



Characterization of Some Portland Limestone Cements in Nigeria Using Fourier-Transform Infrared Spectroscopy (FTIR)

Ibrahim Mohammed Danchuwa¹, Mustapha Zannah Abatcha¹, Yazid Aliyu¹, Mohammed Buba Yawale¹, Walid Haruna Abubakar², Abdulhameed Umar Abubakar^{1*}

¹Department of Civil Engineering,
Modibbo Adama University, Yola, 2076, NIGERIA

²Department of Chemistry,
Gombe State University, Gombe, 127, NIGERIA

*Corresponding Author

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Abstract: In Nigeria, the most widely used cement strength class are 32.5 and 42.5 Portland limestone cement. However, due to lack of awareness, scientists and researchers are reporting the use of PLC as Ordinary Portland Cement (OPC). The study presents the characterization of PLC using absorption spectroscopy as well as oxide composition. Ratio between 7 - and 28 - day strengths of some selected PLCs are presented. It is seen that the Brands investigated conforms to the specifications of Portland composite cements in compliance with ASTM C150 and NIS 444-1. Absorption spectrum for the two brands were similar to that of OPC and exhibited similar characteristics. Concrete strength was within the designed concrete specification, and the ration between the two curing ages is 0.6 for both brands.

Keywords: Portland limestone cement, FTIR, absorption spectroscopy, 28-day strength, concrete, cement

1. Introduction

Portland cement is a hydraulic binder that combines with aggregates in the presence of moisture to produce concrete. This brand of cement is the most used in the world, with composition depending on the intended usage. ASTM C150 (2014) defines Portland cement as cement that hardens by reacting with water but also forms a water-resistant product produced by pulverizing clinker consisting of one or more of the forms of calcium silicates. The binding quality of Portland cement paste is due to the chemical reaction between the cement and water (Yahaya et al., 2014). Portland cement is a mixture of many chemical compounds, the major ones make up 90 % or more of the weight: tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrite (Neville, 2005).

Concrete versatility increases according to variables such as quality of constituent materials (cement aggregates, water and admixtures), skill of the manufactures, management placement procedures and environmental issues reported that in concrete, aggregates and paste are the major factors that affect the strength of concrete (Abdullahi, 2015; Raheem & Bamigbove, 2013). The lower the porosity of the cement paste, the stronger the concrete. The degree of cement hydration which is a function of water to cement ratio has a direct impact on the porosity and consequently on the strength. The richness of the mix is one of the factors that affect the rate of strength development in concrete mix

*Corresponding author: abdulhameed.umar@mau.edu.ng

and is a direct function of the quality and quantity of the cementitious material. Knowledge of the rate of reaction between cement and water is important because it determines the rate of hardening. Once the concrete has been placed and finished, rapid hardening is desirable. Gypsum added at the cement mill when clinker is ground, acts as a regular of the initial rate of setting of Portland cement. Other factors that influence the rate of hydration include cement fineness, admixture, and amount of water added and temperature of the material at the time of mixing.

Variability in terms of strength of some Portland cements around the Federal Capital, Abuja was evaluated using the following brands: Burham, Ashaka, Dangote, Elephant, Sokoto and Lion cements. It was reported that the highest strength at 28 days was obtained in Burham cement, and the lowest was in Lion cement (Tsado, 2010). Results of the comparative study of some brands (Ashaka, Sokoto, Dangote, Rhino and Elephant) on physical and compressive strength properties to BS 4550 specification showed the brands satisfied the requirements. However, Elephant cement fell short of the target strength for a cement with 32.5N strength class at 28 days (Joseph et al., 2014).

In the work of Ibadode et al., (2017), a comparative study of compressive strengths and densities of concrete with some brands (Dangote, Eagle, Ibeto, Lafarge, Purechem and Unicem) of Portland cements in Nigeria. Results of 28-day strengths for grade M10 and M15 concretes were higher than the recommended values for British Standard Institution (BSI). A similar result was reported for Dangote, Elephant, Burham, Diamond and Purechem, with the highest strength recorded by Dangote cement at 28 days and the lowest by Purechem (Joseph & Raymond, 2014).

