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A COMPARATIVE STUDY ON THE STRENGTH DEVELOPMENT OF LIMESTONE CALCINED CLAY CEMENT MORTAR AND CONCRETE FOR VARIOUS CLAY CALCINATION TEMPERATURES

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This study examines the physical properties as well as the mineralogical property of Limestone Calcined Clay Cement (LC^3) by investigating the effect of Replacement of Cement with Clay at Calcination temperatures of 600℃, 700℃ and 800℃ in relation to the various compressive strength values gotten for 3 days, 7 days, 14 days and 28 days strength respectively. This was established by the partial replacement of 50% proportion of Ordinary Portland Cement (OPC) with 15% limestone, 30% Clay and 5% gypsum, which were calcined at 600, 700°C and 800℃ in oven and the results were compared with OPC. The results show that the strength of 13.37Mpa mortar strength of LC^3 Uzebba 800℃ ($LC^3_{(U800)}$) is lower to the strength of the OPC at the 28th day which was 21.95Mpa. The results show that the strength of 27.16Mpa Concrete Strength of LC3 Uzebba 800℃ ($LC^3_{(U800)}$) is lower to the strength of the OPC at the 28th day which was 36.76Mpa, but speculating an increase in strength at the 90th day to almost equate the strength of OPC. This study will generally incorporate the Design aspects for sustainability which includes; Green design, Eco-design and sustainable product design. This is a test for sustainability and is carried out to nullify and assuage the effect of the large emission of CO_2 during the production of Portland cement.

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Keywords

Cement, Calcined Clay, Strength, and Green design

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1. Introduction

The population of the world is said to reach about 9.8Billion in 2050 and global Gross Development Product is expected to double (United Nations, 2019). This growth would require a large and immeasurable amount of sustainable material for housing and infrastructure. Generally, concrete is the ideal material to meet many of these challenges, since it is of low carbon, extremely durable and made from resources that are extremely available and can incorporate large quantities and volumes of waste in its manufacturing. But cement on the other hand being one of concrete's materials is contributed for the large emission of CO_2 and thus making it a need to find adequate partial replacement material for the cement clinker in order to produce a more sustainable material for the growing population. This emission is very evident in the production of Portland cement in the heating of the limestone in a kiln were large CO_2 emission about 5-9% globally is noted, this in turn leads to a high negative environmental impact. Generally, Portland cement (PC) contains about 95% clinker with 5% gypsum as its constituent; parts of this clinker in Portland cement can be replaced (partial replacement) with a pozzolanic material which could be fly ash or calcined clay (Avet et al, 2018) (Vizcaino et al., 2015).

A good solution to lower CO_2 emission is to replace part of the cement clinker in cement by supplementary cementitious materials (SCMs) which in this context are Limestone calcined clay cement (LC_3). All other popular SCMs have been put into consideration over time but unfortunately, they are in relatively short supply (together with about 50% of cement production) (AFNOR, 2010; Osbaeck et al, 1995) (Indexed et al.,1883) (Ferreiro et al, 2017). Clays which may be activated by calcination (heating) are the only resource available in significant quantities which can extend the supply of this SCMs. (Raverdy et al,. 1980; Fernandez et al, 2011) Calcined clay can replace up to 30% of the clinker and this replacement is often called PPC30. Higher levels of replacement can be achieved, were about 50% of the clinker is replaced and generally having a constituent of 50% clinker, 30% calcined clay, 15% limestone and 5% gypsum; it is often denoted as LC3-50. (Sanchez et al, 2016).

LC^3 is a blend containing combination of calcined clay and limestone in gravimetric ratios of 2:1 (AFNOR et al, 2010); this is why it is called limestone Calcined Clay Cement. The LC^3 technology has the potential to reduce worldwide CO_2 emissions by more than 400 million tonnes per year at minimized cost also (Yuvaraj et al, 2018). With about 50% replacement of cement clinker, 30% CO_2 emission can be considerably reduced. Under comparison of strength, the LC^3 is found to attain 28-day compressive strengths in similitude with that of Ordinary Portland cements (OPCs) (Indexed et al, 1883). Other earlier slightly similar strength as compared with OPCs and much higher strength as compared to Portland Pozzolana cements (PPCs) is observed in the early 1 to 7-day. (Sanchez et al, 2016)

The major aim is to make LC^3readily available, industrially proven to be adequate to successfully replace the OPCs for multi-use in the global cement industry, as it readily proven to be durable against sulphate attack and other failure systems in aggressive conditions.

