Effect of activator concentration on the strength, ITZ and drying shrinkage of fly ash/slag geopolymer concrete

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HIGHLIGHTS
- Fly ash/slag geopolymer concrete was studied by varying 10–16 M activator.
- The total heat released during paste formation was not significantly affected beyond optimum concentration.
- ITZ displayed satisfactory aggregate-paste interface at optimized concentration.
- Impact resistance of concrete increased with age and its compressive strength.
- The drying shrinkage was very small compared with the OPC concrete.

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ABSTRACT
Geopolymerization of fly ash and slag in a single mix was studied at ambient condition with the help of an Isothermal conduction calorimetry. Increasing activator concentration in the mix led to its shortening of the induction time as well as peak position shifting in the calorimetric curves which showed more workability loss and faster setting. Geopolymer concrete made with fly ash/slag composite mix and various activator concentrations was evaluated for its mechanical properties, age hardening and drying shrinkage. The optimum compressive strength of concrete mix was obtained at 14 M activator concentration. The presence of spongy amorphous geopolymer pastes extended onto the aggregate surface as viewed under Field emission scanning electron microscope (FESEM) images supported satisfactory aggregate-paste interface in the concrete. The compressive strength, elastic modulus and impact strength increased and the Poisson’s ratio decreased with increasing activator concentration. Under repeated drop weight impact, the specimens failed between 95 and 224 hammer blows depending on the activator concentrations. Geopolymer concrete gained an early strength compared with the OPC concrete as predicted by the equation specified in ACI 209R-92. The drying shrinkage of samples was ~0.1% only at the age of 6 months. It is concluded that fly ash/slag geopolymer concrete can be satisfactorily produced at room temperature with a desired compressive strength similar to that of OPC concrete.

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1. Introduction

Concrete is the most widely used construction material today and its demand is expected to grow to 18 billion tons a year globally by 2050 [1]. Portland cement, the principal hydraulic binder used in concrete is the product of an industry that is not only energy intensive but also responsible for large scale emission of CO₂ [2,3]. At the same time, the cement industry also faces challenges because of the limited reserves of raw materials especially limestone. The options to meet these challenges are either to lower the clinker factor of the binders as much as possible by adding supplementary cementitious minerals in the cement or to find out new alternative binders to Portland cement to meet the growing demand of concrete [3]. Geopolymer provides one route towards these objectives as it is produced from industrial by-products such as fly ash, slag etc. replacing 100% cement in concrete [4]. It essentially consists of a repeat unit of silicate monomer (-Si-O-Al-O-) synthesized from the reaction of alkali metal hydroxides/silicates and aluminosilicate source materials [5,6]. This binder has a great potential in the concrete industry as it cures and sets to a material with an X-ray amorphous structure and also gains strength more rapidly than the Portland cement.

The use of fly ash as an aluminosilicate source material has been investigated for producing geopolymer concrete because of its huge availability, rich silica and alumina contents and also the low water demand [7–10]. The research findings concluded that the engineering properties of fly ash-based geopolymer concrete are comparable to those of OPC concrete and the design strength can be achieved in a relatively short period of time. However, its use is mainly advocated in the pre-cast concrete industry because the activated fly ash cures at elevated temperature [7,11]. Knowing the necessity of acceptance in mass construction, the work is further extended to develop room temperature cured geopolymer concrete compositions by blending the fly ash with calcium containing compounds especially slag [12,13]. Calcium derived from the slag modifies sodium aluminosilicate (N–A–S–H) gel, partially replacing sodium with calcium to form (Ca-Na)-A–S–H gel [14] which causes rapid hardening of the fly ash-based concrete at ambient temperature. Puertas et al. [15] optimized the activation of fly ash/slag mix (50:50) at 10 M NaOH to produce geopolymer paste with a compressive strength of ~50 MPa at 25°C after 28 days. They stated that slag reacts almost completely whereas the fly ash partially dissolves and precipitates in the reaction process during paste formation. The main reaction product is a hydrated calcium silicate like C-S-H gel with high amount of tetra-co-ordinated aluminium in its structure as well as sodium ion in the interlayer spaces. Chi and Huang [16] used 4% and 6% Na₂O by mass of the fly ash/slag mix (50:50) along with sodium silicate with modulus ratio of 1 for making mortars and found that the mix activated with 6% Na₂O may be considered as the optimum mix design. Viewing the encouraging results of these pastes, Nath et al. [17] produced fly ash/slag geopolymer concrete with a compressive strength of 55 MPa at room temperature and optimized its setting time, workability and strength properties using slag content and activator content as principal variables only. Lee and Lee [18] studied the setting and mechanical properties of fly ash/slag concrete and optimized slag content in the range of 15–20% considering setting, slump and strength. There has been, however, no report available on the effect of activator concentration on the interfacial transition zone (ITZ), impact properties and drying shrinkage of the fly ash/slag geopolymer concrete. Given that the reaction products and microstructures depend on the type and activator concentration used, it is therefore, much needed to understand the behaviour of activated fly ash/slag composite mix as a binder in geopolymer concrete manufacturing.