A study was undertaken in Anambra State, Nigeria on some brands (Bua, Superset, Dangote and Unicem) to evaluate the 28 days' compressive strength of concrete. Workability and strength requirements were met, with Superset and Unicem with the highest and lowest strength in the study (Bert-Okonkwo et al., 2019).

In Nigeria, there are two types of cements available in the market, 32.5N, 32.5R, 42.5N and 42.5R designation for ordinary and high early strength. However, the most common type of cements available is the Portland-limestone cement with strength class 32.5 and 42.5 respectively. Portland limestone cement is produced by the intergrinding or the addition of limestone. This has many benefits because of the lower specific surface area of the limestone (Opoczky, 1992). As such, limestone increases the fineness of the target cement (Hooton et al., 2007). The composition is such that it conforms to BS EN 197-1 (2011) containing between 6 - 20% by mass of limestone with total organic content net exceeding 0.50% by mass of strength class 32.5N. According to [12] BS EN 197-1 allows for cement to contain 5% minor additional components (MAC) which are typically limestone or cement raw meal. In [1], it is reported that "the limestone shall be naturally occurring, consisting of at least 70% by mass of one or more of the mineral forms of calcium carbonate." The standard designate PLC based on total organic content (TOC) as L (not exceeding 0.20% by mass) and LL (not exceeding 0.50% by mass) class, and also, calcium carbonate (CaCO_3) should be greater than or equal to 75% (BS EN 197-1, 2011).

In terms of reactivity, it is reported in (Matschei et al., 2007) as cited in Hooton et al., (2007) that at low concentration, limestone reacts to form carboaluminates phases, contrary to the commonly held believe in the past that it is an inert filler. The work of Tsivilis et al. (1999) reported that in terms of performance, there is no clear agreement on the impact of fine limestone on strength development. However, in terms of water demand, there is a positive effect.

The work of Adewole et al. (2014) highlighted a common problem with early research on concrete and cements with strength class 32.5 and 42.5 where the authors failed to distinguish between Ordinary Portland cement (OPC) and Portland - Limestone cement (PLC) that is commonly available in the Nigerian market. This they attributed to lack of awareness of the different strength grades in the market resulting in their failure to indicate grade/strength class of cement used in their research. Their conclusion being that there is no ordinary Portland cement commercially available in the Nigerian market. A similar assertion was made by Uko (2018) that "there are no OPCs available in Nigeria, and so site practices based on such assumptions are no longer valid. The probability of getting a 25 MPa strength mix ratio using grade 32.5N cement is very close to zero. Portland Limestone Cements in circulation in Nigeria now are blended cement clinker with raw limestone before grinding with gypsum, a retarder."

On that premise, this study is undertaken to characterize the PLC in terms of spectrum analysis, as well as assessing the strength of PLC with strength class 32.5N at 7- and 28-days strength.

2. Materials and Methodology

2.1 Materials

The cement brands selected are the most widely used Portland limestone cements, strength class 32.5N in Nigeria. Coarse aggregates used were crushed granite rock free of dust and other impurities with a maximum nominal size of 20 mm. The fine aggregate used was sharp river sand obtained from a river source. It was free of silt or any deleterious substances, and satisfied the requirements of ASTM C33/C33M-16e1 (2016) and ASTM C136 (2014) respectively. Portable water was used during the manufacturing concrete conforming to BS EN 1008 (2002) specifications.

2.2 Experimental Set -Up/Procedures

The two brands of PLC were used to design a 1:2:4 concrete using cement strength class 32.5N. Sieve analysis was conducted on the river sand used as fine aggregate in conformity with (ASTM C136, 2014), and a 100g sample of

cement was measured for each brand to measure the fineness of the cement sample using 90 μ m. A weighing balance with accuracy to the nearest 0.0001g was used for that purpose. Crushed graded granite aggregate obtained from hardware store with a maximum coarse aggregate size of 20 mm was utilized. Standard Method EDXRF Analyzer was used for the determination of oxide composition of the PLC, and PerkinElmer Spectrum Version 10.03.09 was utilized for FTIR Spectroscopy analysis.

