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Original Article

Levels and quality impacts of additives in cement brands in Nigeria

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ABSTRACT

Ten cement samples (labeled A–J) commonly used for construction works in Nigeria were taken from different parts of the country. Their physicochemical parameters were tested in accordance with the Nigerian Industrial Standard, NIS 445-2003, and other associated standards. From the results obtained, all the samples, except B, indicated presence of various levels of additives. Samples (C, I, H, G, A, J, and F) with additive levels manifesting in the form of loss on ignition (LOI) between 5% and 11.2% were still able to satisfy the standard requirements for strength classes of 32.5N/32.5R and 42.5N/42.5R. None of the 10 samples tested met the standard requirements for strength class of 52.5N/52.5R. However, the study revealed that as from LOI above 9.2%, the LOI increases with decreasing compressive strength. In line with this trend, Sample D with LOI = 14.56% came up with a 28 days compressive strength of 25.9 N/mm^2 which is clearly below the minimum standard requirement of 32.5 N/mm^2. D is, therefore, a sub-standard sample. Application of sub-standard cement in building construction can lead to structural failure.

Keywords: Additives, analysis, cement, parameters, physicochemical, standard

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INTRODUCTION

Cement is derived from the Latin word "*Cementum*," which refers to stone chips, such as those found in Roman mortars, rather than the binding ingredient. This term has lately been broadened to include any material that has adhesive and cohesive capabilities that may link mineral pieces into a compact whole.[1]

Historically, in 1824, a bricklayer of Leeds in Britain named Joseph Aspdin produced an excellent hydraulic cement by burning a mixture that contain proportions of limestone and clay at a high temperature. The product after blended and mixed with water, solidified, and resembled a naturally occurring rock found in the Isle of Portland in England.[2] The addition of gypsum during grinding clinker and the use of high temperatures are two major advancements over the previous product. These functions act as a set retarder for the cement and enable the manufacture of greater lime content silicates, which are required for faster concrete strength development.

Application of additives for cement production has recently become popular among cement manufacturers in Nigeria for obvious reasons. Cement additives are substances that are blended with clinker to increase the grinding efficiency and to improve the performance of finished cement.[3] They are often use for the optimization of the cement properties, the cement grinding process, boosting production quantity, and reducing cost of production. It is quite known that the high level of additive in cement enhances the adhesive property of cement.[4] In spite of its positive impacts, studies have shown that excessive use of additives depletes cement quality and performance.[5,6] This implies that additive up to a limited level can enhance compressive strength and beyond a certain level will lower the compressive strength of the cement. Okoye *et al*.[7] confirmed good results of using limestone additives.

Building collapses have been more common in Nigeria in recent years. In the year 2006, four similar collapses were documented in Ebute Metta, Lagos, resulting in the deaths of over 30 people in residential buildings.[8,9] According to

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Nwankwojike *et al.*,[10] substandard building materials are the biggest cause of building collapse in Nigeria; and because cement is a prominent component in building construction, attention has been directed to periodic quality assessment of cements. The aforementioned situation is exacerbated by the porous nature of Nigeria's borders, which allows for the importation of low-quality cements.[11] As a result, the necessity for regular cement sampling and testing becomes critical. This present study aims to assess, evaluate, and compare the quality of various brands of cement currently accessible in Nigerian markets on this premise. This is necessary to determine whether the cements fulfill the required specifications and to evaluate the role of additives in cement quality.

MATERIALS AND METHODS

Analysis

All the sample analyses were carried out at the Quality Assurance Department of Dangote Cement Plc, Gboko Plant, Benue State, Nigeria. For the analysis, the chemical and physical properties of 10 samples were analyzed in accordance with the Nigerian Industrial Standards (NIS). The apparatus used for this research include X-ray fluorescence analyzer, Le Chatelier's mold, Humidity Cabinet, Vicat Apparatus, Manual Blaine Machine, desiccator, water bath, auto-mixer, compressive strength machine, furnace, oven, platinum crucible, glass rod, beakers, and filter papers. All reagents used for the analysis include sodium carbonate (Na_2CO_3) and hydrochloric acid (HCl).

