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**ORIGINAL RESEARCH ARTICLE**

# COMPARATIVE STUDY OF DURABILITY AND MICROSTRUCTURE PROPERTIES OF CONCRETE MADE WITH DIFFERENT GRADES OF NIGERIAN PORTLAND LIMESTONE CEMENT

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**ABSTRACT**

*It is now becoming apparent that concrete should not only be strong but must also be durable. It is also believed that understanding the microstructure of the concrete will help produce strong and durable concrete, and thus enhance innovativeness in concrete application. This paper compares the durability performance and microstructure development of concrete produced with Portland limestone cement (PLC) grades 32.5R and 42.5R. The durability investigation was accomplished through coefficient of water absorption and sorptivity measurements. The scanning electron microscopy (SEM) was used to investigate the microstructure of the concrete samples. The results show that concrete produced with cement grade 42.5R showed higher durability potential than the ones produced with grade 32.5R using 28 days curing period. Similarly, it was also observed that concrete specimens produced with 32.5R and with water/cement ratio of 0.40 have better durability index at the later dayscuring of 90 days in comparison to grade 42.5R concrete specimens. It can be concluded that concrete produced using grade 42.5R cement has potential for better durability performance in relation to concrete produced with 32.5R.*



## 1.0 Introduction

Concrete industry in Nigeria has witnessed a large turn-out of research papers on the Portland limestone cement concrete. However, most of these works focus on either strength comparative analysis, or strength development investigations of concrete made from the available grades of cement (Adewole et al., 2015; Joel and Mbapuun, 2016). Other studies such as Ezeokonkwo and Anyanechi (2015) and Olonode et al. (2015) focused their attention on ascertaining the quality of cement grades. The available grades of cement in Nigeria are grades 32.5 and 42. 5 (in the classes of N, representing ordinary early strength; R, representing high early strength, and S, representing standard strength). Literatures documenting research works on durability characteristics and microstructures of concrete produced with Portland limestone cement are scarce. It is true that until recently, developments in cement and concrete technology have concentrated on achieving higher strengths on the assumption that strong concrete is durable (Neville, 2011). It is now known that, for many conditions of exposure of concrete structures, both strength and durability have to be considered explicitly at the design stage.

However, according to Hilal (2016), in order to achieve desirable strength and good durability requirements to prevent concrete deterioration, reducing the capillary porosity and a substantial reduction in the total porosity as well as reduction gel porosity are necessary. This leads to changes in the C-S-H structure, from porous to more crystalline phase. It is established that the microstructure of concrete (cementitious paste, pore structure, and interfacial transition zone between the cement paste and the aggregate) has a significant role in affecting the strength of concrete. Its effect on the durability of concrete is also overwhelming. This is because microstructure describes the porosity, permeability, and pore size distribution of concrete. Thus, when durability of concrete is considered, the quality of concrete within the context of low permeability comes into the play. This is because, with the exception of mechanical damage, all the adverse influences on durability involve the transport of fluids through the concrete. Thus, durability of concrete is closely interwoven with the microstructure of concrete. Proper understanding of this, is therefore, necessary to provide design engineers with a greater knowledge of concrete behaviour, so that they can optimise the use of the material aspects of concrete in their design. This is more so, when sustainability and environmental issues are becoming important in structural concrete design and construction.

Understanding the microstructure will enable innovativeness in the utilization of unfit, or weak material, as complimentary material. While researchers in Nigeria have given prominence to the study of mechanical properties of concrete produced with Nigerian Portland limestone cement, little attention has been given to its durability and microstructure characteristics. Thus, the aim of this investigation is to carry out comparative evaluation of concrete made with cement grades 32.5 R and 42.5 R in relation to durability and microstructure. Specifically, microstructural examinations using scanning electron microscopy (SEM) and water-based durability investigations on concrete samples would be conducted. The choice of water-based assessment of durability characteristics is informed by the observation made by the Institution of Civil Engineers (ICE, 2019) that water could be responsible for deterioration in buildings and other infrastructures.