A mix containing PLC, natural sand and crushed rock aggregate was designed for a compressive strength of about 25 MPa at 28 days with a slump range of 25-75 mm. Workability tests in the form of compacting factor to BS EN 12350-4 (2019), and slump tested to ASTM C143 (2016), respectively. Fresh concrete mixes were casted in 150mm cube steel moulds, covered and placed in the curing room based on the specifications of ASTM C192 (2015), and then stripped and placed in the curing tank 24 hours later until testing age. Concrete compressive strength (f_c) was determined at 28 days on 150 mm side cubes in accordance with BS EN 12390-3 (2009) with a loading rate of 0.5 MPa/s.

3. Results and Discussion

Grain size analysis for the fine aggregates utilized is presented in Table 1, and percentage of fineness of the two brands of the cement (X and Y) where 31% and 42%. An indication that the former is finer than the latter, and this conforms with the work of Tsvivilis et al. (2003) on intergrinding with limestone that the entire size distribution is affected at higher limestone content.

Table 1 - Sieve analysis for fine aggregate

Sieve sizes (mm)	9.5	4.75	2.00	1.18	0.60	0.30	0.15	0.075
Percentage Passing	100	99	89	73	42	14	3	1

Chemical composition analysis for the two brands presented in Table 2 indicated they conform to the specification of [1] for composite cements with the exception of loss on ignition, which is slightly above the maximum permissible limit. Conformity was also observed with NIS 444-1 (2003). This has been corroborated by Adewole et al. (2014).

Table 2 - Chemical composition analysis of brands used

Major Components	Brand X	Brand Y	ASTM C150	NIS Standard
SiO ₂	20.61	20.60	20 min	18 - 24
Al ₂ O ₃	3.68	4.65	6.0 max	2.6 - 8.0
Fe ₂ O ₃	4.58	3.02	6.0 max	1.5 - 7.0
CaO	60.80	62.68	A	59 - 67
SO ₃	2.83	2.96	3.0 max	≤ 3.5
LOI	3.12	2.95	3.0 max	≤ 5.0

^ANot applicable

Workability is presented in the form of slump and compacting factor in Table 3. Results obtained are within the designed values. This is in line with medium workability as defined by Neville (2005). For the same water/cement ratio, it is seen that Brand X mix is more fluid than Y.

Table 3 - Workability

Type of Portland limestone cement 32.5N	Water/cement ratio	Average Slump(mm)	Average Compacting factor
Brand X	0.5	51	0.88
Brand Y	0.5	38	0.74

To analyses and characterize the different phases of PLC commonly available, spectroscopy was conducted on the Brands utilized. This is a graphical fingerprint showing intensity (%T) against wave number (cm⁻¹). The left-hand side of the graph shows the functional group region, while the right-hand side is called “fingerprint region.”

In Fig. 1 and 2, two brands of Portland limestone cements were tested using FTIR spectroscopy. Brand X had a peak of 3844 cm⁻¹ while Brand Y 3444 cm⁻¹. The wide absorption peak in Brand X is an indication of large amount of water molecule Shrestha (2018). Functional group resulting from the absorption peaks of the brand is O - H. Absorption peaks in the range of 2360 - 2325 cm⁻¹ are carbonate radicals as reported in Moenke (1974). This is captured in both brands.

In Brand X, a small diminished absorption peak is noticeable at 1621 cm⁻¹ corresponding to 24 %T, but absent in Brand Y. This has been attributed to the H - O - H deformation mode due to absorbed molecule of water. Absorption peak in the range of 1600 - 1700 cm⁻¹ is normally attributed to bending mode of H₂O molecule as explained earlier. Absorption peak in the region of 2060 - 1600 cm⁻¹ are characterized by silicates.

Peaks in the range of 1194 - 1122 cm⁻¹ in Brand X and 1101 cm⁻¹ in Brand Y is due to sulfate ions, while calcium carbonate polymorphs in the range of 1504 - 1422 cm⁻¹ in Brand X and 1427 cm⁻¹ in Brand Y are visible. This has been corroborated by Hughes et al. (1995).