Sample Collection

Ten different cement brands labeled A–J were purchased randomly from Nigerian building material markets. A 2.5 kg sample of each brand was measured, parceled into an airtight container, and taken for analysis.

Chemical Analysis

The instrumental method of chemical analysis was employed in investigating the chemical properties of the samples rather than the classical method to obtain more reliable results and to minimize human errors. The chemical parameters determined include CaO, Al_2O_3, SiO_2, Fe_2O_3, insoluble residue, and loss on ignition (LOI).

Bogue formula[12] was used to compute clinker components such as tricalcium silicate or alite (C_3S), dicalcium silicate or belite (C_2S), tricalcium aluminate (C_3A), and tetracalcium aluminoferrite (C_4AF) for each of the 10 samples.

$(C_3S) = 4.07(CaO)–7.60(SiO_2)–6.72(Al_2O_3)–1.43(Fe_2O_3)–2.85(SO_3)$

$(C_2S) = 2.87(SiO_2)–0.754(C_3S)$

$(C_3A) = 2.65(Al_2O_3)–1.69(Fe_2O_3)$

$(C_4AF) = 3.04(FeO_3)$

In addition, some vital quality control parameters were computed as follows:

Silica ratio, $SiR = \frac{SiO_2}{Al_2O_3 + Fe_2O_3}$ (1)

Alumina ratio, $AlR = \frac{Al_2O_3}{Fe_2O_3}$ (2)

Lime saturation factor,

$$\text{LSF} = \frac{CaO - 0.7(SO_3)}{2.8\ (SiO_2 + 1.2\left(Al_2O_3\right) + 0.65(Fe_2O_3)} \quad (3)$$

Physical Analysis

Physical tests covered are sieve, Blaine, setting time (initial and final), soundness, and compressive strength tests. These tests were carried out using NIS.[13]

RESULTS

Extracts from NIS[13] for ordinary Portland cement are also indicated in Tables 1 and 2.

The results of physical properties of the 10 cement brands are shown in Table 3 while Table 4 shows the chemical properties.

DISCUSSION

Compressive strength in cement is the measure of its adhesiveness to the solid particles with which it is mixed. The NIS for cement clearly specifies three classes of cement, namely, 32.5N/32.5R, 42.5N/42.5R, and 52.5N/52.5R (where N represents the class with ordinary early strength and R represents class with higher early strength).

Table 1: Physical properties requirements given as characteristic value

Strength class	Compressive strength N/mm²				Initial setting time (min)	Soundness (expansion) mm
	Early strength		Standard strength			
	2 days	7 days	28 days			
32.5N	-	≥16.0	≥32.5	≤52.5	≥75	≤10
32.5R	≥10.0	-				
42.5N	≥10.0	-	≥42.5	≥62.5	≥60	
42.5R	≥20.0	-				
52.5N	≥20.0	-	≥52.5		≥45	
52.5R	≥30.0	-				

Source: Nigerian Industrial Standard, 2014

Table 2: Chemical properties requirements given as characteristic value

Property	Test reference	Cement type	Strength class	Requirements (%)
Loss on ignition	NIS 445: 2003	Type I Type III	All	≤5.0
Insoluble residue	NIS 445: 2003	Type I Type III	all	≤5.0
Sulfate content (as SO_3)	NIS 445: 2003	Type I Type II Type IV Type V	32.5N 32.5R 42.5N	≤3.5
			42.5R 52.5N 52.5R	≤4.0
		Type III	All	

Source: Nigerian Industrial Standard, 2014

Nine out of the 10 samples satisfied the minimum compressive strength requirement of 10 N/mm^2 in 2 days. Sample D which achieved only 1.5 N/mm^2 in 2 days is, therefore, considered to be substandard. In the same vein, only Sample D could not scale through the 7 days compressive strength specification of 16.0 N/mm^2; D recorded only 9.8 N/mm^2 in 7 days.