## 2.0 Materials and Method

### 2.1 Materials and Mix Proportions

The following materials were used; cement, fine aggregate, coarse aggregate, and water. The cement used was Portland limestone cement (PLC) classified as CEM II by NIS 444-1 (2014). Two graces of cement, namely, 32.5 R and 42.5 R, being the only ones available in the Nigeria market were used. River sand was used as the fine aggregate. The sand was obtained from river FUOYE, a river flowing adjacent to the Ikole-Ekiti Campus of Federal University, Oye-Ekiti, Nigeria. The sand was dried and all contaminants removed. The portion of the materials which passes through sieve size 4.75 mm but retained in sieve size 2.36 mm was collected and stored. An active quarry in Ikole town, where the Engineering Faculty of the University is situated, served as the source of the coarse aggregate. In accordance to the stipulations of BS 8110 (1997), the maximum aggregate size was limited to 20 mm. Portable water, from the University borehole, was used for this investigation. The concrete mix design method adopted by BS EN 206 (2013), in combination with the recommendations by the Council for the regulation of Engineering in Nigeria COREN (2017), were used to obtain the mix design. The slump loss of between 30 – 60 mm for a target mean 28-day cube compressive strength for 25 N/mm2 was adopted for this investigation. The mix proportion is presented in Table 1.

Table 1: Mix Proportion for the Investigation

| Grade | W/C Ratio | Mix Designation | Cement ($kg/m^3$) | Sand ($kg/m^3$) | Gravel ($kg/m^3$) | Water ($kg/m^3$) |
|---|---|---|---|---|---|---|
| 32.5 | 0.4 | M14 | 343 | 686 | 1372 | 137 |
| | 0.5 | M15 | 343 | 686 | 1372 | 172 |
| | 0.6 | M16 | 343 | 686 | 1372 | 206 |
| 42.5 | 0.4 | M24 | 343 | 686 | 1372 | 137 |
| | 0.5 | M25 | 343 | 686 | 1372 | 172 |
| | 0.6 | M26 | 343 | 686 | 1372 | 206 |

Concrete ingredients were batched by weight, thoroughly mixed, and cast into cube moulds (100 x 100 x 100 mm). The cube specimens were demoulded after 24 hours and moist cured until the day of testing at 28 and 90 days of curing. The specimens for microscopic investigation were taking from the crushed 28-day samples.

## 2.2 Experimental Investigations

### *2.2.1 Characterization of Materials*

In order to characterize the naturally-occurring materials used for this investigation, some preliminary investigation was done to determine some of their properties. Physical properties like: specific gravity, moisture content, water absorption, dry density, bulk density and sieve analysis, were carried out on the aggregates. No preliminary investigation was performed on the cement used. This is because the seal of the Standard Organization of Nigeria was conspicuously displayed on the cement bags that were bought in the open market for this investigation. This was considered a sufficient proof that it has been manufactured to conform to NIS 444-1 (2014).

### *2.2.2 Durability Investigation*

The durability assessment of the concrete samples was accomplished through two tests, namely: (i) coefficient of water absorption test and (ii) sorptivity test.

#### *2.2.2.1 Coefficient of Water Absorption Test*

A measure suggested by Powers (1983), in which coefficient of water absorption is considered as a measure of permeability of water was used for this investigation. This test measures the rate of up take of water by dry concrete samples in a period of 1 hour.  Concrete cube specimens with dimensions 100 x100 x 100 mm were used for this test. The specimens were cured and tested at 28 and 90 days. The concrete specimens were preconditioned by drying in an oven at 105o C for three days until constant weight was reached and then allowed to cool in a sealed container for three days. Since it is necessary that water flows in one direction only in this test, the other sides of the concrete samples were coated silicone sealant. Then the samples were kept partially immersed to a depth of 5 mm at one end in a vertical position, while the rest of the sides were kept exposed to the laboratory air. This configuration to assess the rate of water absorption is shown in Figure 1.

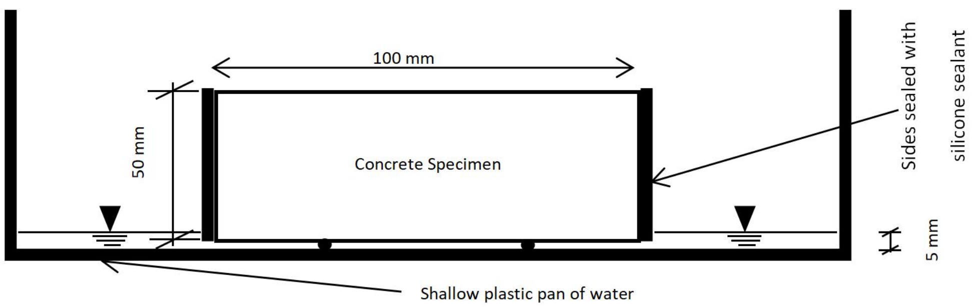


Figure 1: Set up for the measurement of Coefficient of water absorption and sorptivity of Concrete specimens (adaptation from Ganesan et al., 2008).

