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# Characterization of Nigerian Cement Slurries for Potential Usage in Oil and Gas Well Cementing and Construction Purposes

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**ABSTRACT**

This research characterized some Nigerian cements for potential industrial usage. Tests were performed on three types of cement slurry recipes. Compressive strength test was done using compressive strength machine (Hydraulic machine), Practical procedure according to API, 2010. setting time test. (initial and final) was performed with the aid of vicat apparatus. Practical procedure according to ASTM standards vol.04.01. and standard consistency test was performed at room temperature and 65% humidity. The compressive strength development for day 28 was 156.35, 136.05, and 178.98 psi for Dangote cement slurry (DACS), BUA cement slurry (BUCS) and Sokoto cement slurry (SOCS) respectively. Compressive strength developed more in the SOCS than in the other samples because the $C_3S$ percent in it was higher than in the other samples. $C_3S$ is responsible for early compressive strength development between 1-28 days. In DACS the $C_2S$ percent is high which developed compressive strength more after 28 days. Setting time at 20 $^0C$ and consistency showed 216, 285 and 156 min; with 54%, 54%, and 55% for DACS, BUCS and SOCS respectively**.** SOCS probably has lesser gypsum in it than DACS and BUCS. Hence, to optimize the setting time of the cement, less quantity of gypsum is the required.

**Keywords:** Cement, characterization, slurry, calcium trioxocarbonate (IV).

## INTRODUCTION.

Cement is a finely ground powder which, when mixed with water forms hardening paste of calcium silicate hydrate. Cement is used in mortal (to bind bricks or stones together) and concrete (bulk rock-like building material made from cement aggregate, sand and water) [1].

According to Morgan [2], portland cement is a hydraulic product made by burning and grinding a mixture of calcareous and argillaceous materials, such as limestone and clay, limestone and shale, chalk and clay or limestone and iron blast furnace slag. Portland cement is made up of the four major clinker materials, namely tricalcium silicate ($C_3S$), Dicalcium silicate ($C_2S$), tricalcium aluminate ($C_3A$) and tetracalcium aluminoferrite ($C_4AF$) to which 3-5% gypsum ($CaSO_4.2H_2O$) is added [3-6] Chemically. CaO, $SiO_2.Al_2O_3$ and $Fe_2O_3$ constitute 80% of portland cement.

The raw materials contained in the portland cement are lime, alumina and iron oxide. These oxides react to form compounds. The relative proportions of these oxides compositions have a great influence on the various properties of the cement [7]. Manufacturing of portland cement started in 1824 when Joseph Aspidin obtained a patent on a hydraulic cement he called portland cement because it has a color of a stone quarried on the isle of Portland, off the British coast. His method involved pulverizing and burning of a proportionate mixture of lime and clay into clinker, which was ground into the product known as portland cement. This cement had greater strength property than the natural cement produced by John Smeaton which was made by plain crushed limestone. Portland cement today as it was in Aspidin's days, is a predetermined and carefully proportioned chemical combination of calcium, silicon, iron and aluminum. It is the first true portland cement and since then has remained popular or dominant

The different types of ASTM cement show similarities to the API Portland cement in terms of production and usage [8]. Table 1 shows comparison of ASTM and API Portland cements.

Table 1: Comparison of ASTM and API Portland Cement [7]

| ASTM cement | Classes of API Cement | Comment |
|---|---|---|
| Type 1 | Class A | Same as Class A |
| Type 11 | Class B and G | Same as Cclass B, similar to Class G |
| Type 111 | Class C | Same as Class C |
| Type 1V | Class G | Very similar to Class G (Class G meets specifications of type V) |

The problem of using local cement in operational activities has being a serious concern to the field of engineers and designers. Research indicates that locally manufactured cements are compatible with oil well cements additives at 27 and 66 $^{0}$C [8] These can be used as an alternatives to portland cement in oil and gas cementing operations. However, premature gelation's of tests result are phenomenal at 66 $^{0}$C for entirely local manufactured cements. Additionally, they possess low viscosity and low thickening time.