A sharp peak at 917 cm⁻¹ in Brand X and 921 cm⁻¹ in Brand Y are characterized by C₃S spectrum due to stretching Si - O. this is closely followed by a band of 713 to 455 cm⁻¹ in X and 712 - 520 cm⁻¹ in Y respectively. C₃A spectra is represented at 917 - 657 cm⁻¹ and 874 - 656 cm⁻¹ for Brand X and Y respectively. IR Spectra for Brand X and Y PLC is similar to that of OPC, and agree with results presented in Shrestha (2018) and is corroborated by Fernández-Carrasco et al. (2012) for the main compounds (C₂S, C₃S, C₃A, and C₄AF) of Portland cements.

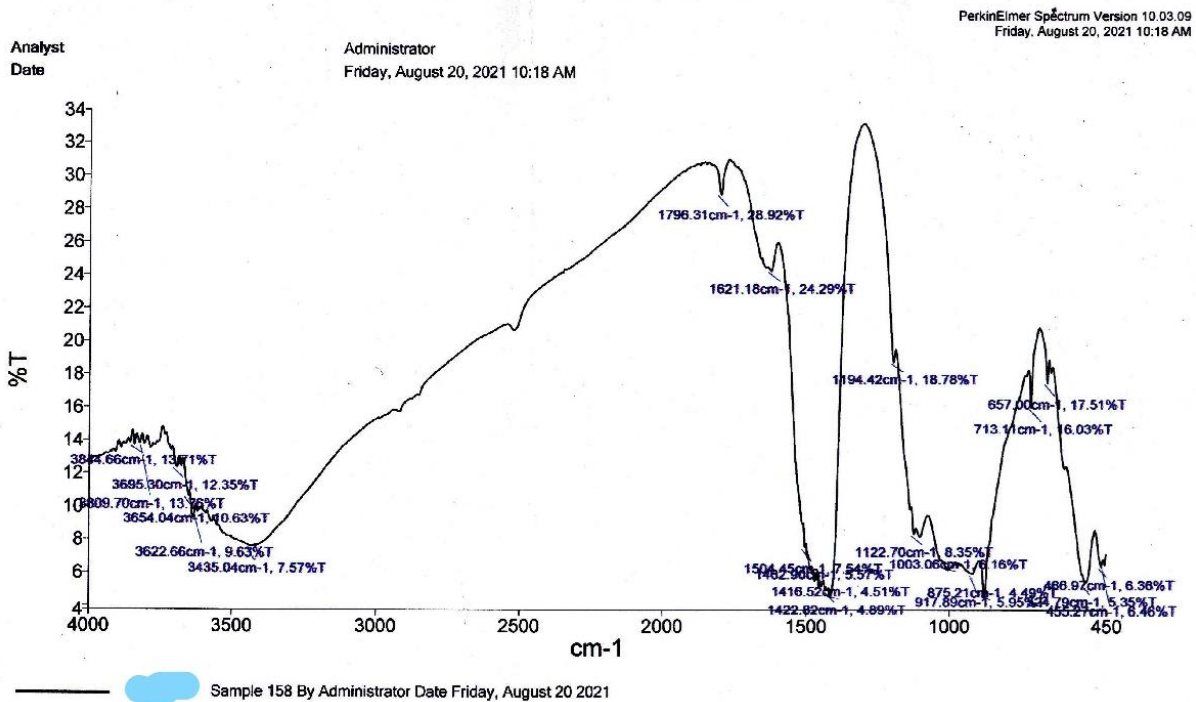


Fig. 1 - FTIR of Brand X

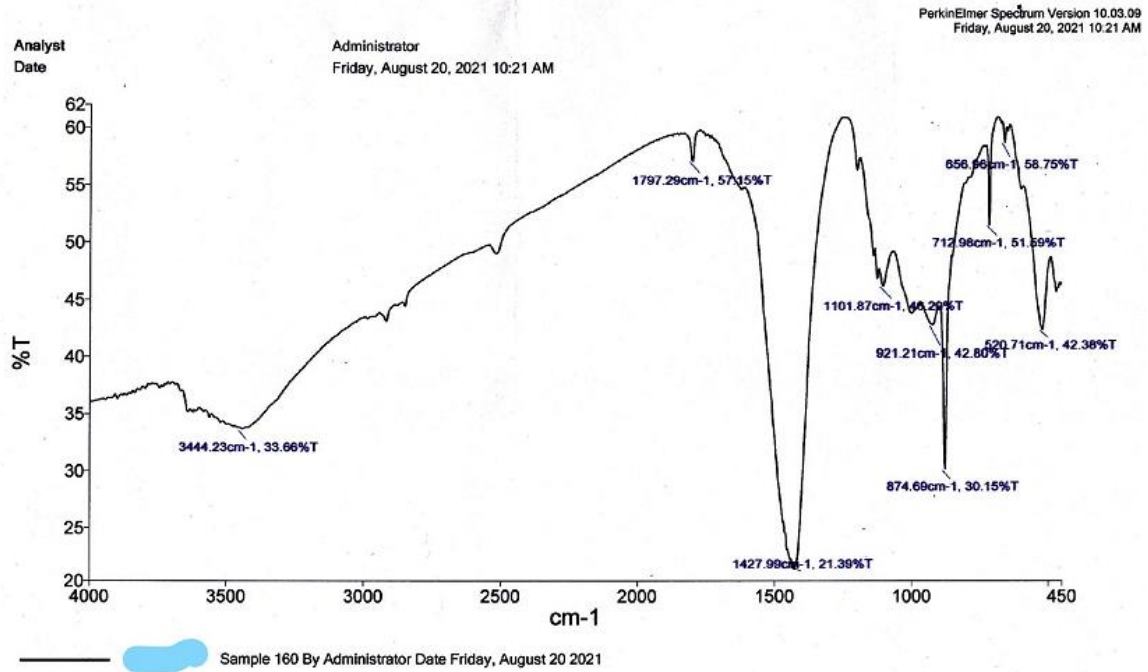


Fig. 2 - FTIR of Brand Y

Table 4 to Table 7 show the average compressive strength for both cement grades at the ages of 7 and 28 days respectively. Difference between the two brands in terms of strength for both curing ages are 2.7 MPa and 4.12 MPa with Brand X having higher strength in each case. The range of sample size for 7 days curing age are 2.33 MPa and 1.49 MPa, and at 28 days, 0.29 MPa and 1.62 MPa for Brand X and Y respectively. It can be seen that in some cases, the results are much closer to each other, for example 28 days’ strength in Brand