2. Methodology

The materials used for this study includes; Ordinary Portland Cement (OPC); which conforms to the BS EN 197-1:2000 (Sanchez et al, 2016) requirements, Limestone ($CaCO_3$), Gypsum; plays major role in workability ($CaSO_4.2H2O$), Calcined Kaolin (Obtained Uncrushed in its natural form from various locations in Delta and Edo State (Irri- Delta state, Ugboroke-Delta state. Kaolin samples were also gotten from Uzebba, Okpella and Ikpeshi), calcination done at the lab using the oven at 600℃,700℃ and 800℃, crushed and calcined), Well graded river Sand (Fine aggregate), Laboratory standard Slaked lime; used more as a curing agent for the mortar cubes, Crushed granite which was gotten from quarries in Edo state and Water.

The Mortar cubes were cast, using the 2 inch or 50mm cubes as per (Kett, 2009), Standard Test Method for Compressive Strength of Hydraulic Cement Mortars. A prefabricated moulds of size 50 x 50 x 50mm were used for producing the test sandcrete cubes. Mechanically mix was done in accordance with the procedure given in Practice C 305. Determine flow in accordance with procedure given in Test Method C 1437. Cast cubes were cured in large buckets with a mixture of slaked lime and water until the age of test required. Test were carried at 3days, 7days, 21days and 28days. In this investigation, OPC was replaced with Limestone, Gypsum and Calcined clay in the following proportions: 50%OPC30%Clay15%Limestone5%Gypsum. Control samples were prepared with 100% OPC. Test was done for Uzebba600$LC^3_{(U600)}$, Uzebba700$LC^3_{(U700)}$, Uzebba800$LC^3_{(U800)}$, Irri700$LC^3_{(IRRI700)}$, Irri800$LC^3_{(IRRI800)}$, Ugboroke700$LC^3_{(UGB700)}$, Ugboroke800$LC^3_{(UGB800)}$, Ikpeshi700$LC^3_{(IKP700)}$

The Concrete cubes were also cast, using 100 x 100 x 100mm prefabricated moulds, mechanically mix using mix ratio 1:1.82:2.95 (Cement: fines: coarse) with cement-water ratio of 0.55. Cast cubes were cured in water tanks without the use of slaked lime, but already used curing water. Tests at 3days, 7days, 14days and 28days were carried out. In this investigation, OPC was replaced with Limestone, Gypsum and Calcined clay in the following proportions: 50%OPC30%Clay15%Limestone5%Gypsum. Control samples were prepared with 100% OPC. Control samples were prepared with 100% OPC. Test was done for LC^3_{Uzebba} ($LC^3_{(U700)}$ & $LC^3_{(U800)}$), $LC^3_{Ikpeshi}$ ($LC^3_{(IKP700)}$ & $LC^3_{(IKP800)}$), $LC^3_{Okpella}$ ($LC^3_{(OKP700)}$ & $LC^3_{(OKP800)}$). The Calcined clay samples used for the concrete test were those that performed well in the sandcrete analysis. The following tests were conducted:

2.1. Sieve Analysis for Fine Aggregate

This test was carried out on the Fine aggregate gotten in order to fully ascertain the percentage passing on the 600micro-meter sieve to be used for the mix design. The test has to do with the use of 9 set sieves arranged in the ascending order (75µm, 150µm, 212µm, 300µm, 425µm, 600µm, 1.18mm, 2.00mm & 2.36mm) 100g of the fine aggregate is measured off the mass volume of river fine sand and placed on the top sieve. Gentle shaking of the set sieves will allow for gravitational pull of the sample to occur, as the sand passing through the sieve hole to be retained on all the sieves. The retained samples were removed from all sieves, measured and recorded.

2.2. Slump Test

This test was accurately done on the concrete with and replacement with different clay sample locations using the slump apparatus which was well cleaned and greased and concrete poured in the cone to fill 3 layers each of which tamped 25times before proceeding to the next layer. Slump Cone is gently removed and the difference between the top of concrete and top cone gives the slump value of the concrete which was measured with a tape and recorded. The slump is carried out as per procedures mentioned in ASTM C143 in the United States.

2.3. Compression Test

All compressive tests were done on the MATEST compressive testing machine. Crushing load for each cube was gotten from the crushing machine screen and recording. The load was applied to the specimen faces that were in contact with the true plane surfaces of the mold. The specimen was carefully placed in the testing machine below the center of the upper bearing block. The load rate of the compressive machine was applied at a relative rate of movement between the upper and lower platen to a loading of 200N/s for the sandcrete but for the concrete a loading rate of 10KN/s was used.

