

Flexural Properties of Blended Mortars Incorporating Limestone Overburden Clay

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Received: 14TH OCT 2025

Reviewed: 2ND NOV 2025

Accepted: 17TH NOV 2025

ABSTRACT

Overburden clays are abundant in limestone quarries which are mostly stripped at a high cost to expose the underlying limestone and carted away to dumpsites. These clays when calcined at specific temperatures can be used as substitutes for clinker in the production of cement. The substitution of clinker with calcined clays has great economic and environmental benefits by reducing greenhouse gas emissions and well as cost of burning clinker. This study investigated the effects of calcination temperature of limestone overburden clay on the flexural properties of blended mortars, to determine its suitability as a partial replacement material for clinker in cement production. Mortar samples were cast using Lab cement produced with clinker substituted with limestone overburden clay calcined at temperatures of 500°C, 600°C, 700°C and 800°Cs, with replacement levels of 30%, 50% and 70% respectively. Mechanical properties were determined at curing ages of 1day, 3days, 7days, 28days, and 90days respectively. Results were compared to performance of CEM 1 which was the control. 600°C was found to be the optimum calcination temperature with flexural strengths of up to 5.9N/mm² at 30% replacement and 90days curing, compared to CEM 1 which had strengths of 7.8N/mm². The oxide composition of the clay had a combined sum of SiO₂, Al₂O₃ and Fe₃O₂ amounting to more than 70% of the total oxides, making it a good natural pozzolan. Water absorption was on average of 8%, less than 10% which qualifies as good mortar.

Keywords: Limestone, Cement, CEM I, Flexural strength, Mortar, Overburden clay.

I. INTRODUCTION

Housing deficit in most developing countries has hindered growth and kept poverty on the high side. One of the reasons for this housing deficit is the high cost of building materials which has been a threat to both construction industry and people aspiring to own houses (Akanni et al., 2014). The building materials industry contributes significantly to global carbon dioxide content in the atmosphere, with cement production accounting for 5% of global carbon dioxide emissions (Jaskulski et al., 2020). According to Kamau et al., (2018), the highly energy-intensive processes that are involved in the production of cement contribute about 7 to 10% to the total global man-made carbon dioxide (CO₂), which is the main cause of global warming; also economically expensive. It has been found through research

that cement is the most utilized construction material in the world, and only second to water as the most consumed commodity (Kamau et al., 2018).

The use of limestone calcined clay cement (LC³) as binders with replacements levels up to 40% have shown a reduction in total CO₂ emission by up to 38% compare to conventional Portland cement (Hazarika et al., 2025). Abdullah et al., (2023) on their part stated that CO₂ emissions in cement production can be reduce by up to 30% with the technology of LC³ which involves a blend of up to 50% clinker, 30% calcined clay, 15% limestone and 5% gypsum. Adjustment in the blend can further reduce CO₂ emissions by up to 40%, resulting in energy and cost efficiencies, as well as savings in terms of greenhouse emissions.

Clay is a widely spread material in the world, cheap and easily accessible (Neiber-Deiters et al., 2019). It is a type of soil which is composed of very fine particles usually silicates of aluminium and magnesium. Dynamis, a Brazilian company owned by Loesche in 2021, the production of cement based on the clinker as the main raw material comes at a high cost in thermal and electrical energy consumption for industries, in addition to being one of the main responsible factors for the emission of CO₂, a main polluting gases of the atmosphere.

Several studies have shown calcined clay as a sustainable replacement for clinker that can reduce the total costs of cement production. Limestone calcined clay is a clay material obtained from a limestone area, mostly overburden soil on top of limestone that has been burnt in a kiln to a specific temperature in a process of calcination.

The idea of Limestone Calcined Clay Cement (LC³) is based on the observation that cements containing alumina reacts with carbonate phases to produce carboaluminate phases that are hard and crystalline and contribute to the development of the microstructure (Sharma et al., 2021), Akindehinde et al., (2020) posited that for clays to be suitable for clinker replacement, their kaolin content must be up to 30% and above.

In a study by Kaleeswari et al., (2016), Ordinary Portland Cement (OPC) was replaced by 5%, 10%, 15%, and 20% of raw clay. The Compressive strengths of the cement mortar mix 1:4 at 28th day of curing at the various levels of replacement were 29.19, 27.91, 26.79 and 20.58Mpa respectively.

