related to the total open porosity, while the kinetics of the process depends primarily on the distribution of the pore sizes (Ernesto., 1999). Granite samples were cut into small cubes of approximately 70 g each. Samples were rinsed with distilled water in order to eliminate powdered material from their surfaces, and then oven-dried at 60 °C for 24 h. The samples were then placed in a desiccator until weights of samples were stable. Known weights of the cube-shaped rock samples were placed in a flask container filled with water until the samples were totally

immersed. After 24 h and 48 h samples were measured (weight) and water absorption percentage was calculated using Equation (1) below (Teutonico., 1998):

$$\frac{\Delta M}{M_0} \% = \frac{M_m - M_0}{M_0} \times 100 \quad (1)$$

Where:

$\frac{\Delta M}{M_0} \% =$ percentage water absorption capacity

$M_m =$ the weight of the wet sample at time t_m (g)

$M_0 =$ the weight of the dry samples (g)

Molecular bonds in the samples were studied with Fourier transform infrared (FT-IR) spectrometry using PerkinElmer's FT-IR Spectroscopy Software. Pore size, pore volume and surface area were determined using a Micromeritics ASAP-2020 surface area and porosity analyser by application of the Brunauer-Emmett-Teller (BET) technique.

2. 2. 2. Geological Property

The geological properties of the rock samples were determined in terms of their chemical composition; the chemical classification of the rock sample was done by means of the total alkali vs. silica (TAS) plot for plutonic rocks (after Rollinson., 1993). The alkali and silica content was determined using XRF for five samples.

3. Results and Discussion

Paper The XRF results shown in Table. 1 indicate that the same oxides were present in the five rock samples with slight variations in their composition. SiO₂ was the predominant oxide with average weight percent (wt. %) of 70.94 ± 0.32, followed by Al₂O₃: 19.16 ± 0.50; K₂O: 4.04 ± 0.20; Na₂O: 3.08 ± 0.56; CaO: 1.33 ± 0.11; Fe₂O₃: 0.94 ± 0.06; TiO₂: 0.08 ± 0.02; MnO: 0.05 ± 0.01; SO₃: 0.05 ± 0.01; P₂O₅: 0.02 ± 0.00.

The XRF shows that no toxic oxide was present in the rock samples; this can be ascribed to the fact that there were no industrial activities in the environment. These oxide composition values were used to plot the total alkali (Na₂O+ K₂O) vs. silica (SiO₂) (TAS) plot of the granite (plutonic rock) samples (Rollinson., 1993). Fig. 2. shows TAS plots; the oxide composition of the five samples fell into the categories of granite. A similar plot of the average weight percent of silica and total alkali is also in agreement with this result.

The FT-IR spectrum of the rock sample (Fig. 3) shows Si-O stretching at 1 100 cm⁻¹, 819 cm⁻¹, 798 cm⁻¹, 779 cm⁻¹, and 695 cm⁻¹ which is in agreement with the findings of Vaculikova and Plevova (2005) and Swann et al. (2011) for quartz. The predominant functional group in the rock sample was Si-O which agrees with the XRF average composition of SiO₂ as 70.94 wt. %.

Fig.4 presents the XRD pattern of the rock sample processed with JCPDS; analysis revealed the presence of albite (Na(AlSi₃O₈)), quartz (SiO₂), and mica, respectively. It was found that there was a good correlation between the XRD pattern and the XRF composition analyses. The mineralogical and chemical analyses were in good agreement and showed that the sample is a granite rock (Vijayalakshmi et al., 2013). A triclinic, monoclinic and hexagonal crystal structure was found for albite, mica and quartz, respectively.

In Fig.5, the results of the petrographic study of the rock sample shows a medium texture and the sample is composed mainly of three primary minerals which are quartz, these are minor occurrences of secondary minerals which have a strong association with albite. Petrogenetic relationships of these minerals indicate that albite crystallized first followed by quartz and phlogopite being the last mineral to crystallize. Inclusions within quartz (Fig. 5 A) indicate that albite began to form before

quartz. Quartz inclusions in phlogopite (Fig. 5 B) indicate that quartz crystallized before phlogopite. Texturally quartz and phlogopite are generally interstitial to albite (Fig.5C).