3. Results and Discussion

The compressive test results obtained for the mortar gave unsatisfactorily results and formed no basis of comparison on samples. The test results were thus used as a mode of determining which sample had good performing kaolin content on the clay sample used for the concrete investigation.

Sieve Analysis for Fine Aggregate

The result from the sieve analysis is shown in the table below:

Table 1: Sieve Analysis Results

Sieve Size	Retained (g)	Passing in (g)	Passing in (%)
2.36mm	5.0	95	95
2.00mm	6.8	88.2	88.2
1.18mm	17.3	70.9	70.9
600µm	26.8	44.1	44.1
425µm	8.0	36.1	36.1
300µm	12.3	23.8	23.8
212µm	15.0	8.8	8.8
150µm	4.2	4.6	4.6
75µm	3.0	1.6	1.6

From sieve analysis carried out on the fine aggregate, it was seen that a percentage passing of 44.1% on sieve number 600µm.

Slump Test (Workability)

Table 1 presents the slump test results obtained for concrete samples with 100% OPC and Replacements from various locations of Clay sample. Concrete mix design specified workability at 30-60mm with a coarse aggregate size of 20mm.

Table 2: Table Showing Slump Result and Water-Cement Ratio

Samples	**Composition**	**Slump (mm)**	**Water-cement content**
Control	100% OPC	50	0.55
Ikpeshi 700	50% OPC, 30%Ikpeshi Clay @ 700° *calcination temp*., 15% Limestone, 5%Gypsum	30	0.55
Ikpeshi 800	50% OPC, 30%Ikpeshi Clay @ 800° *calcination temp*, 15% Limestone, 5%Gypsum	15	0.55
Uzebba700	50% OPC, 30%Uzebba Clay @ 700° *calcination temp*, 15% Limestone, 5%Gypsum	30	0.55
Uzebba 800	50% OPC, 30%Uzebba Clay@ 800° *calcination temp*, 15% Limestone, 5%Gypsum	0	0.55
Okpella 700	(50% OPC, 30%Okpella Clay@700° *calcination temp*, 15% Limestone, 5%Gypsum	30	0.55
Okpella 800	(50% OPC, 30%Okpella Clay @800° *calcination temp*, 15% Limestone, 5%Gypsum	0	0.55

From the values obtained, the control (OPC) had a slump value of 50mm hence, highly workable since it falls within the mix range of 30-60mm. lower values are seen from the LC3 to show a decrease in the workability of the OPC due to the replacement. Higher calcined temperature showed reduced slump value and even attains zero slump at 800℃ for Uzebba and Okpella.

3.3. Compression Test (Sandcrete)

The results gotten from the mortar tests formed no real base of comparison and thus, but was used as a guide to determine which sample was performing well and which was not performing well. The performing samples were used to carry out the concrete test while the non-performing sample discarded forthwith. The Table below shows the Results obtained.

Table 3: Plot for Uzebba (50% OPC, 30%Uzebba Clay., 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average Compressive Strength

	Strength (Mpa) OPC	Strength (Mpa) at 600C	Strength (Mpa) at 700C	Strength (Mpa) at 800C
3 Days	9.04	3.22	7.946	6.89
7 Days	14.96	7.69	16.41	13.27
21 Days	14.09	5.17	14.4	7.91
28 Days	21.95	8.57	8.03	13.37

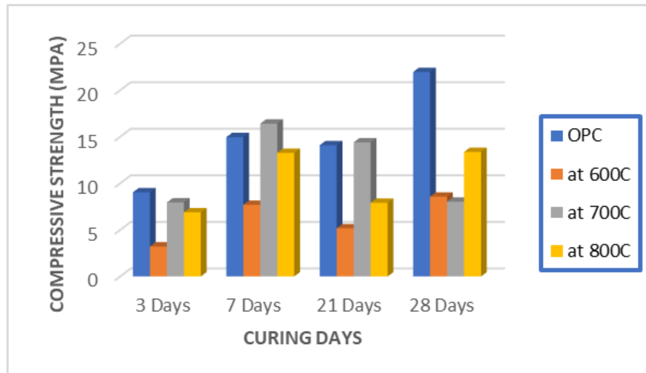

Figure 1: Bar chart showing Strength at different Curing days of OPC and Uzebba at 600, 700 and 800 °C

