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Development Performance Evaluation of Hybrid Alkaline Activator for Fly Ash-Based Geopolymer Concrete

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Abstract

Geopolymer concrete (GPC) has been identified as a possible low-carbon substitute for ordinary Portland cement (OPC) concrete as it makes use of industrial wastes like fly ash as the source of aluminosilicate precursor, which is activated through alkaline activation without the need for hydration through clinker. One problem that continues to hinder the widespread application of geopolymer concrete using fly ash is the requirement for ambient curing process, workability control, and the high cost and danger involved in using traditional single component activators like sodium hydroxide (NaOH). This paper focuses on the development of hybrid activator consisting of NaOH, sodium silicate (Na_2SiO_3) and partially replacing GGBS (ground granulated blast furnace slag). Five formulations have been designed with different concentrations of NaOH (8M-14M), Na_2SiO_3 to NaOH ratio (2.0-3.0), activator content (5-12% mass) and curing conditions (ambient and oven curing 60-80 °C). Tests conducted include fresh and hardening properties such as workability (slump test), setting time, compressive strength at 7, 28 and 56 days as well as microstructural investigations through Scanning Electron Microscopy (SEM). From the experiments, it was found that the optimal performance was attained by a hybrid activator with 12M NaOH, 2.5 Na_2SiO_3/NaOH ratio and 10% activator content in combination with partial GGBS addition (Mix M5), attaining 28-day compressive strength of 51.8 MPa under ambient curing conditions, which surpassed those of purely fly ash mixes under heat curing. It can be seen from the results that hybridization of activator system is an effective way towards ambient curing of high compressive strength geopolymer concrete.

1. Introduction

The production of cement is behind seven to eight per cent of all carbon dioxide emissions worldwide due mainly to the calcination of limestone and the energy-intensive clinker production stage. With the increasing demand for construction materials, especially in developing countries, there have been increased efforts to find low-carbon binders. One solution to this problem comes in the form of geopolymer concrete – first introduced by Davidovits during the 1970s–80s. Aluminosilicate-containing wastes produced from various industries – such as fly ash, GGBS, metakaolin or rice husk ash – are activated using an alkaline medium, resulting in a three-dimensional amorphous-to-semi-crystalline polymer structure known as the geopolymer gel, which bonds the aggregates in a fashion similar to C-S-H in OPC concrete.

Fly ash, which is one of the products of coal burning in thermal power plants, is especially suitable for geopolymer production because of its availability, cheapness, and the fact that fly ash is diverted from the waste process and thus prevents the burdening of landfills. Nevertheless, low-calcium (class F) fly ash geopolymers usually need high-temperature curing (between 60 °C and 90 °C) in order to be able to obtain adequate strength within realistic time frames, because geopolymerization does not progress at the required rate at room temperatures when there is no enough calcium present for C-S-H/C-A-S-H formation at the same time.

The alkaline activator system remains the key tool in the hands of mix designers for regulating the properties of geopolymer concrete both in the fresh state and after hardening. Sodium hydroxide only provides the hydroxide ions needed for the dissolution of the glassy matrix of fly ash but possesses poor potential for the acceleration of polycondensation reactions. On the other hand, sodium silicate provides soluble silicon ions for developing a compacted gel structure and thus helps in obtaining increased compressive strength; however, sodium silicate has a greater viscosity than sodium hydroxide, is costly, and excessive use causes poor workability and efflorescence. Hybrid activator technology, a combination of NaOH and Na_2SiO_3 in an optimized ratio along with a calcium-rich by-product in addition to fly ash as a precursor, aims to utilize the beneficial chemistry of both systems: alkali activation of aluminosilicate structure and calcium gel formation, the latter being efficient even at ambient temperature.

An experimental study is carried out in this paper to examine the formulation, performance evaluation and testing of such hybrid alkali activators used in geopolymer concrete using fly ash. In particular, the aims of this research include: (i) the formulation and characterization of hybrid activators consisting of NaOH, Na_2SiO_3 and partial substitution of slag; (ii) the effects of the molarity of the activator, silicate/hydroxide ratio and dose on fresh and hardened properties in terms of workability, setting time and compressive strength under ambient and heat curing; and (iii) the microscopic mechanism of strength development by SEM studies.

2. Literature Review

Many experiments have been done to analyze activator development in fly ash geopolymer system. The initial works of Hardjito and Rangan highlighted the basic effect of molarity of NaOH and ratio of Na_2SiO_3/NaOH on compressive strength; the strength of geopolymers increased with increase in molarity of NaOH to 14–16M after which increase in strength started declining or showed adverse effect. Many studies have further proved that there is always an ideal Na_2SiO_3/NaOH mass or volume ratio ranging from 2.0 to 2.5 that leads to an optimum balance between dissolution rate and gel densification in case of Class F fly ash geopolymer system.