X where all the data are within 1 MPa. This limits the variability in terms of data, as concrete is a material that is known to have some form of variability due to heterogeneity of the constituents’ materials. An interval plot was produced from the results of the Confidence Interval in Fig. 3 presented in Table 8 - 9 generated from the mean and standard deviation of the sample.

Table 4 - Compressive strength of Brand X at 7 days curing age

S/N	Weight Kg	Volume M ³	Density Kg/m ³	Load KN	Area mm ²	Compressive strength, N/mm ²
1	8.194	3.375x10 ⁻³	2427.8	387.59	22500	17.23
2	8.299	3.375x10 ⁻³	2458.9	390.5	22500	17.36
3	8.279	3.375x10 ⁻³	2453.0	405.51	22500	18.02
4	8.287	3.375x10 ⁻³	2455.4	389.03	22500	17.29
5	8.408	3.375x10 ⁻³	2491.3	440.07	22500	19.56
Average	8.263		2457.23	402.54		17.89

Table 5 - Compressive strength of Brand Y at 7 days curing age

S/N	Weight Kg	Volume M ³	Density Kg/m ³	Load KN	Area mm ²	Compressive strength, N/mm ²
1	8.258	3.375x10 ⁻³	2446.8	343.45	22500	15.26
2	8.140	3.375x10 ⁻³	2411.8	326.78	22500	14.53
3	8.208	3.375x10 ⁻³	2432.0	346.93	22500	15.42
4	8.459	3.375x10 ⁻³	2506.4	362.58	22500	16.11
5	8.718	3.375x10 ⁻³	2583.1	328.97	22500	14.62
Average	8.357		2476.06	341.74		15.19

