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Research Article

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Strength Characteristics of Concrete Produce with Partial Replacement of Cement with Calcined Halloysite

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Abstract: Cement is the most commonly used binding material in construction. However, its high cost and heat of hydration have led to research on alternative materials for partial replacement in concrete. This study investigates the rheological and strength properties of concrete incorporating calcined halloysite (CH) as a partial substitute for cement. Concrete samples were prepared with 5% to 20% CH replacement by weight of Portland Elephant Supaset cement of grade 42.5N. Tests were conducted to determine the chemical composition, workability, compressive strength, water absorption, and flexural strength. The concrete cubes were tested at 7, 14, 21, and 28 days. Results showed that CH is a good pozzolanic material, with a combined SiO_2, Al_2O_3, and Fe_2O_3 content of 83.90%, Workability decreased with increasing CH content, indicating lower slump and compacting factor. Compressive strength improved with CH replacement, with significant gains observed up to 28 days as a result of pozzolanic activities of calcined halloysite which densifies the concrete medium and reduces pore size. Water absorption increased with higher CH content. The flexural strength of beams also improved with CH addition. It was concluded that 5% and 10% CH replacement meet the British Standard strength requirements for structural applications.

Keywords: Calcined Halloysite, pozzolanic material, Compressive strength, Portland Elephant Supaset cement and flexural strength.

1. Introduction

Halloysite is a kind of 1:1 clay mineral having a special nanosized tubular morphology and pore structure, [1]. Halloysite is usually found in soils formed from volcanic deposits, particularly volcanic ash and glass. It is a common clay mineral in the Andisol soil order. The demand for cost-effective building materials is essential in meeting the growing need for adequate housing worldwide. The increasing prices of traditional construction materials continue to be a major challenge, particularly for low-income communities. This has driven interest in locally sourced alternatives that offer both functionality and affordability in urban and rural settings. Various natural and industrial by-products, such as earthen plaster, have been investigated for construction applications [2]. The accumulation of industrial waste, agricultural residues, and other by-products poses serious environmental concerns, especially regarding disposal and pollution. The construction sector plays a crucial role in addressing these issues by incorporating such materials as fillers in concrete production. If these fillers exhibit pozzolanic properties, they can enhance concrete performance while allowing for greater cement replacement [3]. Utilizing these materials effectively can provide both environmental and economic advantages. Since the invention of cement material, research has continued in the field of construction to make structures more resistant, more durable and also more economic. [4]. Cement production significantly impacts the environment due to its high energy demands and CO_2 emissions, creating an urgent need for sustainable alternatives. One promising solution is the partial replacement of Portland cement with pozzolanic materials [5] Pozzolans, whether natural or artificial, contain amorphous silica that reacts with calcium hydroxide (CH) to generate additional cementitious calcium silicate hydrate (C-S-H), improving concrete's strength and durability. Among these materials, calcined halloysite (CH) stands out as a highly reactive alumino-silicate clay mineral formed through thermal processing. Similar to kaolin, halloysite is rich in silica and alumina and is naturally abundant. When exposed to temperatures around 600°C, halloysite transitions from a crystalline to an amorphous phase, significantly increasing its reactivity in cement-based applications [6]. Despite Nigeria's vast deposits of halloysite minerals, research on its potential as a supplementary cementitious material remains limited. This study investigates the viability of using calcined halloysite as a partial cement replacement, analyzing its impact on the rheological properties and strength development of concrete.

2. Methodology

Materials Used

The halloysite used for this research was obtained from Ewekoro, Ogun State. To ensure purity, it was carefully collected to avoid contamination with sand or other foreign materials. The coarse aggregate consisted of granite with a maximum size of 12mm, while the fine aggregate was clean and free from impurities. Both aggregates were sourced from a local supplier in Ibadan, Oyo State, Nigeria. Portland Supaset cement served as the primary binder, and the mixing water was drawn from a borehole. The water was clear and devoid of visible impurities.

Preparation of Calcined Halloysite

The halloysite sample was subjected to calcination in a furnace at 600°C, following the method outlined by [7]. The heating process continued until a noticeable color change occurred, confirming successful calcination. Once cooled, the calcined halloysite was finely sieved using a 75µm sieve to ensure uniform particle size for optimal reactivity with Portland Supaset Cement. A chemical analysis test was then performed to determine its composition.

Sieve Analysis

The calcined halloysite (CH) was collected and weighed before being ground into finer particles using a milling machine. The ground material was then sieved to ensure uniform particle size. Sieve analysis was conducted manually in the Soil Mechanics Laboratory at the Civil Engineering Department, Ajayi Crowther University oyo, oyo state as shown in figure 1, using a set of sieves with a 1.4µm diameter. The sieved sample was weighed, and the particle size distribution was determined. A grading curve was then plotted to analyze the particle size characteristics.

Figure 1: Sieve Analysis on calcined halloysite (CH)

Production of Calcined Halloysite (CH) Concrete

The manufacturing of calcined halloysite cement concrete was conducted at the Civil Engineering Laboratory, Ajayi Crowther University oyo, oyo-state. The process involved batching the concrete mix in precise proportions, followed by casting the prepared specimens for further testing and analysis.

