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Improvememnt of Post-Fire Mechanical Properties of Concrete Using Calcinated Kaolin (Metakaolin)

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Abstract- Fire exposure significantly alters the mechanical and durability properties of concrete, often leading to substantial reductions in structural capacity. This study investigates the improvement of residual mechanical properties of concrete exposed to elevated temperatures through partial replacement of ordinary Portland cement with calcined kaolin (metakaolin, MK). Concrete specimens were produced with 0%, 2.5%, 5%, 7.5%, and 10% metakaolin replacement by weight of cement. The water-cement ratio was varied appropriately to maintain workability as MK content increased. Specimens were cured for 28 and 56 days and subsequently exposed to heat loads of 200°C, 400°C, 600°C, and 800°C. Residual compressive strength, mass loss, and water absorption were determined to evaluate post-fire performance. Results indicate that compressive strength improved at ambient conditions with increasing MK content, with optimum performance observed between 7.5% and 10% replacement. At elevated temperatures, MK-modified concrete demonstrated superior residual strength retention compared to control specimens, particularly at 600°C and 800°C. Mass loss and water absorption increased with temperature across all mixes; however, mixes containing higher MK percentages exhibited comparatively reduced deterioration, attributed to pore refinement and reduced calcium hydroxide content. Specimens cured for 56 days consistently showed better residual performance than 28-day specimens, highlighting the importance of hydration maturity in thermal resistance. Correlation analysis revealed an inverse relationship between water absorption and residual compressive strength, confirming that microstructural densification significantly enhances fire resistance. The findings support the use of metakaolin as an effective supplementary cementitious material for improving the thermal stability and post-fire structural integrity of concrete. The study contributes to performance-based fire design by emphasizing material performance enhancement rather than reliance solely on prescriptive measures such as concrete cover thickness.

Keywords— Metakaolin, elevated temperature, residual compressive strength, fire resistance, mass loss, water absorption, performance-based design.

I. INTRODUCTION

Concrete is the most widely used construction material worldwide. This is due to its excellent mechanical properties, durability, and versatility in structural applications. However, at instances of exposure to elevated temperatures during fire events, concrete undergoes complex physicochemical changes capable of compromising the safety and integrity of structures. Among these changes are dehydration of hydration products, thermal cracking, strength degradation, and in severe cases, explosive spalling, particularly in high-strength and ultra-high-performance concretes (UHPC). These effects raise serious concerns regarding the post-fire safety, serviceability, and rehabilitation of reinforced concrete structures.

Recent years have witnessed a series of devastating fire outbreaks that highlight the urgent demand for improved fire-resistant concrete. For instance, the 2024 Valencia residential complex fire in Spain demonstrated rapid fire spread in modern buildings, while the deadly 2025 Wang Fuk Court blaze in Hong

Kong's Tai Po district—considered the city's deadliest since World War 2, claimed numerous lives and engulfed multiple high-rise structures amid bamboo scaffolding. Similarly, the catastrophic 2025 Palisades and Eaton wildfires in Los Angeles destroyed over 16,000 buildings, including concrete-framed homes and infrastructure, amid extreme conditions that tested material limits. These incidents illustrate how fires can compromise structural integrity, leading to collapse, loss of life, and enormous economic impacts.

At elevated temperatures, the decomposition of calcium hydroxide ($Ca(OH)_2$) and calcium silicate hydrate (C–S–H) phases plays a dominant role in strength deterioration. The dehydration of portlandite typically occurs between 400°C and 600°C, producing calcium oxide which may rehydrate upon cooling, causing volumetric expansion, cracking, and spalling. Consequently, conventional concrete often exhibits severe residual strength loss and compromised structural integrity after fire exposure (Khoury, 2024). This has prompted extensive research into material modifications aimed at improving concrete's fire resistance and post-exposure mechanical performance.

One effective strategy for enhancing concrete performance at elevated temperatures is the incorporation of supplementary cementitious materials (SCMs). Among these materials, metakaolin (MK)—a highly reactive aluminosilicate obtained from the controlled calcination of kaolin clay—has gained increasing attention. Metakaolin exhibits strong pozzolanic activity, reacting with calcium hydroxide released during cement hydration to form additional C–S–H and calcium aluminate hydrates. This reaction refines the pore structure, reduces the amount of free portlandite, and improves the density and stability of the cement matrix (Zhang et al., 2024).