Table. 1. Chemical Composition of Rock Samples

Oxides	Sample 1 (%)	Sample 2 (%)	Sample 3 (%)	Sample 4 (%)	Sample 5 (%)
Na ₂ O	3.38	3.32	3.33	3.30	2.08
MgO	0.19	0.20	0.18	0.21	0.11
Al ₂ O ₃	18.80	18.93	19.16	18.89	20.03
SiO ₂	71.25	71.04	70.92	71.08	70.42
P ₂ O ₅	0.02	0.01	0.02	0.02	0.02
SO ₃	0.04	0.05	0.04	0.06	0.04
K ₂ O	3.93	3.96	3.94	3.96	4.40
CaO	1.32	1.27	1.25	1.30	1.52
TiO ₂	0.07	0.08	0.06	0.09	0.11
MnO	0.04	0.04	0.06	0.06	0.05
Fe ₂ O ₃	0.91	0.94	0.89	0.92	1.04
Rb ₂ O	0.01	0.01	0.01	0.01	0.01
SrO	0.02	0.02	0.02	0.02	0.02

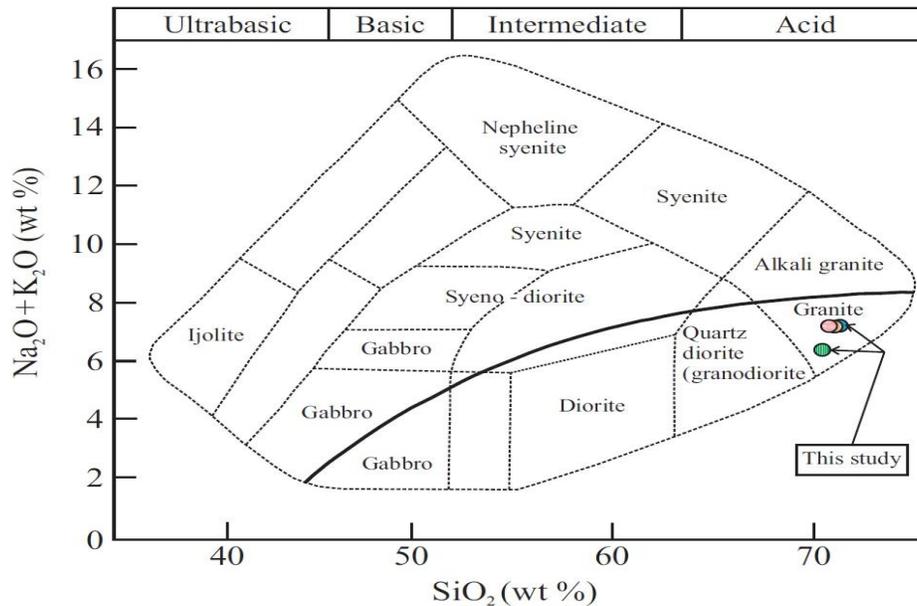


Fig. 2. Total Alkali vs. Silica (TAS) plot for plutonic rocks showing the field in which the samples for the current samples plot. The curved solid line subdivides the alkali from sub-alkali rocks. (After Rollinson, 1993).