The quantity of water absorbed during the first 60 min by the concrete samples was calculated. Coefficient of water absorption values of concrete specimens after 28 and 90 days of moisture curing were determined using the Equation 1 as suggested by Ganesan et al., 2008.

$$K_a = [\frac{Q}{A}]^2 \times \frac{1}{t} \tag{1}$$

#### *2.2.2.2 Sorptivity Test*

According to Neville (2011), sorptivity is a material property which characterizes the tendency of a porous material to absorb and transmit water by capillarity. For the sorptivity test, the configuration, cube specimens and sample preparation used for coefficient of absorption as

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shown in Figure 1, were also used. The difference is that, in this case, the tests were conducted at selected times 0, 2, 4, 8, 10, 20, 30, 60, 90 and 120 minutes. At the selected times, the samples were removed from the water, excess water blotted off with a damp paper towel and then the sample was weighed. The samples were then replaced in the water for the selected time period. Then the gain in mass per unit area over the density of water was plotted versus the square root of the elapsed time. The cumulative water absorption (per unit area of the inflow surface) is believed to increase as the square root of elapsed time (t). The slope of the line of best fit of these points was taken as the sorptivity value. The sorptivity values of blended concrete specimens after 28 and 90 days of moisture curing were calculated by the following formula (Equation 2) suggested by Stanish et al. (1997).

$$i = \frac{S}{\sqrt{t}} \tag{2}$$

### 2.2.3 Microstructural Investigations

In order to carry out microstructural investigation of the concrete samples, a JEOL Scanning electron microscopy (SEM) equipment with energy dispersive spectrometry capabilities shown in Figure 2, was used. The equipment model comes with energy dispersive spectrometer (EDX), and allows high resolution identification of elements and compounds present in prepared 2-D cross-sections of aggregate samples to be obtained. The SEM studies were conducted on broken samples of concrete samples cured for 28 days in order to investigate their morphological structures. The samples were collected from the innermost core of the concrete cubes after crushing. Each of the samples are then prepared into appropriate size that fit into the specimen chamber of the SEM equipment. Samples were then freeze-dried, cut and polished and examined on the backscattered electron (BSE) mode of the SEM equipment. The range of scale used in SEM analysis was 5µm with the resolution of x 10000.


Figure 2: The SEM Equipment used for Microstructural Investigation of Samples

## 3.0 Results and Discussion

## 3.1 Materials Characterization

Some of the properties of the aggregates are shown in Table 2.

Table 2: Some Physical Properties of the Aggregates

| | Sand | Gravel |
|---|---|---|
| Specific Gravity | 2.63 | 2.67 |
| Bulk Density (kg/m3) | 1666.67 | 1641.67 |
| Water Absorption (%) | 2.00 | 2.00 |
| Moisture Content (%) | 0.00 | 0.00 |
| Coefficient of curvature (Cc) | 0.88 | 0.98 |
| Coefficient of uniformity (Cu) | 3.00 | 2.43 |

It was observed that the specific gravities of the sand and gravel are in the range of 2.63 and 2.67 respectively. Specific gravities of natural aggregate according to Gambhir (2013) should be between 2.5 and 2.8. Thus, both the sand and gravel used in this investigation can be considered as natural aggregate. Additionally, the densities of the aggregate ranged from 1280 to 1920 kg/m$^3$; for water absorption, it is 0 to 8% and the moisture content is 0 – 2% for sand and 0 - 10% for gravel. All these vales obtained for density, water absorption and the moisture content of both the sand and gravel fell between the ranges used in normal concrete (ACI, 1999). Also, the coefficient of curvature for both are close to 1, thus indicating that both are well-graded, while the coefficient of uniformity of less than or equal to 4 recorded for both sand and gravel suggest that both are uniformly graded (Iowa, 2020). Overall conclusion of all of these is that the materials are good for concrete production.