In the field of blended precursors, in which GGBS or some other type of calcium-containing industrial waste has been partially used to replace fly ash, there have been many studies. As the use of GGBS leads to an increase in amount of calcium and thus C-A-S-H gel forms alongside N-A-S-H gel in fly ash geopolymer system; thus, these geopolymer systems attain high compressive strength without need of heat curing. Far fewer efforts have been made to study the hybrid activator concept as an integrated variable, whereby the chemistry of the activator and the precursors used are considered interdependent variables instead of independent ones. This paper seeks to fill that void by assessing various combinations of activators for both ambient and heat curing, with one combination utilizing the hybrid activator and partially substituting with slag.

3. Materials and Methods

3.1 Materials

The Class F fly ash that was low in calcium content and conformed to ASTM C618 was obtained from the thermal power plant. The chemical composition of the fly ash oxide in terms of its XRF analysis is presented in Table 1 below. Partial substitution of the precursor for ground granulated blast furnace slag that conformed to the specification limits was done in the case of Mix M5. Local river sand (fineness modulus 2.6) and crushed granite coarse aggregates (maximum nominal size of 12.5 mm) that fulfilled the grading criteria were used. The alkali activating solution was made up of commercial grade sodium hydroxide pellets (98% purity) and commercially available sodium silicate solution whose SiO_2/Na_2O modulus was around 2.0.

Oxide	% by mass	Oxide	% by mass
SiO_2	54.8	K_2O	1.2
Al_2O_3	28.4	TiO_2	1.6
Fe_2O_3	6.9	MgO	0.9
CaO	2.3	Loss on Ignition	1.4
Na_2O	0.5	$SiO_2+Al_2O_3+Fe_2O_3$	90.1

Table 1: Chemical Composition of Fly Ash (XRF Analysis)

3.2 Mix Design

Five different mixtures of geopolymers made using geopolymer concrete were prepared based on variation in molar concentration of NaOH, ratio of Na_2SiO_3/NaOH, amount of total activators as a percentage of total mass of binders, and curing method which is illustrated in Table 2. Mixes from M1 to M4 were based on 100% fly ash as the only source and were oven cured at either 60 °C or 80 °C for 24 hours after a resting period of 24 hours at room temperature, which is a routine procedure in laboratories for the preparation of heat-cured fly ash geopolymers. However, mix M5 included 15% GGBS by mass of total precursors and was cured at ambient temperature (about 27 ± 2 °C).

Mix ID	NaOH Molarity	Na_2SiO_3/NaOH Ratio	Hybrid Activator Dosage (%)	Curing Temp (°C)
M1	8M	2.0	5	60
M2	10M	2.5	7	60

Mix ID	NaOH Molarity	$Na_2SiO_3/NaOH$ Ratio	Hybrid Activator Dosage (%)	Curing Temp (°C)
M3	12M	2.5	10	80
M4	14M	3.0	12	80
M5 (Hybrid + slag)	12M	2.5	10 + 15% GGBS	Ambient

Table 2: Mix Proportions and Curing Regimes

3.3 Specimen Preparation and Testing

The alkali activator solution for each mix was prepared at least 24 hours before casting to ensure that the heat-producing dissolution process reached stability. The fly ash (and GGBS when used) was dry-mixed with the fine and coarse aggregates for about two minutes before adding the solution of the activator and mixing for an additional four minutes to obtain a homogeneous mixture. The workability of the fresh concrete was tested by the standard slump test immediately after mixing. Cubes measuring 150 mm per side were cast in three layers, compacted appropriately, and the initial and final setting times of the mixture were measured using the Vicat apparatus modified for geopolymer paste. The cubes were de-moulded after 24 hours and then subjected to their respective curing processes for compressive strength testing on days 7, 28, and 56 using the standard cube crushing method through a calibrated compression testing machine.

4. Results and Discussion

4.1 Fresh Properties

Slump, indicating workability, reduced steadily with the increase in molarity of NaOH and $Na_2SiO_3/NaOH$ ratio, as presented in Table 3 below. This is explained by the high viscosity of high-concentration solutions of sodium hydroxide and sodium silicate which causes an increase in internal friction of the fresh mix leading to an increase in the rate of dissolving and formation of early gels, hence decreasing its workability. The mix with the highest molarity (14M) and the highest $Na_2SiO_3/NaOH$ ratio (3.0) – M4 – had the lowest slump of 70 mm, thus approaching workability limit of placement without superplasticiser. Although Mix M5 contains GGBS, which is known to enhance setting through reaction with calcium, the mix had moderate workability of 110 mm slump implying that 10% activator concentration is reasonable in view of the combined precursor reactivity.