Nowadays cementing operations are modified through the use of additives to economically meet most of the job conditions [9]. Besides temperature and pressure, other factors such as porous formation, corrosive fluid environment and over pressured formation fluids must be taken into consideration in primary cement design. By having adequate additives incorporated in cement slurry design, a wide range of conditions could be accommodated.

This study is to characterize cement for operational activities. Therefore, for local cements to be used effectively for cementing high temperature operations, stringent quality control during their manufacturing process is imperative. This work has been limited to the testing of the setting time, and the compressive strength of three local cements. Other cement tests such as gas migration, fluid viscosity were not considered. The aim of this research is to characterize local cement slurry and compare it with quality standards (i.e. physical and chemical) by the American Petroleum Institute (API) that can make it serve in oil well cementing. With particular interest in the setting time, and compressive strength.

The objectives of this research include to:

i. Use representative number of local cement samples,

ii. Measure the compressive strength of the local cements,

iii. Establish the compressive strength trend of both cements,

iv. Measure the thickening time of both cements and also, identify the causes of low thickening time in Nigerian local cement.

## MATERIALS AND METHODS

### *Materials*

Three brands of cement available on the Nigerian market which are commonly used by Nigerians for construction purposes were purchased from retail outlets. Sokoto cement was purchased in Angwa rukuba market, Nabor 1, Jos North local government of Plateau State, Nigeria; BUA and

Dangote cements were purchased at Jootar market in Ukum local government area of Benue State, Nigeria. Silica clay was obtained, sun dried and ground in Jootar, using clean grinding stones. Silica gel and potassium chloride were obtained in Wukari. Sharp sand and distilled water were sourced from Dangote cement company, Gboko plant in Benue State, for the preparation of cement slurry.

The following equipment were used to carry out the experiment: Electronic weighing balance, Disc mill/vibrating cup mill machine, Automatic mortar mixers, Compacting machine, Humidity cabinet, and Plunger machine.

**Cement slurry preparation and formulation**

Three cement slurries were formulated and were labeled with the following notations: DACS (Dangote cement slurry), BUCS (BUA cement slurry) and SOCS (Sokoto cement slurry).

All the three formulated cement slurry have the same design with respect to additive concentration. Table 2 shows materials adopted for the study.

Table 2: Materials used with their quantities

| S/No | Research material | Quantity in grams (g) |
|---|---|---|
| 1 | Cement | 130 |
| 2 | Silica clay | 160 |
| 3 | Silica gel | 159 |
| 4 | KCl | 1 |
| 5 | Sand | 1350 |
| 6 | Water | 280 |

To formulate the cement slurries, the following steps were taken:

- 1 g of potassium chloride was weighed into a clean container
- Then 159 g of silica flour and 160 g of silica clay were added
- The mass was then added into 130 g of DAC with 280 g of water and 1350 g of sand.
- The procedure was carried out on Bua, Dangote and Sokoto cements.

Tests carried out on the formulated cement samples.

**Compressive Strength Test**

Compressive strength machine (Hydraulic machine) was used to perform the compressive strength test. After the cement mortar of about 50 mm was cured for a day, it was subjected to a compressive load, until failure. It took more than 20 s, at a temperature of about 23 $^{0}$C [11, 10].

**Setting Time Test (Initial and final)**

The test was performed with the aid of a vicat apparatus. The test was conducted at a temperature of about 20 $^{0}$C ± 2$^{0}$C. This is done to determine the time for the solidification of the plastic cement paste. Initial and final setting times are regarded as the stiffening state of the cements [12]. The test was performed at room temperature of 20 $^{o}$C and humidity of 65%.

**Standard Consistence Test**

The test was performed at room temperature of 20 $^{o}$C and humidity of 65%, by following the steps:

1. 400 grams of cement was weighed
2. It was placed on a non-porous plate.
3. Measured volume of water was added.
4. It was mixed by hand with two trowels for 4 min second.
5. The mould was placed on a glass plate.
6. The mould was filled within 15 seconds.
7. The paste was cut and smoothed quickly and gently
8. The mould was centrally placed under the movable plunger of cement consistency test meter.
9. The plunger was lowered gently into contact with the surface of the cement paste and then released.