### 3.2 Coefficient of Water Absorption

The results of the coefficient of water absorption are presented in Table 3.

Table 3: Coefficient of Water Absorption for the Concrete Specimens

| Water/Cement Ratios | Ka (m$^2$/s) x 10$^{-8}$ | | | |
|---|---|---|---|---|
| | 28 days | | 90 days | |
| | 32.5R | 42.5R | 32.5R | 42.5R |
| 0.40 | 5.2 x $10^{-9}$ | 2.01 x $10^{-10}$ | 2.5 x $10^{-9}$ | 3.73 x $10^{-9}$ |
| 0.50 | 7.72 x $10^{-9}$ | 7.72 x $10^{-10}$ | 1.9 x $10^{-9}$ | 2.78 x $10^{-10}$ |
| 0.60 | 7.72 x $10^{-9}$ | 2.78 x $10^{-10}$ | 4.9 x $10^{-9}$ | 1.51 x $10^{-10}$ |

At 28 days, the values of coefficient of water absorption obtained for cement grade of 42.5R were lower than that of grade 32.5R at all the water/cement ratios. Since coefficient of water absorption is a measure of permeability, it can be said that concrete specimens produced with cement grade of 42.5R are less permeable in comparison to concrete produced with cement grade 32.5R. This can be due to the fact that, at the early ages, more C-S-H gel are produced by concrete with cement grade 42.5R, which has a densifying effect. However, at 90 days curing period, concrete specimens produced with grade 32.5R cement displayed low permeability at water/cement ratio of 0.40 than that of concrete produced with grade 42.5R cement. At the water/cement ratios of 0.50 and 0.60, the concrete specimens produced with grade 42.5R cement have smaller values of coefficient of water absorption, hence low permeability. Furthermore, it can be seen from Table 3 that the observed values of coefficient of water absorption reduced with curing age. This suggested that prolonged curing of concrete samples resulted in more production of C-S-H gel with pore-refining effects leading to reduction in permeable voids.

### 3.3 Sorptivity

Sorptivity test measures the rate of absorption of water by capillary suction of unsaturated concrete placed in contact with water; no head of water exists (Hall, 1989; Dias, 2000 and Neville, 2011). The results of the sorptivity investigation are presented in Table 4.

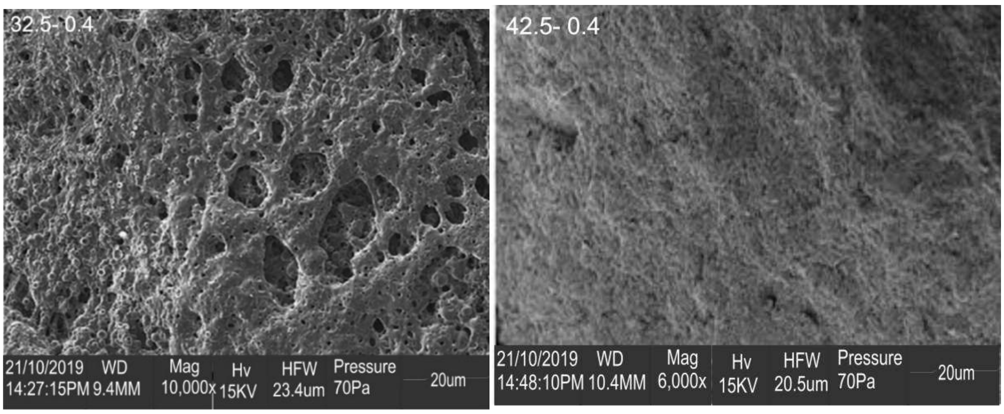
Table 4: Sorptivity for the Concrete Specimens ($10^{-2}$ mm/s$^{1/2}$)

| Water/Cement Ratio | 28 days | | 90 days | |
|---|---|---|---|---|
| | 32.5R | 42.5R | 32.5R | 42.5R |
| 0.40 | 0.048 | 0.097 | 0.073 | 0.058 |
| 0.50 | 0.034 | 0.026 | 0.032 | 0.018 |
| 0.60 | 0.032 | 0.022 | 0.031 | 0.015 |