Shah et al., (2018) in their research on cement replacement with limestone calcined clay pozollans (LCCP) in mortar at 10%, 15%, 20%, 30% and 50% respectively, showed similar or higher compressive strength compared to OPC.

According to Elimbi et al., (2011), kaolinite clays used in production of Geopolymer cement had higher compressive strengths at calcination temperature of between 500 and 700 °C (11.9 and 36.4N/mm²). At higher temperatures of calcination the compressive strength drops. The setting time varied between 130 and 40 min for clay fractions calcined between 500 and 700 °C, and began to increase above 700 °C.

Abdulqader et al., (2023), in their research on LC³ with white clay (WC) and Yellow clay (YC) from Saudi Arabia and Ukrainian clay (UC), they had the best result with UC at 30% clinker substitution, with 7days having 25Mpa, 28days having about 32Mpa, and 90days with about 40Mpa.

Porosity as a property of any material affects its durability. The more the porosity, the more the level of moisture absorption. On the other hand, the lower the porosity of a material, the lower the amount of moisture it absorbs. High

moisture absorption has a negative effect on durability. Clay materials tend to absorb more water, however, good and durable mortar mixes should have water absorption well below 10% by mass (Bohan et al, 2011). According to Chai et al (2019), water absorption has an opposite relationship with the compressive strength.

Limestone Calcined Clay cement (LC³) were discovered to have a higher setting time than ordinary Portland cement (OPC), especially final setting time (Mwiti et al., 2018).

Clay soils are very fine soils that exhibit shrinkage and swelling due to the presence of trapped voids which tend to reduce their specific gravity. It is therefore expected that at calcination, the trapped air will be released, resulting in improved specific gravity (Mac-Eteli and Sopakirite 2021). Most of the existing related studies focussed on compressive strength, hence the need to explore flexural strength in this study.



FIGURE 1: OVERBURDEN BEING STRIPPED TO EXPOSE LIMESTONE

II. MATERIALS AND METHODS

The materials used for this research included:

- i. Limestone overburden clay
- ii. Portland Clinker
- iii. Gypsum

Production of lab cement

The lab cement (CEM I) was produced using a Lab Ball Mill with a characteristic strength of 42.5MPa and a total of 5kg produced at once. The control cement was produced with 90% clinker and 5% gypsum.

Other lab cements were also produced with clays calcined in a locally made kiln to temperatures of 500°C, 600°C, 700°C and 800°C respectively, by keeping the gypsum addition at 5%, while clinker was substituted with calcined clay was 30%, 50% and 70% respectively by weight of clinker. Table 1 shows the composition of materials for lab cement production. A total of Twelve cement samples were produced for calcined clay addition at various calcination temperatures and replacement levels, as well as One sample for CEM 1 which was the control.

TABLE I
LAB CEMENT PRODUCTION AT VARIOUS LEVELS OF CLINKER REPLACEMENT WITH CALCINED CLAY

MATERIAL	CONTROL (CEM 1)	30% REPLACEMENT	50% REPLACEMENT	70% REPLACEMENT
Clinker (kg)	4750	3325	2375	1425
Calcined Overburden Clay (kg)	0	1425	2375	3325
Gypsum (kg)	250	250	250	250
TOTAL (kg)	5000	5000	5000	5000

TABLE II
XRF OXIDES COMPOSITION OF CEM I AND CALCINED OVERBURDEN CLAY

OXIDES MATERIAL	Silicon Dioxide SiO ₂	Aluminium Oxide Al ₂ O ₃	Ferric Oxide Fe ₂ O ₃	Calcium Oxide CaO	Magnesium Oxide MgO	Sulphur trioxide SO ₃	Potassium Oxide K ₂ O	Sodium Oxide Na ₂ O	LOI
Control (CEM I)	19.53	5.06	3.16	62.07	2.60	2.51	1.01	0.14	2.06
Calcined Overburden clay	73.59	10.47	5.74	0.51	0.11	0	0.62	0	7.6



FIGURE 3: LAB CEMENT PRODUCTION IN A LAB BALL MILL



FIGURE 4: MORTAR PRISMS CASTING

Flexural properties of mortar prisms

Mortar prisms measuring 40x40x160mm were cast for CEM 1 and other cement samples with 450g of binder and 1350g of fine aggregate in a 3-in-one mould, and water to binder (w/b) ratio of 0.5. The cast prisms were de-moulded after 24hrs and placed inside a curing tank for 1day, 3days, 7days, 28days and 90days curing. Three prisms were produced for each sample with a total of 195 prisms produced.