Recent studies have shown that metakaolin-modified concrete demonstrates superior performance under thermal loading compared to ordinary Portland cement concrete. The reduction in calcium hydroxide content significantly limits thermally induced microcracking and mitigates explosive spalling, thereby improving residual compressive strength after high-temperature exposure. Experimental investigations conducted by Liu et al. (2025) revealed that concrete containing metakaolin retained higher residual mechanical properties after exposure to temperatures up to 800°C due to enhanced microstructural stability and improved thermal resistance. Similarly, Ahmad, Khan, and Al-Shehri (2024) reported that metakaolin incorporation delayed strength degradation and reduced mass loss in heat-damaged concrete specimens.

Furthermore, the sustainability benefits of metakaolin align with current global efforts to reduce carbon emissions associated with cement production. Partial replacement or addition of cement with calcined kaolin contributes to lower clinker demand while simultaneously improving durability and fire performance. Despite these advantages, the effectiveness of metakaolin depends strongly on factors such as calcination temperature, dosage, and exposure duration at elevated temperatures, which are not yet fully standardized in fire-resistant concrete design.

Therefore, this research focuses on the improvement of residual mechanical properties of concrete at elevated temperatures using calcined kaolin (metakaolin). By investigating the influence of metakaolin addition on strength retention after thermal exposure, the study aims to contribute to the development of performance-based fire-resistant concrete materials suitable for modern structural applications.

The significance of this research lies in advancing performance-based fire engineering (PBFE) for concrete structures, moving beyond the limitations of traditional prescriptive design approaches that rely heavily on standard concrete cover thickness as a proxy for fire resistance. Conventional prescriptive codes typically prescribe minimum cover values to delay temperature rise and protect reinforcement in fire scenarios, yet such provisions do not adequately account for the complex interactions between material degradation, heat transfer, and structural behavior under real fire conditions. This can lead to non-optimal or unsafe designs, as demonstrated by recent research showing that prescriptive methods may underperform in extreme fire conditions and fail to ensure structural integrity during and after fire events (Gernay et al., as cited in Johns Hopkins Univ. study, 2026).

Performance-based design, in contrast, prioritizes actual material and structural performance, allowing engineers to explicitly assess how concrete mixes—such as those modified with metakaolin—respond to fire through residual strength, deformation capacity, and failure modes. This shift enables designs that are quantifiably safer and more resilient, rather than simply compliant with fixed cover requirements. For example, performance-based methods have been shown to produce structures that maintain integrity under fire exposures that would compromise prescriptively designed members, offering enhanced protection of life and property and potentially lowering lifecycle costs through optimized material use (Johns Hopkins Univ. study, 2026). Furthermore, international fire engineering research emphasizes the importance of rigorous performance assessment and validation via advanced computational models and experimental data to support performance-based standards (NIST, 2025).

By focusing on concrete behaviour and residual mechanical properties after thermal exposure rather than fixed cover measures, this study contributes to a more rational, evidence-based fire safety design framework. Such an approach aligns with modern structural fire engineering goals of explicitly defining safety objectives, validating performance outcomes, and enabling flexible, optimized structural designs that reflect real fire hazards and material responses. The findings of this research will provide critical data that can inform code development, material selection, and fire-resilient design strategies for reinforced concrete structures in fire-prone environments.

II. Materials and Method

Kaolin used in this study was obtained from Afao Community, Ikere Local Government Area, Ekiti State, Nigeria. Ordinary Portland Limestone Cement (CEM II), natural river sand, and crushed limestone with a nominal maximum size of 10 mm were used for concrete production. Chemical reagents required for pozzolanic activity determination were purchased from Ogbete Market, Enugu, Nigeria. Experimental work was carried out at the University of Ibadan and the Projects Development Institute (PRODA), Nigeria.

1. Preparation of Metakaolin

The raw kaolin was purified to remove impurities, dried, milled, and calcined to produce metakaolin. Prior to calcination, the optimum activation temperature was determined using the Modified Chapelle test (AFNOR-NF P 18-513, 2010). Samples were calcined at temperatures between 600 and 800 °C at 25 °C intervals, and the temperature corresponding to the highest pozzolanic activity was selected for subsequent production.

The pozzolanic activity of the produced metakaolin was evaluated using the Modified Chapelle method (AFNOR-NF P 18-513, 2010). One gram of metakaolin was reacted with 2 g of CaO in 250 mL of distilled water for 16 h at 85 ± 5 °C under continuous stirring. A blank sample without metakaolin was prepared under identical conditions. After cooling, the remaining calcium hydroxide was determined by titration with 0.1 M HCl using phenolphthalein as an indicator. Pozzolanic activity was expressed as milligrams of $Ca(OH)_2$ fixed per gram of metakaolin.