The setting time decreased inversely with the increase in molarity and slag content: M1 (8M without slag) had the longest initial setting time (approximately 220 minutes), while M5 (containing GGBS) had shorter setting time (approximately 95 minutes).

4.2 Compressive Strength Development

Mix ID	7-Day (MPa)	28-Day (MPa)	56-Day (MPa)	Slump (mm)
M1	14.2	22.6	24.8	140
M2	21.5	34.1	37.0	120
M3	29.8	46.3	49.5	95
M4	27.1	42.0	45.2	70
M5 (Hybrid + slag)	33.6	51.8	55.4	110

Table 3: Compressive Strength and Slump Results

Table 3 provides the results for the compressive strengths, and a positive correlation can be observed from these results in terms of the effect of increasing activator concentration on compressive strength, until the case of Mix M3 (12M, $Na_2SiO_3/NaOH$ = 2.5, 10%, 80 °C) which had the highest compressive strength value (46.3 MPa after 28 days) of all the fly ash mixes that were made purely using the fly ash and without any supplementary binders. Mix M4, with a higher concentration (14M) and $Na_2SiO_3/NaOH$ ratio (3.0) than Mix M3, yielded slightly lower compressive strength value (42.0 MPa).

Perhaps most importantly, Mix M5, characterized by the use of a moderate dosage of hybrid activator and 15% substitution of GGBS, provided the highest strength results at all ages, in the absence of heat treatment – 33.6 MPa at 7 days and 51.8 MPa at 28 days, outperforming even Mix M3 under heat treatment. It supports the hypothesis that calcium-induced gel formation with the aid of the hybrid $NaOH-Na_2SiO_3$ system is a valid alternative to heat input in terms of activation, providing a solution to the production of ambient-cured geopolymer concrete suitable for casting. The growth of strength recorded between 28 and 56 days for all mixes (5-8%) demonstrates further geopolymerization and calcium silicate hydrate formation processes for mixes with OPC, if needed.

4.3 Microstructural Analysis

Analysis of SEM images for Mix M3 (pure fly ash, heat curing) showed that there was a fairly dense and homogenous gel matrix with partially reacted spheres of fly ash incorporated in the geopolymer matrix, which is similar to the characteristic structure of N-A-S-H gels found in previous studies on fly ash geopolymers. In contrast, Mix M5 (hybrid activator + GGBS, ambient curing) showed a denser and more continuous microstructure due to the presence of few unreacted particles and fewer microcracks owing to the presence of both N-A-S-H and C-A-S-H gel structures, which occupy the capillary spaces more efficiently compared to the individual gels.

4.4 Discussion of Activator Design Implications

It may be said with confidence that activator hybridisation occurs on two levels – chemical hybridisation of the activator solution (comprising hydroxide and silicate compounds), and material hybridisation of the precursor matrix (using fly ash along with a calcium-rich by-product). While the former – chemical hybridisation of the activator (Mixes M1-M4) – allows for obtaining noticeable improvements in the strength values through concentration increase until the optimum is reached, it cannot solve the crucial problem of the thermal curing dependence characteristic of pure Class F fly ash systems. The latter – material hybridisation, demonstrated in Mix M5 – solves the issue, offering an independent-of-curing-temperature process mechanism. From the practical and economical point of view, the relatively low 15% GGBS substitution employed in this research provides an optimal solution: while allowing for ambient curing and improvement in strength values, it does not decrease significantly the environmentally-friendly characteristics related to high amount of fly ash.

5. Conclusion

The current research has investigated a hybrid alkaline activator approach towards fly ash geopolymer concrete, studying the effect of NaOH concentration, ratio of Na_2SiO_3 /NaOH, activator content, and precursor mixture on both fresh and hardened properties. The main conclusions include the following: higher concentrations of NaOH and the ratio of Na_2SiO_3 /NaOH increase the compressive strength to an optimal point (12 M NaOH and ratio 2.5), above which further improvement ceases to be observed, but decreasing workability continues to be observed; 15% substitution of fly ash by GGBS in combination with a medium content of a hybrid activator ensures ambient curing and the attainment of compressive strength (51.8 MPa at 28 days) higher than the one achieved through heated curing of neat fly ash blends; and the microstructure analysis shows that such higher performance is due to the presence of complementary N-A-S-H and C-A-S-H gels. The results indicate that the implementation of hybrid activation-batched precursor approach constitutes a reasonable method in the effort to utilize fly ash geopolymer concrete in cast-in-place projects, where ambient curing is a practical requirement. Further research should focus on the durability properties of hybrid activated geopolymer concrete – such as sulfates resistance, chloride penetration, and carbonation – along with environmental and economic aspects related to different slag substitutions ratios.

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