Flexural strength test was carried out of each mortar prism to determine mechanical strengths of mortar prisms.

Setting time

Initial setting times (IST) and final setting time (FST) of pastes were carried out with a Vicat Apparatus and an automatic Toni Technik ToniSET machine. A total of 500g of sample was used to prepare the past at various trials of water to binder ratio and poured into a conical plastic mound with top diameter 70mm, bottom diameter 80mm and height of 40mm, placed on top of a glass disc base. The water content that allowed the plunger travel only between 4mm to 8mm in the paste was the water demand or consistency for the paste. The consistent pastes were place in the automatic Toni Technik ToniSET machine which reads and record the IST and FST.

III. RESULTS AND DISCUSSION

Results obtained from the experimental work of this project were compared to the result from the same test done on CEM I

Flexural strength strength showed an increase from 500°C to 600°C, and then dropped at 700°C and 800°C for 7 days, 28 days and 90 days respectively. There were similar strength behaviours at 50% and 70% replacements, indicating that 600°C is the optimum calcination temperature for the overburden clay obtained at the quarry site.

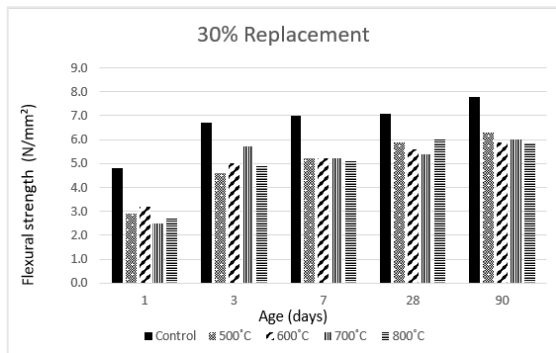


FIGURE 5: 30% REPLACEMENT FLEXURAL STRENGTH

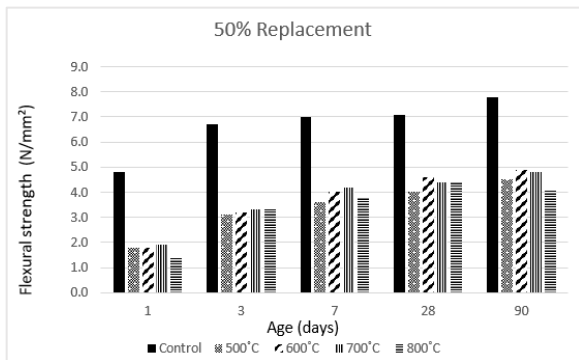


FIGURE 6: 50% REPLACEMENT FLEXURAL STRENGTH

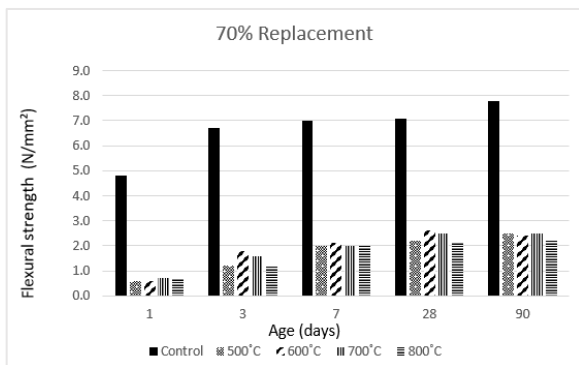


FIGURE 7: 70% REPLACEMENT FLEXURAL STRENGTH

Specific gravity (SG)

From figure 8, CEM I has a higher specific gravity of 2.93 than calcined overburden clays which have values ranging from 2.4 to 2.5. In contrast to the findings of Mac-Eteli and Sopakirite (2021) which concluded that SG increases with increase in calcination temperature, the close values of the calcined clays indicated that the calcination temperature did not have much effect on the specific gravity of the overburden clay at the quarry site.

