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(RESEARCH ARTICLE)

Reactivity of calcined chikoko clay and its influence on the strength properties of concrete

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Abstract

This study investigates the reactivity of calcined Chikoko clay and its influence on the mechanical performance of ordinary Portland cement concrete. Locally sourced Chikoko clay, an aluminosilicate material, was calcined at controlled temperatures of 200, 400, 600, and 800°C and incorporated into concrete at replacement levels of 5, 10, 15, and 20% by mass. The clay was characterized using X-ray fluorescence (XRF), X-ray diffraction (XRD), thermogravimetric analysis (TGA), BET surface area analysis, Fourier-transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) to assess its chemical composition, mineralogical transformation, and microstructural features. Concrete specimens were tested for compressive strength, splitting tensile strength, and modulus of elasticity at curing ages of 7, 28, 56, and 90 days. Results show that both calcination temperature and replacement level significantly influence concrete strength, with optimal performance observed at 10% replacement. The highest 90-day compressive strength of 52.3 MPa was achieved at 800°C, indicating strong pozzolanic reactivity of thermally activated clay. Excessive replacement (>15%) reduced strength due to dilution of the cementitious content. Microstructural analysis confirmed denser pore structures and enhanced hydration in calcined clay blends. The findings demonstrate that calcined Chikoko clay is a highly reactive supplementary cementitious material capable of improving long-term concrete strength while offering potential for sustainable, low-carbon concrete production.

Keywords: Chikoko clay; Calcination; Pozzolanic reactivity; Concrete strength

1. Introduction

Concrete remains the principal construction material used worldwide, and its global demand continues to rise as urbanisation accelerates. The material's performance and versatility are tied closely to Portland cement, yet cement production accounts for an estimated 7–8 percent of global carbon dioxide emissions (Scrivener et al., 2018; Miller et al., 2016). This environmental burden has intensified the search for low-carbon alternatives capable of either partially replacing cement or enhancing long-term durability. Supplementary cementitious materials (SCMs) such as fly ash, silica fume and metakaolin have been widely explored for their ability to improve microstructural development and reduce permeability in concrete systems (Fernández-Jiménez & Palomo, 2007; Siddique & Khan, 2011, Taha, 2009).

Calcined clays, particularly those containing kaolinite, have emerged as one of the most promising and globally available SCMs. When heated to moderate temperatures, kaolinitic clays undergo dehydroxylation and transform into amorphous aluminosilicates with strong pozzolanic reactivity (Ambroise et al., 1994; Tironi et al., 2013). Recent large-scale projects such as LC^3 (Limestone Calcined Clay Cement) have demonstrated the capacity of calcined clays to reduce clinker content significantly while maintaining mechanical and durability performance (Antoni et al., 2012; Bishnoi et al., 2014). These developments show that calcined clays can play a strategic role in lowering carbon intensity, especially in regions where industrial by-products like fly ash or slag are scarce.

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Chikoko clay is a naturally occurring clay deposit found across several regions of West Africa, including the Niger Delta. Preliminary geological surveys indicate that these deposits contain appreciable proportions of kaolinite, fine-grained silts, and aluminosilicate minerals suitable for activation through thermal treatment. Although studies by Ottos and Nyebuchi (2018), Otoko (2014), and Orumu and Overo (2020) have explored the behavior of various tropical clays after calcination, systematic research focusing specifically on Chikoko clay remains limited. Little is known about its mineralogical transformation during calcination, the degree of amorphous phase formation, or its long-term reactivity in cementitious matrices.

Pozzolanic reactivity is crucial for assessing the potential of any calcined clay to participate in cement hydration reactions. Activated aluminosilicates react with calcium hydroxide released during cement hydration, forming additional calcium silicate hydrate (C–S–H) and other binding phases that refine pore structure, enhance strength and reduce ion transport (Wild et al., 1996; Rashad, 2013, Ottos, & Nyebuchi, 2018). Given the increasing evidence that local materials can significantly improve performance in aggressive environments, it becomes necessary to investigate how calcined chikoko clay may contribute to mitigating chloride-induced deterioration, one of the most serious durability concerns in reinforced concrete structures (Neville, 2011; Thomas et al., 2012).