Batching of Calcined Halloysite Admixture

A weighing balance, along with a head pan, shovel, concrete cube mould, and tamping rod, was used for the batching process. The calcined halloysite cement concrete was composed of a mixture of halloysite ash, Portland Supaset cement, sand, granite, and water. The materials were batched by weight in kilograms, ensuring accurate proportions. The mixing process involved replacing 5%, 10%, 15%, and 20% of OPC with calcined halloysite (CH). Initially, the cement and CH were mixed together, followed by the addition of fine and coarse aggregates. A control sample was prepared without calcined halloysite for comparison. Table 1 presents the material proportions used for producing calcined halloysite cement concrete.

Table 1: Mixed proportion for concrete samples

% replacement of CH	Weight of cement	Weight of CH	Water cement ratio (WCR)
0	12.64	0	0.60
5	12.17	0.61	
10	11.87	1.13	
15	10.45	1.43	
20	10.65	2.72	

Preparation of concrete and casting of specimens

The mix ratio used for the concrete was 1:2:4, with a water-cement ratio of 0.60 to achieve the desired compressive strength. Cubic samples of 150 × 150 × 150 mm³ were cast to evaluate compressive strength as shown in figure 2, after curing for 7, 14, 21, and 28 days. After curing, the concrete cubes were removed from the water tank and allowed to rest for 45 minutes before undergoing compression testing. To ensure uniform distribution while placing the mixed concrete into the moulds, a scoop was moved around the mould's edges. Each layer was compacted with 35 strokes using a tamping rod, and the surface was leveled with a trowel. The samples remained in the moulds for 24 hours, after which they were demoulded and submerged in water for proper hydration at 7, 14, 21, and 28 days. Additionally, concrete cubes of the same size were prepared for the water absorption test.

Figure 2: Cube samples

Workability Test

Workability tests were carried out on the samples. These are: slump test and compacting factor test.

Slump test

Slump test was carried out using the slump cone 300mm high as shown in figure 3. The cone was placed on a smooth horizontal surface with the smaller opening at the top. The concrete were poured in three layers; each layer was tamped 35 times with standard steel rod of 16mm diameter. Immediately after filling, the cone was lifted and the decrease in the height of the slump was measured and recorded.

Figure 3: Slump Test

Compacting Factor Test

Compacting factor test was carried out by using the compacting factor apparatus. The upper hopper was filled with concrete in such a way that no compaction occurred, the bottom clamp was released so that concrete will falls to lower hopper. The bottom door of the lower hopper was then released so that concrete falls to the cylinder; excess concrete was cut off. Compacting factor was by dividing weight of concrete in the cylinder to the weight of fully compacted concrete.

$$\text{C. F} = \frac{\text{Weight of partially compacted concrete}}{\text{Weight of fully compacted concrete}} \qquad \text{Equation 1}$$

Absorptivity Test

This test was conducted on concrete samples to determine the rate of water absorption. The samples were initially immersed in a curing tank for 25 days. As shown in Figure 4, the cubes were submerged in water for curing. After this period, the samples were removed and left to dry for 48 hours, then re-immersed in water for an additional 2 days. The weight of the concrete was measured after 28 days of curing and again after the additional 2-day curing period. The difference in weight represents the water absorption rate.

Figure 4: Cube Curing

Compressive Strength test

The concrete cube for this project were batched, mixed and cured for 7, 14, 21 and 28 days at 0, 5, 15, 20 and 25 days. At the end of curing period, the concrete cubes produced were subjected to compressive for assessing residual strength.

Flexural Strength Test

The flexural test of concrete was used to evaluate the strength of concrete in a beam. It tests the ability of unreinforced concrete beam or slab to withstand failure in bending. The specimen was prepared by filling the concrete into the mould in three layers and was tamped 35 blows after each layer. The specimen mould used was 150 x 150 x 300 mm. It was demould after 24 hours and tested at 14 and 28 curing ages. Load was applied continuously without shock till the point of failure. The flexural strength determined by:

$$\text{Fr} = \frac{\text{Pl}}{\text{bd}^2} \qquad \text{Equation 2}$$

Where: b = width of specimen; d = failure point depth; l = Supported length; P = maximum load (KN)

3. Results and Discussion

Chemical Composition of Calcined Halloysite (CH)

The chemical composition of calcined halloysite (CH) was analyzed and compared with that of Ordinary Portland Cement (OPC). The results are presented in Table 2, showing the key oxides present in both materials.

Table 2: Chemical composition of Calcined Halloysite (CH) compared with cement

Oxide	Content %	
	Cement	**Calcined Halloysite**
Al_2O_3	4.9	39.50
SiO_2	20.1	44.2
TiO_2	0.2	0.093
K_2O	0.4	-
CaO	65	0.756
Na_2O	0.2	
MgO	3.1	0.024
MnO	0.02	
Fe_2O_3	2.5	0.081
Loss on ignition	8.8	12

The chemical analysis of calcined halloysite (CH) indicates that the combined content of SiO_2, Al_2O_3, and Fe_2O_3 is 83.90%, which exceeds the 70% benchmark for pozzolanic materials as outlined by ASTM standards. This confirms its effectiveness as a supplementary cementitious material. However, the CaO content in Ordinary Portland Cement (OPC) is significantly higher than that of CH. These findings are consistent with the research by [8, 9, 10, 11, 12 & 13].