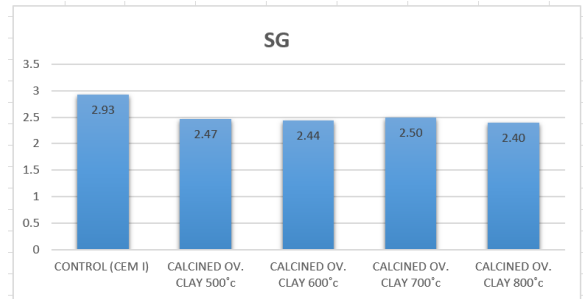


FIGURE 8: SPECIFIC GRAVITY FOR CEM I AND CALCINED OVERBURDEN CLAYS

Setting time

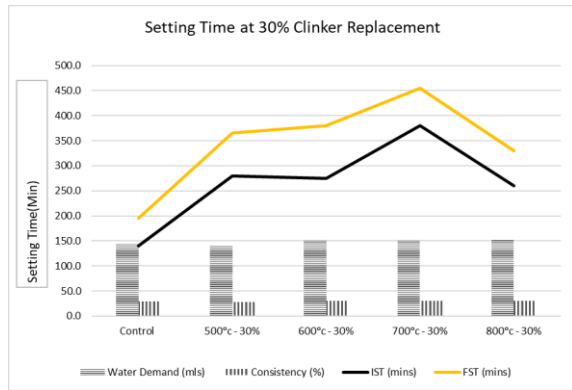
From figure 9, the water demand was relatively steady at 30% replacement at about 150mls, 50% replacement at 165mls and 70% replacement at 175mls, while the CEM I was 145mls, and 140mls for 30% replacement for 500°C. The consistency for CEM I and 30% of 500°C was 29.8% and 28% respectively, while other calcination temperatures and different percentage replacement were relatively steady ranging from 30% to 35%.

Initial setting time for CEM I was 140mins, while 30% replacements varied between 260mins to 280mins, except 700°C which was higher at 380mins for 30% replacement. Initial setting time for 50% and 70% replacements ranged between 300 and 380mins except for 50% of 800°C that was low at 270mins. All these were far higher than that of the CEM I.

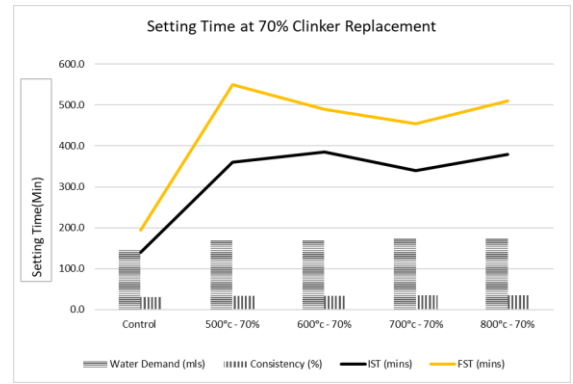
Final setting time for CEM I was 195mins, while that for all replacements at different calcination temperatures was far above 300mins. 500°C- (30% and 50%), 600°C -30%, 700°C -50% and 800°C- (30% and 50%) all had final setting time of between 330mins and 380mins. 500°C – 70% and 800°C – 70% had final setting time as much as 500mins.

The expansion for the control was 0.5mm, while all other samples had expansions between 0.5mm to 1.0mm except for 800°C – 70% that was 2mm.

In comparison to the result obtained by Abdulqader et al., (2023), Ukrainian clay (UC30) with IST of about 170mins and FST of about 350mins, 600°C -30% had higher values, with IST of 275mins and FST of 380mins.

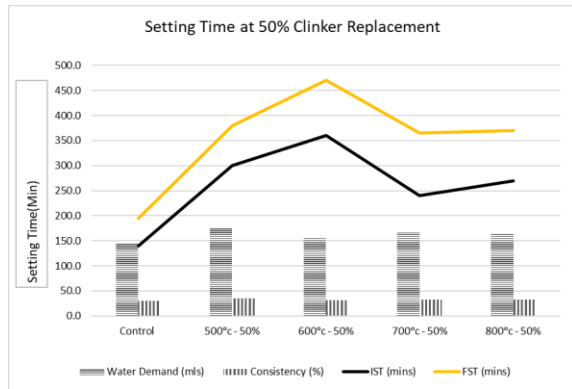


(a)



(c)

Figure 9: Setting time (a) 30% replacement (b) 50% replacement (c) 70% replacement



(b)

Durability

From figure 10 below, the CEM 1 had a water absorption of 6.3%. 30% replacements had up to 7% water absorption, 50% replacements had about 8% water absorption, while 70% replacements had up to 9% water absorption. This meant that water absorption increased with increase in clay content.

No significant difference in water absorption across different calcination temperatures between 500°C and 800°C, all the samples had water absorption less than 10%, meeting the requirement of good mortars (Bohan et al, 2011).