All the brands (except D) satisfy the requirements for class 32.5N/32.5R. Furthermore, all the brands (except D and F) comply with the requirements for cement next higher class: 42.5N/42.5R. It is significant to note that none of the brands tested qualify for the highest grade of cement: 52.5N/52.5R as shown in Figure 1.

The levels of additives in cement are usually detected from the LOI.[14] For a sample of cement, the value of percentage LOI complies with the specification of NIS if the value of LOI is ≤5.0%. It is quite known that additive in cement enhances the adhesive property of cement and reduces the influence of C_2S.[4] This implies that additive up to a limited level can enhance compressive strength and beyond a certain level will lower the compressive strength. Okoye *et al*.[7] confirmed good results of using limestone additives.

Based on the above understanding, it is shown in Figure 2 that Sample D with the highest level of LOI (14.56%) has the lowest 28 days compressive strength (25.9 N/mm^2). Furthermore, this very low compressive strength does not meet up to the requirement of the lowest class of cement (32.5N/32.5R). On the basis of compressive strength, Sample D was already identified as sub-standard. Now, this quality failure is attributed to excessive use of additive.

The cement sample with its LOI next to Sample D is sample F (LOI = 11.22%). Sample F fell short of the requirements

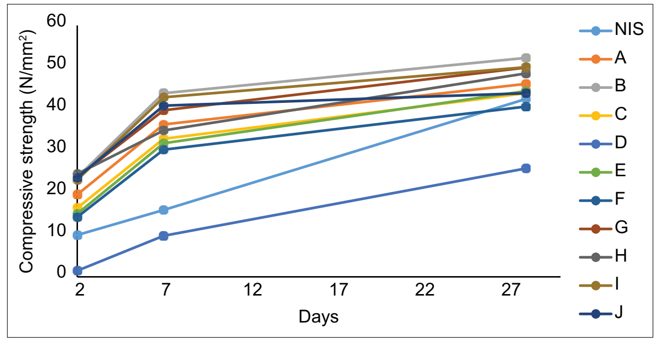

Figure 1: Graph of compressive strength of cement samples versus days

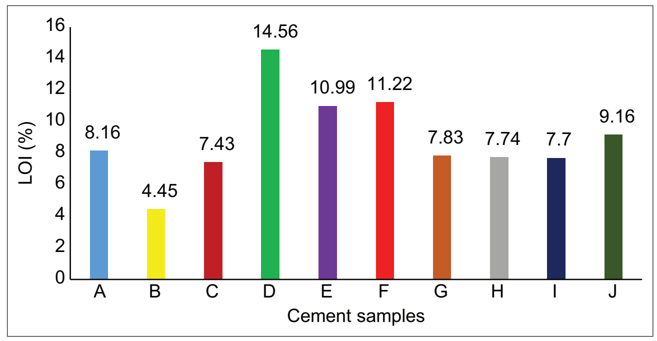

Figure 2: %Loss on ignition of cement brands

for strength class 42.5N/42.5R. This limitation in the quality of cement brand F is attributed to the high level of additives. All other eight cement brands satisfied the requirements for strength classes 32.5N/32.5R and 42.5N/42.5R. None of the 10 brands met the requirements for strength class 52.5N/52.5R. This work confirms the findings of Sam *et al*.[15] and Nwankwojike *et al*.[10] that the cement brands sold in Nigeria met most of the physicochemical parameters recommended by NIS but should not be used for heavy weight constructions such as story buildings, bridges, and skyscraper building.

A close look at the test results of the remaining eight brands of cement will suggest categorizing them into two groups with Sample D also belonging to the second group, as shown in Tables 5 and 6, respectively.