Table 6 - Compressive strength of Brand X at 28 days curing age

S/N	Weight Kg	Volume M ³	Density Kg/m ³	Load KN	Area mm ²	Compressive strength, N/mm ²
1	8.344	3.375x10 ⁻³	2472.3	656.41	22500	29.17
2	8.260	3.375x10 ⁻³	2447.4	662.90	22500	29.46
3	8.434	3.375x10 ⁻³	2498.9	658.82	22500	29.28
4	8.492	3.375x10 ⁻³	2516.1	670.09	22500	29.78
5	8.364	3.375x10 ⁻³	2498.2	661.20	22500	29.39
Average	8.379		2486.58	661.88		29.42

Table 7 - Compressive strength of Brand Y at 28 days curing age

S/N	Weight Kg	Volume M ³	Density Kg/m ³	Load KN	Area mm ²	Compressive strength, N/mm ²
1	8.118	3.375x10 ⁻³	2405.3	553.04	22500	24.58
2	8.122	3.375x10 ⁻³	2406.5	587.51	22500	26.20
3	8.008	3.375x10 ⁻³	2372.7	562.41	22500	25.00
4	8.125	3.375x10 ⁻³	2407.4	559.45	22500	24.86
5	8.137	3.375x10 ⁻³	2410.9	584.15	22500	25.96
Average	8.102		2400.56	569.31		25.30

Table 8 - 95% confidence interval of the mean for 7 days

Factor	N	Mean	StDev	95% CI
Brand X 7 Days	5	17.892	0.985	(17.033, 18.751)
Brand Y 7 Days	5	15.188	0.645	(14.329, 16.047)

Table 9 - 95% Confidence interval of the mean for 28 days

Factor	N	Mean	StDev	95% CI
Brand X 28 Days	5	29.416	0.231	(28.868, 29.964)
Brand Y 28 Days	5	25.320	0.715	(24.772, 25.868)

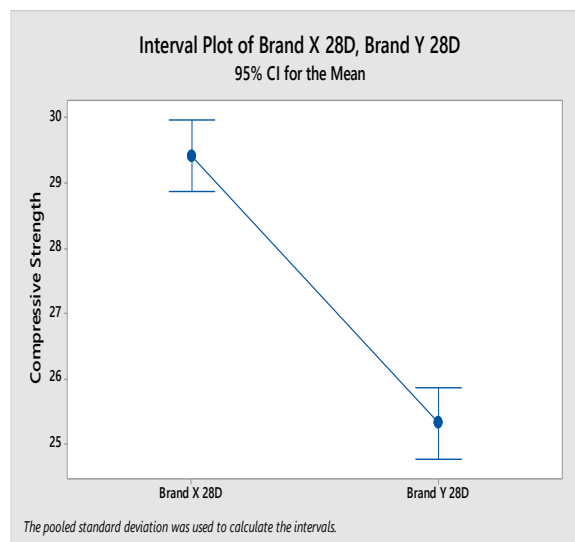


Fig. 3 - Interval plots of 95% CI of the mean for Brand X and Y at 7 and 28 days

In Table 10 - 13, descriptive statistics is presented, and the Standard Error of the Mean (SE Mean) describes the phenomenon showing the degree of variability of the sample mean. Sample results in Table 4 presented the highest SE Mean (0.441) due to the variation between the maximum and minimum values in Table 8. This effect is well depicted in Fig. 4a and 4b where the it could be seen that the median in Brand X at 7 days and Brand Y at 28 days are very close to the maxima in each case. On the other hand, in Brand Y at 7 days and Brand X at 28 days, both are at an intermediate point, close to the mean of the sample data. Generally, boxplots show agreement or closeness or spread within the range.

Table 10 - Descriptive statistics for 7 days Brand X

Variable	N	N*	Mean	SE Mean	St Dev	Minimum	Q1	Median	Q3	Maximum
7 days	5	0	17.892	0.441	0.985	17.230	17.260	17.360	18.790	19.560

Table 11 - Descriptive statistics for 28 days Brand X

Variable	N	N*	Mean	SE Mean	St Dev	Minimum	Q1	Median	Q3	Maximum
28 days	5	0	29.412	0.100	0.224	29.170	29.225	29.390	29.610	29.760