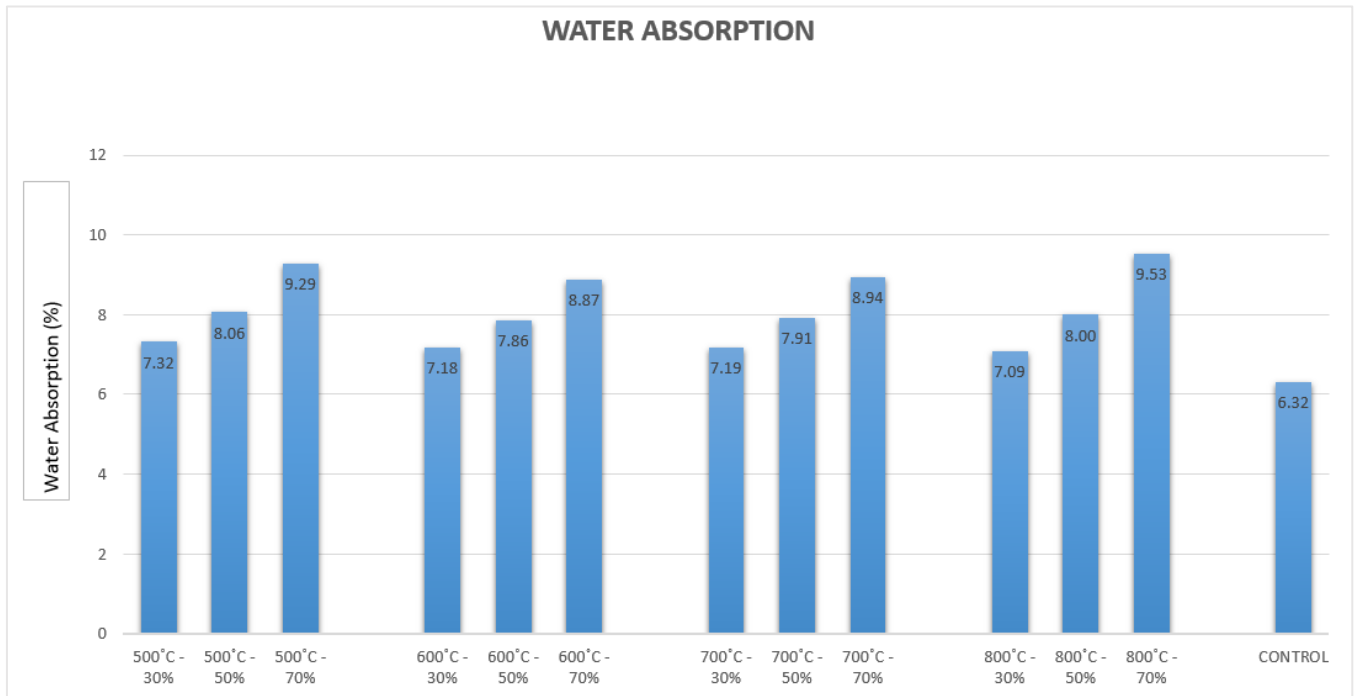


FIGURE 10: WATER ABSORPTION

IV. CONCLUSION

Based on the various tests carried out on the limestone overburden clay, the following conclusions could be reached.

Up to 30% replacement gave a flexural strength of between 5.9N/mm² and 6.3N/mm² for 500°C as against 7.8N/mm² for the control sample. 600°C calcination also gave a good flexural strength of 5.9N/mm² at 30% replacement. The flexural strength had a marginal increase from 5.9N/mm² at 600°C calcination to 6.0N/mm² at 700°C, then dropped again to 5.9N/mm² at 800°C, all at 30% replacement at 90 days.

The best mechanical performance was obtained at 600°C calcination and 30% replacement. The results suggest that overburden clays could replace clinker up to 30% for structural concrete and up to 50% for concrete with low structural significance. Also, 600°C was found to be the ideal calcination temperature from overburden clays in this limestone quarry.

Chemical oxides composition

The Calcined Overburden Clay satisfied the requirement of Natural Pozzolans, with the combined sum of SiO₂, Al₂O₃ and Fe₃O₂ amounting to more than 70% of the total oxides content. The control sample (CEM 1) on the other hand had very high CaO content and relatively low SiO₂.