Calculations

Mass of Ca(OH)2 fixed

$$= 2 * \frac{V_1 - V_2}{V_1} * \frac{74}{56} * 1000$$

Where:

- V1 is the volume of 0.1M HCl necessary for the 25 ml of the solution obtained by the blank sample.
- V2 is the volume of 0.1M HCl necessary for the 25 ml of the solution obtained by the reaction of the metakaolin. The results are expressed in mg Ca(OH)2 fixed by the metakaolin.

Blank Reaction

$$1000 > \frac{56V_1}{2}$$

Titration Reaction

$CaO + 2HCl \rightarrow CaCl_2 + H_2O$

$Ca(OH)_2 + 2HCl \rightarrow CaCl_2 + H_2O$

The result was expressed in mg Ca(OH)2 fixed by the metakaolin. This indicates the non-reacted lime

2. Aggregate Preparation

Fine aggregate (natural river sand) and coarse aggregate (crushed limestone) were washed to remove deleterious materials and air-dried to a saturated surface-dry condition in accordance with BS EN 1097-6:2022. Particle size distributions were determined through sieve analysis following BS EN 12620:2022 for fine aggregate and ASTM C33/C33M-23 for coarse aggregate to ensure compliance with grading requirements. The selected limestone aggregate was considered appropriate because of its relatively low thermal expansion and improved fire resistance compared with siliceous aggregates.

3. Concrete Batching and mixing

Concrete mixtures were prepared using the weight batching method in accordance with BS EN 206:2023 and ASTM C192/C192M-23. Metakaolin was incorporated as a partial replacement of Portland limestone cement at predetermined replacement levels by mass. Due to the high fineness and increased water demand associated with metakaolin, the water-to-cement ratio was adjusted for each mixture to achieve comparable workability. Workability was assessed using the slump test following BS EN 12350-2:2019 and ASTM C143/C143M-23. Water content was adjusted until each mix attained a slump comparable to that of the control concrete.

4. Determination of hardened concrete properties

Concrete specimens were water-cured at 20 ± 2°C until the specified testing ages before being subjected to physical, thermal, and mechanical evaluations.

5. Mass Loss Determination

Mass loss was determined to evaluate thermal degradation following exposure to elevated temperatures. Oven-dried specimens (105 ± 5 °C) were weighed before heating and after cooling to room temperature following fire exposure. Percentage mass loss was calculated from the difference between the initial and final masses (M1 and M2 respectively) in accordance with BS EN 12390-7:2019 and RILEM TC 129-MHT recommendations. Mass loss was used to assess dehydration and thermal deterioration of the concrete.

$$\%\ \text{mass loss} = \frac{M_1 - M_2}{M_1} * 100$$

6. Water Absorption Test

Water absorption was measured following BS EN 12390-7:2019 and ASTM C642-23. Oven-dried specimens were immersed in water for 48 h, after which the saturated surface-dry mass was recorded. Water absorption was calculated as the percentage increase in mass relative to the oven-dried mass. The results provided an indication of pore refinement and durability, with lower absorption values indicating improved matrix densification due to the pozzolanic action of metakaolin.

7. Fire (Elevated Temperature) Test

Fire performance was evaluated in accordance with ISO 834-1:2020 and RILEM TC 200-HTC recommendations. Concrete specimens were heated in an electrically controlled furnace at a rate of approximately 5–10°C/min to target temperatures of 200, 400, 600, and 800°C. Each temperature was maintained for sufficient time to ensure uniform heat distribution throughout the specimen before natural cooling within the furnace. Surface cracking, colour changes, and spalling were visually documented following thermal exposure.

8. Compressive Strength Test

Compressive strength tests were performed according to BS EN 12390-3:2019 and ASTM C39/C39M-24. Both unheated and thermally exposed specimens were tested using a calibrated compression testing machine. Compressive strength was calculated as the maximum failure load divided by the loaded cross-sectional area. Residual compressive strength was expressed as a percentage of the corresponding ambient-temperature strength and used to evaluate the effectiveness of metakaolin in improving the post-fire mechanical performance of concrete.