Table 12 - Descriptive statistics for 7 days Brand Y

Variable	N	N*	Mean	SE Mean	St Dev	Minimum	Q1	Median	Q3	Maximum
7 days	5	0	15.188	0.289	0.645	14.530	14.575	15.260	15.765	16.110

Table 13 - Descriptive statistics for 28 days Brand Y

Variable	N	N*	Mean	SE Mean	St Dev	Minimum	Q1	Median	Q3	Maximum
28 days	5	0	25.320	0.320	0.715	24.580	24.720	25.000	26.080	26.200

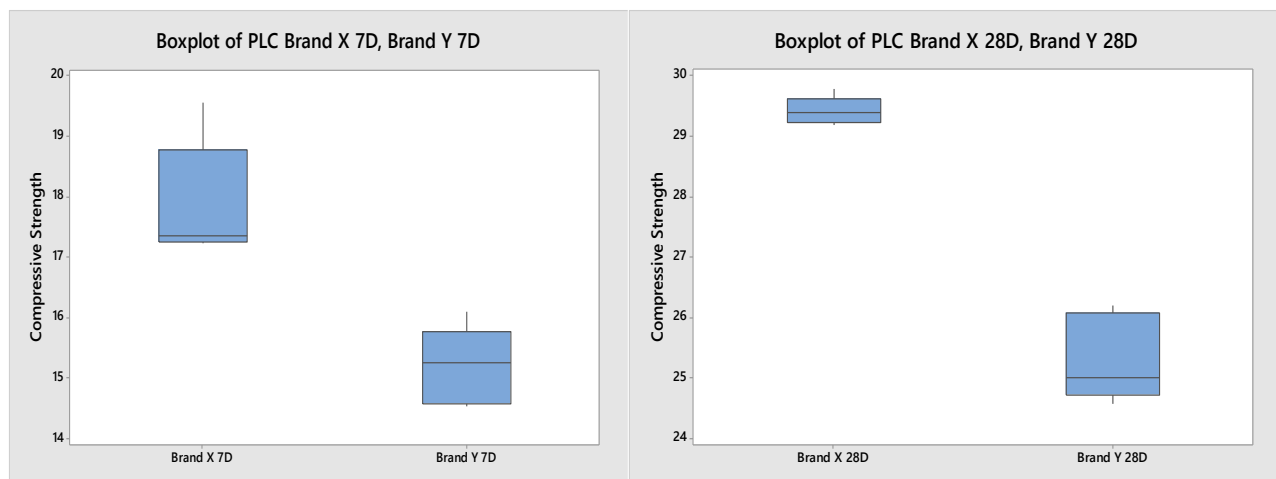


Fig. 4 - Boxplots for Brand X and Y at 7 and 28 days

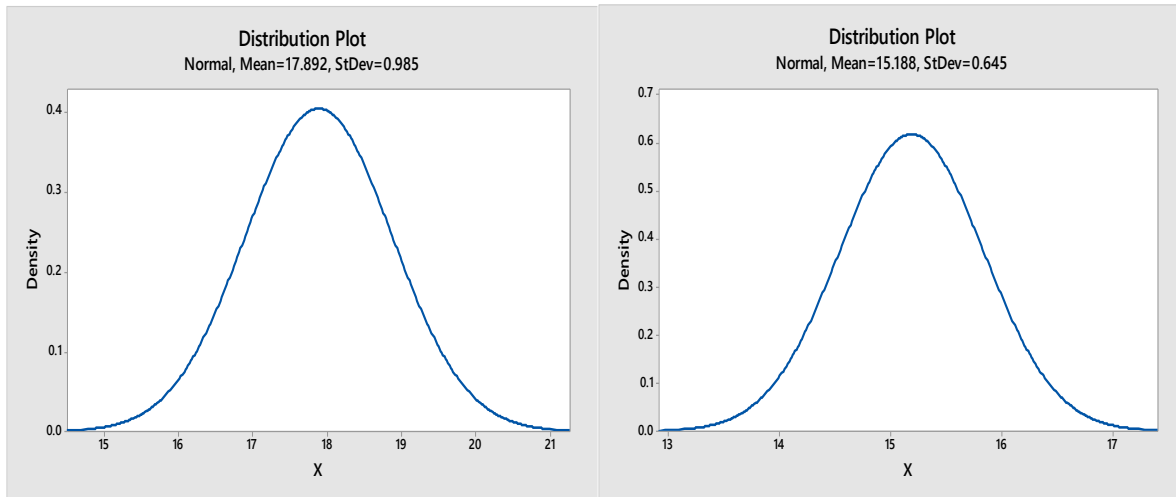


Fig. 5 - Probability distribution plot for Brand X and Brand Y cements mean compressive strength at 7 days curing age

