

EXPERIMENTAL ANALYSIS OF GEOPOLYMER CONCRETE FOR DIFFERENT CONSTRUCTION WORKS

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ABSTRACT: *The use of Portland cement in concrete construction is under critical review due to high amount of carbon dioxide gas released to the atmosphere during the production of cement. In recent years, attempts to increase the utilization of fly ash to partially replace the use of Portland cement in concrete are gathering momentum. Most of this by-product material is currently dumped in landfills, creating a threat to the environment. Geopolymer concrete is a 'new' material that does not need the presence of Portland cement as a binder. Instead, the source of materials such as fly ash, that are rich in Silicon (Si) and Aluminium (Al), are activated by alkaline liquids to produce the binder. Hence concrete with no Portland cement. This thesis reports the details of development of the process of making fly ash-based geopolymer concrete. Due to the lack of knowledge and know-how of making of fly ash-based geopolymer concrete in the published literature, this study adopted a rigorous trial and error process to develop the technology of making, and to identify the salient parameters affecting the properties of fresh and hardened concrete. As far as possible, the technology that is currently in use to manufacture and testing of ordinary Portland cement concrete were used. Fly ash was chosen as the basic material to be activated by the geopolymerization process to be the concrete binder, to totally replace the use of Portland cement. The binder is the only difference to the ordinary Portland cement concrete. To activate the Silicon and Aluminium content in fly ash, a combination of sodium hydroxide solution and sodium silicate solution was used. Manufacturing process comprising material preparation, mixing, placing, compaction and curing is reported in the thesis. Napthalene-based superplasticiser was found to be useful to improve the workability of fresh fly ash-based geopolymer concrete, as well as the addition of extra water. The main parameters affecting the compressive strength of hardened fly ash-based geopolymer concrete are the curing temperature and curing time, the molar H₂O -to-N a 2 O ratio, and mixing time. Fresh fly ash-based geopolymer concrete has been able to remain workable up to at least 120 minutes without any sign of setting and without any degradation in the compressive strength. Providing a rest period for fresh concrete after casting before the start of curing up to five days increased the compressive strength of hardened concrete.*

I. INTRODUCTION

After wood, concrete is the most often used material by the community. Concrete is conventionally produced by using the ordinary Portland cement (OPC) as the primary binder.

The environmental issues associated with the production of OPC are well known. The amount of the carbon dioxide released during the manufacture of OPC due to the calcination of limestone and combustion of fossil fuel is in the order of one ton for every ton of OPC produced. In addition, the amount of energy required to produce OPC is only next to steel and aluminium. On the other side, the abundance and availability of fly ash worldwide create opportunity to utilise this by-product of burning coal, as partial replacement or as performance enhancer for OPC. Fly ash in itself does not possess the binding properties, except for the high calcium or ASTM Class C fly ash. However, in the presence of water and in ambient temperature, fly ash reacts with the calcium hydroxide during the hydration process of OPC to form the calcium silicate hydrate (C-S-H) gel. This pozzolanic action happens when fly ash is added to OPC as a partial replacement or as an admixture. The development and application of high volume fly ash concrete, which enabled the replacement of OPC up to 60-65% by mass (Malhotra 2002; Malhotra and Mehta 2002), can be regarded as a landmark in this attempt.

II. FLY ASH-BASED GEOPOLYMER CONCRETE

In this work, fly ash-based geopolymer is used as the binder, instead of Portland or any other hydraulic cement paste, to produce concrete. The fly ash-based geopolymer paste binds the loose coarse aggregates, fine aggregates and other un-reacted materials together to form the geopolymer concrete, with or without the presence of admixtures. The manufacture of geopolymer concrete is carried out using the usual concrete technology methods. As in the OPC concrete, the aggregates occupy the largest volume, i.e. about 75-80 % by mass, in geopolymer concrete. The silicon and the aluminium in the low calcium (ASTM Class F) fly ash are activated by a combination of sodium hydroxide and sodium silicate solutions to form the geopolymer paste that binds the aggregates and other un-reacted materials.

