INDIAN INSTITUTE OF TECHNOLOGY GUWAHATI
SHORT ABSTRACT OF THESIS

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Thesis Title: A study on influence of control parameters on strength, durability and microstructure of fly ash-ground granulated blast furnace slag based geopolymer mortar and concrete

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SHORT ABSTRACT

Ordinary Portland cement (OPC) has been extensively used as the primary binding material in the preparation of concrete. However, over last few decades, Portland cement manufacturing industry has become one of the major contributors of emission of large volume of CO_2 during the manufacturing process. On the other hand, the management of industrial waste in limited landfill space creates a massive challenge for the developing countries. To mitigate these issues, since last few decades, extensive research work has been undertaken to explore new alternate and more sustainable construction materials. In this line, there is a widespread use of industrial wastes in the production of concrete. Geopolymer concrete (GPC) has gained popularity as a potential alternative of Portland cement concrete due to its better mechanical properties and environmental benefits. The major challenges associated with the geopolymer composites are the variations in the physicochemical properties of precursor materials depending upon their sources, requirement of heat curing for the development of geopolymer composites with low calcium bearing precursor materials, limited availability of efficient and systematic mix design methodologies etc. Furthermore, the limited research works on durability performance of geopolymer composites in various aggressive environment is hindering the practical application of this material.

To address the aforementioned challenges associated with development of geopolymer composites, the present study aims to develop geopolymer mortar (GPM) and geopolymer concrete (GPC) by investigating the influence of wide range of control parameters on fresh, mechanical, microstructure, and various durability properties of fly ash-GGBS based geopolymer composites. In this study, Taguchi-Grey relational analysis (GRA) method was used to arrive at the optimal combination of control parameters (mix parameters) of geopolymer mortar and geopolymer concrete with respect to multiple properties simultaneously. In this research work, the materials used for preparation of GPM and GPC mixes were class F fly ash and ground granulated blast furnace slag (GGBS) as precursor materials, a combination of sodium hydroxide (NaOH) solution (SH) and sodium silicate (Na_2SiO_3) solution (SS) as alkaline solution, river sand, and coarse aggregates of 20 mm MSA (maximum size of aggregate) and 10 mm MSA (in case of GPC mixes). Two types of sulfate solution such as sodium sulfate (Na_2SO_4) and magnesium sulfate ($MgSO_4$) solution and two types of acid solution such as sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) solution

were used to investigate the resistance of GPM against sulfate and acid attack. Sodium chloride (NaCl) was used as the source of chloride ions while examining the effect of chloride ions on consistency, compressive strength, and steel rebar corrosion in geopolymer concrete (GPC).

The experimental program was carried out in two different series i.e., GPM series and GPC series, and in each series, various experiments were conducted in two different phases. In 1st phase of GPM series, Taguchi-GRA method was implemented to investigate and optimize the influence of control parameters on fresh (setting time, and flowability), mechanical (compressive strength), and durability properties (water absorption, apparent volume of permeable voids, sorptivity, and resistance against sulfate and acid attack) of geopolymer mortar mixes simultaneously. The considered control parameters and their levels were GGBS replacement level: 15%, 30%, and 45%, water-to-geopolymer solids (W/GPS) ratio: 0.31, 0.33, and 0.35, molarity of NaOH solution: 10 M, 12 M, and 14 M, and sand-to-binder (S/B) ratio: 1.5, 2.0, and 2.5. Subsequently, the verification experiments were performed on the mix combination of optimized level of control parameters derived from the multi-response optimization by Taguchi-GRA method. The GPM cube specimens of size 50 mm were prepared for compressive strength test at the age of 7, 28, 90, and 180 days of ambient curing, water absorption and apparent volume of permeable voids (AVPV) test at 28 days, and sulfate and acid resistance test for an exposure period of 26 weeks. In addition, cylindrical GPM specimens of size 100 mm diameter × 50 mm height were prepared for sorptivity test at 28 days. All fly ash-GGBS based geopolymer mortar specimens were prepared under ambient condition. The resistance of GPM specimens against sulfate and acid attack was evaluated in terms of change in compressive strength after 26 weeks of immersion in different sulfate solutions (3% Na_2SO_4, 6% Na_2SO_4, 3% $MgSO_4$, 6% $MgSO_4$) and acid solutions (0.31 mol/l H_2SO_4, 0.62 mol/l H_2SO_4, 0.31 mol/l HCl, and 0.62 mol/l HCl). In 2nd phase of GPM series, another set of mix proportion of GPM was designed by varying two most influential control parameters i.e., GGBS replacement level and S/B ratio in the optimized GPM mix derived from the 1st phase of GPM series. Subsequently, all the experiments (except setting time), which were performed in the 1st phase of GPM series were carried out again on the mixes in 2nd phase of GPM series. It may be noted that in 2nd phase of GPM series, the resistance of GPM specimens against sulfate and acid attack was evaluated in terms of visual observation, change in weight, and change in compressive strength up to 26 weeks of immersion in different sulfate and acid solutions. In both phases of GPM series, the microstructural evolution of GPM mixes during strength development at different ages of ambient curing and after exposure against different sulfate and acid solutions were evaluated through XRD, EDS, FESEM, and FTIR spectroscopy analyses.