Table 4: Plot for Irri (50% OPC, 30%Irri Clay., 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average Compressive Strength

	Strength (Mpa) OPC	Strength (Mpa) at 700C	Strength (Mpa) at 800C
3 Days	9.04	5.76	7.55
7 Days	14.96	7.22	11.74
21 Days	14.09	7.53	11.2
28 Days	21.95	12.34	11.23

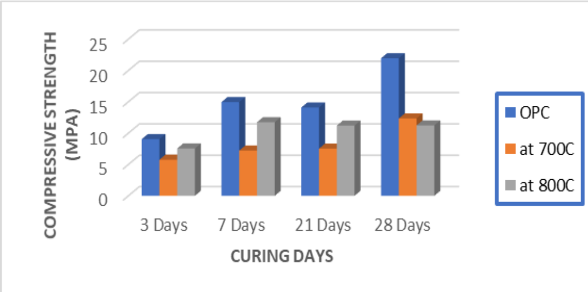

Figure 2: Bar chart showing Strength at different Curing days of OPC and Irri at 700 and 800 temperatures.

Table 5 Table of plot For Ugboroke (50% OPC, 30%Ugboroke Clay., 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average Compressive Strength

	Strength (Mpa) OPC	Strength (Mpa) at 700C	Strength (Mpa) at 800C
7 Days	14.96	6.5	6.22
28 Days	19.47	8.83	10.64

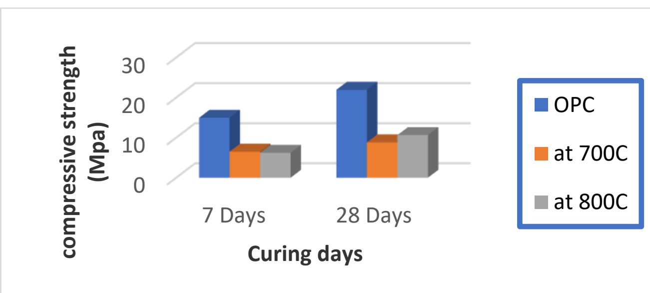

Figure 3: Bar chart showing Strength at different Curing days of OPC and Ugboroke at 700 and 800 temperatures

Table 6 Table of plot For Okpella (50% OPC, 30% Okpella Clay, 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average Compressive Strength

	Strength (Mpa) OPC	Strength (Mpa) at 700C
3 Days	9.04	6.25
7 Days	14.96	9.43
28 Days	21.95	19.23

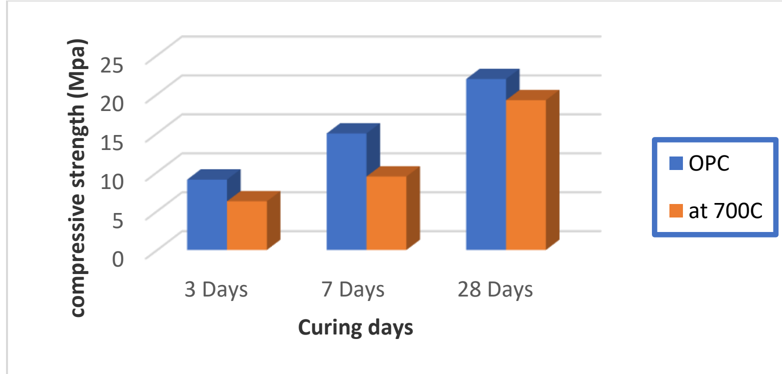

Figure 4: Bar chart showing Strength at different Curing days of OPC and Okpella at 700 degrees

Table 7 Table of plot For Ikpeshi (50% OPC, 30%Ikpeshi Clay., 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average Compressive Strength

	Strength (Mpa) OPC	Strength (Mpa) at 600C	Strength (Mpa) at 700C
7 Days	14.96	11.44	11.27

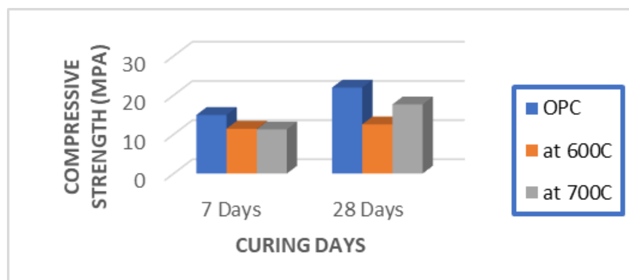

Figure 5 Bar chart showing Strength at different Curing days of OPC and Ikpeshi at 600 and 700°C

The compressive test results obtained for the mortar gave unsatisfactorily results and formed no basis of comparison on samples. The test results were thus used as a mode of determining which sample had good performing kaolin content on the clay sample used.