III. LITERATURE REVIEW

The trading of carbon dioxide (CO₂) emissions is a critical factor for the industries, including the cement industries, as the greenhouse effect created by the emissions is considered to produce an increase in the global temperature that may result in climate changes. The 'tradeable emissions' refers to the economic mechanisms that are expected to help the countries worldwide to meet the emission reduction targets established by the 1997 Kyoto Protocol. Speculation has arisen that one ton of emissions can have a trading value about US\$10 (Malhotra 1999; Malhotra 2004).

The climate change is attributed to not only the global warming, but also to the paradoxical global dimming due to the pollution in the atmosphere. Global dimming is associated with the reduction of the amount of sunlight reaching the earth due to pollution particles in the air blocking the sunlight. With the effort to reduce the air pollution that has been taken into implementation, the effect of global dimming may be reduced, however it will increase the effect of global warming (Fortune 2005). In this view, the global warming phenomenon should be considered more seriously, and any action to reduce the effect should be given more attention and effort. The production of cement is increasing about 3% annually (McCaffrey 2002). The production of one ton of cement liberates about one ton of CO₂ to the atmosphere, as the result of de-carbonation of limestone in the kiln during manufacturing of cement and the combustion of fossil fuels (Roy 1999).

The contribution of Portland cement production worldwide to the greenhouse gas emission is estimated to be about 1.35 billion tons annually or about 7% of the total greenhouse gas emissions to the earth's atmosphere (Malhotra 2002). Cement is also among the most energy-intensive construction materials, after aluminium and steel. Furthermore, it has been reported that the durability of ordinary Portland cement (OPC) concrete is under examination, as many concrete structures, especially those built in corrosive environments, start to deteriorate after 20 to 30 years, even though they have been designed for more than 50 years of service life (Mehta and Burrows 2001).

The concrete industry has recognized these issues. For example, the U.S. Concrete Industry has developed plans to address these issues in 'Vision 2030: A Vision for the U.S. Concrete Industry'. The document states that 'concrete technologists are faced with the challenge of leading future development in a way that protects environmental quality while projecting concrete as a construction material of choice. Public concern will be responsibly addressed regarding climate change resulting from the increased concentration of global warming gases. In this document, strategies to retain concrete as a construction material of choice for infrastructure development, and at the same time to make it an environmentally friendly material for the future have been outlined (Mehta 2001; Plenge 2001).

In order to produce environmentally friendly concrete, Mehta (2002) suggested the use of fewer natural resources, less energy, and minimise carbon dioxide emissions. He categorised these short-term efforts as 'industrial ecology'. The long-term goal of reducing the impact of unwanted by-products of industry can be attained by lowering the rate of material consumption. Likewise, McCaffrey (2002) suggested three alternatives to reduce the amount of carbon dioxide (CO₂) emissions by the cement industries, i.e. to decrease the amount of calcined material in cement, to decrease the amount of cement in concrete, and to decrease the number of buildings using cement.

IV. EXPERIMENTAL PROGRAM

The main objectives of the preliminary laboratory work were:

- to familiarize with the making of fly ash-based

geopolymer concrete;

- to understand the effect of the sequence of adding the alkaline activator to the solid constituents in the mixer;
- to observe the behaviour of the fresh fly ash-based geopolymer concrete;
- to develop the process of mixing and the curing regime; and
- to understand the basic mixture proportioning of fly ash-based geopolymer concrete.

The preliminary laboratory work revealed the following:

MIXING

It was found that the fresh fly ash-based geopolymer concrete was dark in colour (due to the dark colour of the fly ash), and was cohesive. The amount of water in the mixture played an important role on the behaviour of fresh concrete. When the mixing time was long, mixtures with high water content bled and segregation of aggregates and the paste occurred. This phenomenon was usually followed by low compressive strength result of hardened concrete.

