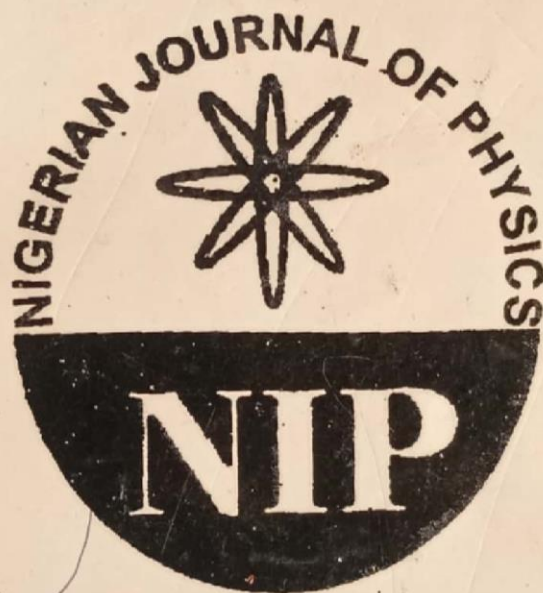

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Structural and qualitative analysis of solid minerals (marble) in selected locations in Nigeria

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Abstract

Marbles are formed from recrystallization of limestone or dolomite during metamorphism and occur as a coarse grained rocks composed of interlocking calcite crystals. The structural and porosity analysis of some selected Nigerian marbles were investigated with a view to establishing crystal configuration and constituents of the marble occurrences in the areas considered. The samples were collected from five different locations in Nigeria namely; Igbeti, Oyo State; Olayinka, Kwara State; Jakura, Kogi State; Biribiri, Niger State and Takalafia, Nassarawa State. The samples were examined by X-ray diffraction which confirmed the presence of CaCO_3 in all the samples. Other constituents present are Calcium Magnesium, Calcium Magnesium Carbonate and Magnesium Carbide. The average grain sizes of the Samples are 32.15, 31.42, 32.72, 31.47 and 30.95 nm from Igbeti, Olayinka, Biribiri, Takalafia and Jakura respectively. The samples were further examined by density profiling to determine the porosity of the marble samples. The comparison of the dry and wet density of the samples in percentage change in density revealed the porosity of the all the samples and it was deduced that the marble sample has different pore sizes due to their formation processes. The porosity for the samples are as follow: Igbeti (6.9%), Biribiri (62.4%), Takalafia (24.9%), Olayinka (10.3%) and Jakura (13.8%).

Keywords: Marble, XRD, Porosity, Grain Size, Location and Calcium Carbonate.

Introduction

Nigeria as a nation is blessed with abundant solid minerals resource distributed fairly across her states. According to reports by the Nigerian Geological Survey Agency, Nigeria has some 34 known major mineral resources deposit distributed in locations across the country. Exploration in Nigeria for solid minerals such as Tin, Niobium, Lead, Zinc, and Gold goes back for more than 90 years and the exploration of marble started in the 1970's.

Constituents and Characteristics of Marble

Marble is a metamorphic rock consisting of predominantly fine to coarse grained recrystallized calcite and/or dolomite usually with a granoblastic, saccharoidal texture. It is any crystallized carbonate rock including true marble and certain types of limestone (Orthomarlble) that will take a polish and can be used as architectural or ornamental stone. Marble results from the metamorphism of limestone or dolostone. This metamorphic process causes a complete recrystallization of the original rock into an interlocking mosaic of calcite, aragonite and/or dolomite crystals. The

temperatures and pressures necessary to form marble usually destroy any fossils and sedimentary textures present in the original rock (Deane, 2013).

A carbonate can be regarded as a marble if internal deformation and recrystallization have been sufficient to deform severally or to obliterate sedimentary features and fossils and the non calcareous constituents of the rock are in the form of graphite, chlorite, mica, tremolite or other typically metamorphic minerals. The supporting evidence is the character of the associated detrital rocks which are commonly slate, schist, meta-quartzite and gneiss (Bates, 1969).

The main mineral of most marble is calcite and marbles contain several percent of other minerals of non-silicates such as quartz, graphite, hematite, limonite and pyrite and the common silicates are mica, chlorite, tremolite, wallastonite, diopside and hornblende.