The samples in Table 5 (i.e., C, I, H, G, A, and J) have LOI ranging roughly from 7.4% to 9.2%. All their chemical properties satisfy all the NIS standard requirements. Their physical properties are also in compliance. Based on the levels of compressive strengths attained, the brands listed in this table satisfy strength classes of 32.5N/32.5R and 42.5N/42.5R only. It is interesting to note that within this range of additive application (represented by the quoted LOI range), the samples did not show any definite pattern of strength development

Table 3: Physical properties requirements given as characteristic value

S/N	Physical properties	A	B	C	D	E	F	G	H	I	J
1	Initial setting time (min)	121	118	120	124	127	124	127	128	134	125
2	Final setting time (min)	221	218	122	127	230	228	201	210	220	200
3	Blaine (m^2/kg)	4110	4220	4250	3940	3910	3730	3870	3820	3820	3750
4	Sieve residue (45 μm %)	12.00	3.20	2.00	14.80	6.80	7.60	8.80	6.40	7.60	7.20
5	Compressive strength (2 days N/mm^2)	19.7	23.8	16.5	1.5	15.1	14.3	23.8	24.6	23.2	23.7
6	Compressive strength (7 days N/mm^2)	36.4	43.9	33.0	9.8	31.9	30.4	39.8	35.0	42.9	40.9
7	Compressive strength (28 days N/mm^2)	46.1	52.3	43.9	25.9	42.0	40.7	50.0	48.6	50.1	43.9
8	Le Chatelier's expansion (mm)	0.50	0.25	1.00	0.00	0.25	0.50	1.25	0.50	0.25	0.00

Source: Laboratory analysis, 2021

Table 4: Chemical properties of 10 cement samples

S/N	Chemical properties	A	B	C	D	E	F	G	H	I	J
1	LOI%	8.16	4.45	7.43	14.56	10.99	11.22	7.83	7.74	7.70	9.16
2	IR%	1.00	0.76	1.13	1.23	1.09	1.13	1.25	1.29	1.22	1.13
3	SO_3%	1.44	1.39	1.16	1.90	1.13	0.99	1.78	1.63	1.65	1.54
4	CaO%	62.10	63.90	61.41	56.44	59.61	59.89	61.25	60.86	61.34	60.29
5	SiO_2%	18.93	19.20	18.70	17.86	18.48	18.20	18.26	18.60	18.68	18.80
6	Al_2O_3%	5.40	5.71	5.52	4.87	5.24	5.17	5.52	5.61	5.35	5.12
7	Fe_2O_3%	2.97	4.58	4.64	3.14	3.47	3.41	4.11	4.27	4.06	3.97
8	Total%	100	100	100	100	100	100	100	100	100	100
10	SiR	2.26	1.86	1.84	2.23	2.12	2.12	1.90	1.88	1.98	2.07
11	AlR	1.82	1.25	1.19	1.55	1.51	1.52	1.34	1.32	1.32	1.29
12	LSF	0.99	0.99	0.98	0.95	0.98	1.00	0.99	0.97	0.98	0.97
13	C_3S%	64.29	65.25	60.78	51.37	58.79	63.01	62.50	57.92	61.20	58.08
14	C_2S%	5.85	5.91	7.84	12.53	8.70	4.72	5.27	9.71	7.46	10.15
15	C_3A%	9.29	7.39	6.79	7.60	8.04	7.95	7.66	7.66	7.32	6.85
16	C_4AF%	9.02	13.93	14.11	9.54	10.54	10.35	12.51	12.97	12.35	12.06

Source: Laboratory analysis, 2021, IR: Insoluble residue, LOI: Loss on ignition

Table 5: Cement brands with LOI of 7.4–9.2%

Samples	C	I	H	G	A	J
LOI%	7.43	7.70	7.74	7.83	8.16	9.16
Compressive strength (minimum standard)	16.5	23.2	24.6	23.8	19.7	23.7
2 days: N/mm^2 (≥10)						
7 days: N/mm^2 (≥16)	36.4	42.9	35.0	39.8	36.4	40.9
28 days: N/mm^2 (≥32.5)	43.9	50.1	48.6	50.0	46.1	43.9

Source: Laboratory analysis, 2021, LOI: Loss on ignition

(increasing or decreasing) as shown in Figure 3 although they all met the standard.

