



Influence of calcium content on durability of geo polymer mortar

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Abstract : In recent times, interest in research stimulated in the field of geopolymerisation because of its environmentally friendly nature. Despite extensive research conducted on various aspects of geopolymerisation, especially in enhancing the properties of resultant binders, a number of questions remain to be answered. The role of calcium in geopolymerisation is one of them. In this study an attempt is made to understand the effect of exterior calcium on durability aspects of geopolymer mortar. Experimental work is done by partially replacing fly ash (FA) with optimum 10% calcium hydroxide (CH) for alkaline liquid ratio (AL/FA) of 0.5 at 10M of sodium hydroxide (NaOH). Hence a comparative study is done with optimum 10%CH and 0%CH, Cube strength is monitored against durability properties such as water absorption, resistance to acids, sulphate and chloride attacks up to 3 months (90 days).

Keywords : Geopolymer mortar, Calcium content, Compressive strength, Durability, Acid, Chloride and Sulphate attack.

Introduction

Geopolymer is a cementitious material, manufactured from an aluminosilicate precursor activated in a high alkali medium. From the term geopolymer, it should not be concluded that polymers are used to manufacture geopolymer concrete. Source material like Fly Ash, Metakaolin clay, Rice Husk Ash etc, rich in silica and alumina belong to geological origin. The polymerization process involves a fast chemical reaction under alkaline conditions on silicon-aluminium minerals that results in a three dimensional polymeric chain and ring structure. The ultimate structure of geopolymer depends largely on the ratio of Si to Al (Si:Al), with the materials having a ratio of Si:Al between 2 to 3.5 for use in concrete application. A critical feature is that water is added only for workability and this water does not become a part of geopolymer structure.

In other words, water is not involved in the chemical reaction and is expelled during curing and drying. In the hydration process of OPC, the resultant products are predominantly calcium silicate hydrate (C-S-H) gel and calcium hydroxides. Whereas in the case of geopolymer, these do not form. CSH is a gel of hydrated CaO-SiO₂, which normally contributes mechanical strength to cement. The major difference between geopolymers and Portland cement in terms of their chemical composition is calcium. It is not essential for calcium to be present in any part of a basic geopolymeric structure. In contrast, the formation of three dimensional amorphous alkali aluminosilicate networks which attributes the binding properties to geopolymeric gel in terms of their elemental

composition is calcium. The presence of calcium in fly ash in significant quantities could interfere with the polymerisation setting rate and alters the microstructure. If excess calcium is added, some forms of C-S-H gel will be obtained. But it has significantly lower Ca/Si ratio than the CSH gel formed from hydration of Ordinary Portland Cement.

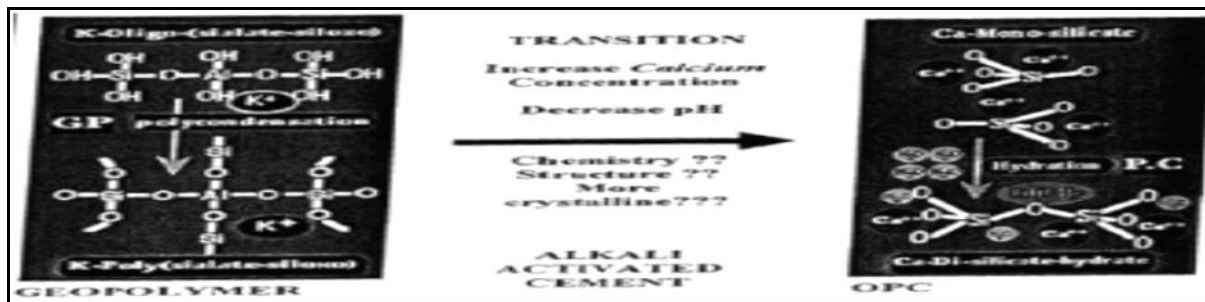


Figure 1. Conceptual Model of Relationship between Geopolymers and OPC.