Communication with Davidovits (2002), suggested that it is preferable to mix the sodium silicate solution and sodium hydroxide solution before adding it to the solid constituents. He also suggested that the sodium silicate liquid obtained from the market usually is in the form of a dimer or a trimer, instead of a monomer, and mixing it together with the sodium hydroxide solution assists the polymerisation process. When this suggestion was followed, it was found that the occurrence of bleeding and segregation ceased.

The effects of water content in the mixture and the mixing time were identified as test parameters in the detailed study (see Chapter 4). From the preliminary work, it was decided to observe the following standard process of mixing in all further studies.

- Mix sodium hydroxide solution and sodium silicate solution together prior to adding to the dry materials.
- Mix all dry materials in the pan mixer for about three minutes. Add the liquid component of the mixture at the end of dry mixing, and continue the wet mixing for another four minutes.

Curing

Geopolymer concrete specimens should be wrapped during curing at elevated temperatures in a dry environment (in the oven) to prevent excessive evaporation. Unlike the small geopolymer paste specimens, which can easily be wrapped by placing a lid on the mould, a suitable method was needed for large size geopolymer concrete specimens. Extensive trials revealed wrapping of concrete specimens by using vacuum bagging film is effective for temperatures up to 100°C for several days of curing. To tighten the film to the concrete moulds, a quick lock seal (Figure 3.5) or a twist tie wire (Figure 3.6) was utilized. The later was used in all further experimental work due to its simplicity and economics.

Preliminary tests also revealed that fly ash-based geopolymer concrete did not set immediately at room temperature. When the room temperature was less than 30°C, the setting did not occur at least for 24 hours. Also, the handling time is a more appropriate parameter (rather than setting time used in

the case of OPC concrete) for fly ash-based geopolymer concrete.

V. EXPERIMENTAL RESULTS

The experimental results are presented and discussed. Each of the compressive strength test data points plotted in various graphs or stated in various Tables corresponds to the mean value of the compressive strengths of five test concrete cylinders in a series. The standard deviations are plotted on the test data points as the error bar.

In Section 4.2 of the Chapter, the effects of various salient parameters on the compressive strength of fly ash-based geopolymer concrete are discussed. The parameters considered are as follows:

1. Ratio of activator liquid-to-fly ash, by mass
2. Concentration of sodium hydroxide (NaOH) solution, in Molar
3. Ratio of sodium silicate solution-to-sodium hydroxide solution, by mass
4. Curing temperature
5. Curing time
6. Handling time
7. Addition of super plasticiser
8. Rest period prior to curing
9. Water content of mixture
10. Dry curing versus steam curing
11. Mixing Time
12. Age of concrete

In all cases, low calcium (ASTM Class F) fly ash from Batch I, Batch II or Batch III (Section 3.2.1) was used. The mass of aggregates (Section 3.2.3) was approximately 75 to 80 percent of the mass of the entire mixture.

Section 4.3 of the Chapter presents the measured elastic constants, while Section 4.4 describes the stress-strain relations in compression for different grades of fly ash-based geopolymer concrete. Section 4.5 and 4.6 report the indirect tensile strength and the density of the fly ash-based geopolymer concrete respectively.

Temperature history during curing at elevated temperature was measured, and the results are reported in Section 4.7. The Chapter ends with Section 4.8, where a mixture design process for fly ash-based geopolymer concrete is proposed.

In all, twenty-six different Mixtures were made to study the effect of various parameters. The details of these Mixtures are given in Table 4.1 and Table 4.2, and the properties of the Mixtures are presented in Tables 4.3 to 4.8. In Tables 4.1 and 4.2, the mass of each component of a Mixture is given in terms of kg per cubic metre of concrete.

