

## Quality Assurance of Available Portland Cements in Nigeria

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### Abstract

The notable demand for the want of cement in our time has stimulated increment in cement production and increment in manufacturers to be had in our day. These in turn amongst others have brought about failure of buildings and other construction members. As a result, the need to ensure the quality of available cement in circulation is of utmost paramount. This research work studies the available Portland lime cement of grade 42.5 only, in Nigeria from such company as company A, company B, and company C. The qualities and properties of the Portland lime cement from every of the above-named companies were investigated and as compared. Properties like Fineness, Setting Time, Chemical composition, Specific gravity, Consistency, Loss of ignition, Strength (compressive strength, and flexural strength), Micro structural analysis were determined according to relevant BS EN 197-1 (2011) and (ASTMC) 150-92 standards. These tests were carried out with the usage of scanning electron microscope, X-ray fluorescence spectrometer, Vicat apparatus, compressive strength machine, consistency apparatus and Chatelier flask, among others. The results showed that the chemical composition mostly of CaO, SiO<sub>2</sub>, AL<sub>2</sub>O<sub>3</sub> and MgO in the cement +are within the acceptable limit of 60.0-67.0, 17.0-25.0, 3.0-8.0 and 0.1-4.0 respectively in the BS 4550: Part 3 (1979). The results of the compressive strength acquired for all the cement have been a way beneath the standardized result expected for grade 42.5. These could partly be traced to the cement micro-structure as Company C with the best morphology exhibits better compressive strength than others. Improvement on the cement morphology by the manufacturers could lead to compressive strength improvement. It was also discovered that one of the important factors that would have contributed to the low compressive strength was excessive amount of silt/clay content material inside the sand used.

**Keywords:** Quality; Portland cement; Cement morphology; Compressive strength.



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### 1. Introduction

Quality in general can be defined as a measure of being free from defects, deficiencies and significant variations. Quality of cement in addition to the definition stated above can be said to be the totality of cement features, characteristics and performance output to satisfy specific customer need, users' requirement and the relevant standards specification (ISO 8402, 2015). Cement is one of the most vital construction materials in circulation today Faleye *et al.* (2009); Hani (2011); Ezeokonkwo and Anyanechi (2015). It is one of the important ingredients in concrete. Concrete usage cuts across so many disciplines, its ability to perform excellently well when combined with other construction materials as well as ability to conform to any shapes among others gives it a lead over other construction materials. The strength of any concrete structure depends upon the constituents of the concrete which in turn depends on the cement quality. To attain the desired strength of concrete and to increase the durability of any structure made with it, good quality cement should be ensured.

The quality of cement varies from place to place because of such a lot of reasons like material properties, kiln temperatures, fineness upon grinding, cement structures, chemical composition, among others. Ezeokonkwo and Anyanechi (2015). These variations will definitely affect the concrete properties and performance made from different cements. Past researchers reported that tricalcium aluminate (C<sub>3</sub>A) and alkali content of cement have been found to have dominant effect on the drying shrinkage of concrete (Johannes *et al.*, 2005). Likewise, Dale *et al.* (2008) checked the effect of fineness of cement on early strength of concrete, and observed that the coarser cement exhibit compressive strengths properly below the ones of the finer ones at every age tested with little heat of hydration, which results in a notably lower semi-adiabatic temperature rise.

MacGinley and Choo (1990), observed that failure of concrete structures is usually attributed to incorrect selection of material amongst other factors. Cement, a major binding material in making concrete, influences the quality of the concrete. Then, the quality of cement in civil Engineering construction should not be taken with levity, as it involves many lives, be it private building, bridges, roads constructions, e.t.c Allahverdi and Mahinroosta (2014).

Comparative quality evaluations of cement brand used, investigated by Olonade *et al.* (2015) in Nigeria, shows a fall in the expected results of the compressive strength obtained from various cement brand. They then recommended that the choice of cement brand to be used should be based on the expected strength requirements. It is pertinent that materials used in building construction should be regularly subjected to quality checks. This, will

undoubtedly, encourage the use of best quality materials thereby forestalling negative consequences such as building collapse or poor-quality products in the industries. Alteration of cement quality begins in production stages, from proportion of raw materials, to the degree burning of clinker, to the percentage of gypsum, finally to the grinding.

The aim of this study, therefore, is to evaluate the quality assurance of the available Portland limestone cement in Nigeria in terms of chemical composition, micro-structural analysis and compressive strength of the resulting concrete among others.

## 2. Materials and Methods

The materials used in this research work includes Portland lime cement of grade 42.5 from three different company's products represented as (cement A, cement B, and cement C), coarse aggregates, and fine aggregates with water.

### 2.1. Consistency Test

Normal consistency test was carried out to measure the wetness or fluidity of the cement. 300g of cement powder was measured in electronic compact scale, 25% of 300g was calculated and weigh to be the mass of water. Stop watch, 75g of water, trowel, and mixing tray were used for the test. The various cement were thoroughly mixed, poured into the vicat mould, with the surface properly dressed. The consistency needle was then released to touch cement in the mould. The consistency of the various cements were then measured in accordance with [BS 1370 \(1979\)](#).