In 1st phase of geopolymer concrete (GPC) series, Taguchi-GRA method was used to investigate and optimize the effect of different control parameters on fresh (setting time, and consistency), and mechanical (compressive strength) properties of GPC mixes simultaneously. The considered control parameters along with their individual levels were GGBS replacement level: 15%, 30%, 45%, and 60%, W/GPS ratio: 0.28, 0.29, 0.30, and 0.31, molarity of NaOH solution: 8 M, 10 M, 12 M, and 14 M, binder content: 375 kg/m^3, 400 kg/m^3, 425 kg/m^3, and 450 kg/m^3, and SS/SH ratio: 1.5, 1.75, 2.0, and 2.25. Afterward, the verification experiments were carried out on the optimized GPC mix. In 2nd phase of GPC series, the effect of chloride ions on consistency, compressive strength, corrosion behaviour of steel reinforcement, and chloride content of selected fly ash-GGBS based GPC mixes, and their corresponding fly ash based GPC mixes were examined. To investigate the effect of chloride ions, NaCl of different concentrations i.e., 1.5% and 3.5% by weight of geopolymer solids were added during the preparation of GPC mixes. The GPC cube specimens of size 150 mm were prepared for compressive strength test in both phases of GPC series. Further, cylindrical reinforced GPC specimens of size 72 mm diameter and 200 mm height with a centrally embedded steel bar were prepared for electrochemical measurements (corrosion potential, and corrosion current density by linear polarization resistance measurement) in the 2nd phase of GPC series. All fly ash-GGBS based GPC specimens were prepared under ambient condition whereas the fly ash based GPC specimens, after preparation, were subjected to 48 hours of rest period followed by oven curing at temperature of 80° C for 48 hours. The microstructural changes (through XRD, EDS, FESEM, and FTIR analyses) in GPC mixes were evaluated and correlated with strength development as well as rebar corrosion in geopolymer concrete mixes.