VI. SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

The major conclusions, and some recommendations for future research. When this study started in 2018, the published literature contained only limited knowledge and know-how on the process of making fly ash-based geopolymer concrete. Most of the literature dealt with the use of metakaolin or calcined kaolin as the source material for making geopolymer paste and mortar. Moreover, the exact details regarding the mixture compositions and the process of

making geopolymers were kept undisclosed in the patent and commercially oriented research documents. With the generic information available on geopolymers, a rigorous trial-and-error method was adopted to develop a process of manufacturing fly ash-based geopolymer concrete using the technology currently used to manufacture OPC concrete. In order to reduce the number of variables in this trial-and-error approach, the study was restricted to low-calcium (ASTM Class F) dry fly ash obtained from Collie Power Station in Western India, and to the type of aggregates used locally to make OPC concrete. After some failures in the beginning, the trial-and-error method yielded successful results with regard to manufacture of low-calcium (ASTM Class F) fly ash-based geopolymer concrete. Once this was achieved, tests were performed to quantify the effect of the salient parameters that influence the short-term properties of fresh and hardened geopolymer concrete. In the following Sections, the outcomes of the study are summarised.

MANUFACTURING PROCESS

Material Preparation

Aggregates used in the manufacture the fly ash-based geopolymer concrete were in a saturated surface dry (SSD) condition. The aggregate selection and proportion were in accordance with the current practice used in making OPC concrete. The alkaline liquid consisted of a combination of sodium silicate solution and sodium hydroxide solution. The sodium silicate solution was purchased from a local supplier. The sodium hydroxide solution was prepared by dissolving the solids, purchased from a local supplier in flakes or pellets form, in water. Both the solutions were premixed the day before use. The alkaline liquid was mixed with the super plasticiser, if any, and the extra-added water, if any, to prepare the liquid component of the geopolymer concrete mixture.

MIXING, PLACING AND COMPACTION

The aggregate and the fly ash were mixed dry in a pan mixer for about three minutes. The liquid component of the mixture was then added to the solids particles, and mixing continued for another four minutes in most cases.

The fresh fly ash-based geopolymer concrete remained workable up to at least two hours without any sign of setting and degradation in compressive strength. The fresh geopolymer concrete could easily be placed, compacted, and finished in moulds in that time. In all these operations, the equipment and the facilities currently used for OPC concrete were used. For cylinder specimens of 100x200 mm, the mixture was cast in three layers. Each layer received 60 manual strokes, and vibrated for 10 seconds on a vibrating table. In some cases, the common internal needle vibrator was also utilised to successfully compact the fly ash-based geopolymer concrete.

CURING

After finishing, the test specimens were covered by a vacuum bagging film. Curing at an elevated temperature was achieved either in the dry curing environment in an oven, or in the steam curing chamber, for a specified period of time.

After curing, the concrete specimens were allowed to cool down in the moulds to avoid drastic change in the environment for at least six hours. After releasing from the moulds, the test specimens were left to air dry in the ambient conditions in the laboratory until the day for testing.

TEST SPECIMENS AND TEST VARIABLES

The test specimens in this study were mainly of 100x200 mm cylinders; larger size 150x300 mm cylinders were used to measure the indirect splitting tensile strength.

The concentration of sodium hydroxide solution was in the range between 8 M and 16 M. The sodium silicate solution-to-sodium hydroxide solution ratio by mass was in the range of 0.4 to 2.5; for most Mixtures, this ratio was 2.5. The solution-to-fly ash ratio by mass was approximately 0.35 in most cases, except for the Mixtures with extra-added water.

In order to study the effect of mixture composition on the compressive strength of fly ash-based geopolymer concrete, the test variables were the H₂O-to-Na₂O molar ratio in the range between 10.00 and 14.00, and the Na₂O-to-SiO₂ molar ratio between 0.095 and 0.120. These ranges of variable were selected after several trials. Outside these ranges, geopolymer concrete mixtures were either too dry for handling or too wet causing segregation of aggregates. For these ranges of variables, the water-to-geopolymer solids ratio by mass in the geopolymer paste varied from 0.17 to 0.22. The mass of naphthalene sulphonate-based super plasticiser varied from 0% to 4% of the mass of fly ash. Workability was measured by the conventional slump test. The influence of water content on the slump value was also studied by varying the mass of extra water added to a reference mixture in the range of 0 to 26.5 kg/m³.