At 28 days of curing, with the exception of concrete samples with water/cement ratio of 0.40, the values rate of absorption of water obtained for grade 42.5 R specimens were lower than that of grade 32.5 specimens. Also, the values of the rate of absorption of water reduced with water/cement ratio for all the concrete specimens. This is ostensibly due to higher water content in concrete samples at higher water/cement ratios. At 90 days of curing, the values of the rate of water absorption for concrete samples produced with grade 42.5R were lower than that produced with grade 32.5 R specimens at all the water/cement ratios. Furthermore, the sorptivity values at 90 days reduced with water/cement ratios for all the specimens, due to higher water content at higher water/cement ratios. It also ought to be observed that sorpitivy at 90 days were lower than sorptivity values at 28 days for all the specimens. It is known that prolong curing results in the densification of the concrete matrix, thus inhibiting the rate of water absorption by the specimens.

### 3.4 Microstructure of Concrete Specimens

The scanning electron microscopy (SEM) images of the concrete specimens at 28 days of curing are shown in Figure 3. Microstructure is usually explained in terms of distribution of pores, sizes of pores and interconnectivity of the pores. Figure 3 shows the images of the microstructure of concrete produced with cement grades 32.5 R and 42.5R at the same water/cement ratio. It could be observed that the microstructure of concrete produced with grade 32.5R at 0.40 water/cement ratio consisted of big pores or voids in comparison to grade 42.5R concrete. This suggests that concrete produced with grade 32.5R are likely to be porous and less durable than concrete produced with grade 42.5R. This observation is consistent with the results of permeability measured through the rate of water absorption test as presented in Table 3. This pattern can also be observed in the images of the microstructure of concrete samples produced with water/cement ratio of 0.50 for both grades of cement. In particular, the very large pores, from the images of concrete produced with grade 32.5R cement is very glaring. There is also an evidence in the microstructure of weak interfacial zone (ITZ) between the vicinity of aggregate and the hydrated products. This ITZ is the weakest region in concrete due to its higher porosity resulting from the poor packing of cement particles (Mehta and Monteiro, 2006). Although, the images of concrete samples produced with grade 42.5 R cement and with water/cement ratio of 0.60, showed a microstructure consisting of large pores, there is evidence that the pores are not connected. Figure 4 showed the EDX spectrum of the samples from which the elemental counts presented in Table 5 was extracted for clarity. Silicates and calcium are the constituents of the strength-forming products of hydration. Their counts, through the EDX spectrum and the ratio of calcium to silicate are strength indicator. From Table 5, the ratio $\frac{C}{S}$ for concrete produced with 32.5R grade of cement increase marginally with increase in water/cement ratio. However, for concrete produced with grade 42.5R cement, the ratio increases with water/cement ratio.