The Irri and Ugboroke–Warri sample showed considerable reduced strength at later days, this deterioration in strength showed that the sample had low kaolin content and when calcined, the activation energy wasn't high enough to give satisfactorily results.

The LC_3 Uzebba 800°C at 28days strength in comparison with the control OPC shows progressive results almost equating in strength with an average compressive strength value of 13.37Mpa and 21.95Mpa in the later and former samples respectively, Speculations shows from past tests that, this sample would greatly supersede OPC at much longer times say, 90 days.

Compression Test (Concrete)

Analysis of the compressive strength for the concrete cube specimens of design mix of control and 600°c, 700°c, and 800°c temperature for 3, 7, 14, and 28 days and with loading rate of the compressive machine set at 10KN/Sec. are summarized in the results given in the tables below.

Table 1: Plot for Uzebba Sample (50% OPC, 30%Uzebba Clay., 15% Limestone, 5%Gypsum) in Comparison with OPC Control Using Average Compressive Strength.

	OPC	at 700C	at 800C
3 Days	20.76	12.09	13.5
7 Days	27.82	15.06	20.83
14 Days	29.98	20.49	27.51
28 Days	36.76	21.03	27.16

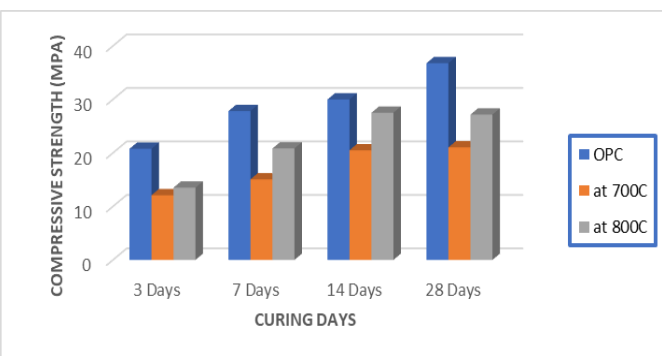

Figure 6: Strength at different Curing days of OPC and Uzebba at 700 and 800 Centigrades

Table 9: Plot for Ikpeshi (50% OPC, 30% Ikpeshi Clay, 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average

	OPC	at 700C	at 800C
3 Days	20.76	12.52	16.28
7 Days	27.82	11.19	22.55
14 Days	29.98	16.53	19.01
28 Days	36.76	19.59	28.93

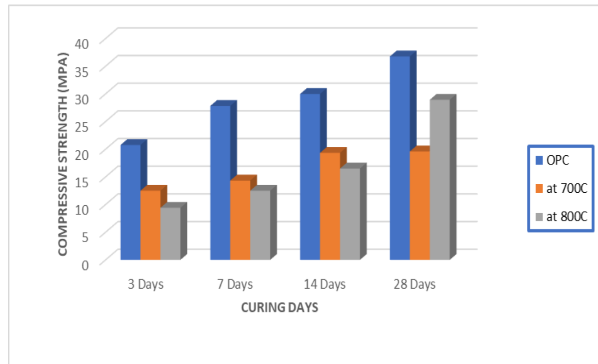

Figure 7: Strength at different Curing days of OPC and Ikpeshi at 700 and 800°C

Table 10: Plot for Okpella (50% OPC, 30%Okpella Clay., 15% Limestone, 5%Gypsum) Sample in Comparison with OPC Control Using Average

	OPC	at 700C	at 800C
3 Days	20.76	12.5	9.41
7 Days	27.82	14.33	12.5
14 Days	29.98	19.34	16.5
28 Days	36.76	20.09	16.66

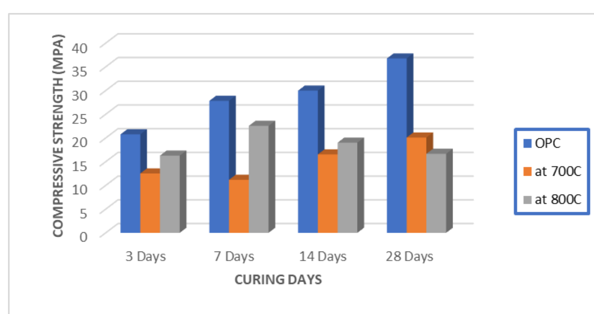

Figure 8: Strength at different Curing days of OPC and Okpella at 700 and 800°C

From the Figures and Tables above, Concrete Cube of 100mm size were prepared with LC3 and OPC using a mix ratio for Grade 20 as, 1: 1.82: 2.95 and tested on kaolinite clay from 3 different samples at 2 different calcined temperatures which are 700℃ and 800℃.

