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Assessment of Physio-Chemical Properties of Cement Slurry for Offshore Well Completion

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***Abstract**: This study is aimed at examining physio-chemical properties of some local cements that may be suitable for well bore completion. American Society for Testing and Materials (ASTM) and British Standards Institution (BSI) are used as standards for this study. Four major cement samples using various physio-chemical methods were analysed; three were locally sourced and one imported (Class-G Cement). Results of all the analysis showed that the locally produced cements, i.e., Elephant, Eagle and Dangote cements differ in their physio-chemical properties with reference to ASTM and BSI standards. This involves testing for the chemical composition, mechanical and rheological properties of slurry of four cement samples. The results obtained from the experiments indicates that the properties (standard consistency test, compressive strength and setting time) of two of the local cements (Eagle and Dangote) are at variance with the Class G cement (which acts as control) but Elephant cement shows properties similar to the class G cement. On the other hand, all the local cement samples have similar chemical composition (CaO, Fe_2O_3, Al_2O_3, MgO, etc) with the Class G cement. Laboratory tests and simulations should be carried out repeatedly to ensure the cement slurry can withstand actual well conditions.*

Keywords: Physico-chemical, Cement slurry, well completion, setting time, compressive strength.

1. INTRODUCTION

Cements may be defined as adhesive substances capable of uniting fragments or masses of solid matter to a compact whole [6]. Cement functions by forming a plastic paste when mixed with water, which develops rigidity (sets) and steadily increases in compressive strength (hardness) by chemical reaction with the water (hydration). Cement is a material made by burning limestone and clay which after being wetted becomes hard like stone and it is used for building, construction and so on. It can also be defined as hydraulic cement produced by pulverizing Portland cement clinker usually containing calcium sulphate [1]. Historically cementing materials have played an important role in providing shelter for mankind. The production of cement has played a key role as a construction material throughout the history of civilization. It is very important in the construction of buildings, bridges, tunnels, to mention a few [5, 10]. Ordinary Portland cement (OPC) is the mostly consumed cement in the construction industries due to its diversified applications. It could be noted that it contributes significant impacts on social, urban and economic development of human society but it is one of the elements responsible for carbon dioxide emission [9]. The results of the chemical parameters of two indigenous Portland cement brands sold in Nigeria indicated that the chemical composition conform well to international standards and that majority of the Portland cement sold in Nigeria are better used for structures involved with low loading [13]. The study of rheological properties attempts to determine the intrinsic fluid properties; mainly viscosity, which is necessary to determine the relationships between the flow rate (shear rate) and the pressure gradient (shear stress) that causes the movement of a fluid. Drilling and completion are major steps in oil and gas production and occur after the seismic investigations. The major conventional operations include drilling the well, placing the casing, cementing and completion of the wellbore. The rheological behaviour of oil well cement (OWC) slurries must be optimized to achieve an effective well cementing operation. Cement forms a strong, almost impermeable barrier between the casing and formation which prevents gas and fluids from migrating through the wellbore and provides support for the casing [2]. This study is aimed at examining properties of four (4) portland cement based on their chemical composition, physical and rheological characteristics which will serve as guide for users for different purposes. These samples are: Dangote cement, Eagle cement, Elephant cement and G-class Cement.

2. MATERIALS AND METHODS OF STUDY

Experimental Procedure

Mixing and preparing cement slurry

The cement slurries were prepared using a high-shear blender type mixer with bottom driven blades. First, the weighed cement and solid admixture were dry mixed in a bowl for about 30 s, and subsequently mixed in a blender at a slow speed for 15 s so that chemical admixtures could be thoroughly dispersed in the water. Manual mixing was conducted for 15 s and a rubber spatula was used to recover material sticking to the wall of the mixing container to ensure homogeneity. Finally, mixing resumed for another 35 s at high speed. This mixing procedure was strictly followed for all cement slurries. All mixing was conducted at a controlled ambient room temperature of 23±1°C. The prepared slurry was then placed into the bowl of a mixer for preconditioning over 20 minutes at the test temperature (23 °C, 45°C, or 60°C) at a speed of 150 rpm. The total time between the beginning of mixing and the start of the rheological tests was kept constant to avoid the effect of exogenous variables on the results. The rheometer set-up was also maintained constant for all slurries (Figure 1). The concentric cylinder test geometry was kept at the test temperature so as to avoid sudden thermal shock of the slurry.