11. Standard consistence = $\frac{\text{Ml of water added}}{\text{Grams of cement taken}} \times 100$

**RESULTS AND DISCUSSION**

*Compressive Strength*

Compressive strength of the set cements is important as it commonly represents the overall quality of cements. Higher compressive strength generally means lower porosity and increased durability [9]. This is consistent with what is contained in the petroleum industry, where porosity reduces rock strength [1]. Hence, the operators must eliminate porosity effects in the slurry to enable it withstand high pressure and perform properly.

$C_3S$ compound is the chief molecule responsible for early compressive strength development (1-28 days) [12]. The major factor for this is the relative magnitude of CaO compared to $SiO_2$, $Al_2O_3$ and $Fe_2O_3$ in the samples. The best way to optimize local cement compressive strength is to maximize this expression:

$C_3S = 4.07C - 7.6S - 6.72A - 1.43F - 2.85SO_3$ [13]

In Table 3, the steepness of DACS is more compared to SOCS and BUCS. This is because the percentage composition of the compounds such as $C_2S$, and $C_4AF$ which are responsible for hydration rate are more in DACS than the other samples. DAC outperforming the others may also mean that it has good water utility in the short time compared to others. Compressive strength development is more in SOCS than the other samples because the $C_3S$ percent in it is higher than in the other samples. $C_3S$ is responsible for early compressive strength development between 1-28 days [12] In DACS the $C_2S$ percent is high which develop compressive strength more after 28 days. By 28 days, DAC must have fully utilized water during hydration to develop its strength while SOC was still utilizing water for strength buildup.

Table 3: Compressive strength comparison of the three cement samples at 23 ℃

| Time (days) | DACS compressive strength (psi) | BUCS compressive strength (psi) | SOCS compressive strength (psi) |
|---|---|---|---|
| 2 | 45.39 | 43.08 | 45.39 |
| 7 | 83.83 | 81.65 | 79.34 |
| 28 | 156.35 | 136.05 | 178.98 |

The compressive strength of the cement samples increased steadily with time (days).

Table 4 shows the result for setting time at 20 $^0$C and consistency

Table 4: Setting time at 20 $^0$C and consistency

| Sample | Initial Time (min) | Final Time (min) | Consistency (%) |
|---|---|---|---|
| DACS | 83 | 216 | 54 |
| BUCS | 93 | 285 | 54 |
| SOCS | 61 | 156 | 55 |

According to ASTM C191 standard specification the minimum for initial setting time is 45 min [14], and maximum for final setting time is 375 min [15]. Gypsum reduces the speed at which $C_3S$ molecules bind with water in order to set [16]. Setting time is low if there is rapid binding with water., SOCS probably has the lesser gypsum added to it than DACS and BUCS. To optimize the setting time of the cement, lesser quantity of the gypsum is the requirement [1]. The molecule $C_3A$ hydrates rapidly. SOCS has more of it than the other two samples thereby having lower setting time. In other to optimize SOCS set time, a reduction in amount of aluminum oxide and /or increase in iron oxide is required.

To obtain higher compressive strength buildup for high pressure cementing, SOC might work better at late time while DAC might be more efficient at earlier time, based on the results.

Both temperature and water utilization during the hydration process can affect the compressive strength buildup.

## CONCLUSION.

The characterization and evaluation of the selected brands of portland cements have become necessary in order to evaluate the product quality and, hence, the area of application. From the result of the analysis, the compressive strength varies with time. Also, compressive strength increases with rate of hydration. Hence, SOCS and DACS have compressive strength development and setting time property within the recommended limit. However, significant improvement in the local cement are possible by using appropriate measures to step up slurry characteristics. Local cement can be improved with certain molecules such as $C_3S$, $C_2S$ $C_3A$, and oxides such as CaO, $SiO_2$ and $Fe_2O_3$ which increase their compressive strength properties, setting time, rheology making them potentially suitable for onshore and offshore constructions.

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