Only a few marble are dominantly dolomite instead of the calcite and such marbles comprising at alternating beds or masses of calcite and dolomite are undesirable because the two minerals frequently differ in colour, texture,

susceptibility to polish and resistance to weathering (Bates, 1969). Marbles as a group show a wide range of colour. Absolutely pure marble is brilliant white. Shades ranging from very light grey to black are produced by disseminated carbonaceous matter in the form of fine scales of graphite. Green tints result from the presence of chlorite or other silicates. Pink and red marbles owe their colour to finely divided hematite or manganese carbonate and yellow and cream marbles to a content of limonite. The colour may be evenly distributed throughout the rocks, but in many marbles, it is in mottled patterns or in streaks and bands called "veins". Since most marbles are dominantly of calcite, their texture is chiefly a function of the size mutual relations and orientation of the calcite grain. The grains are highly irregular in shape and are interlocked and the intergranular spaces are extremely minute. The grains of most marble show random orientation but in some the crystallographic axes are approximately aligned in a common direction. The cleavage of calcite produces a mosaic of bright reflecting surfaces which allows the rock to take a brilliant polish. On the Moh's scale, the hardness of calcite is 3.0 and that of dolomite is 3.5 to 4.0. Hence, marble is only of moderate hardness. Owing to their tight interlocking crystalline fabric Marbles have a very low porosity which ranges from 0.0002 to 0.5 percent and their solubility on exposed surfaces depends to some extent on the texture and composition but more especially on atmospheric condition.

Moh's Scale

The physical hardness of a mineral is usually measured according to the Moh's scale. This scale is relative and goes from 1 to 10. Minerals with a given Moh's hardness can scratch the surface of any mineral that has a lower hardness than itself. The largest proportion of marble is produced by strong directed pressure and high temperature acting on deeply buried limestone, in other words, by dynamothermal metamorphism. The action of strong unbalanced pressure is plainly shown by the intricate folds into which the marble beds and associated strata have been thrown. The concurrent high temperature is indicated by accessory silicate minerals such as tremolite and diopside which occur only in rock that has undergone intense heating. It is also shown by the fact that marble occurring with schist and gneisses show evidence of a dynamothermal history (Bates, 1969).

Meta-sedimentary rocks represented by quartzite, marble and mica schist of the migmatite gneiss complex (Hockey and Jones, 1964), have been referred to as the older metasediments by Oyawoye (1964) and McCurry (1976). The gneissic complex represents the oldest member of the Precambrian rocks of Northwestern Nigeria and that it evolved by successive sedimentation, deformation, metamorphism and igneous intrusion over a vast period in the history of the basement.

Types and Location of Marble

Marbles are generally classified into types by their location and Composition

Location: The location is generally used to name the marble, this happens mostly with marbles from the western world because they are historically notable. There are other types with varying colour, location and name.

Marble can also be classified into type by their composition. Silicate Poor Marble (White Marble) examples are Igbebi Marble and Isale-Osin Marble, Carbon Rich Marble (Grayish-White Marble) examples are Biribiri Marble and Itoke Marble, Marble with Calc-Silicate gneisses examples are Igarra Marble and Jakura Marble.

Occurrence of Marble in Nigeria

Various Marble deposits have long been known to occur as lensoid bodies within the younger Metasedimentary sequence or the schist belts of the Precambrian domain of Nigeria (McCurry, 1976 and Folami and Ojo, 1991). Notable among these are the Igbebi, Elebu, and Kwakuti marble on the western part and the Uba, Okpella and Jakura marbles on the eastern part of the schist belts. Other smaller deposits have also been reported from Igarra, Ikpeshiu, Ubo Ukpilla, Ososo, and Atte in Edo State, Idoani in Ondo State as well as Osara in Kogi State (Emofurietu and Ekuajemi, 1995). Others include Isale-Osin, Okelute, Oro (Kwara State), Takalafia in the FCT, Toto- Muro Hill in Nasarawa, Kankata (Kaduna State), Itoke (Benue State).

In this work, we obtained marble samples from different locations in Nigeria to confirm the availability of marble in those locations by x-ray diffractometry analysis, Investigate the constituents and composition of the marble in a particular area or location, Investigate the structural nature of the marble in this area which includes the crystal structure and the crystal size, determine the density of the marble samples by displacement method and investigate the porosity of the samples.