### 2.2. Fineness Test

This test was done to check the proper grinding or particle size of the cements, using sieve number 0.075mic, electronic compact scale, pan, sieve shaker machine, spatula, and brush.

1g of cement powder sample was weighed using electronic compact scale termed as  $W_1$ , sieved with sieve number 0.075mic. The weight of the sample retained was obtained and recorded, then the percentage retained was calculated using equation 1. This test was done in accordance with [BS 4550: Part 3 \(1978\)](#)

$$\text{fineness} = \frac{W_2}{W_1} \times 100 \quad (1)$$

### 2.3. Setting Time Test

The Setting time test was carried out to check the amount of time required for the cement (mortar or concrete) to set (harden).

300g of cement powder was measured in electronic compact scale, 25% of 300g was calculated and weigh to be the mass of water. Stop watch, 75g of water, vicat/ring mould, vicat apparatus, trowel, spatulas, 1mm square needle and mixing tray were used for the test. The cement samples were thoroughly mixed, transferred into the vicat mould placed on lightly greased plane of glass with the surface properly dressed. Thereafter, the vicat plunger was released to touch the mortar cement, the depth values the plunger penetrates at given time interval were taken by the scale, and was done in accordance with [BS 4550: Part 3 \(1979\)](#) and [BS EN 197-1 \(2011\)](#).

### 2.4. Soundness Test

This test was carried out to measure the expansivity of cement paste through heating, the use Le-Chatelier apparatus, glass plate, mixing board plate, spatula, and dish.

Samples of cement paste were prepared (cement and water) and transferred into three number of the Le-chart apparatus, with surfaces levelled and properly dressed then kept for 24 hours for setting. Thereafter, they were removed, lengths were measured and placed in boiled water for two hours, then allow to cool and the expansivities were measured. This test was conducted in accordance with [BS 4550: Part 3 \(1978\)](#).

$$\text{Expansivity (mm)} = \text{Length after boiling (mm)} - \text{Length when cool (mm)}.$$

### 2.5. Tests on Aggregates

These tests were done to actually check for strength, toughness, hardness, shape, and water absorption of Aggregates.

#### 2.5.1. Natural Moisture Content Test

Two cans were labelled: A and B, weighed and recorded. Thereafter, the wet sand was poured into two cans, weighed and recorded, then transferred to oven for 24 hours. it was later withdrawn, allow to cool and weighed, in accordance to [BS 812 part 1 \(1975\)](#),

#### 2.5.2. Silt/Clay Content Method

250ml measuring cylinder, water, sand and tray were used to carried out the test. According to [BS 812 part 1 \(1975\)](#), 50ml of water and 1% salt solution were added into the cylinder. Thereafter, sand was poured to make it 100ml. It was then shaken very well and put down for 24 hours to allow it settled. Thereafter the whole height of sand in the bottle was measured and the silt/clay height also.

### 2.5.3. Particle Size Distribution

500g of the fine aggregate was weighed and oven dried. The sieves for the analysis were stacked in their order increasing aperture size. The sample was transferred into the sieve stack at the topmost sieve and vibrated vigorously for about 10 – 15 minutes. The sieves were carefully separated and the weight of soil retained on every sieve was weighed and recorded. Percentage retained and Percentage passing on every sieve were determined using equation 1 and 2 respectively in accordance with B.S 812 part 1 1971,

$$\% \text{ Retained} = \frac{\text{Mass retained (g)}}{\text{Total mass (g)}} \times 100 \quad (2)$$

$$\% \text{ Passing} = 100 - \% \text{ Retained} \quad (3)$$

### 2.5.4. Bulk Density Test

Cylinder of height (H) = 34mm. diameter (D) = 32mm, weight (W) = 25g, tapping rod, shovel, and a piece of glass were used to carry out the test. The Cylinder was filled with water and the amount of water was weighed as (V). Thereafter, cylinder was filled with aggregate sample in 3 layers, such that each layer was subjected to 25 blows using a tamping rod, the surface of the sample was leveled then weighed and recorded as (W). The bulk density was obtained using equation with respect to BS 812 part 110 (1990).

$$\text{Bulk density} = \frac{\text{Wt of sand (W)}}{\text{Volume of mould (V)}} \quad (4)$$

### 2.5.5. Specific Gravity Test

Specific gravity test was done in accordance with BS 812 part 110 (1990). The measuring bottle was weighed as  $M_1$ , dry sand was transferred into it and weighed as  $M_2$ , water was added and weighed as  $M_3$ . Then leave for 24 hours to settle, after  $M_4$  is known to be weight of bottle plus weight of water.