III. RESULTS AND DISCUSSION

1. Result of Compressive Strength Development

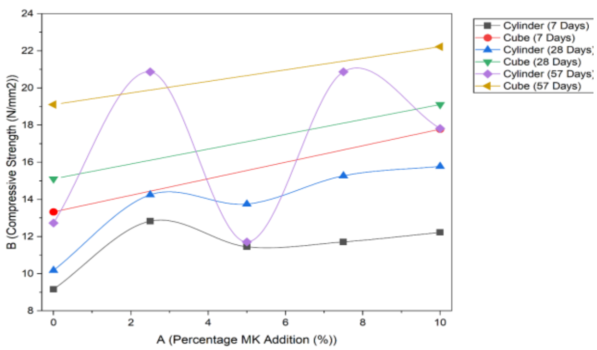

Fig 3.1 Compressive strength of samples at 200C

The results presented in Figure 3.1 illustrate the variation of compressive strength of both cube and cylindrical concrete specimens with increasing percentage replacement of cement with calcined kaolin (metakaolin) at different curing ages (7, 28, and 56 days). The general trend observed indicates that compressive strength increases with increasing metakaolin content up to 10%, with more pronounced strength enhancement at later curing ages.

The moderate improvement observed at 7 days is primarily attributed to the filler effect of metakaolin and its high nucleation potential, which accelerates cement hydration by providing additional sites for calcium silicate hydrate (C–S–H) formation. As hydration progressed to 28 and 56 days, the pozzolanic reaction became increasingly dominant, consuming calcium hydroxide generated during cement hydration to produce secondary C–S–H gel. This process refined the pore structure, reduced porosity, strengthened the interfacial transition zone (ITZ), and consequently enhanced the load-bearing capacity of the concrete. Similar improvements in compressive strength of metakaolin-modified concrete have been reported by Zhang et al. (2024), Ahmad et al. (2024), and Liu et al. (2025), who attributed these gains to matrix densification and improved microstructural integrity.

Cube specimens consistently exhibited higher compressive strengths than the corresponding cylindrical specimens throughout the study. This behaviour is expected because cube specimens experience greater lateral restraint during loading, resulting in higher measured strengths, whereas cylindrical specimens provide a more uniform stress distribution and are therefore considered a more representative measure of the in-situ compressive behaviour of concrete (ASTM C39/C39M, 2024). Nevertheless, both specimen geometries demonstrated similar trends in strength development with increasing metakaolin content, confirming that the beneficial effect of metakaolin is independent of specimen geometry. Overall, the results demonstrate that partial replacement of cement with approximately 10% metakaolin significantly improves the ambient compressive strength of concrete, providing a denser and more mechanically stable matrix that forms the basis for enhanced post-fire performance.

2. Mass Loss at Elevated Temperatures

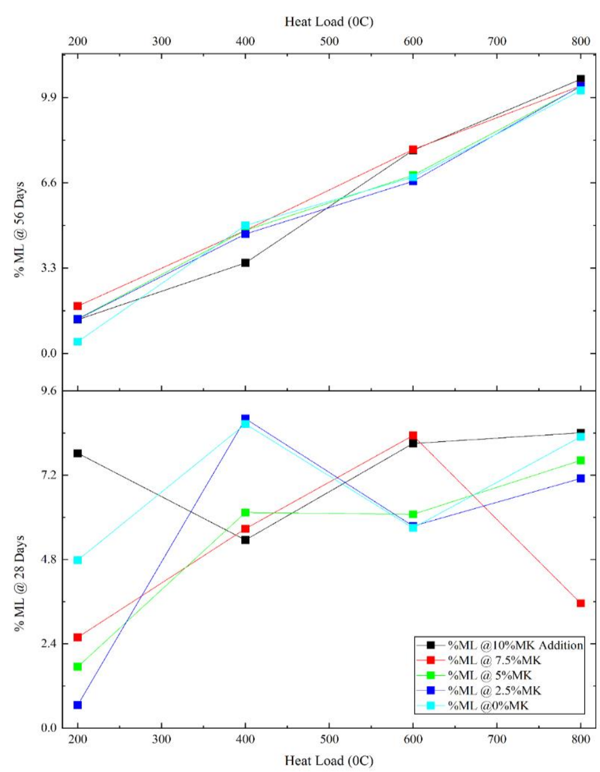

Figure 3.2 Comparison of mass loss for various additions of metakaolin and varying heat load