Specific gravity

The close values of the calcined clays indicated that the calcination temperature did not have much effect on the specific gravity of the overburden clay at the quarry site

Setting time

The water demand was relatively steady at 30% replacement at about 150mls, 50% replacement at 165mls and 70% replacement at 175mls, while the control was 145mls, and 140mls for 30% replacement for 500°C. IST for the different replacement percentages was as high as 380mins compared to the control with IST of 140mins. The FST for the replacement was even very high ranging from 330mins to 550mins compared to 195mins for the control. Additives may be required to improve the IST and FST of the cement produced with clinker substitution with calcined overburden clay.

The expansion for all replacements and calcination temperatures was between 0.5mm and 1mm, except for 800°C – 70% which had 2mm. Expansion for the control was 0.5mm.

References

- [1] A. Elimbi, H.K. Tchakoute, D. Njopwouo, (2011) Effects of calcination temperature of kaolinite clays on the properties of geopolymers cements, Journal of Construction and Building Materials Volume 25, Issue 6
- [2] Abdullah, Anand Kumar, Abhinav Panwar, Kumar Ayush (2023), Development and Experimental Investigation On Lime Calcined Clay Cement. International Research Journal of Modernization in Engineering Technology and Science
- [3] Akindehinde A, François A, and Karen S, (2020) The Influence of some calcined clays from Nigeria as clinker substitute in cementitious systems. Case Studies in Construction Materials.
- [4] Amrita Hazarika, Liming Huang, Sigurdur Erlingsson, Klaartje de Weerd, Ingemar Lofgren, Sahar Iftikhar, Arezou Babaahmadi. (2025). Characterization, activation and reactivity – A case study of Nordic volcanic materials for application as Supplementary Cementitious Materials
- [5] Axel Neiber-Deiter, Sebastian Scherb, Nancy Beuntner, Karl-Christian Thienel, (2019) Influence of the calcination temperature on the properties of mica mineral as a suitability study for the use as SCM. Applied Clay Science Vol. 179
- [6] Bohan N, Mohamed S, (2011) Laboratory Study of Water Absorption of Modified Mortar, e-Journal of Civil Engineering Vol. 2 (1)
- [7] Calcined clays - Understand what it is and its benefits in Cost Reduction in Cement Production by, Dynamis, a Brazilian company owned by Loesche, (2021)
- [8] Chai Teck Jung, Tan Cher Siang, Tang Hing Kwong, Koh Heng Boon. (2019), Compressive Strength and Water Absorption of Mortar Incorporating Silica Fume
- [9] H. D. Mac-Eteli, S. Sopakirite (2021) Experimental Analysis on the Effect of calcination on the Index and Engineering Properties of Clay Soil. Saudi Journal of Civil Engineering
- [10] John Kamau, Ash Ahmed, Paul Hirst and Joseph Kangwa (2018), Suitability of Anthill Soil as a Supplementary. European Journal of Engineering Research and Science, Vol. 3, No. 7
- [11] Kaleeswari. G, Dhanalakshmi. G, and Manikandan. N, published (2016) Clay as a Partial Replacement of Cementitious Material in Cement. International Journal of Advanced Research in Biology Engineering Science and Technology (IJARBEST), Vol. 2, Issue 3,
- [12] Marangu J. Mwiti, Thiong'o J. Karanja, Wachira J. Muthengia (2018), Properties of activated blended cement containing high content of calcined clay. Heliyon 4 (2018)

- [13] Marwan Abdulqader, Hammad R. Khalid, Mohammed Ibrahim, Saheed K. Adekunle, Mohammed A. Al-Osta, Shamsad Ahmad, Muhammad Sajid (2023), Physicochemical properties of limestone calcined clay cement (LC3) concrete made using Saudi clays. *Journal of materials research and Technology*.
- [14] Meenakshi Sharma, Shashank B, Fernando M, and Karen S, (2021), Limestone calcined clay cement and concrete: A state-of-the-art review, *Journal of Cement and Concrete Research* Volume 149, November 2021
- [15] P. O. Akanni, A. E. Oke, and O. J. Omotilewa. (2014). Implications of Rising Cost of Building Materials in Lagos State Nigeria. *Open Access Journals*.
- [16] Roman Jaskulski, Daria Jó'zwiak-Nied 'Zwiedzka and Yaroslav Yakymchko (2020) Calcined Clay as Supplementary Cementitious Material. *Open Access Journals*.
- [17] Vineet Shah, Satya Medepalli, Geetika Mishra, Sreejith Krishnan (2018), Influence of cement replacement by limestone calcined clay pozzolan on the engineering properties of mortar and concrete. *Advances in Cement Research*, Volume 32, Issue 3, 31 October 2018, Pages 101-111