Chemical Analysis on Cement

Chemical analysis of major components of cement slurries was carried out. Some of the components includes: SiO_2, CaO, Fe_2O_3, Al_2O_3, MgO, SO_3, as well as compounds such as K_2O, Na_2O, Ca_2O_2, MnO_2, and other impurities that make up the remaining balance.

Loss on Ignition Test

Approximately 1.0 g of sample was weighed in a platinum crucible at a temperature of 25 °C, capacity of 25 - 30 ml. This material was heated in a muffle furnace at a temperature between 900 - 1,000 °C, cooled and weighed (W_1). The loss in weight was checked by a second heating at same temperature for 5 min and the content re-weighed. This process was repeated until a constant weight was attained (W_2). The loss in weight was recorded as the loss in ignition. Percentage loss on ignition was calculated as follows: $W = W_1 – W_2$,

% loss of ignition = $\frac{(W\ X\ 100)}{W_1}$, where W = weight of sample taken.

Determination of Oxides in Cement Silica

0.5 g of the cement sample was mixed thoroughly with 0.5 g of NH_4Cl in a 50-ml beaker covered with a watch glass and 5 ml of HCl was cautiously added by allowing the acid to fill to the brim. Then 1-2 drops of HNO_3 was added to the mixture after the chemical reaction has subsided and stirred with a glass rod. The beaker was set on a steam bath for digestion for 30 minutes with occasionally stirring and breaking up of any lumps to facilitate complete decomposition of the cement (Fig. 2). A medium-textured filter paper is fitted to the funnel and the jelly-like mass of silicic acid is filtered as completely as possible without dilution and the solution is allowed to drain through. The beaker was rinsed with hot HCl (1:99) 2 to 3 times and then with 10 -12 small portions of hot water. Each portion is allowed to drain through completely before adding another. The filtrate was reserved for determination of ammonium hydroxide group. The filter paper and residue was transferred into weighed platinum crucible, W1. This material was dried and ignited first slowly without inflaming until the filter paper was charred and finally at 1,100-1,200°C for 1 hr in a muffle furnace. This material was cooled in desiccators and weighed, W_2. The SiO_2 obtained contains small impurities which were treated in the crucible with 2 ml of water, 2 drops of H_2SO_4 (1:1) and 20 ml of HF and cautiously evaporated to dryness. The residue was heated at 1,050 - 1,100°C for 5 minutes, and cooled and weighed, W_3. The difference between this weight and the weight previously obtained represents the weight of SiO_2.
Calculations: Silica (%) = $(((W_1 – W_2) + W_3)/W) \times 100$,
Where: W_1 = weight of silica and insoluble impurities; W_2 = weight of impurities; W_3 = weight of silica recovered from iron and aluminum oxide; W = weight of sample taken.

Figure 1: Brookfield Programmable Rheometer.

Figure 2: Heating Mantle with Stirrer.