Figure 3.2 compares the percentage mass loss of concrete containing varying proportions of metakaolin after exposure to temperatures ranging from 200 to 800°C at curing ages of 28 and 56 days. As expected, mass loss increased progressively with increasing temperature for all concrete mixtures, reflecting the combined effects of moisture evaporation, dehydration of hydration products, and progressive deterioration of the cementitious matrix. The relatively low mass loss observed at 200°C is primarily associated with the evaporation of free and physically bound water, whereas the marked increase beyond 400°C corresponds to the decomposition of calcium hydroxide and the gradual destabilization of calcium silicate hydrate (C–S–H). At temperatures approaching 800°C, severe microstructural degradation, including decalcification of C–S–H and the development of interconnected microcracks, contributed to the highest mass losses, consistent with established thermal degradation mechanisms in cement-based materials (Khoury, 2024; Liu et al., 2025).

The incorporation of metakaolin significantly influenced the thermal response of the concrete. Mixtures containing approximately 7.5–10% metakaolin exhibited lower and more gradual mass loss than the control concrete, particularly at elevated temperatures. This behaviour is attributed to the pozzolanic reaction of metakaolin, which consumes calcium hydroxide to form additional C-S-H gel, thereby reducing the amount of thermally unstable portlandite while simultaneously refining the pore structure. The denser microstructure restricts moisture migration, minimizes internal vapour pressure, and delays thermally induced cracking, resulting in improved thermal stability (Ahmad et al., 2024; Zhang et al., 2024).

Curing age also affected the thermal performance of the specimens. Although 56-day concrete generally exhibited slightly greater cumulative mass loss at the highest temperatures due to the larger quantity of hydration products available for dehydration, the rate of deterioration was more gradual than that observed at 28 days. This finding suggests that prolonged curing enhanced the pozzolanic reaction and matrix densification, thereby improving resistance to thermal degradation. Since mass loss is closely associated with microstructural damage and residual strength, the reduced mass loss observed in metakaolin-modified concrete indicates superior preservation of structural integrity after fire exposure. These findings support the suitability of metakaolin as a supplementary cementitious material for improving the post-fire durability and mechanical performance of concrete in fire-resistant structural applications (Gernay et al., 2024).

3. Water Absorption

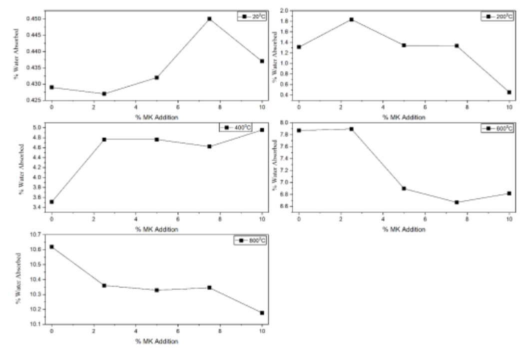

Figure 3.3 Water absorption

The results of figure 3.3 demonstrate that water absorption increased progressively with increasing temperature for all concrete mixes, indicating progressive deterioration of the pore structure and microstructural integrity following thermal exposure. Nevertheless, metakaolin (MK)-modified concretes consistently exhibited lower water absorption than the control mix, particularly at higher replacement levels, highlighting the beneficial role of MK in enhancing concrete durability after fire exposure. These observations are consistent with the established relationship between water absorption, pore connectivity, and permeability.

At ambient temperature (20°C), the incorporation of MK produced only slight variations in water absorption, with moderate replacement levels reducing absorption because of the combined filler effect and pozzolanic reaction. The ultrafine MK particles refined the interfacial transition zone (ITZ), while the consumption of calcium hydroxide generated additional calcium silicate hydrate (C-S-H), resulting in a denser cement matrix with reduced capillary porosity. Similar improvements in pore refinement and reduced permeability have been reported by Zhang et al. (2024).

Exposure to 200-400°C caused a noticeable increase in water absorption owing to the evaporation of free water, differential thermal expansion, and the onset of microcracking. Although dehydration of C-S-H and other hydration products further increased pore connectivity, MK-modified concretes exhibited smaller increases than the control, suggesting improved thermal stability. This agrees with Ahmad et al. (2024), who reported enhanced resistance to permeability changes in MK-blended concrete under moderate thermal exposure.

At 600-800 °C, water absorption increased substantially because of portlandite decomposition, severe degradation of C-S-H, and extensive microcrack formation (Liu et al., 2025). However, concretes containing 7.5-10% MK showed comparatively lower absorption than the control. This behaviour is attributed to the reduced calcium hydroxide content resulting from the pozzolanic reaction, thereby minimizing thermally induced pore expansion and preserving residual matrix integrity (Zhang et al., 2024). Overall, the findings indicate that MK effectively mitigates fire-induced deterioration by refining the pore structure and enhancing the residual durability of concrete, particularly under severe thermal exposure.