This study addresses these knowledge gaps by examining the thermal activation of chikoko clay, evaluating its pozzolanic reactivity and assessing its influence on the mechanical and durability performance of concrete. The work expands current understanding of locally sourced SCMs and aims to demonstrate that calcined chikoko clay can support more sustainable construction practices in chloride-prone environments. By integrating material characterisation, strength testing and chloride-related durability assessments, the study provides comprehensive insight into the applicability of this underutilized resource for low-carbon and resilient concrete production.

2. Materials and methods

2.1. Materials

The materials for this study were selected to investigate the influence of calcined *Chikoko* clay (CKC) on concrete properties. The primary binder was Ordinary Portland Cement (OPC) Grade 42.5N, conforming to ASTM standards, which served as the baseline for comparison with CKC-blended mixes.

Chikoko clay, a deltaic clay sourced from a local deposit in the Niger Delta's Delta State, was processed into a reactive supplementary cementitious material. The processing involved air-drying, crushing, sieving, calcining, and pulverizing the clay for use as a partial cement replacement.

The fine aggregate consisted of natural river sand obtained from the Gbanatoru community in Yenagoa, Bayelsa State, which was pulverized to ensure a uniform particle size distribution. The coarse aggregate was locally sourced crushed granite with a maximum nominal size of 20 mm, providing the primary structural skeleton for the concrete mix. Potable water, free from deleterious impurities, was used for all mixing and curing operations to maintain the integrity of the concrete.

2.2. Calcination Procedure

The raw Chikoko clay was transformed into a reactive supplementary cementitious material through a controlled calcination process. Initially, the clay was oven-dried at 105°C to remove free moisture, then milled and sieved through a 75 μm mesh to ensure uniform particle size and eliminate coarse impurities. The calcination stage was performed in an electric muffle furnace under a precisely controlled thermal regime. Guided by thermogravimetric analysis, the heating rate was set at 10°C per minute, reaching an optimal temperature range of 700–800°C, with a holding period of 1.5 hours as shown in Figure 1.

Figure 1 Sample Preparation and Calcination Process

This procedure ensured complete dehydroxylation of the clay minerals, activating their pozzolanic properties while preventing recrystallization that could reduce reactivity. After calcination, the material was allowed to cool gradually to ambient temperature within the furnace to minimize thermal shock. The calcined clay was subsequently pulverized using a ball mill to achieve a fine powder with high specific surface area. The final product, designated as Calcined Chikoko Clay (CKC), was stored in airtight containers to prevent moisture absorption and carbonation, preserving its reactivity until incorporation into concrete mixtures.

2.3. Characterization of raw and Calcined Clay

The following characterisation methods were employed:

- XRF for chemical composition
- XRD for mineralogical identification
- TGA and DTG for thermal transformation behavior
- BET surface area analysis
- Particle size distribution
- FTIR spectroscopy
- SEM for Morphological evaluation

2.4. Mix Design and Specimen Preparation

Five concrete mixes were prepared for this study, including one control mix and four mixes incorporating 0%, 5%, 10%, 15%, and 20% Calcined Chikoko Clay (CKC) as partial replacement of Portland cement. All mixes were designed with a water-to-binder ratio of 0.50 to ensure workability and consistency. Concrete cubes and cylindrical specimens were cast for the determination of compressive strength, splitting tensile strength, and modulus of elasticity. After casting, the specimens were demolded and cured in water at 23°C. Strength tests were conducted at curing ages of 7, 28, 56, and 90 days to evaluate the development of mechanical properties over time.

2.5. Mechanical and Microstructural Testing

- Compressive strength according to ASTM C39
- Splitting tensile strength according to ASTM C496
- Modulus of elasticity according to ASTM C469
- SEM analysis of hydration products
- TGA quantification of calcium hydroxide in hardened paste

3. Results and Discussion

3.1. Characterization of calcined Chikoko Clay

XRF results showed that the combination of silica and alumina exceeded the threshold usually associated with good pozzolans. XRD patterns confirmed the disappearance of kaolinite peaks after calcination, replaced by a diffuse hump

associated with an amorphous phase as shown in Table 1. TGA confirmed the completion of dehydroxylation around 700°C. BET surface area increased significantly after calcination, indicating enhanced reactivity.