Combined Ferric Oxide and Alumina:

According to [1], 5 ml of HCl was added to the filtrate reserve after separating silica. Few drops of methyl red indicator were added and treated with NH_4OH (1:1) until the color of the solution becomes distinctly yellow; a drop is added in excess for confirmation. The solution containing the precipitate was heated to boiling for about 50 to 60 s and was allowed to settle down for 5 m. This material was filtered using medium textured filter paper and hot solution of ammonium nitrate (20 g/l) was used in washing the precipitate for at least 2-3 times. The filtrate was set aside and the precipitate was transferred alongside the filter with the same beaker where the first precipitation was effected. The precipitate was dissolved by using hot HCl (1:2) and stirred by thoroughly macerating the paper. The solution was diluted to about 100 ml and re-precipitation was carried out. The solution was filtered and the precipitate was washed with about four 10 ml portion of hot NH_4NO_3 solution (20 g/l). The filtrates were combined, set aside and reserved for the determination of CaO. The precipitate was placed in a weighed platinum crucible and the papers were slowly heated until they charred. This material was finely ignited to constant weight at 1,050 1,100°C.

Calculations: Combined $Al_2O_3 + Fe_2O_3$ (%) = (Si x 100)/W, where: Si = weight of crucible; W = weight of the sample taken.

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Ferric Oxide:

The solution (100 ml) from silica filtrate (made up to 250 ml) was cooled to 10 °C. A freshly prepared and filtered 5% aqueous cupferron solution was slowly added. These were rigorously stirred until no further formation of brown precipitate. After thoroughly stirring it, this material was filtered through Whatmann filter paper No.41 and the precipitate was washed several times with 10% by volume of HCl containing 1.5 g of cupferron per litre and twice with 5 ml of ammonia solution to remove excess cupferron and finally washed once with water. The precipitate in a weighed platinum crucible was slowly charred and finally ignited to constant weight at 1,000 °C [4].
Calculation: Fe (%) = (Weight of precipitate x 100) / (Weight of samples).

Magnesium Oxide:

The filtrate after removal of CaO was acidified with HCl and concentrated to about 150 ml. Approximately 10 ml of ammonium hydrogen phosphate (25% w/v) was added to the solution and cooled to 10 °C by placing it in ice cold water. NH_4OH was added with constant stirring until becoming yellow and 1-2 ml in excess. The solution was kept in a cool atmosphere, overnight and filtered through Whatman filter paper No.42. The precipitate was washed with 10 g NH_4NO_3 + 20 ml NH_4OH and diluted to 100 ml. In a weighed platinum crucible, the precipitate with filter paper was placed and slowly heated to char the paper without inflaming it and finally ignited until a constant weight is obtained at a temperature of 1,100-1,200 °C.
Calculations: MgO(%) = Weight of Ignited Residue x 0.362 x 100)/W, where: W = Weight of sample; conversion factor of $Mg_2P_2O_7$ to MgO = 0.362.

Sulphate (as SO_3):

1.0 g of cement sample accurately weighed to four decimal places was taken and 25 ml of cold water and 5 ml of HCl was added while stirring the mixture vigorously. The solution was heated for complete decomposition of sample and diluted to 50 ml. Solution was digested for 15 m at a temperature just below boiling. This material was filtered through a medium textured paper and washed thoroughly with hot water. The filtrate was diluted to 250 ml and heated to boiling, 10 ml of hot $BaCl_2$ (100 g/l) was added and boiling was continued until the precipitate was well formed. The solution was digested for 8 hr at temperature just below boiling and the volume of solution was maintained between 225-260 ml by adding water when necessary. The solution was filtered through a retentive paper and washed thoroughly with hot water. The filter paper with precipitate in a weighed platinum crucible was charred without inflaming and finally ignited at a temperature of 800-900 °C until constant weight was attained, then it was cooled in a desiccator and weighed at SO_3.
Calculations: SO_3(%) = (Weight of Ignited Residue x 0.343 x 100)/W, where: W = Weight of the sample; conversion factor for $BaSO_4$ to sulphate (SO_3) = 0.343, i.e., the molecular ratio of SO_3 to $BaSO_4$.