The strength at 3, 7, 14, and 28 days were measured as shown in Figures above. The test result for 28days is pending; hence discussion will be based on 14days. The results show that the strength of concrete with higher calcining temperature gives higher results for LC^3_{Uzebba} ($LC^3_{(U800),}$ $LC^3_{(U700)}$) and $LC^3_{Ikpeshi}$ ($LC^3_{(IKP800),}$ $LC^3_{(IKP700)}$) as seen from the values above. Okpella on the other hand gives higher compressive strength values at 700℃ as compared to 800℃, $LC^3_{(OKP800),}$ $LC^3_{(OKP700)}$) at 3, 7 and 14days strengths when compared.

According to studies done, the temperature to Compressive strength test for Clay has been proven to be justifiable in comparison with the Ikpeshi sample. The LC_3 Ikpeshi 800℃ at 28 days strength in comparison with the control OPC shows progressive results almost reaching target strength of grade 30 with an average compressive strength value of 28.93Mpa and 36.76Mpa in the later and former samples respectively, Speculations shows from past tests that, this sample would greatly reach the targeted strength at the 90days strength. But Progress reduction in strength is observed for the Uzebba 800℃ sample which reduced from 27.51Mpa to 27.16Mpa due to leaching as a result of curing in water. Curing in Air, would achieve better results. The bar chart below shows the Compressive strength results gotten from the study done by the LC3 team at Cuba and Switzerland by the Ecole Polytechnique Federale De Lausanne.

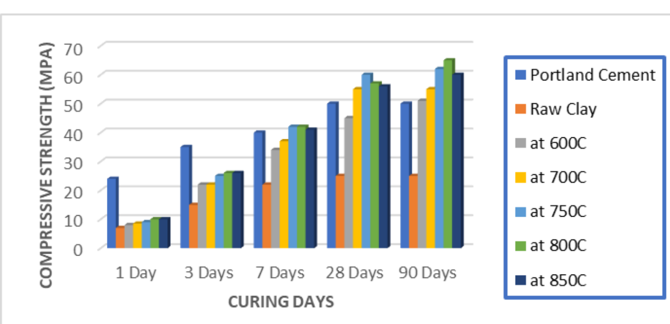

Figure 9: Strength at different Curing days of PC, raw clay & Calcined Clay at different temperatures (Ecole Polytechnique Federale De Lausanne Switzerland, Cuba).

4 Conclusion

Giving the vast availability of the sample, as it abounds in different locations in Nigeria and in the world, it can be used as a sustainability material to promote and strike a balance between Economic, Social and Environmental issues and development that will lead to the attainment of the United Nation's sustainable Development goals as stipulated in UN circular of September, 2015. (Goal3 (Good Health & Well-being), Goal13 (Climate Action), Goal9 (Industry, Innovation and Infrastructure), Goal11 (Sustainable Cities and Communities)) The experimental study hoped to reach a breakthrough to the replacement of 50% Cement with Limestone, Calcined Clay and Gypsum. And comparing the strength development of the mixes and the control (100% OPC) Concrete and Sandcrete after 28days strength development has been reached.

The mortar Test following the methodology, gave unsatisfactory results. This unsatisfactory result gotten from the mortar strengths in comparison to the control may be due to a whole lot of reasons as earlier discussed. As the temperature increased, a much greater strength was expected but this led to a retrogression of strength with respect to expectations.

The mortar test results were used to streamline the test samples by discarding the Irri and Ugboroke samples. The Uzebba, Ikpeshi and Okpella sample used for the Concrete test gave satisfactory results with the data gotten currently.

The concrete test results demonstrated from the performance of Ikpeshi 800 (LC3) fulfills the aim of the experiment as greater strength will be observed in terms of serviceability for later days and in terms of economy. Curing in Air for concrete samples will produce much higher strength and reduce the factor of leaching in concrete. Initial economic feasibility assessment demonstrates LC3 production attractiveness. LC3 has high sustainability as well as serviceability to be used in concrete mixes and structural building projects.

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