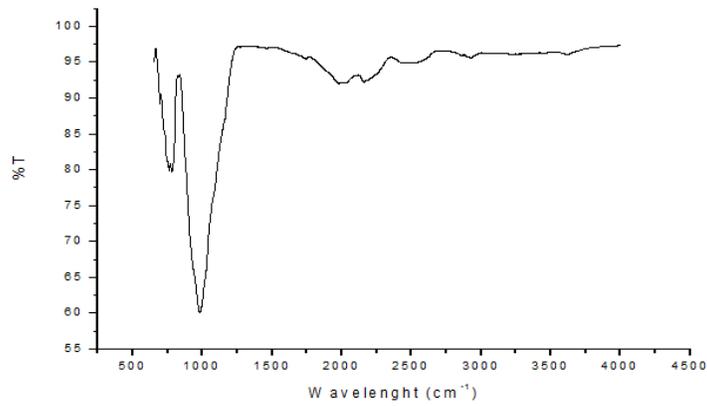


Fig. 3. FTIR spectrum of rock sample

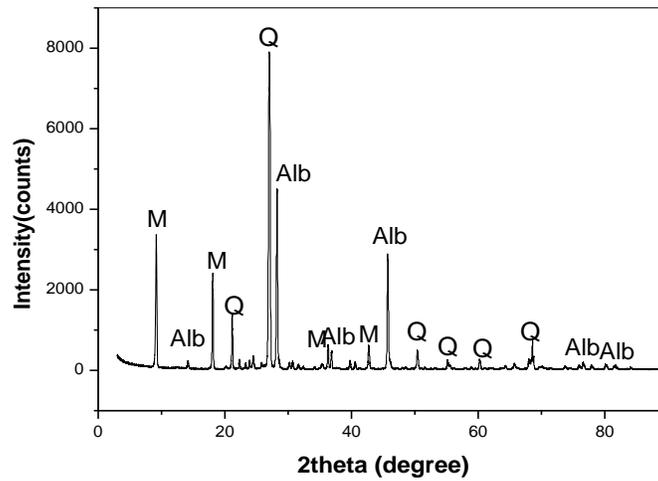


Fig. 4. XRD diffractogram of the granite rock showing the presence of Quartz (Q), Albite (Alb), and Mica (M)

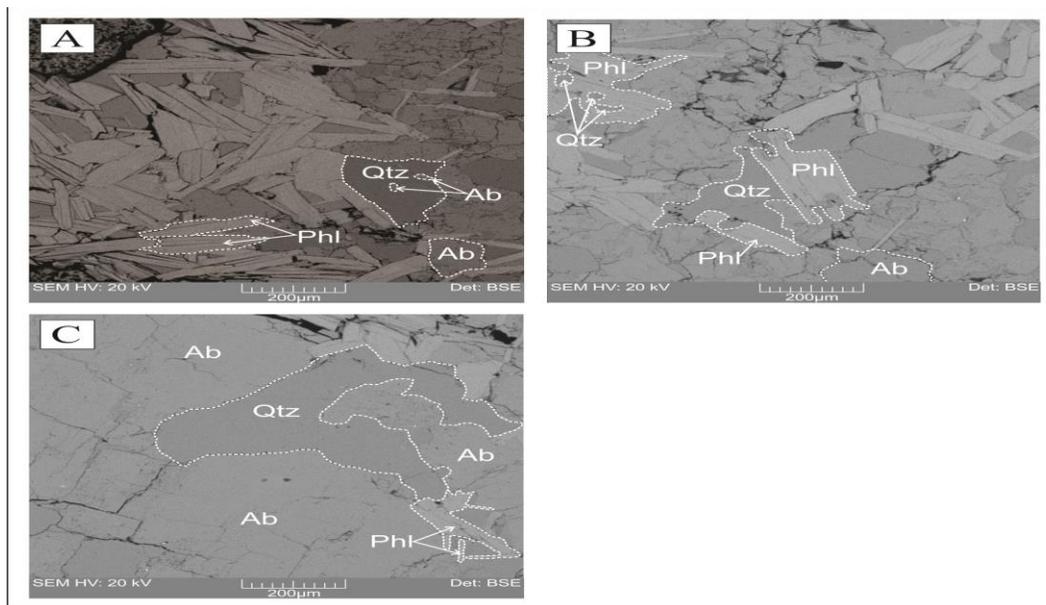


Fig. 5. Backscattered electron microphotographs showing the dominant granitic textures found in the sample. A) Phlogopite interstitial to albite and quartz. B) Phlogopite with inclusions of quartz. C) Quartz and phlogopite interstitial to albite. Note: Ab = albite, Phl = phlogopite and Qtz = quartz.

Table. 2 show the summary of BET analysis: surface area was 1.404 m²/g; pore width was 18.064 nm; and pore volume was 0.006 cm³/g. The pore volume and pore width were low, and therefore high energy will be required for water molecules to permeate the rock mass. This was confirmed by the water absorption capacity of the granite rock sample. Water absorption is usually expressed as weight percent, which is a measure of the weight of water that the stone has absorbed, compared to the weight of the sample. The water absorption capacity of the sample was 1.2 ± 0.2 wt. %. Water absorption can be a useful tool to categorise various granites or stones in construction. Natural stone with high water absorption capacity can be used for interior wall applications while those with low water absorption capacity can be useful for exterior walls, kitchen walls, floor tiles, bathroom/toilet walls, etc.