The Specific Gravity was calculated using Equation 5.

$$\text{Specific gravity (G}_s\text{)} = \frac{W_2 - W_1}{(W_2 - W_1) - (W_3 - W_4)} \quad (5)$$

Where;

$W_1$  = mass of measuring bottle

$W_2$  = mass of measuring bottle + dry soil sample

$W_3$  = mass of measuring bottle + water + dry soil sample

$W_4$  = Weight of measuring bottle + water (full)

### 2.5.6. Aggregate Crushing Value Test (ACV)

A steel cylinder, plunger, base plate, tamping rod, weighing balance, IS sieves of sizes 12.5mm, 10mm and 2.36mm, compression testing machine, and dual gauge, were used to carry out this test.

Cylinder was positioned on base plate and weighed, as  $W_1$ . The sample was filled in 3 layers, such that each layer was subjected to 25 blows using a tamping rod. The surface of the sample of aggregate was leveled and weighed as  $W_2$  then plunger was inserted so that it rests horizontally on its surface as care was taken to ensure that the plunger does not jam in the cylinder. The cylinder with the test sample and plunger was positioned on the loading platform of the compressive testing machine and loaded to a uniform rate so that a total load of 250kN was reached in 10 minutes. The load was released and the sample was removed from the cylinder, then sieved using 2.36 mm sieve. Thereafter, fraction passing the sieve was weighed and recorded as  $W_3$ . The procedure was repeated in three times and ACV was obtained using equation 3.4. with respect to BS 812 part 110 (1990).

$$\text{ACV} = \frac{W_3}{W_2 - W_1} \times 100\% \quad (6)$$

### 2.5.7. Aggregate Impact Value Test (AIV)

A steel cylinder, plunger, base plate, tamping rod, weighing balance, IS sieves of sizes 12.5mm, 10mm and 2.36mm, compression testing machine, and dual gauge, were used to carry out this test.

The cylinder was positioned on base plate and dry granite was fully put into it and weighed as  $A_1$ . Then the sample was empty and again put into it in 3 layers, each layer subjected to 25 blows using a tamping rod. The surface of the sample of aggregate was leveled. Thereafter, it was fixed into the AIV machine. the load was released and the sample was removed from the cylinder and then sieved using 2.36 mm sieve. The fraction passing the sieve was weighed and recorded  $A_2$ . The procedure was repeated in three times according to BS 812 part 112 (1990).

## 2.6. Compressive Strength Test

The compression test was done to measure the compressive strength of concrete of different cement samples. Local sand, 19mm crushed granites, clean water, and concrete moulds, were used to perform the test.

A mix ratio of 1:2:4 and water cement ratio of 0.5 was used to cast the cubes of 150mm×150mm×150mm for 7, 14, 21, and 28 day curing periods in accordance with BS EN 12390-3 (2009).

Figure-1. Cast Concrete cube of Cement A, Cement B and Cement C sampl



## 2.7. Tests on Fresh Concrete

### 2.7.1. Slump Test

Slump test was carried out to assess the workability of the fresh concrete.

Metal mold, frustum of a cone, open at both ends and provided with a handle, having a top internal diameter of 100mm and bottom internal diameter of 200mm with a height of 300 mm and a tamping rod were used for the test.

Fresh concrete were placed in three layers in the slump cone through the slump funnel with the aid of a scoop. Each layer was given 25 blows using the tamping rod. Blows were evenly distributed around the entire surface of the concrete in the cone. The funnel was removed and the surface of the fresh concrete was troweled with the aid of a straight edge. The slump cone was removed in order to get the slump concrete mix and the slump value was measured. *BS 1881: Part 116 (1983)*, prescription.

### 2.7.2. Compacting Factor Test

The degree of compaction is measured by the density ratio of the partially compacted test piece to that of same fully compacted concrete.

Empty cylinder was weighed using a weighing balance and its weight was recorded. The internal surfaces of the hoppers and cylinders were ensured to be smooth, clean and damp so as to allow for free falling of the concrete through the openings. The frame was placed in a position free from vibration in such a manner that it was stable with the axis of the hoppers and cylinders were all lying on the same vertical line. The two trap doors were closed at this time and the locks were used to hold them in place. The sample of the concrete was carefully placed in the top hopper until it was filled to the level of the brim. The upper trap door was then opened for the concrete to fall freely into the lower hopper. The trap door of the lower hopper is opened to allow for the free flow of the concrete into the lower cylinder. The excess concrete remaining at the top of the cylinder was leveled with the top of the cylinder by cutting it off with a hand trowel. The fresh concrete was weighed and recorded as  $m_p$ . The self-compacted concrete was emptied out of the cylinder and the cylinder was refilled with fresh concrete and compacted in three layers and each layer was given 25 blows with the tamping rod. After top layer was compacted, the concrete was leveled with the top of the cylinder and weighed and was recorded as  $m_f$ . The compacting factor value was calculated using Equation 3.5.

$$\text{Compacting factor} = \frac{m_p}{m_f} \quad (7)$$

Where;

$m_p$  = Mass of Partially Compacted Concrete

$m_f$  = Mass of Fully Compacted Concrete

## 3. Results and Discussion

The results obtained from the research output for the various selected cements are presented and discussed under the appropriate subheadings



### 3.1. Physical and Mechanical Properties

The results of the physical and mechanical properties of the selected cements shown in Table 1 indicates that the consistency, fineness, setting times and the loss on ignition for all the selected cements conform with the standard requirement stipulated by NIS 444-1 (2003). The soundness tests did not meet the NIS 444-1 (2003) requirement for any of the cement. The results of the oxide composition are also shown in Table 2 for the selected cements. None of the cement A, cement B, cement C has the same oxide composition according to the results obtained. Their oxide composition differs. However, the compositions of the magnesium Oxide (MgO) and Sulphide (SO<sub>3</sub>) in any of the cement meet the requirement in the code NIS 444-1 (2003).