Workability of Calcined Halloysite (CH) Concrete

The results of the slump and compacting factor tests are shown in Table 3.

Table 3: Workability of Calcined Halloysite (CH) Concrete

% replacement of CH	Slump (mm)	Compacting Factors values
0	107	0.989
5	94	0.975
10	88	0.965
15	80	0.960
20	67	0.960

The slump value declines from 107 mm to 67 mm as the proportion of CH increases. Likewise, the compacting factor decreases from 0.989 to 0.960 with higher CH content. These results indicate that increasing CH content reduces the workability of concrete, suggesting that more water is required to maintain the desired consistency. Figure 5 shows a graphical representation:

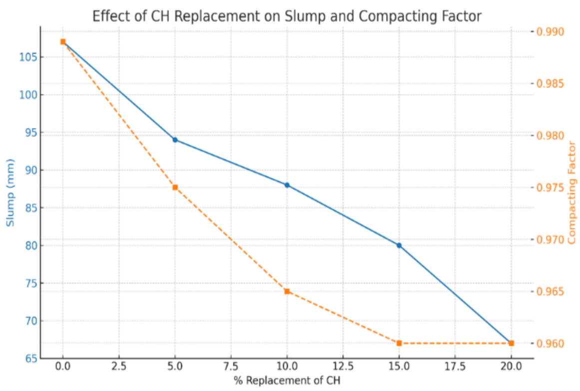

Figure 5: the effect of CH replacement on Slump and Compacting Factor

Water Absorptivity of Concrete

The results from the water absorption test of concrete samples are presented in Table 4. The findings indicate that the permeability value increased from 2.25% to 3.40% as the percentage content of CH increased. This increase is due to the reaction of oxides present in CH, which readily interact with other oxides, especially in the presence of water. However, the concrete remains workable as the water absorptivity is below 3%, aligning with the recommended British specification values.

Table 4: Water absorptivity value

% Replacement of CH	Rate of Absorption %
0	2.35
5	2.42
10	2.76
15	3.15
20	3.35

Compressive Strength of Calcined Halloysite (CH) Concrete

The effect of curing age on the compressive strength of CH concrete is presented in Table 5

Table 5: Compressive Strength of Calcined Halloysite (CH) Concrete

% Replacement of CH	Compressive Strength Value N/mm^2			
	7 days	14 days	21 days	28 days
0	14.98	17.56	21.90	24.97
5	17.12	17.63	22.53	25.95
10	17.76	17.82	22.97	25.30
15	18.15	18.02	23.20	25.95
20	18.59	18.76	23.23	26.12

Furthermore, Figure 6 shows the results that compressive strength generally increases with the curing period but decreases as the CH replacement increases. At 7 days, the strength increased from 14.98 N/mm² to 18.59 N/mm² at 20% CH replacement. A similar trend was observed at 14, 21, and 28 days. These findings indicate that concrete containing CH gains strength more slowly at an early curing age.

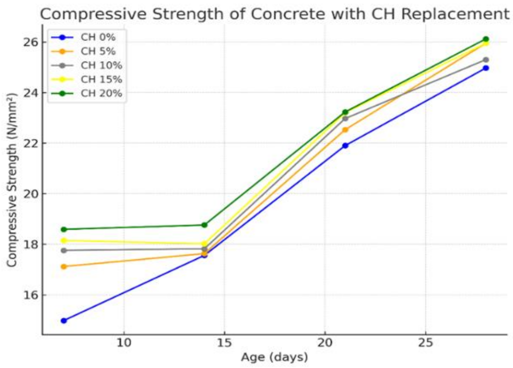

Figure 6: Compressive Strength of Concrete

Flexural Strength of Concrete Beam

The results from the flexural strength test on hardened concrete at 21 and 28 days of curing are presented in Table 6. At 21 days, the beam's strength increased from 2.32 to 2.89 N/mm² as the percentage replacement of CH increased. This may be due to the reaction between water and the oxides of CH, contributing to strength development. At 28 days, the beam's strength reached 3.67 N/mm², exceeding the recommended value for 0% replacement. The significant increase in strength of CH concrete is attributed to the pozzolanic reaction of CH, which enhances the binding properties of the cementitious matrix.

Table 6: Flexural Strength of Calcined Halloysite (CH) Concrete

% replacement of CH	Flexural strength value	
	21 days	28 days
0	2.32	3.02
5	2.48	3.14
10	2.54	3.24
15	2.76	3.35
20	2.89	3.67

4. Conclusions

Compressive strength improved with CH replacement, with significant gains observed up to 28 days as a result of pozzolanic activities of calcined halloysite which densifies the concrete medium and reduces pore size. Water absorption increased with higher CH content. The flexural strength of beams also improved with CH addition. It was concluded that 5% and 10% CH replacement meet the British Standard strength requirements for structural applications.

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