Calcium Oxide (CaO):

According to [1], the filtrate (200 ml) obtained in the determination of the ammonium hydroxide group was acidified by using 5 ml of HCl. A few drops of methyl red indicator and 30 ml of warm ammonium oxalate (50 g/l) were added to the solution. The solution was heated to a temperature of 70-80 °C. NH_4OH (1:1) was added with constant stirring until it changes colour from red to yellow. The solution was allowed to stand for 1 hr with occasionally stirring during the first 30 m. The solution was filtered using a retentive paper and was washed moderately with cold ammonium oxalate (1 g/l). The filtrate was reserved for the determination of MgO. The precipitation and filter paper were transferred to the same beaker in which precipitation was first made. The oxalate was dissolved in 50 ml of hot HCl (1:4) and the filter paper was macerated. The mixture was diluted to 200 ml with water and few drops of methyl red indicator were added together with 20 ml of ammonium oxalate. The solution was heated nearly to boiling and calcium oxalate was precipitated again by neutralizing the acid solution with NH_4OH (1:1) until distinctly changing the colour to yellow. The solution was combined with that already obtained for the determination of MgO. The precipitate was dried in a weighed covered platinum crucible and the paper was charred without inflaming. Finally, the tightly covered crucible was heated in a muffle furnace at a temperature of 1,100 to 1,200 °C, cooled in a desiccator and weighed as CaO. The ignition was repeated until a constant weight was obtained.
Calculation: CaO (%) = Weight of Precipitate x 100)/W, where: W = Weight of the sample taken.

Test for Physical Properties of Cements

Standard Consistency Test

According to [4,10], 400g of cement sample was weighed and spread out on a steel plate for about 30 m to cool to the temperature of the mixing room (27±50 °C). 30% water content of the mass of dry cement was added as a start. The mixture was mixed for 4±0.25 m by using a trowel to give a paste and was immediately transferred into the mould laying on the steel plate. The top of the mould was smoothened off as quickly as possible with the aid of the trowel. The mould and paste were placed under the plunger in the vicat apparatus and the plunger lowered gently to contact the surface of the paste. This material was released quickly and allowed to sink into the paste. The scale reading of the vicat apparatus was noted after 1 m and recorded. If the plunger penetrates to a point 5 to 7 mm above the bottom of the mould, the water-cement ratio is taken as the consistency, if not, a new water-cement ratio is taken and the procedure repeated.

Compressive Strength Test:

The ELE mixing bowl and paddles were wiped clean with a damp cloth and different grades of cement, sand and aggregates in the ratio of 1:2:4 were weighed into the mixing bowl. 10% weight calculated as tap water was added and mixing was carried out until a cement mortar of homogenous consistency was obtained. The concrete was turned in the bowl over 3-4 times by using the scoop and trowel. 12 pieces of clean 150 x 150 x 150 mm cube mould were set on the working bench and all bolts were tightened using the spanners. The base of the mould was greased with a thin film of petroleum jelly and also the joints of the two halves. As quick as possible, the concrete mixture was scooped into each mould (to half the depth) in a single layer. The concrete mixture was tamped in each mould with 25 strokes of the tamping bar spread uniformly over the cross-section of the mould and exerting the same force in each case by raising the bar about 30 mm above the surface of the concrete and guiding it as it is then allowed to fall under its own weight. More mixture was scooped into each cube being completely filled to the brim. This material was tamped again 35 times for each mould. The assembled mould was placed on a vibrating machine and well secured in place, a suitable hopper was used to facilitate filling. The mould was vibrated on a jolting machine for 2 m. Afterwards, the cubes were covered with an impervious sheet to avoid evaporation and were allowed to cure at room temperature for 24 hr. The cubes were removed from their mould after 24 hr and were marked for identification. This material was then immersed in clean water for curing (Figure 3). The cubes were brought out of the water in due time and their surface was carefully wiped clean of water and adhering loose sand. The DMO Samuel Denison Compression machine was switched on 15 m before use. The weights of the cubes in air were determined by using a spring balance. Each cube was then placed inside the crushing machine centralizing it by using the scribe lines of the four locating pins on the bottom plates and with the marked face of the cube to the front of machine. The Perspex guard was closed and the 'Enter' sign pressed on the machine (Figure 4).