Table-1. Physical and Mechanical Properties of The Selected Cements

S/N			Cement A	Cement B	Cement C	NIS 444-1 (2003) Requirement
1	Consistency (min)		12	13	15	30min
2	Fineness (%)		1.0	1.2	3.4	≥10%
3	Setting time (min)	Initial Time	1:32min	1:14min	60:00min	≥60
		Final Time	9:00hr	10:00hr	9:00hr	≥600
4	Soundness		1.56	2.32	2.21	≥10
5	Loss on ignition (LOI)		2.16	1.48	1.22	5-Maximum

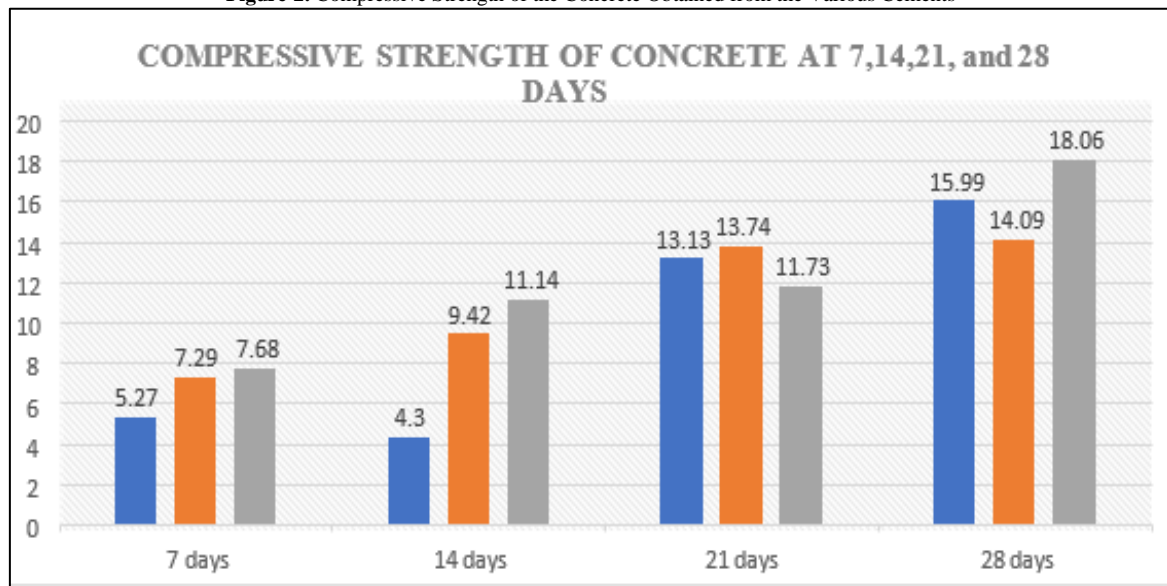
Table-2. Results of the Oxide Composition for the Selection Cements

S/N		Results			NIS 444-1-2003 Requirement
		Company A	Company B	Company C	
1	Silicon Oxide (SiO <sub>2</sub> )	20.25	17.93	20.28	
2	Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	4.54	4.32	5.25	
3	Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> )	4.06	3.70	2.30	
4	Magnesium Oxide (MgO)	1.52	2.68	2.06	≤4
5	Calcium oxide (CaO)	62.75	61.97	62.82	
6	Sulphide (SO <sub>3</sub> )	1.09	0.32	1.72	≤3
7	Potassium Oxide (K <sub>2</sub> O)	0.24	0.18	0.64	
8	Sodium Oxide (Na <sub>2</sub> O)	0.16	0.07	0.40	
11	Titanium Oxide (TiO <sub>2</sub> )	0.07	0.05	0.03	

### 3.2. Compressive Strength

The results of average compressive strength tests of concrete cubes made with cement A (blue), cement B (orange) and cement C (grey) are shown in figure 2 for 7days, 14days, 21days and 28 days of curing. Cement C has the highest compressive strength while cement A has less but not the lowest compressive strength at 28 days of curing. This could be partly traced to the high amount of Silicon oxide (SiO<sub>2</sub>) and Calcium oxide (CaO) present in these cements. The compressive strength results obtained for all the cements are lower than the minimum expected results of 13.5N/mm<sup>2</sup> at 7 days and 20N/mm<sup>2</sup> for 28 days for M20 grade concrete (IS 456, 2000).