Table 1 Chemical composition of raw and calcined chikoko clay (wt. %)

Oxide	Raw clay	Calcined clay
SiO_2	48.2	49.5
Al_2O_3	32.6	33.1
Fe_2O_3	5.4	5.6
CaO	1.3	1.4
MgO	0.9	1.0
K_2O	0.7	0.7
Na_2O	0.5	0.5
Loss on ignition	10.4	3.0

3.2. Pozzolanic Activity

The Frattini test placed calcined chikoko clay in the reactive zone. Chapelle test values indicated high lime fixation capacity. The strength activity index at 28 days exceeded 100 percent for the 10 and 15 percent replacement levels is illustrated in Table 2.

Table 2 Pozzolanic activity indices

Test	Requirement	Result
Strength activity index at 28 days (%)	≥75	102 (10 percent replacement)
Lime fixation (Chapelle test) (mg CaO/g)	≥700	760
Frattini test	Must fall below solubility curve	Passed

3.3. Compressive Strength Development

The influence of calcined chikoko clay on the compressive strength of concrete was examined at 7, 28 and 90 days. Table 3 to summarizes the results, while Figure 2 to illustrate the strength development trend. The results show that early-age compressive strength (7 days) declined progressively with increasing chikoko clay content. This behaviour is expected, as pozzolanic reactions are typically slower and depend on longer curing periods.

Table 3 Compressive Strength of Concrete without Calcined Chikoko Clay (MPa)

Temperature (0°C)	Replacement (%)	Comprehensive strength (N/mm^2)			
		7 days	28 days	56 days	90 days
	0	14.7	28.3	33.3	36.1
	5	16.3	30.0	37.5	39.3
0	10	20.6	33.0	39.1	44.8
	15	16.9	30.0	39.3	45.1
	20	10.1	21.1	26.2	33.8

Table 4 Compressive Strength of Concrete with low Calcined Chikoko Clay (MPa)

Temperature (0°C)	Replacement (%)	Comprehensive strength (N/mm²)			
		7 days	28 days	56 days	90 days
200	0	14.7	28.3	33.3	36.1
200	5	21.8	34.1	38.7	45.1
200	10	23.7	39.3	44.1	45.9
200	15	20.3	34.0	35.8	38.5
200	20	18.3	29.9	30.9	33.0
400	0	14.7	28.3	33.3	36.1
400	5	25.9	38.1	42.2	46.5
400	10	28.5	40.7	44.5	47.0
400	15	25.4	36.5	38.6	41.5
400	20	22.6	32.4	33.9	35.0

Table 5 Compressive Strength of Concrete with High Calcined Chikoko Clay (MPa)

Temperature (0°C)	Replacement (%)	Comprehensive strength (N/mm²)			
		7 days	28 days	56 days	90 days
600	0	14.7	28.3	33.3	36.1
600	5	29.8	37.8	43.9	49.2
600	10	33.5	43.3	46.9	50.3
600	15	32.3	41.9	44.2	44.6
600	20	27.7	34.7	40.8	42.5
800	0	14.7	28.3	33.3	36.1
800	5	35.0	43.0	46.2	46.8
800	10	37.4	46.8	47.6	52.3
800	15	36.7	44.8	48.4	49.6
800	20	30.3	40.3	41.3	44.4

The strength patterns reported in Tables 3 to 5 show a progressive enhancement in compressive strength as the chikoko clay undergoes calcination and as the replacement level is optimized. Table 1 provides the baseline behaviour of concrete incorporating uncalcined chikoko clay. At 7 days the control mix achieves 14.7 MPa and reaches 36.1 MPa at 90 days. The inclusion of uncalcined clay produces inconsistent performance. For instance the 10 percent mix rises to 20.6 MPa at 7 days and 44.8 MPa at 90 days, yet the 20 percent mix decreases sharply to 10.1 MPa at 7 days and only 33.8 MPa at 90 days. This irregularity supports the inference that raw chikoko clay contributes little pozzolanic reactivity and functions mainly as a filler.