The role of calcium in ancient concrete could possibly include : (a) The formation of the traditional calcium silicate hydrate (C-S-H). (b) Participation in the geopolymerisation in forming Ca-geopolymer and, (c) Bridging the bonding between the calcium silicate hydrate and geopolymers or many others. However, other research indicates that the presence of both CSH and geopolymer gel in a geopolymer could have beneficial effects on strength because the CSH phase act like micro-aggregates for the geopolymer gel and forms a denser and more uniform binder¹. More research needs to be conducted to understand the effects of composition and microstructure on mechanical properties of both the geopolymer gel and the CSH phases.

Experimental Investigation

Materials

The materials used in this study are low calcium fly ash as source material, calcium hydroxide, sand, alkaline liquids (Sodium Hydroxide and Sodium Silicate), and water.

Fly Ash used was procured from the Ramagundam thermal project. Fly ash passing from 90 micron sieve was used with a specific gravity 2.3. The chemical composition of fly ash was obtained by XRF method of analysis at Indian Institute of Chemical Technology, Tarnaka, Hyderabad. According to IS 3812-1981, the maximum and minimum quantities of chemical compounds present in the fly ash were also checked². Clean and dry river sand available locally passing through IS 2.36 mm sieve was used for casting all the mortar specimens considered in this study. The grading of sand used conforms to Zone-II of IS: 383 -1970.

The alkaline liquid used was a combination of sodium silicate solution and sodium hydroxide solution. The sodium silicate solution (Na₂O= 15.9%, SiO₂=31.4%, and water=52.7% by mass) was purchased from a local market in bulk. The sodium hydroxide (Na OH) in flakes or pellets form with 97%-98% purity was also purchased from a local market. The NaOH flakes were dissolved in water to make the solution with required molarity. Calcium hydroxide in a powdered form was purchased from the local suppliers. Potable water free from any impurities and organic materials conforming to IS 456-2000(19) was used for diluting NaOH flakes^{3,4}.

Mix Proportion

Ratio of Fly ash to Fine aggregate 1:2.5 was selected after doing trail mixes with 70.6mm cube size , which reflected very flowmortar for 2.2 and very stiff for a ratio 3. Ratio of sodium silicate solution-to-sodium hydroxide solution is kept as 2.5; Molarity of NaOH is 10M for alkaline liquid-to-fly ash 0.5. Durability studies are carried out for two types of mix cases ie, G0-mix with 0% CH and 100% fly ash, G10-mix with 10%CH and 90% fly ash

Geopolymer Synthesis and curing

For mortar sample preparation, Sodium hydroxide solution and sodium silicate solution are mixed together 24 hours prior to adding to the dry materials. Fly ash and CH were mixed thoroughly until a uniform mix, as far as possible, was produced. The alkaline mix was subsequently stirred with the dry mix to form a paste and mixed for a further 3 minutes to ensure homogeneity. Sand was gradually added until a uniform mixture was formed, which was then poured into cube specimens and vibrated. The sample specimens were left with the moulds in open air in the ambient temperature ranging from 38⁰ C to 40⁰C for 24 hours. Then they were demoulded and placed in shade for further chemical attack^{5,6,7}. Improvement in setting time with addition of CH was observed compared to only fly ash based geopolymer⁸.

Durability Studies

Experimental investigations were carried out on the geopolymer mortar test specimens of fly ash to sand ratio of 1:2.5 with fluid to binder ratio as 0.5, 10M to ascertain the durability-related properties. A total of one forty four numbers of samples were cast for G0 and G10 grade at 10M of NaOH. The visual appearance, residual compressive strength and change in mass were observed on the 28th day, 56th day and 90th day of immersion, and readings were noted. The surface of the cubes were cleaned, weighed and tested in the compression testing machine.

Normal geopolymer mortar and optimum calcium added geopolymer mortar cubes were cast to test durability against sulphate, acid, chloride attack and water absorption. Observations were recorded on the fourth, eighth and thirteenth weeks after immersion into the solutions. Eighteen numbers of mortar cubes for G0 grade and eighteen numbers of mortar cubes for G10 grade were cast for each case, and immersed in 5% H₂SO₄, 5% HCL, 5% Na₂SO₄ and 5% NaCl solution^{9,10,11,12}.