Figure-2. Compressive Strength of the Concrete Obtained from the Various Cements



### 3.3. Particle Size Distribution

Table 3 shows the result of the particle size distribution carried out on the fine aggregate. The silt content of the sand is greater than 5% which means it is well graded according to ASTM D3282-09.

Table-3. Particle size distribution (PSD) of the fine aggregate

Sieve Size (Mm)	Weight Retained (G)	%Weight Retained	%Weight Passing
4.00	-	-	100
3.00	13.5	2.70	97.3
2.36	2.8	0.56	96.74
1.7	11.5	2.30	94.44
1.18	19.5	3.90	90.54
0.6	23.9	4.78	85.76
0.5	13.2	2.64	83.12
0.425	58.4	11.68	71.44
0.212	150.6	30.12	41.32
0.150	55.9	11.18	30.14
0.075	26.0	5.20	24.94
Pan	124.7	24.94	00.00

### 3.4. Moisture Content Test

The value of moisture content of 6.2% shown Table 4 indicated that for every 26g of sand measured, 1.612g is the weight of water in it while 24.388g is the weight of dry sand.

Table-4. Data of Moisture of Contents

DESCRIPTION	SAMPLE A	SAMPLE B
Wt of can	20.0g	20.0g
Wt of can + wet sand	46.0g	55.40g
Wt of can + dry sand	44.4g	53.2g
Wt of wet sand	26.0g	35.4g
Average wet sand	30.7	
Wt of dry sand	24.4g	33.2g
Average dry sand	28.8	
Moisture content	6.2%	

### 3.5. Silt/Clay Content

The result of the silt/clay content test indicated that the aggregate is in ratio of 3:1 (sand to clay/silty), the Percentage of silt/clay content = 32.25%, which is not too good for construction purposes as stated by ASTM C 150-92 standards.

### 3.6. Bulk Density

From the result this material is termed to be very good. It within the range 1600 – 2400 values by B.S 812 part 2 1975.

### 3.7. Specific Gravity

From the result, the specific gravity for the granite and fine aggregates were 2.75 and 2.462 respectively. This implies that the granite aggregates are within the acceptable range of 2.55 - 2.80 according to BS 4550: Part 3 (1979) and therefore suitable for construction purpose.

### 3.8. Aggregate Crushing Value

From Table 5, the result of the aggregate crushing value for the coarse aggregate is 28.57% which is within the acceptable limit range of 30% prescribed by BS 812 part 110 (1990). This indicates that the aggregate is suitable for concrete production.

### 3.9. Aggregate Impact Value

From Table 5, the result of the aggregate impact value for the coarse aggregate is 20.77% which is within the acceptable limit range of 30% prescribed by (BS 812 part 112 (1990)). This indicates that the aggregate is suitable for concrete production.

Table-5. ACV and AIV Data

Sample No	Wt <sub>2</sub> Of Sample in Mould	Wt <sub>1</sub> Retain Through Sieve No 2.36	(%)
Average	490.7	140.2	ACV = 28.57
Average	570.87	118.6	AIV= 20.77

### 3.10. Slump Value and Compaction Factor of Fresh Concrete

From the result, the compaction factor values of the fresh concretes for all cement used are almost equal to 1, which are 0.978, 0.942, 0.979 for cement A, Cement B, Cement C respectively. This mean the cement/water is almost the same. This is because of huge silt-clay content in sand, it affected the workability of fresh concrete. Hence, more water is been added when mixed.

### 3.11. Scanning Electron Microscope (SEM) Results

The SEM results in Figure 2 to Figure 9 enable us to established more facts. By comparing the SEM image together, Cement C has more compacted microstructure than the rest. Consequently, Cement C has better morphology and its constituents could easily filled up tiny voids in the resulting concrete made from it. Meanwhile, Cement B and Cement A have larger voids which could lower their interconnection with the other concrete constituents thereby resulting into lower strength and increased porosity of the resulting concrete. The compressive strength results in Figure 2 confirmed this fact.

Figure-2. SEM size of 350 x 766 (Cement A)