A clearer improvement is observed in Table 2 as the clay is calcined at low temperatures. At 200 degrees Celsius the 10 percent replacement level records 23.7 MPa at 7 days and 45.9 MPa at 90 days, which already exceeds the control values. Strength enhancement becomes more distinct at 400 degrees Celsius. At this temperature the 10 percent mix reaches

28.5 MPa at 7 days, 40.7 MPa at 28 days and 47.0 MPa at 90 days. These results reflect the beginning of meaningful activation in the clay. Partial dehydroxylation at 200 degrees Celsius produces moderate reactivity, whereas the higher temperature of 400 degrees Celsius appears sufficient to initiate structural changes in the aluminosilicate network. This leads to improved early hydration and a more reactive surface for secondary gel formation.

Table 3 illustrates the behavior of the clay under high calcination temperatures. At 600 degrees Celsius the 10 percent replacement level attains 33.5 MPa at 7 days, 43.3 MPa at 28 days and 50.3 MPa at 90 days. The rise in both early and later-age strengths indicates the formation of highly reactive phases similar to metakaolin. The effect becomes strongest at 800 degrees Celsius. At this temperature the 10 percent mix records the highest strengths across all conditions, reaching 37.4 MPa at 7 days, 46.8 MPa at 28 days and 52.3 MPa at 90 days. These values exceed those of the control mix and all other replacement–temperature combinations. The 5 and 15 percent mixes also show elevated strengths, although they do not surpass the 10 percent level. The drop observed at 20 percent replacement, where the 90-day strengths fall to 33.0 MPa at 200 degrees Celsius, 35.0 MPa at 400 degrees Celsius, 42.5 MPa at 600 degrees Celsius and 44.4 MPa at 800 degrees Celsius, illustrates the classical dilution effect. At this level the reduction in clinker content outweighs the benefits gained from pozzolanic reaction.

Taken together the values in Tables 1 to 3 reveal that chikoko clay attains full pozzolanic reactivity between 600 and 800 degrees Celsius. The consistent strength peak at 10 percent replacement across all temperature levels provides strong evidence of an optimal balance between clinker reduction and secondary gel formation. Moreover the significant gains in early strength at 600 and 800 degrees Celsius suggest that properly calcined chikoko clay not only contributes to long-term hydration but also enhances early-age microstructural refinement.

3.3.1. Effect of Replacement Level and Reactivity on Calcination Temperature Across Curing Days

The influence of Chikoko clay replacement level and calcination temperature on the compressive strength of concrete is shown in Figures 2–5. For uncalcined clay (0°C), strength behaviour is inconsistent: it increases with replacement up to 10%, achieving a peak 90-day strength of 44.8 MPa, notably higher than the control (36.1 MPa), but declines at 15% and 20% replacement, with the 20% sample dropping to 33.8 MPa. This indicates minimal pozzolanic activity in uncalcined clay, with early gains likely due to microfilling rather than chemical reactivity, while higher substitution levels dilute the cementitious content. Such behavior aligns with observations by Garg et al. (2025) and Dhandapani et al. (2025), who reported that raw or partially activated clays contribute limited strength at early ages, with improvements largely dependent on filler effects rather than chemical reaction.