The range mixing time studied was between two and sixteen minutes. For curing, temperature ranges from 30°C to 90°C were studied. The curing time ranged from four hours to four days, either in the dry curing environment in the oven or in the steam curing chamber. The influence of age at test was studied up to the age of 90 days.

VII. CONCLUSIONS

Based on the experimental work reported in this study, the following conclusions are drawn:

1. Higher concentration (in terms of molar) of sodium hydroxide solution results in higher compressive strength of fly ash-based geopolymer concrete (Table 4.9).
2. Higher the ratio of sodium silicate-to-sodium hydroxide ratio by mass, higher is the compressive strength of fly ash-based geopolymer concrete (Table 4.9).
3. As the curing temperature in the range of 30°C to 90°C increases, the compressive strength of fly ash-based geopolymer concrete also increases (Figures 4.1 and 4.2).
4. Longer curing time, in the range of 4 to 96 hours (4 days), produces higher compressive strength of fly ash-based geopolymer concrete (Figure 4.3). However, the increase in strength beyond 24 hours is not significant.
5. The addition of naphthalene sulphonate-based super plasticiser up to approximately 4% of fly ash by mass, improves the workability of the fresh fly ash-based geopolymer concrete with very little effect on the

compressive strength of hardened concrete (Figure 4.5, 4.6 and 4.7).

6. The slump value of the fresh fly-ash-based geopolymer concrete increases with the increase of extra water added to the mixture (Figure 4.13).

7. The Rest Period, defined as the time taken between casting of specimens and the commencement of curing, of up to 5 days increases the compressive strength of hardened fly ash-based geopolymer concrete. The increase in strength is substantial in the first 3 days of Rest Period (Figure 4.8 and 4.9).

8. The fresh fly ash-based geopolymer concrete is easily handled up to 120 minutes without any sign of setting and without any degradation in the compressive strength (Figure 4.4).

9. As the H₂O-to-Na₂O molar ratio increases, the compressive strength of fly ash-based geopolymer concrete decreases (Figure 4.10).

10. As the ratio of water-to-geopolymer solids by mass increases, the compressive strength of fly ash-based geopolymer concrete decreases (Figure 4.11).

11. The effect of the Na₂O-to-SiO₂ molar ratio on the compressive strength of fly ash-based geopolymer concrete is not significant (Figure 4.12).

12. The compressive strength of heat-cured fly ash-based geopolymer concrete does not depend on age (Figures 4.16 and 4.17).

13. Prolonged mixing time of up to sixteen minutes increases the compressive strength of fly ash-based geopolymer concrete (Figures 4.14 and 4.15).

14. The average density of fly ash-based geopolymer concrete is similar to OPC concrete.

15. The measured values of the modulus elasticity of fly ash-based geopolymer concrete with compressive strength in the range of 40 to 90 MPa, were similar to those of OPC concrete. The measured values are at the lower end of the values calculated using the current design Standards due to the type of coarse aggregate used in the manufacture of the geopolymer concrete (Table 4.11).

16. The Poisson's ratio of fly ash-based geopolymer concrete with compressive strength in the range of 40 to 90 MPa falls between 0.12 and 0.16 (Table 4.10). These values are similar to those of OPC concrete.

17. The stress-strain relations of fly ash-based geopolymer concrete in compression fits well with the expression developed for OPC concrete (Figures 4.19, 4.20, 4.21 and 4.22), with the strain at peak stress in the range of 0.0024 to 0.0026 (Table 4.12).

18. The indirect tensile strength of fly ash-based geopolymer concrete is only a fraction of the compressive strength, as in the case of Portland cement concrete. The measured values are higher than those recommended by the relevant Indian Standard (Table 4.13).

19. Fly ash-based geopolymer mortar does not show any exothermic action, as shown by metakaolin-based geopolymer paste or mortar (Figure 4.24). In spite of this, the fly ash-based geopolymer shows high compressive strength.

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