Materials and Methods

Location of Marble Sample

A sample was taken from each location, their global coordinates as obtained for use in this work are as follows:

- (i) Alaguntan Area, Igbebi In Oyo State
Lat. 8.63°N, Long. 4.08°E
- (ii) Biribiri In Niger State
Lat. 9.01°N, Long. 4.83°E
- (iii) Abanyan Via Olayinka In Kwara State
Lat 8.52°N, Long. 4.91°E
- (iv) Iwasabo Via Takalafia In Nassarwa State
Lat. 8.75°N, Long. 7.61°E
- (v) Jakura In Kogi State
Lat. 9.01°N, Long. 4.8°E

Processing and Characterization of the Samples

The marble samples were crushed separately into particulate form and fine appropriately enough for X-ray diffractometry analysis.

The XRD spectra figures 1-5, were acquired with a diffractometer using CuK α radiation and Magnesium Oxide Carbonate (31-0804), CaCO $_3$, (41-1475, 29-0305, 17-0763 and 05-0586), Calcium Magnesium (13-0450), Magnesium Carbide (01-1138), Calcium Magnesium Carbonate (06-0462 and 43-0697), Calcium Iron Oxide (39-1033) JCPDS standards were used to index the peaks.

Determination of the Porosity of each Sample

Paragnis (1962) has affirmed that the *in situ* density of sub-surface rocks lie between dry and wet densities of the rocks. Thus, high density contrast of dry to wet for a rock could be a signature of high porosity. Consequently the apparent porosity was obtained by an indirect method of density determination. This is done by immersing a dry sample of known mass in water for 48 hours. The percentage increase in mass relative to the original mass of the sample was then determined. This gives wet and dry densities of a given rock.

Results and Discussion

X-ray Diffractometry analysis of the samples

X-ray Diffraction analysis was used as the major tool for identification of phases (Johan, 2011).

Qualitative Analysis of the Samples

The X-ray diffractometer (XRD) pattern for the Olayinka sample is as shown in figure 1. The qualitative analysis revealed that the sample constituents include Calcium Magnesium, Calcium Magnesium Carbonate (Calcite Magnesian) and Calcium Carbonate (Calcite) with hexagonal, rhombohedral and orthorhombic crystal structure lattice system respectively. The XRD pattern for Igbe sample is shown in figure 2, consisting of Calcium Magnesium Carbonate (Calcite, Calcite Magnesian) and Calcium Carbonate (Calcite) with Rhombohedral and Orthorhombic crystal structure respectively. Figure 3 is the XRD pattern of the sample from Biribiri with the following compositions, Calcium Magnesium, Magnesium Carbide and Calcium Carbonate (Aragonite) with hexagonal, hexagonal and orthorhombic crystal structure respectively. The fourth sample obtained from Takalafia revealed a crystalline pattern under XRD analysis as shown in figure 4. This sample contains mainly Calcium Carbonate (Aragonite) with Orthorhombic crystal structure. The last sample was obtained from Jakura. The qualitative analysis which was carried out by pattern matching as done for all the samples, revealed that Calcium Magnesium carbonate (Calcite Magnesian), and Calcium Carbonate (Calcite) are

the main constituents present in this sample, with rhombohedral and orthorhombic crystal structure respectively.

It was observed that all the samples consist of calcium carbonate. In addition to this, Magnesium is another major element present in the marble samples. We also observed the presence of magnesium carbide in the sample from Biribiri.

Structural Analysis of the Samples

The particle size was estimated using Scherrer's equation:

$$g = \frac{\kappa \lambda}{\beta \cos \theta}$$

Where λ , β , and θ represent the wavelength of the X-ray source, the full width at half maximum (FWHM), and the Bragg angle, respectively (Kidowaki, 2012).

Olayinka sample has an average grain size of 31.42 nm. The sample from Igbe sample has average grain size of 32.15 nm. The sample from Biribiri having an average grain size of 32.72 nm. The fourth sample obtained from Takalafia has an average grain size of 31.47 nm. The last sample was obtained from Jakura has an average grain size of 30.95 nm. The marble sample with the highest grain size is that obtained from Biribiri (32.72 nm) and that with the lowest grain size is Jakura sample (30.95 nm) as shown in figure 5.