In the present work, the aim of study is to investigate the effect of activator concentration on the properties of geopolymer concrete made with fly ash/slag composite mix and fluoride admixture. Four concentrations of activator (10, 12, 14 and 16 M) were used in the concrete mix. Different properties such as compressive strength, flexural strength, splitting tensile strength, modulus of elasticity, Poisson’s ratio and drying shrinkage of geopolymer concrete were determined. The impact behaviour of geopolymer concrete was studied under repeated drop weight test according to ACI 544.2R-89 [19]. FESEM was used to correlate deterioration in the mechanical properties of concrete with its interfacial transition zone. A correlation between the elastic modulus and compressive strength was also presented and compared with the heat cured fly ash geopolymer concrete and OPC concrete.

2. Experimental

2.1. Materials

Low calcium fly ash was collected from M/s National Thermal Power Corporation, Suratgarh, India. The Blaine’s fineness of fly ash was ~4099 cm²/g. The sum of SiO₂, Al₂O₃ and Fe₂O₃ content in the fly ash was 89.65% of the total mass. The mean size of fly ash particles was ~26.73 μm and ~82.48% of the fly ash particles were smaller than 45 μm. Ground granulated blast furnace slag was collected from M/s Vizag steel plant, Visakhapatnam, India. The Blaine’s fineness of slag was ~3144 cm²/g. The mean size of slag particles was ~19.19 μm and ~90.69% of particles were smaller than 45 μm. The glassy content and basicity of slag were 1.09 and 0.94 respectively. The particle size distribution of fly ash and slag is shown in Fig. 1. The chemical composition of fly ash and slag is shown in Table 1. River sand (fineness modulus 2.79) and sandstone aggregate (fineness modulus 6.97) were procured locally. Laboratory grade sodium hydroxide in a pellet form (98% purity) and laboratory grade sodium silicate (Na₂O, 8%; SiO₂, 27% and water, 65%) were used as activators. Sodium silicofluoride and polycarbosilic ether based superplastitizer (Glenium 51, BASF) were used as admixtures to control setting and consistency of the mix.

2.2. Casting of specimens

Mix proportioning of the geopolymer concrete carried out as per absolute volume method is given in Table 2. The gradation of fine and coarse aggregates was made with the help of a Fuller’s ideal curve method (Fig. 2). As can be seen in the grading curve, the experimental curve was in part parabolic and then straight. This indicated that graded aggregate imparted maximum density with a harsh or somewhat unworkable mix. Because of this, the excess of paste volume along with fine aggregate was trialed in the mix to improve its workability. The concentration of activator varied from 10 M to 16 M. The sodium hydroxide solution of different molarities was prepared by dissolving sodium hydroxide pellets into water. For example, 10 M activating solution consisted of 10x40 = 400 g sodium hydroxide per liter of solution and so on. Sodium hydroxide and sodium silicate were used in a molar ratio of 1:2.5 as reported by Hardjito et al. [8]. The water-geopolymer solid ratio in the concrete was kept at 0.19 and 0.21.

Fly ash and slag were inter-ground in a ratio of 2:1 as optimized in our earlier work [20]. The dry ingredients consisting of fly ash, slag, river sand and coarse aggregate were mixed together in a pan mixer for 3 min. Thereafter, activating