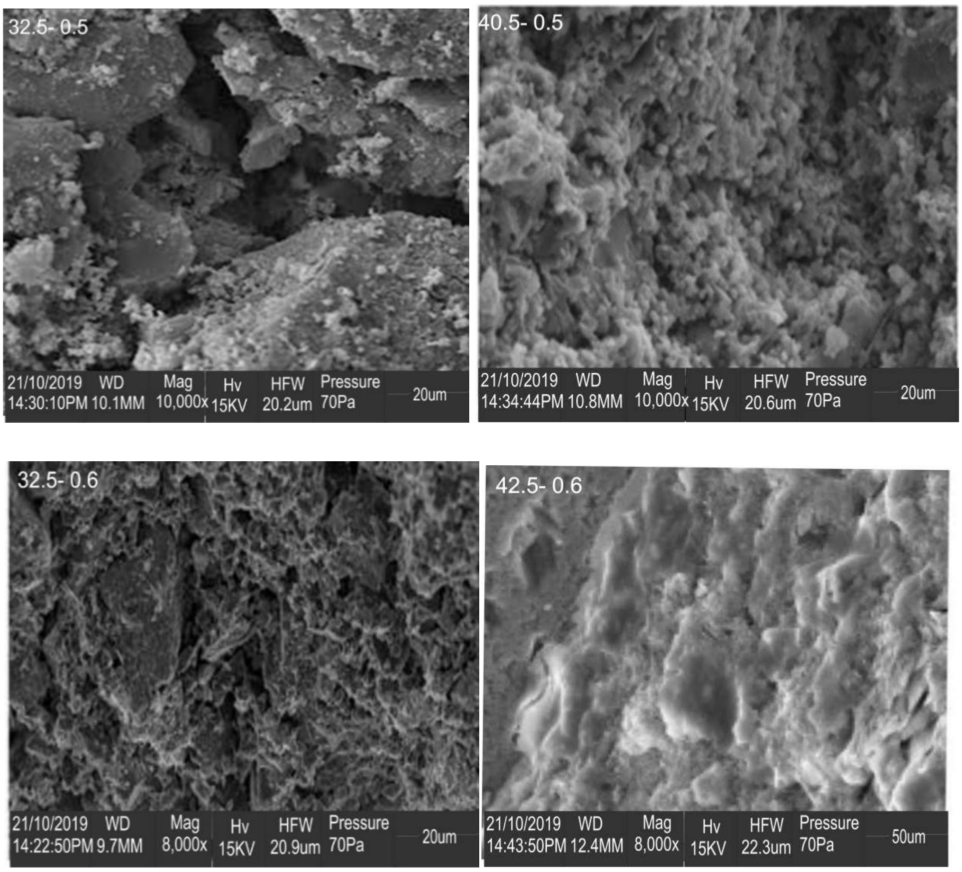
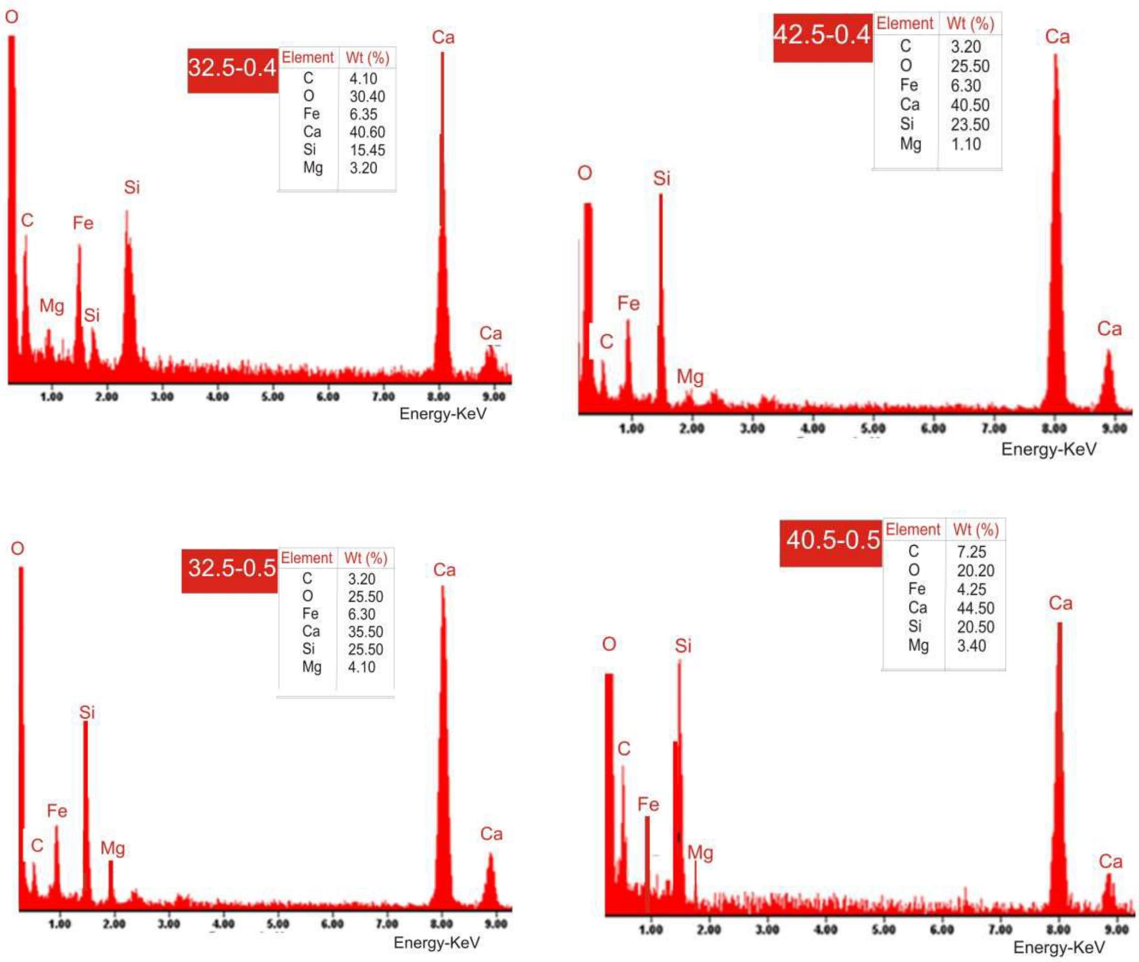



