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Effects of Activated Fly Ash in Low Ordinary Portland Cement

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Abstract: ***This study investigates the effectiveness of various activation strategies- chemical, mechanical, and thermal- for improving the reactivity of fly ash in very low clinker ternary binders. All binders contained 30% OPC, 40% fly ash, and 30% of either metakaolin or limestone as the second supplementary cementitious material (SCM). Compressive strength of mortar cubes was evaluated at 1, 3, 7, 28, and 90 days, and hydration studies were performed using thermogravimetric analysis (TGA) and isothermal calorimetry. Chemical activation was found to provide the highest early-age strength, while mechanical and thermal activations showed more pronounced effects at later ages. The results indicate that the optimal activation strategy depends on the type of co-SCM present in the system, highlighting the importance of multi-SCM design for sustainable low-carbon binders.***

Keywords: fly ash activation, supplementary cementitious materials, low clinker binder, compressive strength, pozzolanic reaction

1. Introduction

The cement industry is one of the major contributors to global CO_2 emissions, with clinker production being the primary source. Reducing the clinker factor through the use of Supplementary Cementitious Materials (SCMs) is a well-established and effective strategy. Commonly used SCMs include slag, silica fume, fly ash, and calcined clays, which not only reduce the carbon footprint of cement production but also improve the mechanical and durability properties of concrete.

Fly ash, a by-product of coal-fired power generation, is among the most widely used SCMs owing to its economic and environmental advantages. However, its pozzolanic nature- requiring calcium hydroxide (portlandite) from clinker hydration to initiate reaction—limits its replacement level to 35% in composite cements as per IS 16415. Beyond this threshold, fly ash negatively impacts binder performance due to insufficient portlandite availability.

To circumvent this limitation, this study explores the use of ternary binder systems containing 30% OPC, 40% fly ash, and 30% of a second SCM (metakaolin or limestone), along with three fly ash activation strategies: chemical, mechanical, and thermal. The goal is to enhance fly ash reactivity so that higher replacement levels can be achieved without compromising early or late-age mechanical performance.

2. Materials and Experimental Methods

2.1 Raw Materials

Four raw materials were used: Ordinary Portland Cement (OPC, Grade 43), Class F fly ash, high-purity limestone, and high-purity metakaolin. Material characterization included particle size distribution (PSD) measured by laser diffractometry (Mastersizer 3000), X-ray fluorescence (XRF) for oxide composition, and Loss on Ignition (LOI) measured at 1000°C for 1 hour.

The PSD analysis showed that fly ash is the coarsest material (d_{50} = 18.4 µm, d_{90} = 82.4 µm), while limestone is the finest (d_{50} = 3.83 µm, d_{90} = 18.1 µm). Metakaolin exhibited intermediate fineness (d_{50} = 6.57 µm, d_{90} = 26.4 µm). OPC had a d_{50} of 17.9 µm and d_{90} of 54.2 µm. The fine limestone particles are expected to promote hydration through the inert filler effect by providing nucleation sites for hydration products.

2.2 Blend Compositions

Ten binder blends were prepared with a fixed ratio of 30% OPC, 40% fly ash (activated or unactivated), and 30% second SCM (metakaolin or limestone). A turbo-mix was used for blend preparation. Table 1 summarizes all blend compositions.

2.3 Activation Methods

Three activation methods were investigated:

Chemical Activation: Based on literature [2,3], three activators were screened—anhydrous sodium sulphate (Na_2SO_4), calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$), and sodium silicate nonahydrate ($Na_2SiO_3 \cdot 9H_2O$). The selected activator was dissolved in mixing water at the time of casting with unactivated fly ash.

Mechanical Activation: Fly ash was ground in a planetary ball mill for 2 hours to increase fineness and amorphous silica content, thereby enhancing reactivity and the inert filler effect [4].

Thermal Activation: Fly ash was calcined at 800°C for 2 hours to reduce residual unburnt carbon. Two cooling strategies were employed: (a) slow cooling inside the furnace to room temperature, and (b) immediate quenching by pouring the hot fly ash into water, followed by oven drying and turbo-mixing to break agglomerates. The quenching method was intended to maximize amorphous (glass) content.

2.4 Test Methods

Mechanical Strength: Mortar cubes (70.6 mm) were cast at W/B = 0.4 using standard sand per IS 650. Compressive strength was measured at 1, 3, 7, 28, and 90 days per IS 4031-6.

Hydration Studies: Thermogravimetric analysis (TGA) and isothermal calorimetry were performed on paste samples. TGA was conducted from ambient temperature to 1000°C; mass loss between 400–500°C was used to quantify portlandite content, and the peak near 800°C indicated carbonate phases [5].

3. Results and Discussion

3.1 Mortar Compressive Strength

Figures 1 and 2 present the compressive strength results for metakaolin and limestone blend series respectively. The key observations are as follows:

Chemical activation (CH-LS and CH-MK) yielded the highest early-age compressive strength across both blend series. This is attributed to the increased alkalinity introduced by the chemical activator, which accelerates OPC hydration and thus early portlandite production. However, strength gain from day 28 to day 90 was only marginal for chemically activated blends, irrespective of the second SCM used.