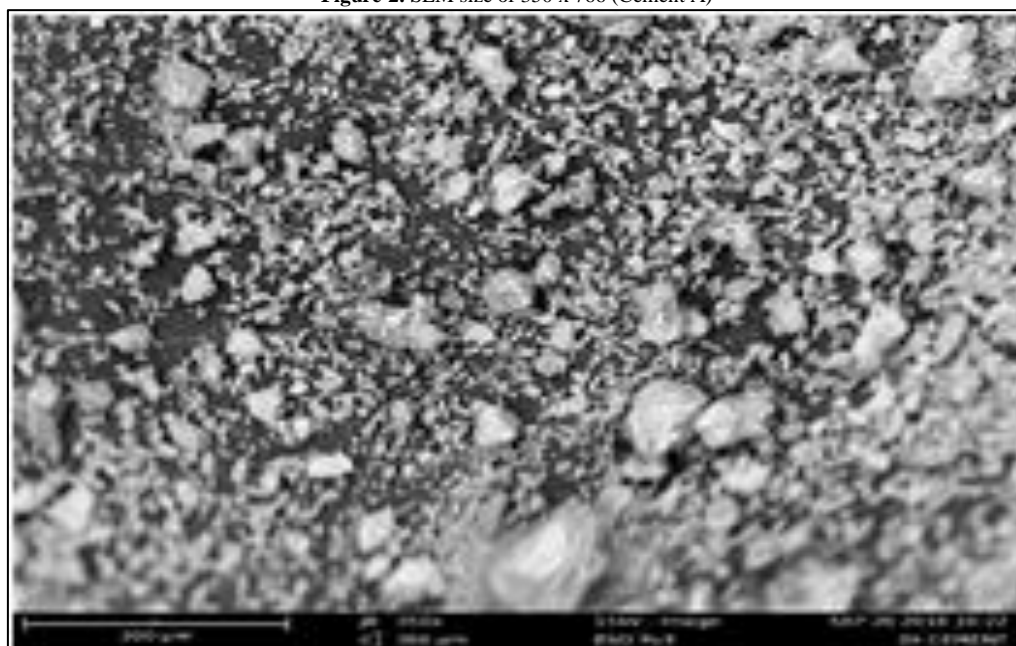
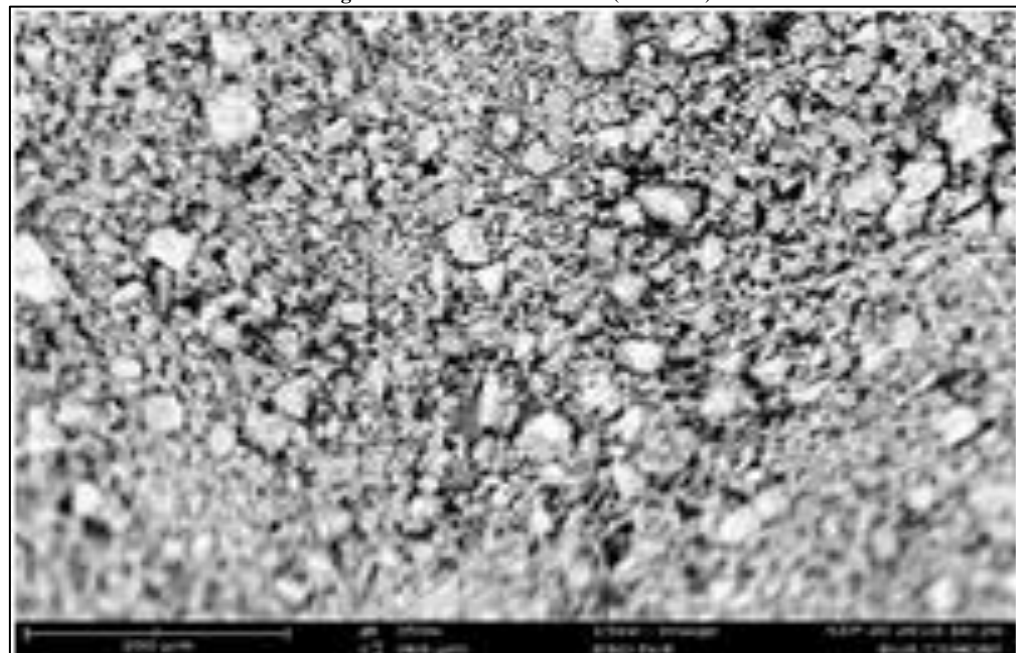


Figure-3. SEM size of 350 x 766 (Cement B)