Porosity of the Samples

Density Comparison of the Marble Samples

From the comparison of the densities for the dry and wet samples, we observed that the sample from Biribiri is the most porous of the sample collected with percentage increase in density of 62.85% which is suspected to be due to the presence of magnesium compounds in the sample. The composition of the Biribiri marble has a lot of impurities thus interstitial defects which was filled with water after soaking for 48 hours. This makes the sample inappropriate for serious construction work and sculptural uses because it is easily susceptible to weathering.

The sample from Igbe is the least porous of the rocks because it is pure calcite, this makes the sample well packed with very little pores which makes it the best sample for several uses and applications. Also the sample from Olayinka in Kwara state is fairly compacted.

The presence of certain minerals in all the analyzed samples influences the physical properties especially the specific gravity and the strength control in the mineralogy. Marble as an industrial raw material is dependent on the degree to which its properties meet manufacturing specification. The higher specific gravity in Biribiri marble could be related to the presence of specific layers of silica or carbide. Another reason for such variation could be that the volume of crystallized dolomitic limestone as

recognized from the peak compared to the remaining analyzed samples. So, as the dolomite replaces the calcite, more pores are being created. The higher the values of these physical parameters, the higher the degree of dolomitization.

The good correlation of these parameters are indications of the the percentage of certain minerals in the marble, the influence of these minerals on the specific gravity and porosity of the marble and strength control on mineralogy.

TABLE 1: The Grain Sizes obtained by Debye Scherrer formula according to Crystal Peaks in the XRD Patterns for each Location

FWHM	2 θ	θ	Cos θ	β Cos θ	Grain Size
(Rad.)	(Rad.)	(Rad.)			(m)
Olayinka					
0,00505	0,4962	0,2481		0,96938	0,0049
0,00505	0,51628	0,25814	0,96687	0,00489	2,962E-8
0,00484	0,82485	0,41243	0,91615	0,00443	3,2678E-8
0,00494	1,04529	0,52265	0,8665	0,00428	3,38216E-8
Igbeti					
0,00505	0,50633	0,25316	0,96812	0,00489	2,95805E-8
0,00481	0,84615	0,42307	0,91183	0,00439	3,29745E-8
0,00495	1,05611	0,52806	0,86379	0,00427	3,38824E-8
Biribiri					
0,00505	0,48294	0,24147	0,97099	0,00491	2,94963E-8
0,00505	0,50039	0,2502		0,96886	0,0049
0,00494	0,74387	0,37193	0,93163	0,0046	3,14729E-8
0,00485	0,81438	0,40719	0,91824	0,00445	3,25275E-8
0,00483	0,83445	0,41723	0,91422	0,00441	3,28135E-8
0,00482	0,84527	0,42264	0,91201	0,00439	3,2962E-8
0,00476	0,98071	0,49036	0,88216	0,0042	3,45011E-8
0,00494	1,06432	0,53216	0,86171	0,00426	3,39795E-8
0,00489	1,08474	0,54237	0,85649	0,00419	3,45417E-8
0,00485	1,12977	0,56488	0,84465	0,0041	3,53104E-8
Takalafia					
0,00503	0,45449	0,22724	0,97429	0,0049	2,95207E-8
0,00478	0,87111	0,43555	0,90664	0,00433	3,34141E-8
Jakura					
0,00487	0,30701	0,1535		0,98824	0,00482
0,00504	0,50685	0,25343	0,96806	0,00488	2,96912E-8
0,0048		0,84807	0,42403	0,91144	0,00437
					3,31124E-8

Table 2: Densities of Dry and Wet Samples resulting in Porosity

Marble Location	Density (Dry) (g/cm ³)	Density (Wet) (g/cm ³)	Average Density (g/cm ³)	Porosity (%)
Igbeti	2.76	2.95	2.855	6.9
Biribiri	2.55	4.14	3.345	62.4
TakaLafia	2.41	3.01	2.710	24.9
Olayinka	2.14	2.36	2.250	10.3
Jakura	2.18	2.48	2.330	13.8