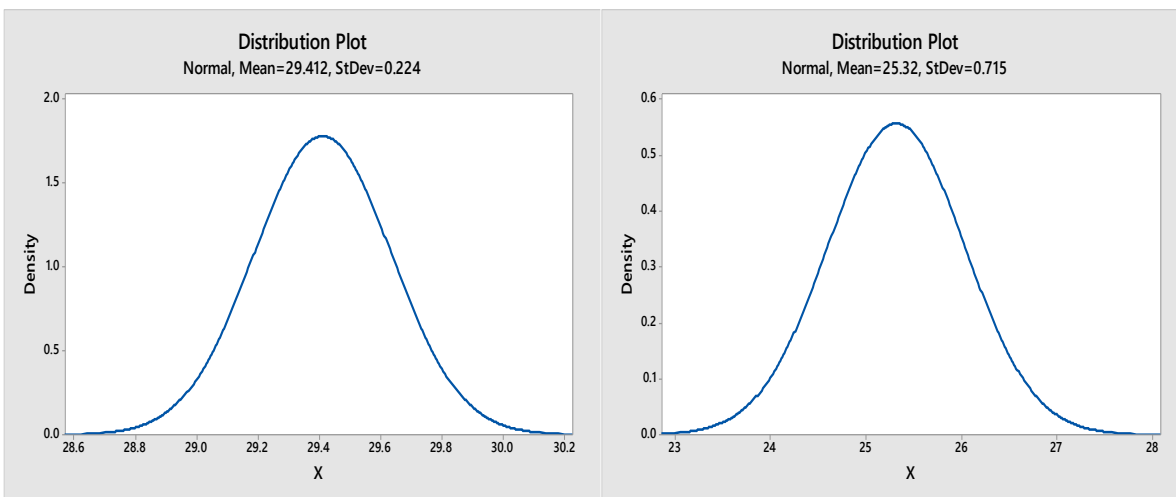


Fig. 6 - Probability distribution plot for Brand X and Brand Y cements mean compressive strength at 28 days curing age

In Fig. 5 - 6, the probability plots are presented to check the normality of the results. It is seen that the bell-shape distribution is evident and validating the results of Table 8 - 9 confidence interval values.

From Table 14, it can be seen that there is an increase of 11.52 MPa and 10.13 MPa in terms of mean compressive strength between the ages of 7 and 28 days curing for Brand X and Y respectively. Brand X has a higher value of compressive strength in each case. The ratio between the two curing ages for each Brand is 0.6.

Table 14 - Mean compressive strength for Portland limestone cements

Type of Portland limestone cement	Mean compressive strength (N/mm ²)	Mean compressive strength (N/mm ²)	Standard cube strength (N/mm ²)	Ratio btw 7 & 28 days	Remark
	7days	28days	28days		
Brand X	17.90	29.42	25.00	0.61	Satisfies min cube strength
Brand Y	15.19	25.32	25.00	0.60	Satisfies min cube strength

4. Conclusions

Two brands of Portland limestone cements were used to design a concrete with a mix ratio of 1:2:4 using cement strength class 32.5N as well as spectrum analysis on a sample of PLC, and the following conclusions were drawn: Portland cements available in Nigeria are indeed PLC based on the chemical/oxide composition analysis satisfying the requirements of composite cements and SON - NIS regulation. Characterization using FTIR spectrum analysis indicated that the functional groups in PLC are similar to those obtained in OPC, and the main oxides are located at similar spectra as in OPC. It is also seen that it is possible to obtain concrete with compressive strength in the range of the designed mix using cement strength class 32.5N. Future study should widen the scope to a large number of cement brands as well as statistical analysis.

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