Results and Discussions

All the specimens recorded loss in weight over the entire duration of exposure 12 weeks. Visual appearance, changes in mass and residual compressive strengths were evaluated and presented respectively.

Visual appearance

Both type G0 and G10 Specimens did not exhibit any noticeable colour change and showed no visible signs of deterioration in sulphuric acid and hydrochloric acid in 4 weeks of immersion. The specimens were seen to remain structurally intact. However, the surface became a little softer as the duration of the test progressed up to 90 days, but could not be easily scratched with finger nails (Figure 1&2). In Figure 3, it can be seen that the visual appearance of the test specimens after soaking in sodium sulfate solution up to 90 days revealed that there was no change in the appearance of the specimens compared to the condition before they were exposed. The test specimens were immersed in 5% sodium chloride solution. The chloride attack was evaluated based on change in mass and change in compressive strength after exposure up to 12 weeks. In Figure 4, the visual appearance of the test specimens is seen.



Figure 1 specimens after 90days in 5% H₂SO₄ Figure 2 specimens after 90days in 5% HCL



Figure 3 specimens after 90days in 5% Na₂SO₄ solution.



Figure 4 specimens after 90days in 5% NaCl solution.

Change in mass

The weight loss in specimens immersed in Hydrochloric acid was less compared to those immersed in sulphuric acid. After 12 weeks of exposure, all the geopolymer mortar specimens of G0 mix invariably lost strength by about 27.54% when exposed to H₂SO₄ and about 15.5% when exposed to HCL. Whereas the G10 specimens had a substantial weight loss of about 29.03% when exposed to H₂SO₄ and 14.9% when exposed to HCL. The weight loss is attributed to the residual compressive strength of specimens is graphically illustrated in Figures 5-6.

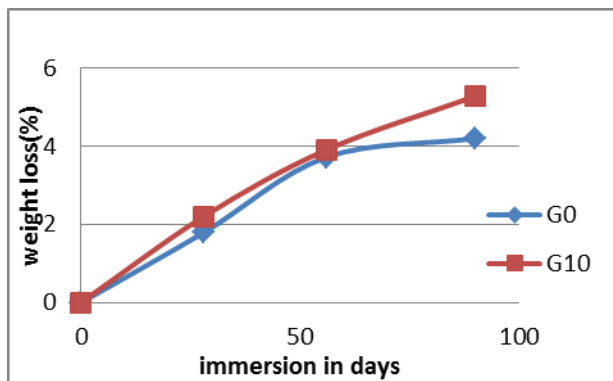


Figure 5 Weight loss of mortar specimen when exposed to 5% H₂SO₄

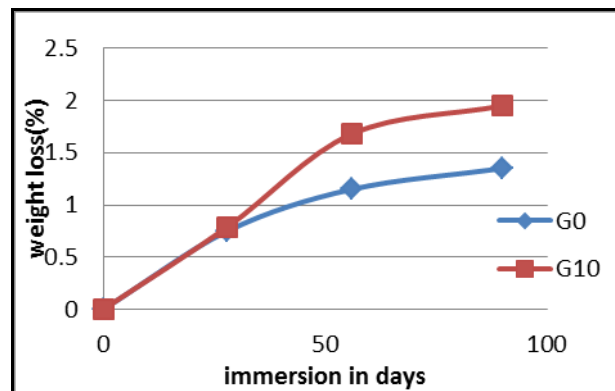


Figure 6 weight loss of mortar specimen when exposed to 5% HCL

The increase in mass of specimens soaked in sodium sulphate solution was approximately 1.5% after 90 days of exposure and in the case of specimens with added calcium; this increase in mass was about 1.2%. For comparison, Figure 7 presents the change in mass of specimens soaked for the corresponding period. It can be seen that there was no reduction in the mass of the specimens, as confirmed by the visual appearance of the specimens in Figure 3.