The samples in Table 6 present cement brands: J, E, F, and D with LOI ranging from 9.2% to 14.56%. Samples J and E

Table 6: Cement brands with LOI of 9.2–14.56%

Samples	J	E	F	D
LOI%	9.16	10.99	11.22	14.56
Compressive strength (minimum standard)				
2 days: N/mm^2 (≥10)	23.7	15.1	14.3	1.5
7 days: N/mm^2 (≥16)	40.9	31.9	30.4	9.8
28 days: N/mm^2 (≥32.5)	43.9	42.0	40.7	25.9

Source: Laboratory analysis, 2021, LOI: Loss on ignition

comply with the standard requirements for strength classes of 32.5N/32.5R and 42.5N/42.5R. Sample F satisfies only the 32.5N/32.5R class. The striking observation in this range of additive application is that beyond 9.2% LOI, the compressive strength consistently decreases as the LOI increases, as shown in Figure 4.

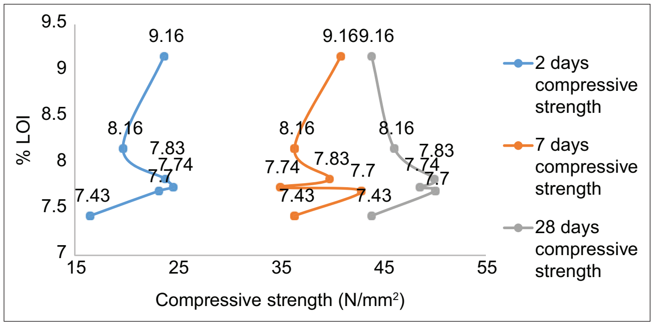

Figure 3: Loss on ignition (7.4–9.2%) versus compressive strength

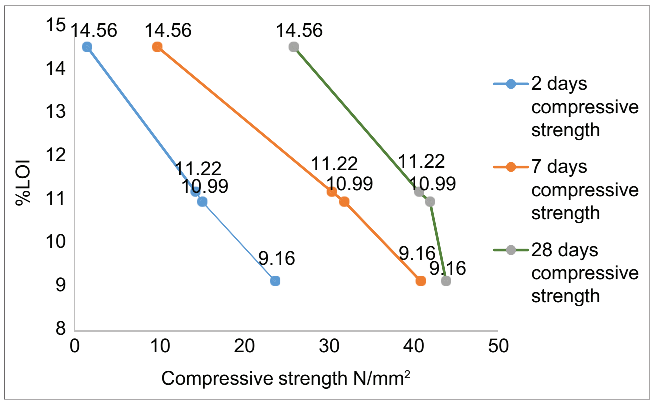

Figure 4: Loss on ignition (9.2–14.56%) versus compressive strength

The message in that, above a certain level of additive application, the higher the additive, the lower the compressive strength. This trend applies to all ages of compressive strength (i.e., 2 days, 7 days, and 28 days).

CONCLUSION

In this work, 10 different brands of cements were sampled from across the country and labeled A–J. Based on the standard strength which is the ultimate parameter for cement quality assessment, the 10 brands of cement can be rated as follows: B>I>G>H>A>E>C>I>F>D. Their physicochemical parameters were tested and studied, with emphasis on the impacts of clinker additives on cement quality. It has been found that beyond 9.2% LOI, compressive strength of cement decreases progressively with increasing LOI, while below 9.2%, there is no definite pattern between LOI and compressive strength. This implies that excessive use of cement additives beyond a certain level depletes cement strength development even when it conforms to other physicochemical specifications. Sample B (having the lowest %LOI of 4.45%) met all specifications for physical and chemical properties. Yet, it did not meet up with the highest strength class of 52.5N/52.5R. This could be due to interplay of other parameters such as clinker phases.

Regular cement testing of this nature is strongly recommended to enlighten the end users and the general public. Building engineers need to collaborate with building materials testing laboratories which can recommend specific cement qualities for specific engineering projects, to minimize structural failures and building collapses.

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