Metakaolin blends consistently exhibited higher early-age strength than corresponding limestone blends. This is due to the high pozzolanic reactivity of metakaolin, which rapidly consumes portlandite. TGA data confirmed the near-complete depletion of portlandite in most metakaolin blends by day 3. Conversely, limestone blends showed significant strength gain from day 28 to day 90, attributed to the ongoing pozzolanic reaction of fly ash facilitated by the preserved portlandite reservoir.

Thermal activation results showed a dependence on the type of co-SCM: the quenched fly ash blend (TH-Q-MK) outperformed the slow-cooled blend (TH-C-MK) in the metakaolin series, while the opposite trend was observed for limestone blends. TH-C-MK was the weakest blend in its series at all ages.

Mechanical activation (MCH-LS and MCH-MK) resulted in marginal early-age strength improvement. Notably, MCH-LS achieved higher 90-day strength than MCH-MK, a reversal of the usual trend where metakaolin blends outperform limestone blends. This suggests that the finer mechanically activated fly ash, in conjunction with limestone's preserved portlandite, supports continued late pozzolanic reaction.

3.2 Thermogravimetric Analysis

TGA and DTG curves were analyzed at 1, 3, and 7 days for reference, chemical, and mechanical blends. In limestone blends, an accelerated and more intense portlandite peak at day 1 was observed for chemical and mechanical blends relative to the reference, confirming accelerated early hydration. This peak diminished by day 7, indicating pozzolanic consumption of portlandite.

In metakaolin blends, a small portlandite peak at day 1 rapidly disappeared by day 3 for the reference and mechanically activated blends, reflecting the high pozzolanic reactivity of metakaolin. In contrast, chemically activated metakaolin blends (CH-MK) maintained a relatively stable portlandite level through days 1, 3, and 7. The increased alkalinity from the chemical activator accelerates OPC hydration, sustaining portlandite production to offset its consumption.

The depletion of portlandite in metakaolin blends by day 7 (except CH-MK) explains the limited late-age strength gain observed in those blends, as insufficient portlandite is available to sustain fly ash pozzolanic reaction beyond early hydration.

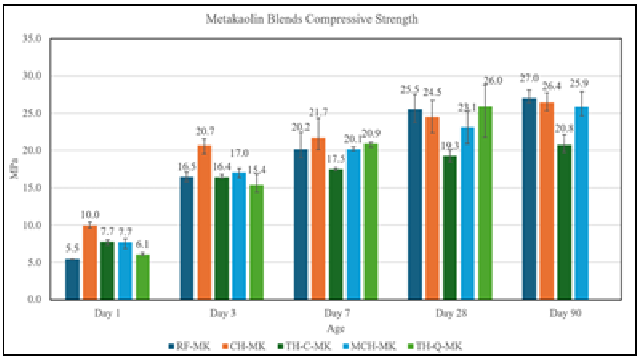

Figure 1: Compressive Strength of Metakaolin Blends

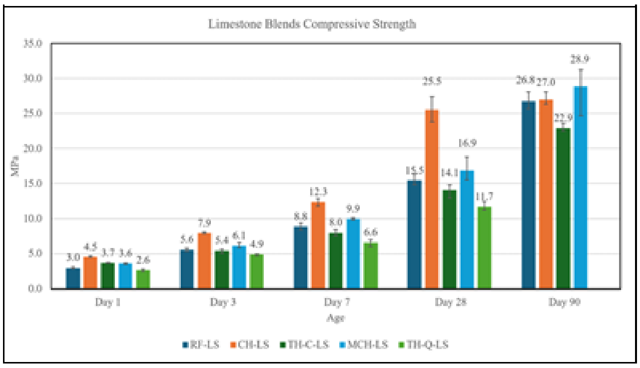

Figure 2: Compressive Strength of Limestone Blends

4. Conclusions

This study evaluated the performance of activated fly ash in very low clinker ternary binders containing 30% OPC, 40% fly ash, and 30% metakaolin or limestone. The following conclusions were drawn:

1) Chemical activation was the most effective method for improving early-age compressive strength in both metakaolin and limestone blend systems. Its effect is predominantly in the early stages of hydration with negligible improvement in later-age strength.
2) Thermal activation performance depended on the co-SCM: quenched fly ash (TH-Q-MK) performed better in metakaolin blends, while slow-cooled fly ash (TH-C-LS) gave better results in limestone blends. The mechanism behind this differential behavior warrants further microstructural investigation.

3) Mechanical activation provided marginal early-age gains but showed more significant improvement at 90 days in limestone blends, suggesting a synergistic effect between increased fly ash fineness and portlandite availability.
4) The optimal activation strategy for fly ash is not universal but depends on the type of co-SCM present. Metakaolin blends, despite higher early strength, show limited late-age gain due to rapid portlandite depletion. Limestone blends support continued fly ash pozzolanic reaction at later ages.

Overall, this study demonstrates the viability of achieving high SCM replacement in low-clinker binders through targeted activation strategies and multi-SCM design. Future work should focus on detailed microstructural analysis to elucidate the hydration mechanisms under different activation conditions.

Number citations consecutively in square brackets [1]. The sentence punctuation follows the brackets [2]. Multiple references [2], [3] are each numbered with separate brackets [1]–[3]. Please note that the references at the end of this document are in the preferred referencing style. Please ensure that the provided references are complete with all the details and also cited inside the manuscript (example: page numbers, year of publication, publisher’s name etc.).

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