Figure 3: The SEM Micrograph of the Concrete Samples at 28 days of Curing



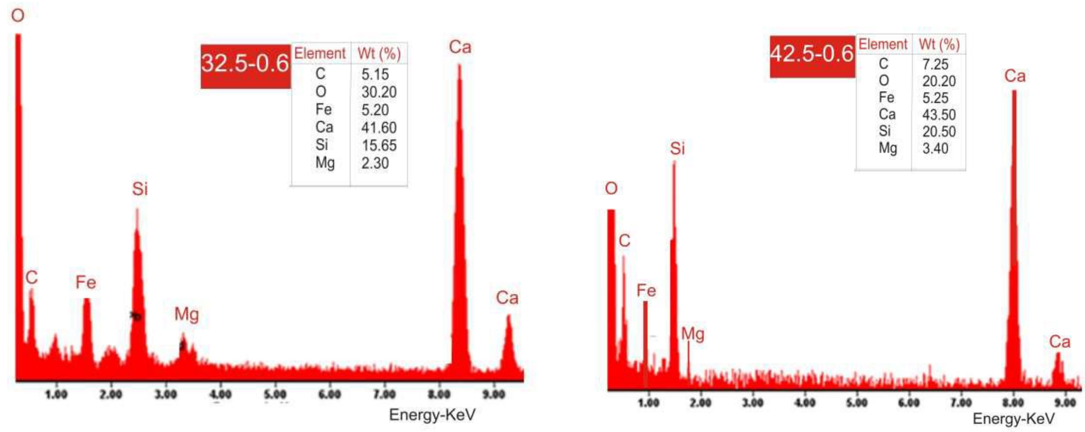



Figure 4: EDX Spectrum of the Concrete samples at 28-day curing

Table 5: The *C/S* Ratio of the Mixes

| Concrete Mix | Mix Designation | Ca (C) | Si (S) | $\frac{C}{S}$ |
|---|---|---|---|---|
| 32.5 | M04 | 40.60 | 15.45 | 2.63 |
| | M05 | 35.50 | 25.50 | 1.39 |
| | M06 | 41.60 | 15.65 | 2.66 |
| 42.5 | M04 | 40.50 | 23.50 | 1.72 |
| | M05 | 43.50 | 20.50 | 2.12 |
| | M06 | 44.50 | 20.50 | 2.17 |

According to Nazari et al. (2011), increasing ratio means increasing strength-forming activities. This conclusion is consistent with the results of sorptivity presented in Table 4.

## 4. Conclusions and Recommendations

### 4.1 Conclusions

From the analysis of the results of this investigation, the following conclusions can be made.
The values of coefficient of water absorption obtained for concrete specimens produced with grade 42.5 R are lower than specimens produced with cement grade 32.5 R at 28-day curing. At 90 days curing age, concrete specimens produced with grade 32. 5R cement had lower coefficient of water absorption at lower water/cement ratio of 0.40. The microstructure of concrete produced with 32.5R are more porous than those produced with grade 42.5R cement at 28-day curing .

### 4.2 Recommendations

The concrete specimens produced with cement grade 32.5R resulted in low durability, especially at the structural age of 28 days curing. Since both of them are available in the Nigeria market and both are equally widely used, the need to improve the durability performance of concrete made with grade 32.5R cannot be overemphasized. The use of highly reactive pozzolans like rice husk ash, that abounds in Nigeria (Joel, 2010, Oyekan and Kamiyo, 2011 and Abalaka, 2013), should be given consideration by incorporating it into concrete mix with cement grade 32.5R. Incorporating pozzolans in the concrete with 32.5R cement grade, according to Büyüköztürk and Lau (2020) will improve the concrete durability performance by strengthening the hydrated cement paste through the adding of mineral additives. This will help in generating a crystalline C-S-H gel with lower gel porosity, compared to a conventional amorphous C-S-H gel. Reduction in the porosity of the paste, which translate to low permeability and improved durability, will result.

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