Figure 3: Water Bath for Curing of Samples

Figure 4: Automatic Digital Compression Testing Machine

Setting Time Determination

Sample of cement paste of standard consistency was prepared and the time of first mixing the water with cement was noted down. A slight excess of paste was immediately transferred into the mould in one layer by using hand trowel. The top of the mould was smoothened and levelled. The mould was placed under the initial set needle of cross-sectional area of 1 mm and the needle was covered gently onto the surface of the paste and was quickly released by allowing it to sink to the bottom. These tasks were repeated several times at regular intervals of 10 m in different positions of mould until the paste has stiffened sufficiently for the needle not to penetrate deeper than 5 mm above the bottom of the mould. The time interval between the addition of water and the initial setting time was recorded. Finally, the needle was replaced with a 1 mm square needle fitted with a metal annular attachment and this probe was allowed to come gently with contact with the surface of the cement paste at an interval of 15 m. The final set was reached when the needle makes an impression on the surface but annular cutting edges fail to do so [4].

3. RESULT AND DISCUSSION

The characterization and evaluation of the available brands of Portland cements have become necessary in order to correctly evaluate the product quality and, hence, the area of application. The experimental results show various oxides and their percentage composition in different cements samples and were presented in form of tables and charts. Their physical and mechanical properties were analyzed to determine its compressive strength, settling time and standard consistency test. The results of the tests carried out on various cement samples are discussed below.

Table 1: Chemical and percentage composition of cement samples

S/N	**PARAMETERS**	**% Composition**			
		Eagle	**Class-G**	**Elephant**	**Dangote**
1	PH	11.18	12.19	10.19	11.19
2	Silicon Oxide %	20.65	20.32	16.23	20.99
3	Aluminum Oxide %	3.80	2.97	4.17	4.18
4	Ferric oxide %	1.90	1.86	2.26	3.30
5	Calcium oxide %	61.08	60.50	60.10	62.50
6	Magnesium oxide %	0.52	0.87	0.50	1.87
7	Sodium Oxide %	0.21	0.27	0.30	0.37
8	Potassium Oxide %	0.65	0.85	0.86	0.92
9	Sulphide %	1.60	0.58	1.60	2.02
10	Loss On Ignition %	2.10	1.05		1.53

Table 2: Result of Standard Consistency Test

Cement Sample	D	G	EA	EL
Consistency (%)	28.00	30.00	29.00	33.00
Mass of Cement (g)	300	300	300	300
Mass of Water (g)	84	90	87	99
Penetration (mm)	32	34	32	35

Table 3: Result of Setting time of cement samples

Cement Sample ID	D	G	EA	EL
Consistency (%)	28.00	30.00	29.00	33.00
Initial setting time (m)	31	35	32	40
Final setting time (m)	480	510	492	576

Table 4: Compressive Strength of cement samples

	Compressive Strength (N/mm^2)			
Samples	**7 Days**	**14 Days**	**21 Days**	**28 Days**
Dangote	26.82	27.25	28.38	22.90
G-Class	20.50	26.44	28.38	28.85
Eagle	24.59	27.25	28.14	29.95
Elephant	23.56	26.93	28.21	30.26