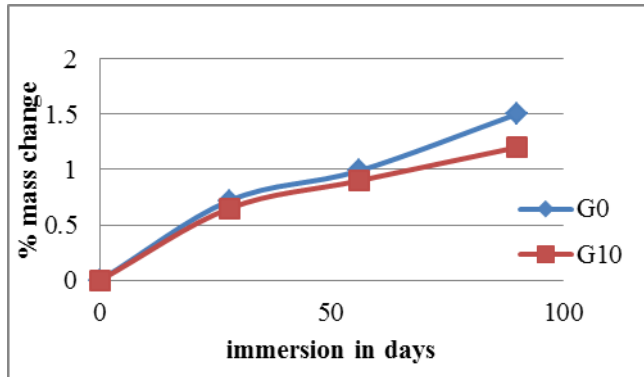


Figure 7 weight gain of mortar specimen when exposed to 5% Na₂SO₄

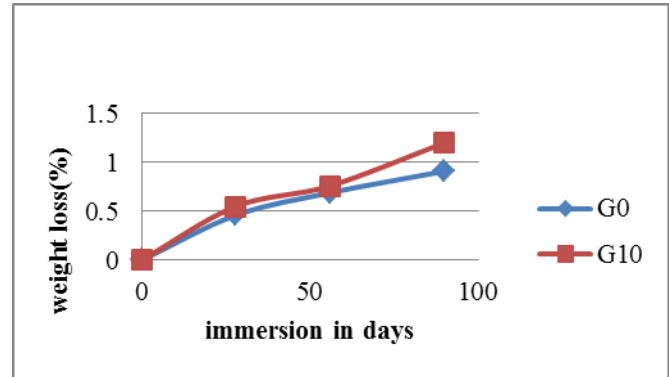


Figure 8 weight loss of mortar specimen when exposed to 5% NaCl

Figure 8 shows the change in mass of G0 and G10 specimens which had lost mass approximately by 0.91% and 1.2% after 12 weeks exposure to sodium chloride solution.

Change in compressive strength

On observation after 12 weeks of exposure, all the Geopolymer mortar specimens of G0 mix invariably had lost strength by about 27.54% when exposed to H₂SO₄ and about 15.5% when exposed to HCL. Whereas the G10 specimens had a substantial weight loss of about 29.03% when exposed to H₂SO₄ and 14.9% when exposed to HCL. The weight loss is attributed to the residual compressive strength of specimens is graphically illustrated in Figure 9-10.

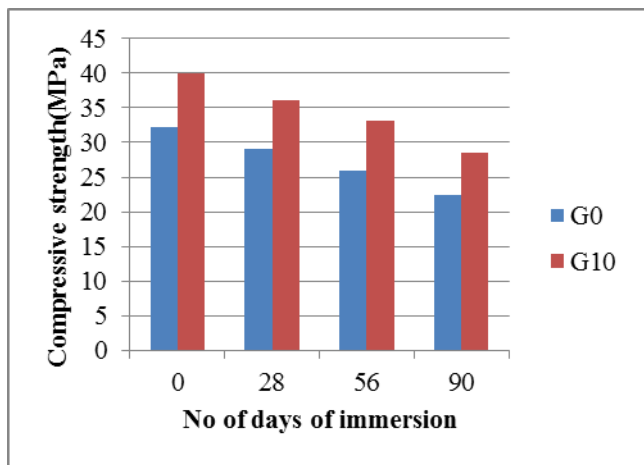


Figure 9 compressive strength variation when immersed in 5% H₂SO₄ solution.

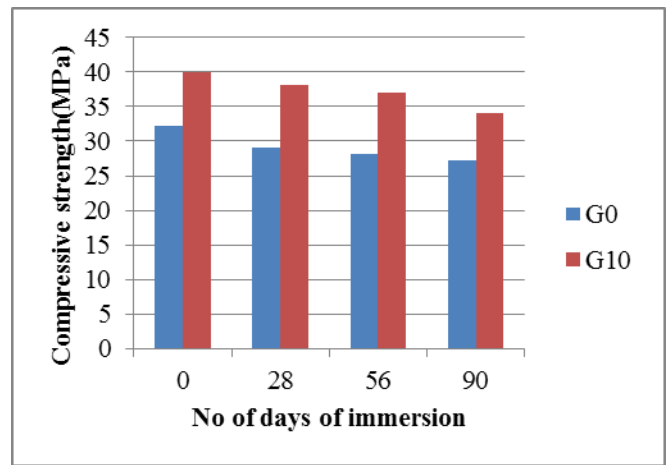


Figure 10 compressive strength variation when immersed in 5% HCL solution.