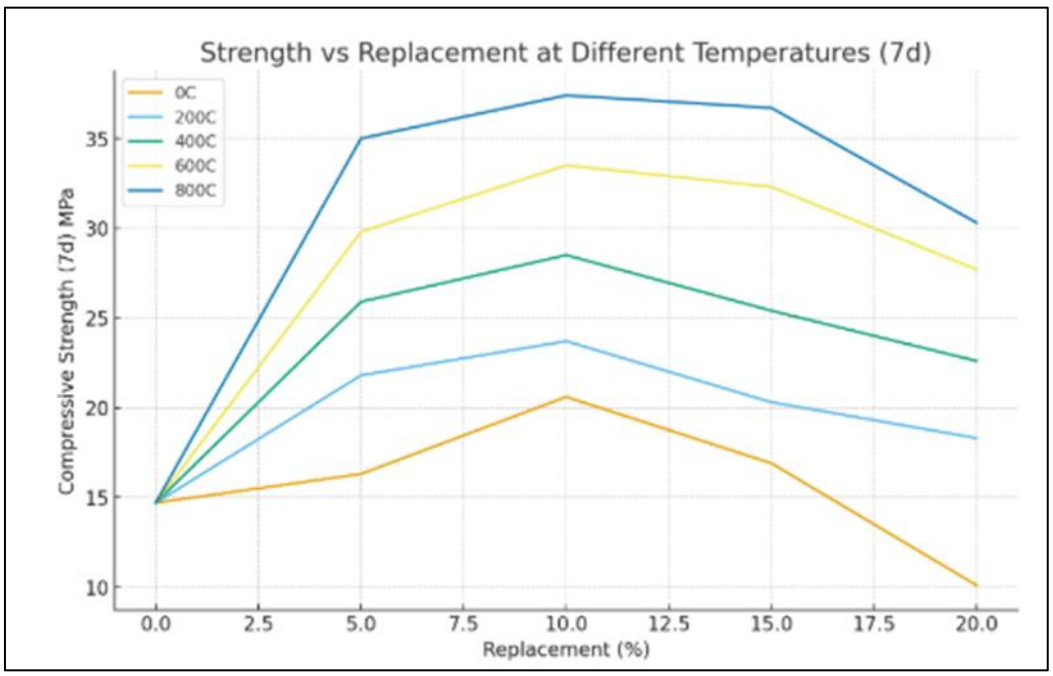

Figure 2 Compressive Strength of Concrete at 7 Days for Varying Chikoko Clay Replacement Levels and Calcination Temperatures

Partial thermal activation at 200°C improves both early- and later-age strengths, with 10% replacement reaching 45.9 MPa at 90 days, reflecting partial activation of clay minerals; however, replacement levels above 15% again reduce strength. This trend is consistent with findings from Vásquez-Torres et al. (2024), who highlighted that low-temperature calcination only partially dehydroxylates clay minerals, resulting in moderate pozzolanic reactivity. At 400°C, strength increases more markedly, with 10% replacement achieving 47.0 MPa at 90 days, suggesting a greater proportion of reactive aluminosilicates has been activated. These results corroborate recent evidence that mid-temperature calcination can significantly enhance early- and later-age mechanical properties (Taha, 2009).

Increasing the calcination temperature to 600°C further enhances pozzolanic reactivity, yielding 50.3 MPa at 90 days for the 10% replacement, consistent with complete dehydroxylation of kaolinitic clay to metakaolin. Maximum performance is observed at 800°C, where the 10% replacement attains 52.3 MPa at 90 days, with 5% and 15% replacements also performing well, although 20% replacement remains suboptimal. These observations support the conclusions of Dhandapani et al. (2025) and Alghamdi et al. (2024), who reported that high-temperature calcination (700–800°C) generates highly reactive amorphous aluminosilicates, resulting in significantly improved long-term compressive strength.

Across all temperatures, several trends are apparent: strength increases with calcination temperature, 10% replacement consistently gives optimal results, all calcined samples outperform uncalcined and control mixes, and excessive replacement (≥15%) reduces performance due to dilution of cementitious material. These findings are in agreement with global studies on calcined clay–cement blends, including LC^3 and other low-carbon concrete systems (Scrivener et al., 2018; RILEM TC 282-CCL, 2022; Cleaner Waste Systems, 2025), confirming that thermally activated Chikoko clay exhibits significant pozzolanic reactivity.

The data demonstrates that optimized calcination and replacement of Chikoko clay not only enhance compressive strength but also provide a pathway for sustainable, lower-carbon concrete production by reducing cement consumption while maintaining or improving mechanical performance.