Table. 2. Summary of BET Analysis

Average pore width (nm)	Average pore volume (cm ³ /g)	Surface area (m ² /g)
18.064	0.006	1.404

The rock sample was crushed into powder to examine surface morphology using SEM with secondary electron (SE) and back scattered secondary electron (BSE) detectors shown in Fig. 6 (A) and (B) respectively. The powdered rock sample shows non-availability of micro/macro pores micrograph within rock sample that can permitting free flow of water molecule, the different colours seen on the micrograph shows distribution mineral within the rock (see Fig. 5 for mineral distribution of the rock sample).

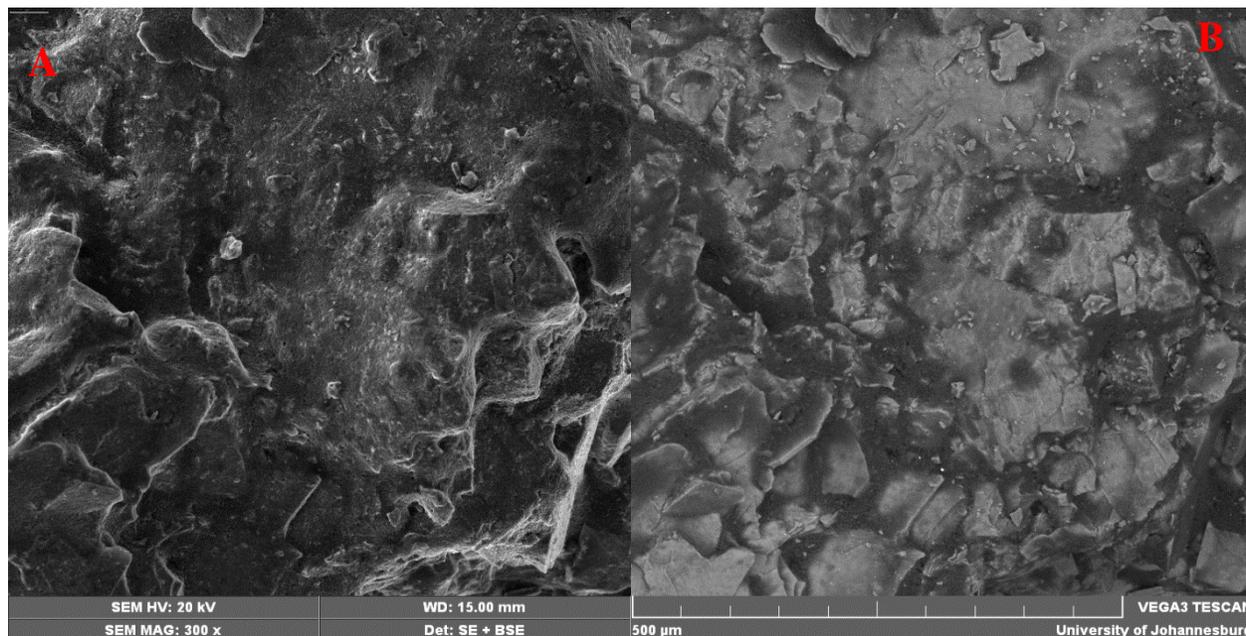


Fig. 6. SEM micrograph of the granite sample using a (A) Secondary Electron (SE) and (B) Backscatter Electron (BSE) detectors showing the surface morphology of the rock powder sample.

The SEM micrograph and EDS analysis is shown in Fig.7. The EDS analyses of the selected spots depict the presence of Na, Mg, Al, Si, K, Fe and O. The EDS results in oxide form were in agreement with the XRF.

The non-availability of micro- and macro-pores confirms the low water absorption capacity of the granite rock sample. On the other hand, the dry density analysis was conducted on the granite sample and it was found to be 2.73 ± 0.03 g/cm³; this value was within the range of granite rock density.