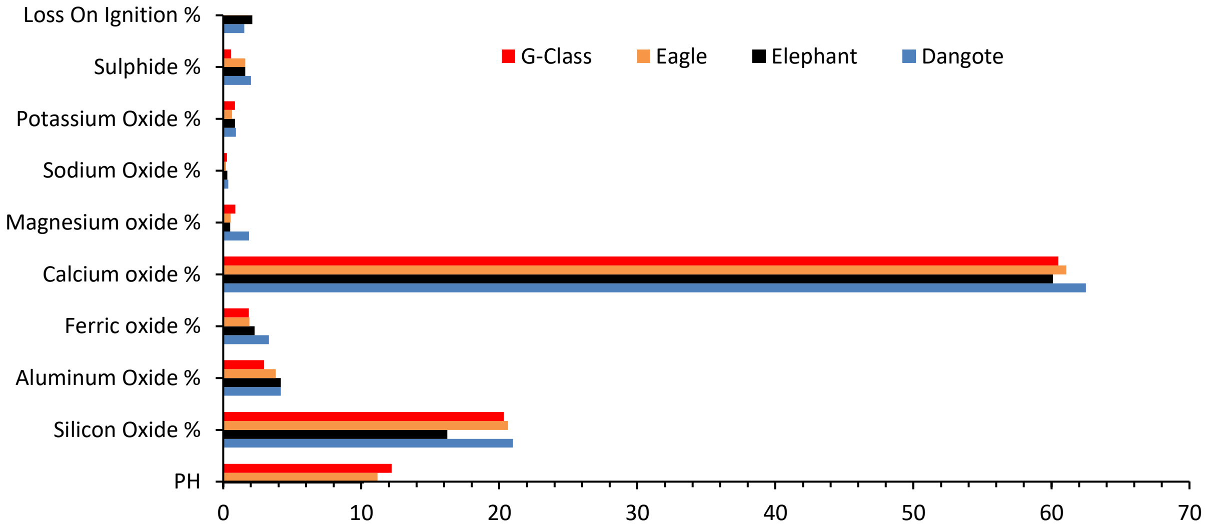

Figure 5: Percentage Composition of Oxides in Cement Samples

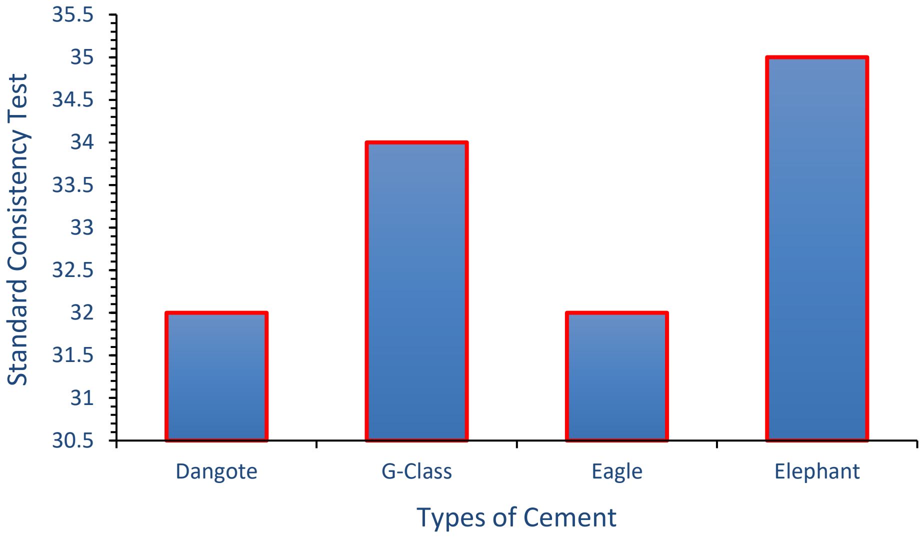

Figure 6: Standard Consistency Test on Cement Samples

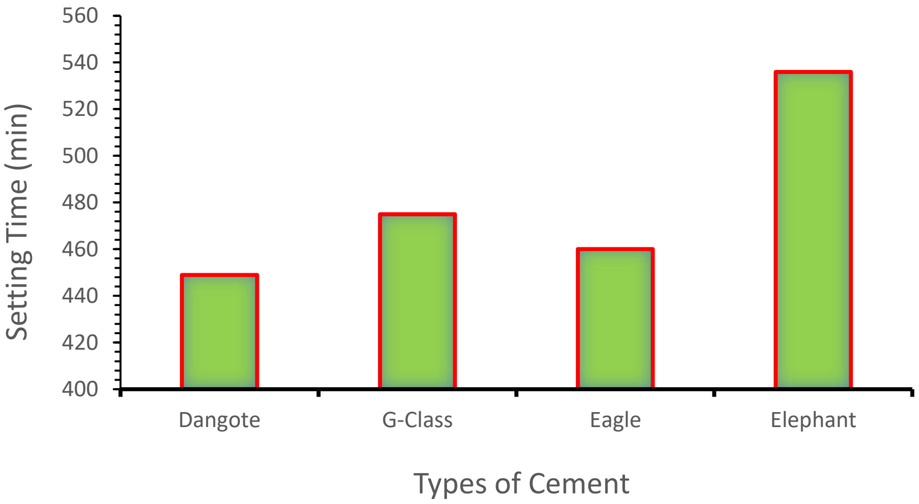

Figure 7: Settling Time of Cement Samples

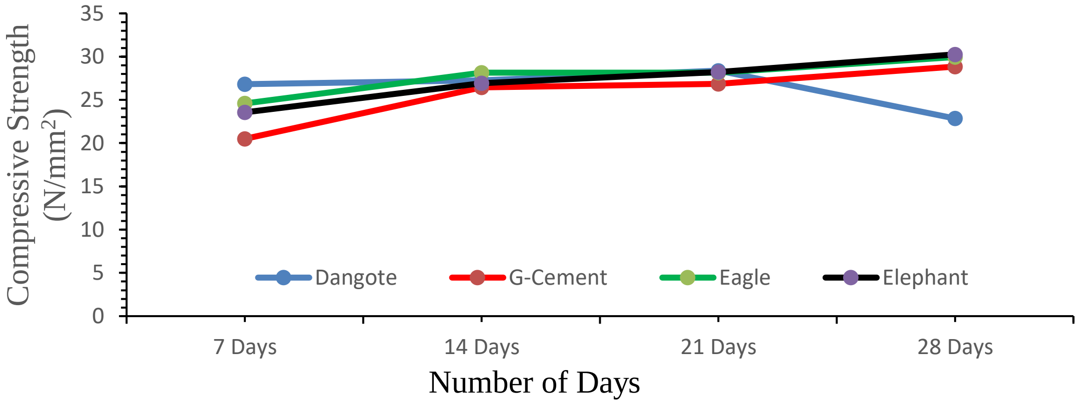

Figure 8: Compressive Strength of Cement Samples

Dangote Cement

Dangote cement has so many varying properties that are comparable with all the cement types. It recorded the highest percentage of composition of oxides (Figure 1) The SiO_2 percentage value was in accordance with the Ordinary Cement of

[4] and thus recorded an average Compressive Strength of 26.82N/mm. The Al_2O_3 of about one percent composition corresponded slightly with type IV of ASTM (1986). The MgO and SiO2 values are of type I and its CaO and SO_3 percentage composition are of type IV. It has the highest uncombined lime, thus resulting in its low CaO. According to its mineralogical composition (Table 1), its C_3S and C_3A corresponded to type I, while its C_2S and C_4AF are type IV and type V, respectively. The low value of C_4AF was observed as a result of the substitution of ferric oxide for Alumina and thus following to an increase in C_3A and a reduction of C_4AF. This observation was ascertained as given in analytical form [12]. The high C_3A value is reflected in the interval between the setting times of 11.38 minutes. This is an indication of poor workability [7]. It is difficult to ascertain the grade of Dangote cement. It is most likely to be a slag Portland cement. This can only be verified with further investigation.