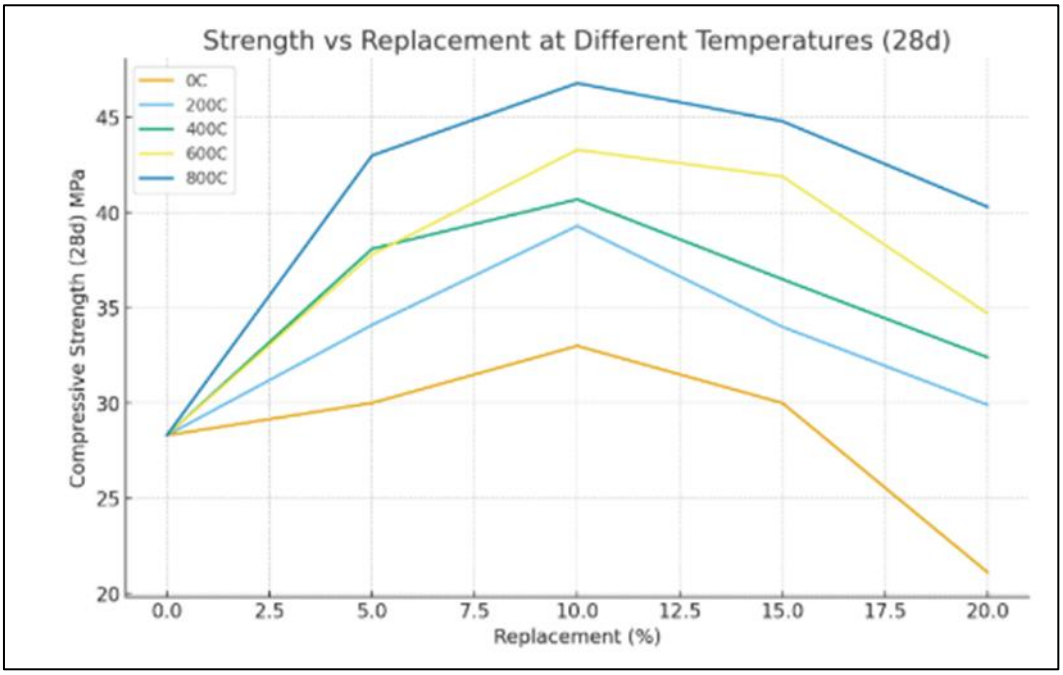

Figure 3 Compressive Strength of Concrete at 28Days for Varying Chikoko Clay Replacement Levels and Calcination Temperatures