4. Residual Compressive Strength at Varying Heat Loads, Metakaolin Content, and Curing Age

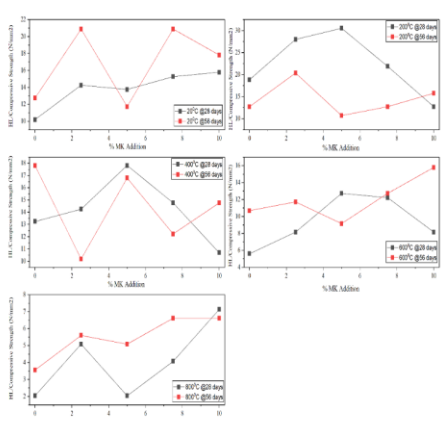

Fig 3.4 Residual Compressive Strength at Varying Heat Loads, Metakaolin Content, and Curing Age

Residual compressive strength decreased progressively with increasing temperature for all concrete mixtures, reflecting the progressive deterioration of the cement matrix under thermal loading. A slight strength enhancement observed at 200°C can be attributed to the evaporation of free water, which reduces pore pressure, and continued cement hydration induced by internal autoclaving. However, strength declined markedly beyond 400°C due to dehydration of calcium silicate hydrate (C-S-H), decomposition of calcium hydroxide (CH), and the development of thermally induced microcracks. At 800 °C, severe degradation of hydration products and extensive crack propagation resulted in substantial strength loss, consistent with the findings of Khoury (2024) and Liu et al. (2025).

Metakaolin (MK) incorporation significantly improved residual compressive strength, particularly at replacement levels of 7.5-10%. At ambient and moderate temperatures (20-400°C), MK enhanced strength through its filler effect and pozzolanic reaction, which refined the pore structure, strengthened the interfacial transition zone (ITZ), and generated additional secondary C-S-H. These mechanisms produced a denser and more durable microstructure. At elevated temperatures (400-800°C), MK-modified concretes retained higher residual strength than the control because the pozzolanic consumption of CH reduced the amount of portlandite available for thermal decomposition, thereby limiting internal cracking. Furthermore, the formation of alumina-rich hydrates and a refined pore network enhanced resistance to thermally induced damage, corroborating the observations of Ahmad et al. (2024) and Zhang et al. (2024).

Curing age also exerted a significant influence on residual performance. Specimens cured for 56 days consistently exhibited higher residual compressive strength than those cured for 28 days, with the difference becoming more pronounced above 400°C. This improvement reflects continued hydration, greater pozzolanic activity, enhanced matrix densification, and reduced capillary porosity, thereby increasing resistance to thermal degradation (Liu et al., 2025). Overall, the results demonstrate a synergistic interaction between MK content, curing age, and thermal exposure, with 7.5-10% MK providing optimum post-fire strength retention. These findings support the application of MK as a supplementary cementitious material for improving the fire resilience and post-fire structural integrity of concrete, consistent with recent performance-based fire engineering recommendations (Gernay et al., 2024).

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IV. CONCLUSION

This study demonstrates that the incorporation of calcined kaolin (metakaolin) significantly improves the post-fire mechanical performance of concrete. The pozzolanic activity of metakaolin refines the pore structure, enhances the interfacial transition zone, and contributes to the formation of additional calcium silicate hydrate (C-S-H) gel, resulting in higher strength and improved durability.

Experimental results indicate that metakaolin-modified concrete retains a greater percentage of its compressive, tensile, and flexural strengths after exposure to elevated temperatures compared with conventional concrete. Although all concrete specimens experience some deterioration due to thermal damage, mixes containing an optimum percentage of metakaolin exhibit reduced cracking, lower permeability, and better structural integrity after cooling. The improvement is most evident at moderate to high temperatures, where the denser microstructure helps limit the extent of fire-induced degradation.

Overall, the use of metakaolin is an effective and sustainable approach to enhancing the fire resistance and residual mechanical properties of concrete. It can increase the resilience and service life of concrete structures exposed to fire while reducing dependence on ordinary Portland cement. Future research should investigate the long-term durability of fire-damaged metakaolin concrete, optimize replacement levels for different applications, and evaluate its performance under real fire scenarios and repeated heating–cooling cycles.

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