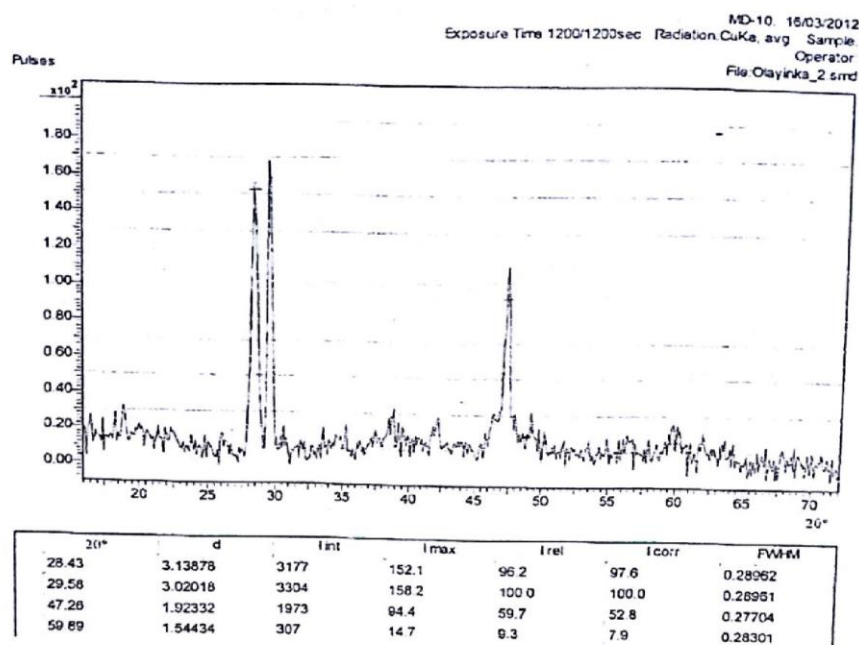


Figure 1: The XRD pattern of Olayinka Sample

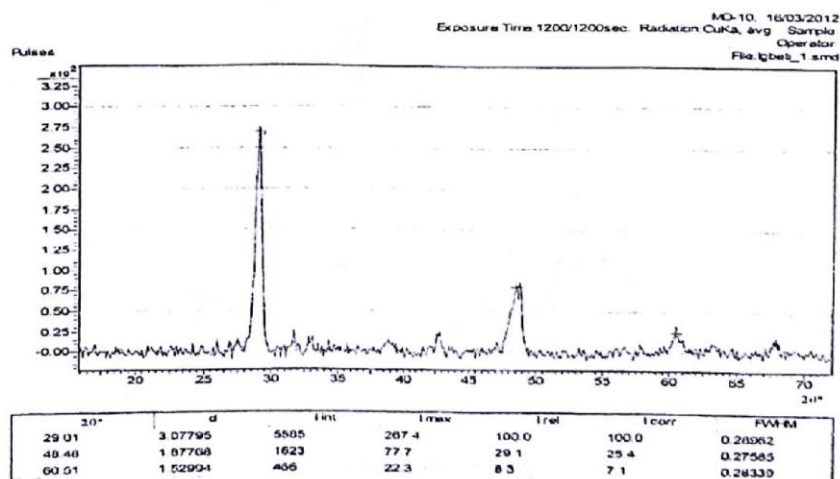


Figure 2: The XRD pattern of Igbeti Sample

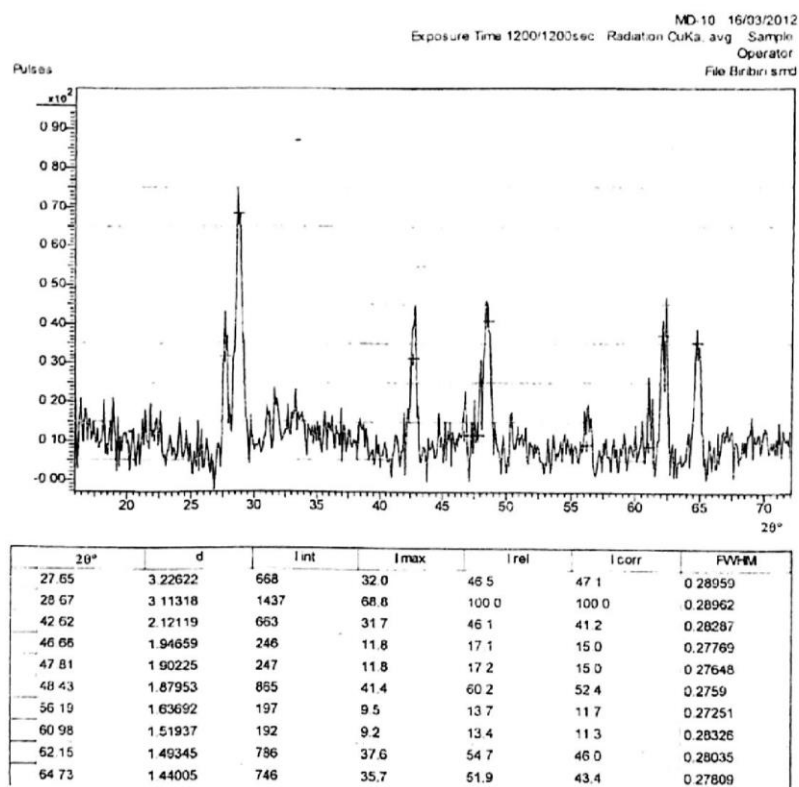


Figure 3: The XRD pattern of Biribiri Sample

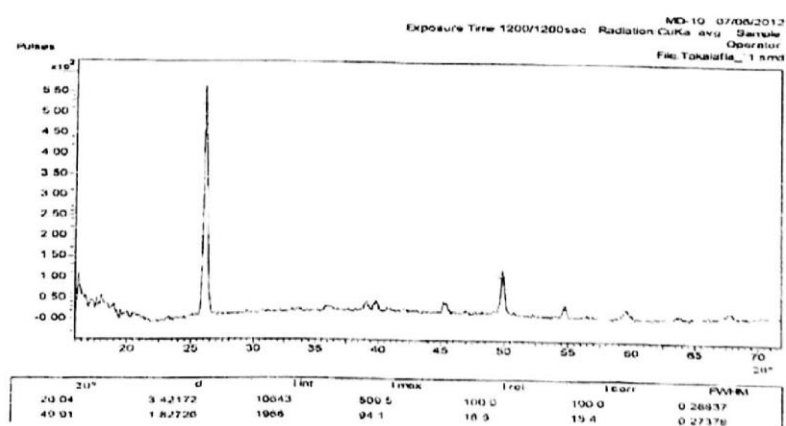


Figure 4: The XRD pattern of Takalafia Sample

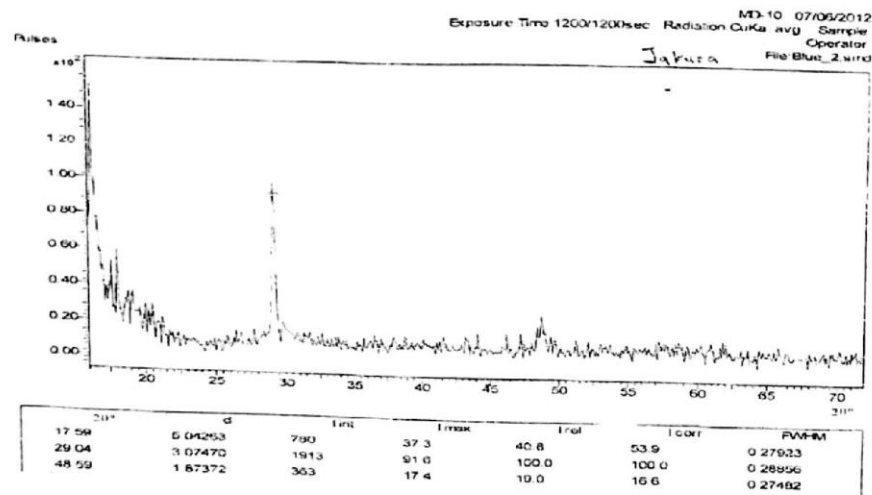


Figure 5: The XRD pattern of Jakura Sample

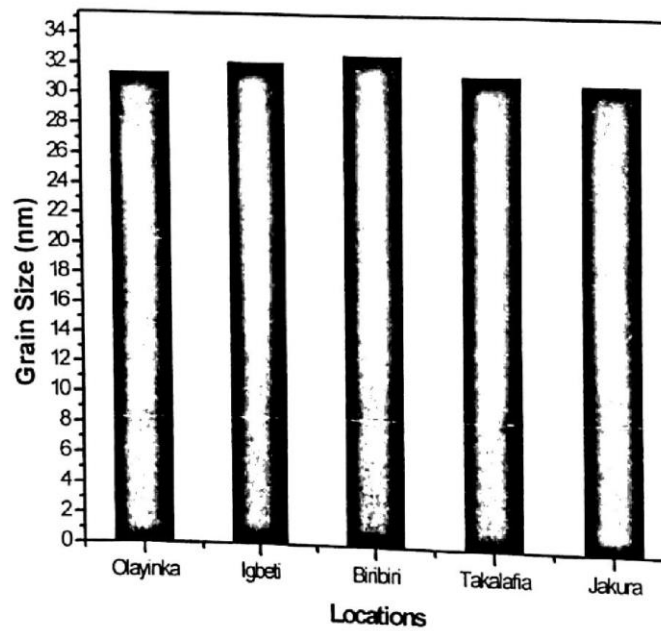


Figure 5: Grain Size against the Sample Locations

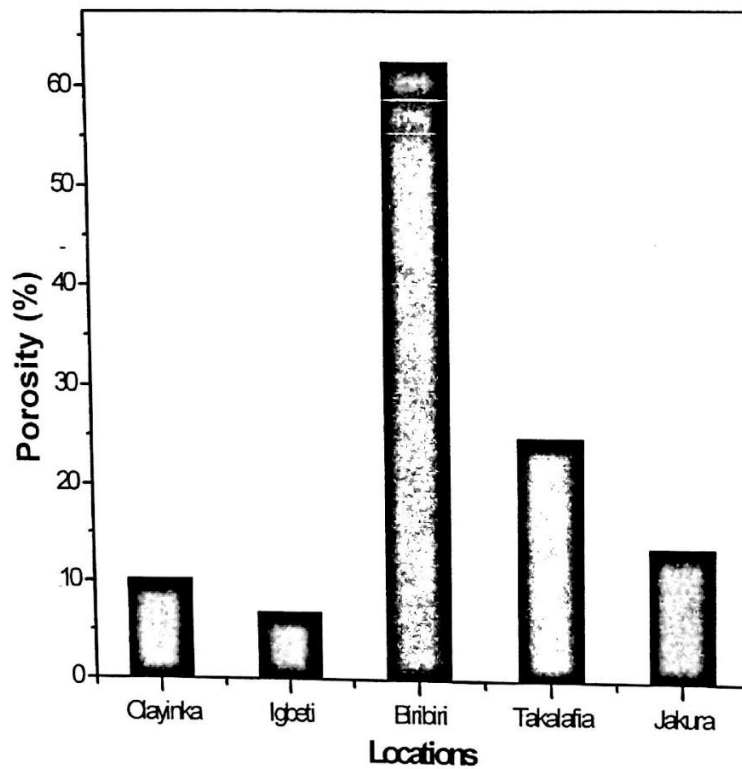


Figure 6: Porosity against the Sample Locations

Conclusion

It has been investigated and affirmed that the selected locations have marble deposits with deep quarrying sites. The XRD analysis showed that the samples are indeed marble containing Calcium Carbonate and other constituents such as Calcium Magnesium, Magnesium Carbide. The grain size of the samples from different locations were determined using Debye Scherrer formula to be 32.15, 31.42, 32.72, 31.47 and 30.95 nm from Igbeji, Olayinka, Biribiri, Takalafia and Jakura respectively. It was observed that there is variation in the grain sizes which is due to peculiar process of formation of each sample. The

comparison of the dry and wet density of the samples in percentage change in density revealed the porosity of the all the samples and it was deduced that the marble sample has different pore sizes due to their formation processes. The porosity for the samples were found to be: Igbeji (6.9%), Biribiri (62.4%), Takalafia (24.9%), Olayinka (10.3%) and Jakura (13.8%). This gives an insight into lattice arrangement and formation processes which will affect uses and application. It was also observed that the presence of other minerals apart from Calcium Carbonate like Calcium Carbide and the large grain size compared with other samples could be the reason for high porosity in Biribiri sample.

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