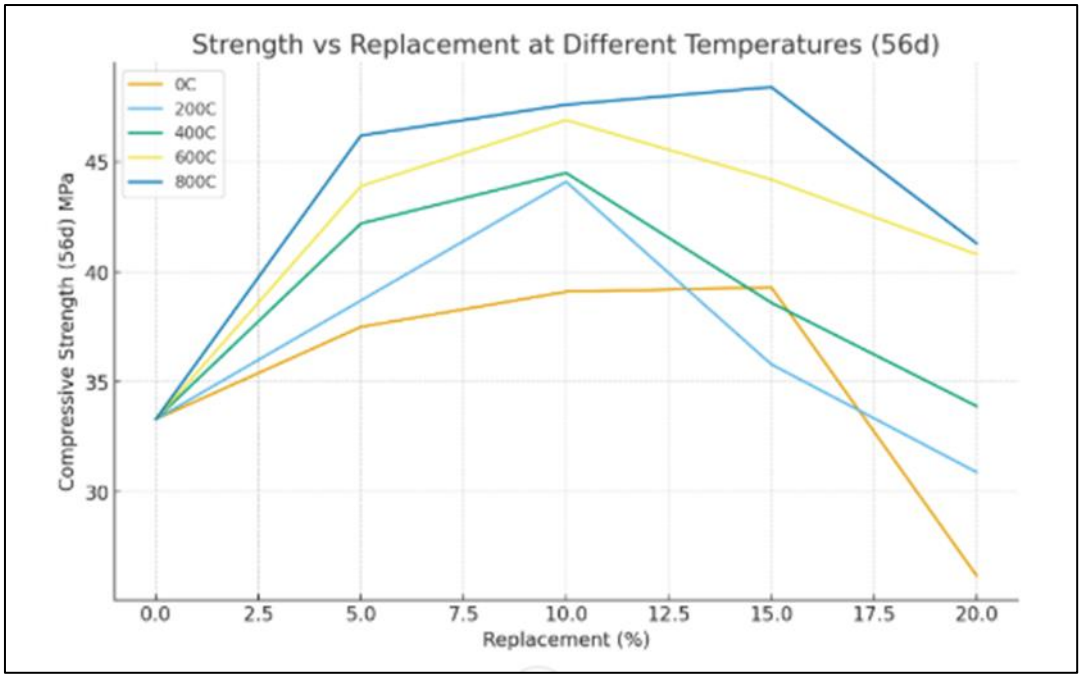

Figure 4 Compressive Strength of Concrete at 56 Days for Varying Chikoko Clay Replacement Levels and Calcination Temperatures

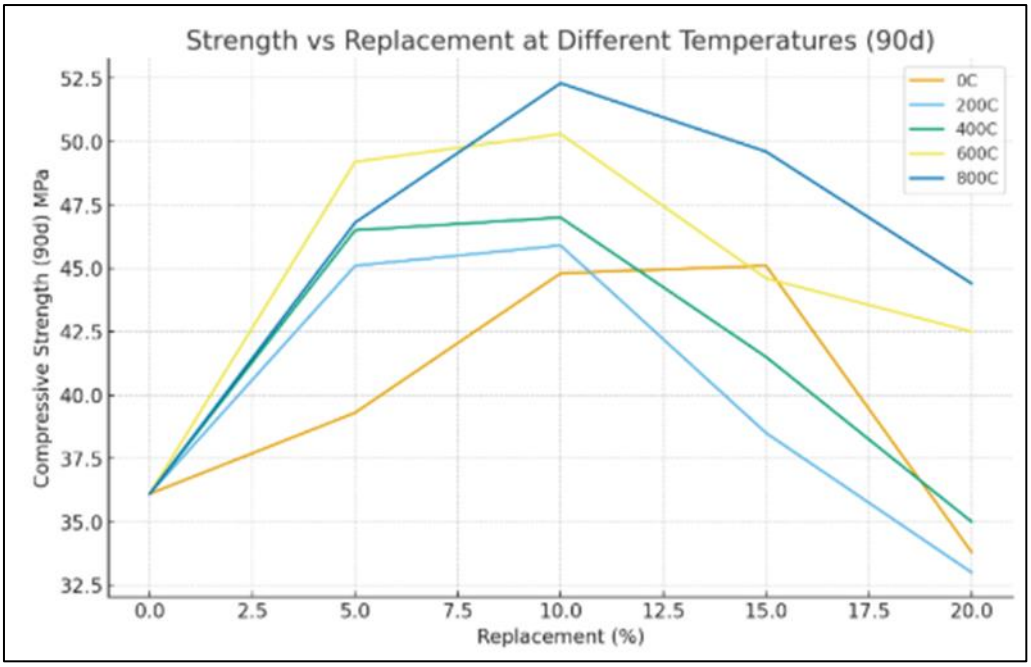

Figure 5 Compressive Strength of Concrete at 90 Days for Varying Chikoko Clay Replacement Levels and Calcination Temperatures

3.4. Splitting Tensile Strength

The results for splitting tensile strength at 28 and 90 days are presented in Table 6. The general trend shows a marginal increase in tensile strength up to 10–15 percent replacement. This suggests that the fine, reactive particles of calcined Chikoko clay enhance the paste–aggregate bond, improve packing density, and reduce micro cracks within the matrix.

At 90 days, the maximum tensile strength of 3.28 MPa occurred at 15 percent replacement. The decline observed at 20 percent follows the same pattern noted in the compressive strength results and aligns with diminishing binder efficiency at higher replacement levels.

The enhancement of tensile strength with calcined clay incorporation can be attributed to the pozzolanic reaction, which generates additional calcium silicate hydrate (C–S–H) gel and refines the pore structure, thereby improving cohesion within the matrix. Strength gains are more pronounced at higher calcination temperatures, particularly between 600–800 °C, due to increased reactivity of the aluminosilicate phases. The results demonstrate that an optimal replacement range of 10–15 percent maximizes tensile performance, while excessive clay content (>15 percent) reduces the efficiency of the cementitious matrix, confirming the importance of balancing pozzolanic reactivity and cement dilution.