Change in compressive strength was determined by testing the specimens after 4, 8, 12 weeks of soaking in sulphate solution. The test data reveals that sodium sulphate solution causes very little reduction in compressive strength in geopolymer concrete specimens. The test results show that exposure of fly ash-based geopolymer mortar specimens to sodium sulphate solution had strength loss of about 5.3% for G0 and 4.21% for G10 samples after 12 weeks of exposure. The test results for various exposure periods are presented in Figure 11

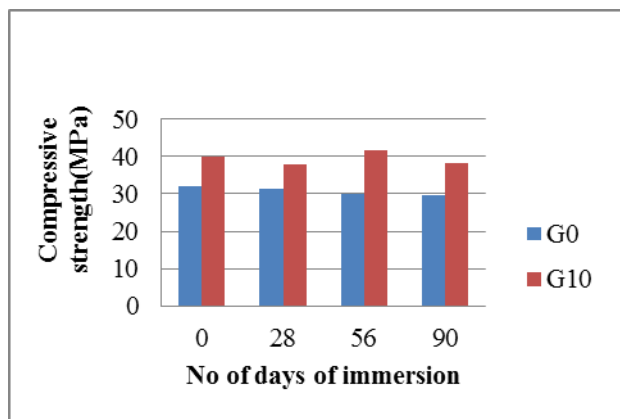


Figure 11 compressive strength variation when immersed in 5%Na₂SO₄solution

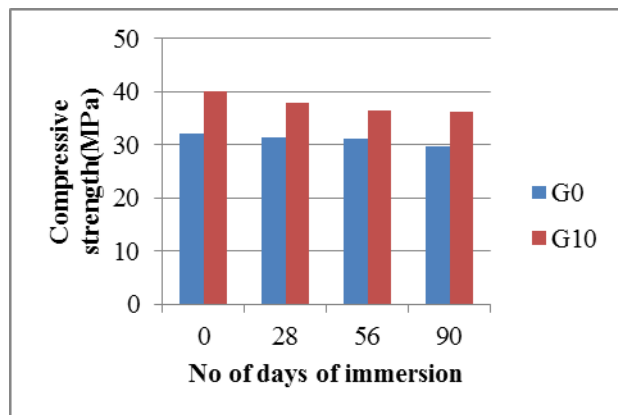


Figure 12compressive strength variation when immersed in 5%NaCl solution.

Figure12 shows the change in compressive strength obtained after 4, 8 and 12 weeks of exposure. Test results have shown that G0specimen exhibited a reduction in compressive strength by 7.82% after 90 days of exposure. Similarly, the G10 specimenshows 9.95% decrease after 90 days of exposure.

Water Absorption Test

The water absorption after 90 days immersion of G0 grade is found to be 2.83% and G10 specimens recorded water absorption of 1.63%.This shows the decrease in water absorption in geopolymer mortar with added calcium.

Conclusions

The compressive strength of some of the series of ambient cured geo-polymer mortar with added calcium is comparable to that of cement mortar, indicating that those mortar combinations can be adopted for making ambient cured structural concrete.

- Water absorption values were found directly related to total porosity of specimens. Mortar specimens, showed a decreasing trend in water absorption with increasing calcium content.
- Addition of calcium had little effect on the durability to acid attack. Deterioration effect of sulphuric acid was found more severe than hydrochloric acid.
- The weight loss was observed to be gradually decreased with increase in calcium content in all the specimens immersed in sodium sulphate and sodium chloride.
- Increased calcium content showed better resistance to sulphate and chloride attack. The specimens in the study showed better performance.

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