From the obtained results, it is observed that GGBS replacement level and S/B ratio significantly influenced most of the studied properties of geopolymer mortar (GPM). The GPM prepared with higher GGBS replacement showed higher compressive strength under ambient condition, whereas the GPM prepared with lower GGBS replacement exhibited improved resistance against sulfate and acid attack. The results of multi-response optimization by Taguchi-GRA method indicated that the GPM mix made with GGBS replacement of 45%, molarity of NaOH solution of 14 M, S/B ratio of 2, and W/GPS ratio of 0.31 showed better performance in relatively more number of properties simultaneously. The variations in peak intensity of the compounds related to N-A-S-H and N-(C)-A-S-H gels formed in this GPM mix were consistent with the variations in compressive strength with ambient curing age as well as with variations in compressive strength of GPM mix in case of exposure to different sulfate solutions. Furthermore, significant decrease in peak intensity of compounds related to aluminosilicate gels, and in atomic Na/Si ratio and Al/Si ratio are consistent with the significant reduction in compressive strength of GPM in case of exposure to acid solutions, which corroborates the depolymerization of aluminosilicate gels in acid exposure condition. From the results of Taguchi-GRA method, the mix parameters along with their individual levels of the optimized GPM mix based on maximum mean grey relational grade (GRG) were 45% GGBS, W/GPS ratio of 0.31, NaOH solution of 14 M and S/B ratio of 1.5. From the result of verification experiments, the optimized GPM mix showed adequate setting time, flow index, and water absorption properties, and higher compressive strength at all ages of ambient curing, and for exposure to different sulfate and acid solutions when compared with other GPM mixes. The flowability of fresh GPM mixes decreased with increase in GGBS content from 15% to 45% in the mixes, whereas it increased with increase in sand-to-binder ratio from 1.5 to 2.5. The compressive strength of GPM mixes made from various fly ash/GGBS blends and S/B ratios mostly increased from 7 days to 180 days, which was supported by the formation of higher amount of N-A-S-H, N-(C)-A-S-H and C-S-H gel in the GPM mixes at later ages of ambient curing as indicated by the results of XRD analysis. The weight gain percentage of GPM mixes exposed to sulfate solutions mostly decreased with increase in GGBS content, whereas the weight loss percentage of the mixes exposed to acid solutions increased with increase in GGBS content and S/B ratio. The GPM mix made with lower GGBS content, i.e., 15% showed maximum gain and minimum loss in compressive strength in case of exposure to sulfate and acid solutions respectively. These variations in compressive strength are consistent with the variations in peak intensity of aluminosilicate gels from XRD analysis, and atomic ratios obtained from EDS analysis of GPM mixes. The GPM mixes made with higher S/B ratio showed improved performance in offsetting the loss in compressive strength when exposed to sulfate and acid solutions. There was more formation of gypsum in geopolymer mortar mix made with higher GGBS content in case of exposure to sulfate and H_2SO_4 solutions as observed from XRD, EDS, and FESEM analyses. In case of exposure to acid solutions, the GPM made with lower GGBS content showed improved stability of geopolymer gels as indicated by the variations in atomic Na/Si, Al/Si, and Ca/Si ratios obtained from EDS analysis.

From the obtained results of geopolymer concrete (GPC) mixes, it is observed that the GGBS replacement had dominant effect on setting time and compressive strength whereas W/GPS ratio significantly influenced the consistency of GPC mixes. The formation of N-(C)-A-S-H gel along with C-S-H gel in GPC mixes as indicated by the XRD patterns contributed to the development of compressive strength of GPC mixes. Based on the results of multi-response optimization by Taguchi-GRA method, the optimized level of control parameters of GPC mix were GGBS replacement of 45%, W/GPS ratio of 0.31, NaOH solution of 14 M, binder content of 450 kg/m^3 and SS/SH ratio of 1.5. The obtained results of verification experiments on the proposed optimized GPC mix confirmed the effectiveness

of Taguchi-GRA approach for determining the optimal combination of mix parameters for the production of geopolymer concrete. The presence of NaCl improved the consistency of fly ash and fly ash-GGBS based GPC mixes. The fly ash and fly ash-GGBS based GPC mixes exhibited lower compressive strength with increase in concentration of NaCl, at all ages. However, the reduction in compressive strength of NaCl admixed GPC mixes decreased with increase in age, which indicates dominant effect of NaCl in hindering the geopolymerization reaction at early age. The fly ash-GGBS based GPC specimens mostly showed less negative corrosion potential (E_{corr}) and lower corrosion current density (I_{corr}) as compared to their corresponding fly ash based GPC specimens. In chloride admixed fly ash-GGBS based GPC mixes, the embedded steel bar mostly exhibited less negative E_{corr} and lower I_{corr} with increase in GGBS content as a result of availability of lower amount of chloride ions in the electrolytic pore solution of concrete surrounding the embedded rebar due to formation of denser microstructure in GPC mix made with higher GGBS content. The results of chloride analysis of GPC mixes at different ages and at rebar level of GPC specimens indicated very less extent of chloride binding in GPC mixes. The fly ash-GGBS based GPC mixes mostly showed higher chloride binding capacity as compared to their corresponding fly ash based GPC mixes at the age of 28 and 360 days and at rebar level of GPC specimens at the age of 600 days as a result of more physical adsorption of chloride ions on calcium bearing aluminosilicate gel (N-(C)-A-S-H) and C-S-H gel. The higher peak intensity of aluminosilicate gels and presence of C-S-H gel observed from XRD analysis, higher atomic Ca/Si ratio obtained from EDS analysis, comparatively denser microstructure observed from FESEM analysis, and shifting of wavenumbers of Si-O-Si(Al) bond toward lower magnitude in FTIR analysis corroborated the improved performance of fly ash-GGBS based geopolymer concrete in terms of higher strength, lower corrosion activity and lower chloride content when compared with fly ash based geopolymer concrete.