Table 6 Splitting Tensile Strength of Concrete at 28 Days (MPa)

Temperature (°C)	Replacement (%)	Splitting Tensile Strength (MPa)
0 (Control)	0	2.97
	5	3.06
	10	3.22
	15	3.06
	20	2.57
	0	2.97
200	5	3.26
	10	3.51
	15	3.26
	20	3.05
	0	2.97
400	5	3.45
	10	3.57
	15	3.38
	20	3.18
600	0	2.97
	5	3.44
	10	3.68
	15	3.61
	20	3.30
800	0	2.97
	5	3.67
	10	3.82
	15	3.73
	20	3.54

3.5. Modulus of Elasticity

Table 6 presents the modulus of elasticity at 28 and 90 days. Stiffness increased gradually up to 10 percent replacement, reaching 27.7 GPa at 28 days and 30.1 GPa at 90 days. This trend reflects the improved microstructural integrity associated with the pozzolanic reaction. Enhancement in stiffness is consistent with the densification of the

cementitious matrix due to the formation of secondary calcium silicate hydrates (Boakye & Khorami, 2025; Garg et al., 2025).

Table 7 Modulus of Elasticity of Concrete at 28 Days (GPa)

Temperature (°C)	Replacement (%)	Modulus of Elasticity (GPa)
0 (Control)	0	25.0
0	5	25.8
0	10	27.0
0	15	25.8
0	20	21.7
200	0	25.0
200	5	27.4
200	10	29.4
200	15	27.4
200	20	25.7
400	0	25.0
400	5	29.0
400	10	30.0
400	15	28.4
400	20	26.7
600	0	25.0
600	5	28.9
600	10	30.9
600	15	30.3
600	20	27.7
800	0	25.0
800	5	30.8
800	10	32.1
800	15	31.3
800	20	29.7

Beyond 15 percent replacement, the modulus declined, confirming that an optimal replacement range exists between 5 and 15 percent. Excessive clay leads to reduced clinker content, which negatively affects the rigidity of the hardened material. Similar trends have been reported in studies on low-carbon cements, where calcined clay replacement above optimal levels resulted in decreased mechanical performance despite ongoing pozzolanic activity (RILEM TC 282-CCL, 2023; Dhandapani et al., 2025).

The positive effect of calcined clay on modulus of elasticity is supported by evidence that thermal activation at 600–800 °C increases pozzolanic reactivity and promotes microstructural densification, resulting in higher stiffness at both early and later curing ages (Alghamdi et al., 2024; Study on the performance of ternary blended cement, 2024). These findings validate the observations in this study that a 10 percent replacement of Chikoko clay achieves optimal modulus values, consistent with global literature on calcined-clay–blended concretes (Calcined clay as a low-carbon cementitious material, 2025; Effect of low-grade calcined clay, 2025).

The trends indicate that appropriate replacement of Portland cement with calcined Chikoko clay can enhance elastic properties, but excessive substitution reduces stiffness due to dilution of the binder matrix.

4. Conclusion

The study demonstrates that Chikoko clay becomes a highly reactive aluminosilicate when calcined within the temperature range of 600–800°C. Concrete incorporating 10–15% calcined Chikoko clay exhibits superior mechanical performance, with the 10% replacement at 800°C achieving the highest 90-day compressive strength of 52.3 MPa. Microstructural analyses confirm enhanced hydration, reduced calcium hydroxide content, and a denser pore structure, consistent with strong pozzolanic activity. Strength improvements are strongly influenced by both calcination temperature and replacement level, while excessive substitution (≥15%) leads to reduced performance due to dilution of cementitious material. These findings establish calcined Chikoko clay as a viable, locally available supplementary cementitious material capable of improving concrete strength and contributing to sustainable, low-carbon construction.

Recommendations

Future research should evaluate the durability performance of CKC-blended concrete, including chloride penetration, sulfate resistance, and carbonation behavior, to confirm long-term serviceability.

Optimization studies could explore combined use with other supplementary cementitious materials to enhance early-age strength and further reduce clinker content.

Compliance with ethical standards

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