Elephant Cement

From Table 1 presented, Elephant cement has some of its composition, i.e., SiO_2, Al_2O_3, Fe_2O_3 and MgO in conformity with Type I Class G Portland Cement of [4]. The SO_3 percentage though low can still be approximated as ordinary Portland cement in fact, this is the least value when compared to the other analyzed cements. The CaO percentage composition was quite low in comparison with its indigenous counterpart as a result of the higher uncombined lime in the clinker. This high CaO-free content could be attributed to improper sintering condition or long storage of the clinker before grinding. This may enhance polymerization of the unstable alite and belite forms into the subsequent release of free CaO [3]. The C_3S and C_2S phases are in the region of type IV whereas the C_3A and C_4AF are of type I. The value of C_3A was quite low (the least), 6.36%, and thus affected the setting time, initial setting was after 40 minutes and final setting was 576 minutes. The presence of C_3A enhances the rate of hydration and thus initial strength development [8]. The physico-mechanical analysis showed that the Elephant brand has the lowest compressive strength of 28.21 N/mm which is consistent with the observed poor consistency and soundness test value [6]. Based on this analysis, it is evident enough to say that Elephant cement lies in the region of type I Portland cement and can be used for general concrete construction. However, structures must not be subjected to loading.

Eagle Cement

In Table 1, the percentage values of SiO_2, Al_2O_3 and Fe_2O_3 fell within the range of type I Portland cement. The MgO, though out of range of [1], still merges well with [4] of Ordinary Cement. On the other hand, the percentage values recommended for CaO, SO_3 and CaO all fell within type IV of Portland cement. The high percentage recorded for free lime is as a result of inefficiency in burning and may be explained as in the case with Elephant cement [4]. The C_3S and C_2S contents tally well with type IV low heat Portland cement, but the C_3A and C_4AF corresponded to type I. Based on the high silica content, Eagle cement has a higher compressive strength and also the relatively high C_3A content will enhance initial strength gain. A compressive strength of 23.56 N/mm2 was recorded which is according to the acceptable limit [4]. The setting time for Eagle cement was having initial setting of 32 minutes and final setting of 492 minutes. This could be attributed to the relatively low gypsum content compared to the high C_3A [1]. Sequel to this, it can be said that Eagle cement lies within the region of type I Portland cement. It can be used for general concrete structures considering the obtained soundness value; such structures could be subjected to loading.

4. CONCLUSIONS

Investigation conducted and experimental results indicate that indigenous portland cement brands, Eagle and Elephant conform in their chemical composition with ASTM standard. The conditions that are present in the borehole have a major impact on the differentiation of physico-chemical properties of the slurry viz-a-viz bonding, hardening and durability. However, their physico-mechanical analysis assessments show considerable deviation from [1,4]. They are better used for structures involved with low loading except in the free-lime percent value. Based on its soundness, it can be subjected to structures involved with loading. The chemical and physico-mechanical properties of Dangote cement fall a little short in standard as compared to other brands analysed. It needs to be further investigated upon. The Class G cement conforms well both in its chemical and physico-mechanical composition with the international standards. The Fe_2O_3 content in Eagle cement is less than 3% and MnO is practically absent in the raw material used. This eliminates their influence on the clinker formation mechanism of alite, belite and calcium-alumino-ferrite solid solutions [11]. Consequently, the poor performance of eagle cement is connected with poor sintering conditions, especially inability to obtain the required sintering temperature and cooling regime [12]. This has allowed the formation of predominantly poor hydrating belite solid solutions. It is recommended that the locally produced cement be used particularly for structures with low loading capacity. The Dangote Portland cement has relatively high Fe_2O_3 content. It is impossible to sinter such cement in a normal rotary kiln. Consequently, microscopic analysis needs to be performed to ascertain the class of cement. This brand belongs to a slag Portland cement. The class G brand has a chemical composition and physio-mechanical properties of a normal Portland cement. Both cements can be used for structures with high loading capacity. Elephant cement shows properties similar to the class G cement. On the other hand, all the local cement samples have similar chemical composition (CaO, Fe_2O_3, Al_2O_3, MgO, etc) with the Class G cement. A classification guideline based on [1,4] was developed to enhance easy assessment of the Nigerian cement. This developed assessment technique can be used to control product quality during manufacturing of cement.

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