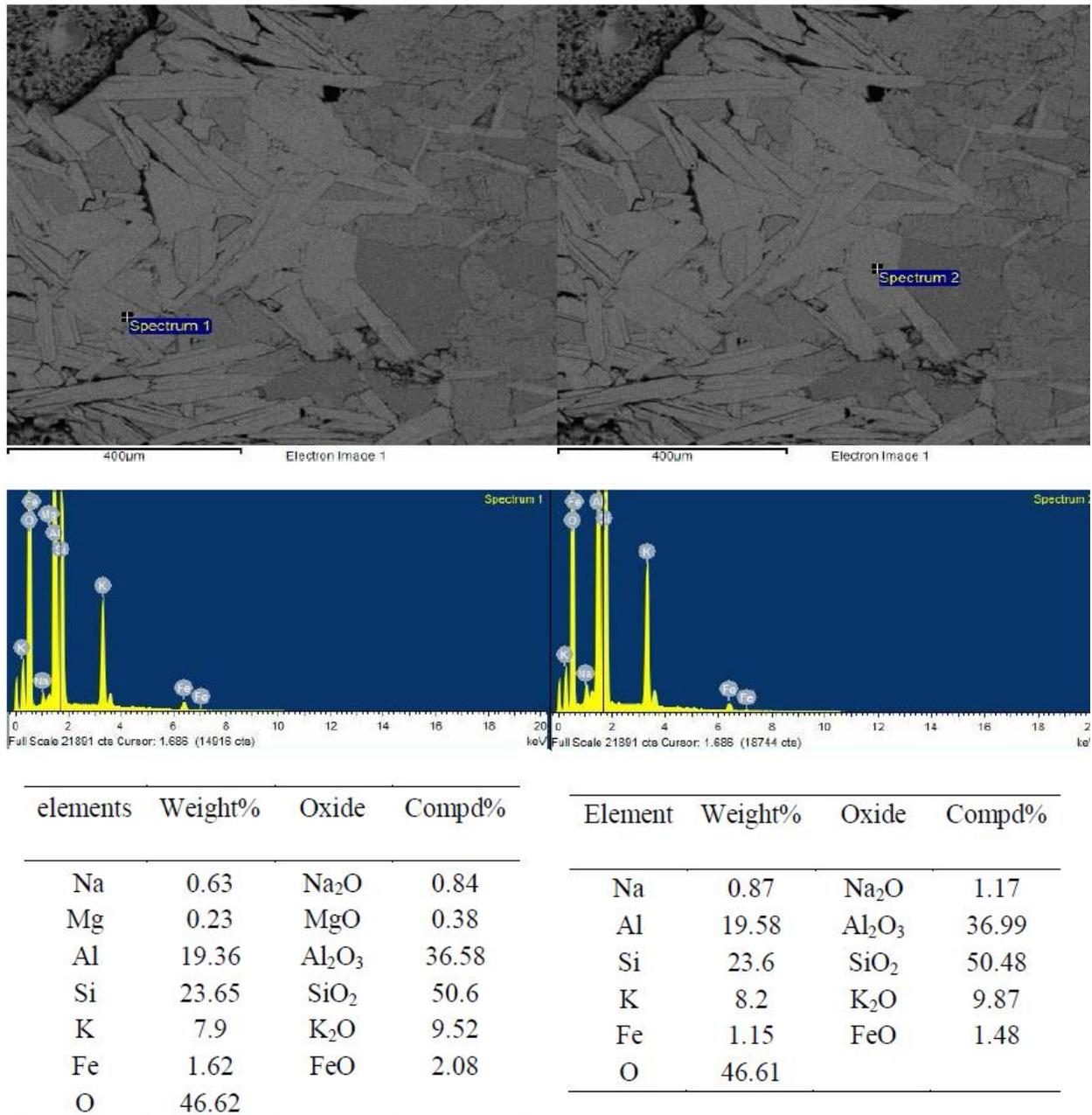


Fig. 7. SEM micrograph and EDS analysis of the selected spots

4. Conclusion

A sample of the unexploited deposit of granite rock in Oluwo Egbeda, Oyo State, Nigeria was characterized. The physicochemical and geological characterization confirms the rock as granite with quartz, mica and phlogopite minerals. The granite has small pore width and volume resulting in low water absorption capacity. The granite rock deposit, if exploited, can be a potentially valuable raw material for exterior walls, interior walls, kitchen tiles, etc. in the building and construction industry.

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