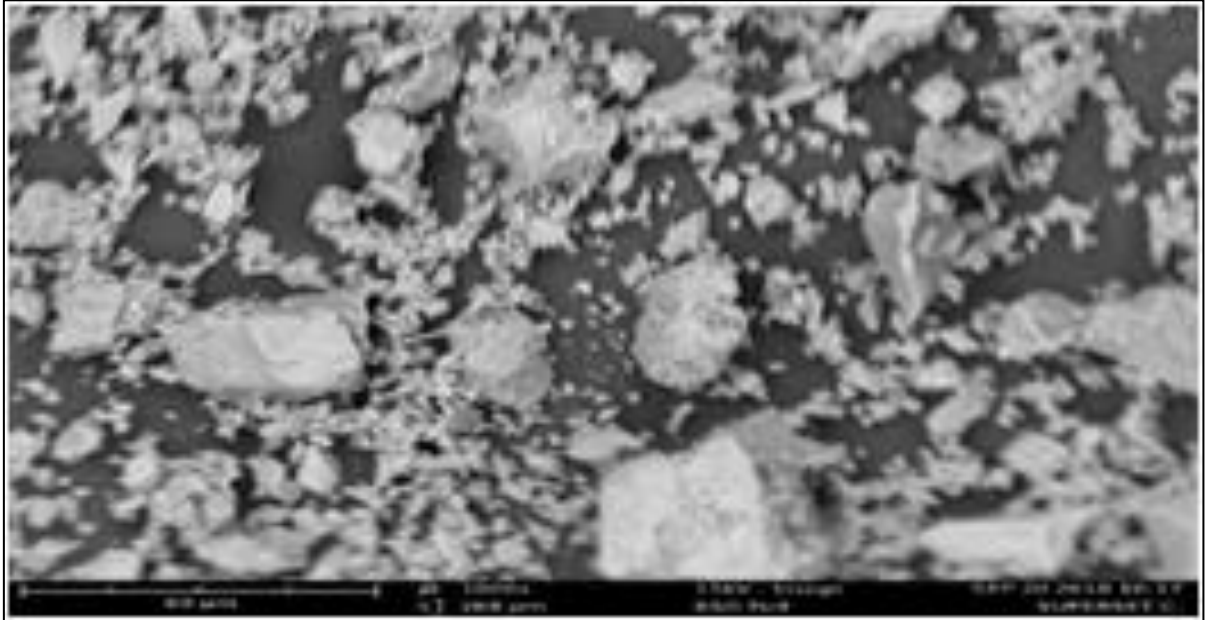




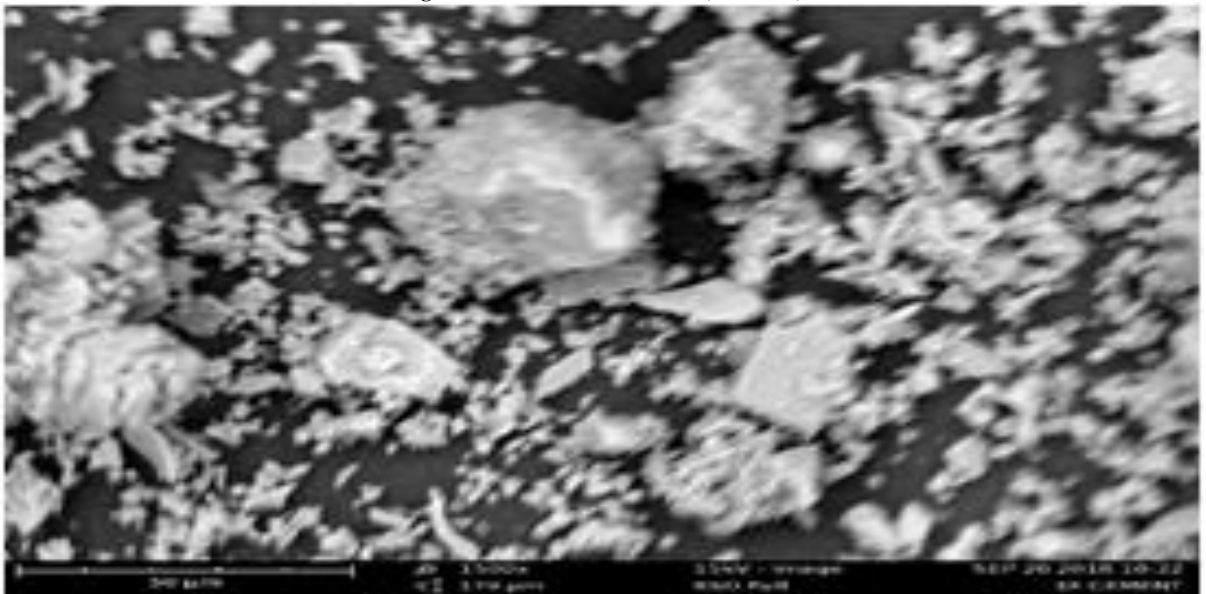




**Figure-6.** SEM size of 1000 x 268 (Cement C)



**Figure-7.** SEM size of 1500 x 179 (Cement A)



**Figure-8.** SEM size of 1500 x 179 (Cement B)

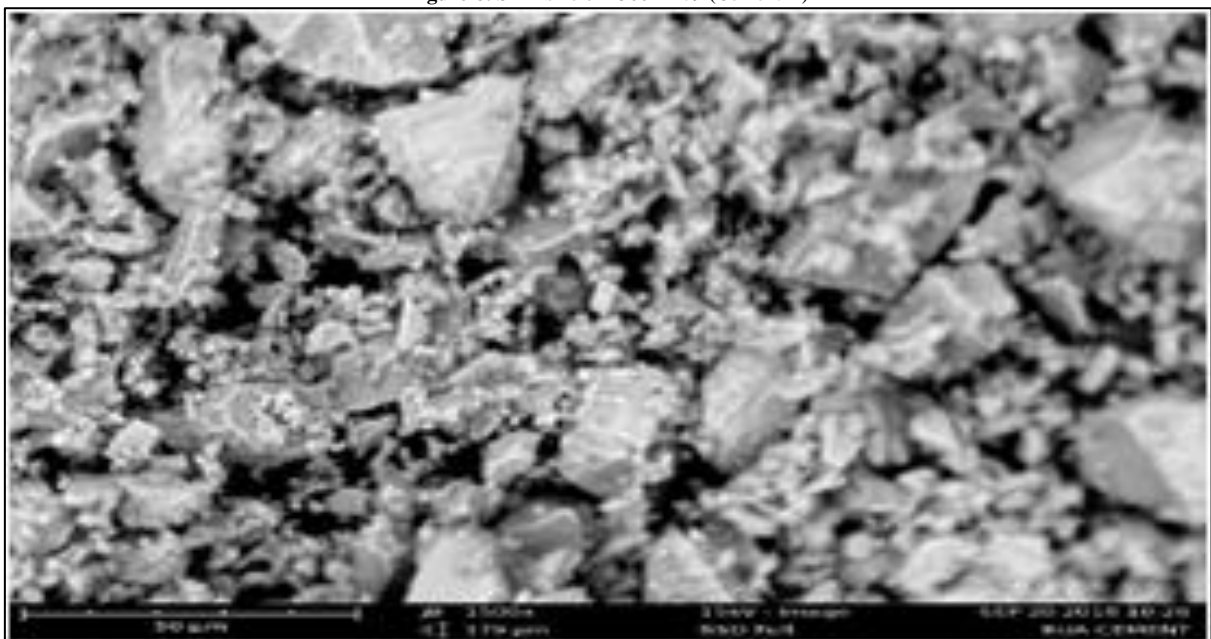
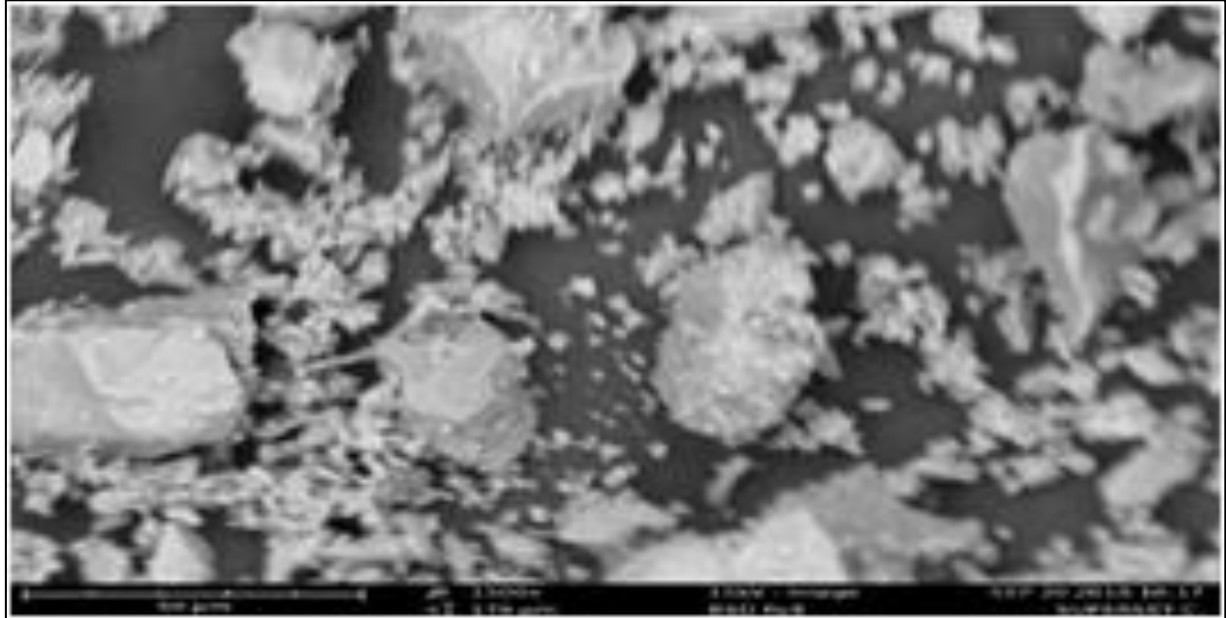


Figure-9. SEM size of 1500 x 179 (Cement C)



#### 4. Conclusions

From the experimental investigations conducted on Cement A, Cement B and Cement C in Nigeria the following conclusions can be drawn:

- Low compressive strengths were recorded for all types of cement investigated. The average compressive strengths at ages 7, 14, 21, and 28 days were below the minimum expected results of 13.5N/mm<sup>2</sup> at 7 days and 20N/mm<sup>2</sup> for 28 days for M20 grade concrete (IS 456, 2000).
- The chemical composition of all cement considered are within the range stipulated by BS EN 197-1 (2011) and BS EN 12 (1989). But cement C has the highest strength, due to the presence of higher reactive Silicon Oxide (SiO<sub>2</sub>) and Calcium Oxide (CaO) as well as having better morphology than others.
- Silt/clay content in the sand could have contributed to the reduction in strength.

#### Recommendations

Based on the results of the research, the following recommendations are made:

- Chemical composition and microstructural tests should be carried out for any cement before using it especially in major construction.
- Compressive strength test should also be conducted prior to construction and during construction building or civil engineering works.

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