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EFFECT OF MIXING PROPORTION ON COMPRESSIVE STRENGTH OF FLY-ASH BASED GEOPOLYMER PASTE AND MORTAR USING TAGUCHI'S METHOD

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Alkali activated pozzolan are known low carbon cementitious binders which can be used to replace cement. The material is also known as geopolymer because of its three dimensional polymeric chain and ring like structure consisting silica and alumina. A common type of pozzolan used is fly ash because of its rich silica content; therefore the term alkali activated fly-ash based binders is adopted. Despite much research and development of this material, there is no specific standard for design mix proportion. This research used the Taguchi's design of experiment method to determine the optimum mix proportion of alkali activated fly ash based cement paste and mortar. Four factors were considered in the tests, silica fume, sand to cementitious ratio, liquid to solid ratio, and percentage of superplasticiser. Tests were conducted on the 9 batches of alkali activated fly-ash based paste and mortar samples to determine the compressive strength under ambient condition. Tests were also conducted to determine the residual strength of the samples after exposed to elevated temperatures. ANOVA analysis of the test results revealed the main factors contribution on the tested properties and led to the determination of the optimum design proportion of the factors considered in these tests.

Keywords: Binders, Cement, Alkaline solution, Residual strength, Taguchi's method.

1 INTRODUCTION

Ordinary Portland Cement (OPC) gives sufficient fire safety for most ordinary applications. However, the quality of OPC cement diminishes at elevated temperatures because of chemical and physical changes (Crozier and Sanjayan 1999). Further, spalling of traditional solid happens in fire which causes a quick layer-by-layer loss of cement cover, conceivably prompting the presentation of the primary fortifications within the concrete to fire. Efforts have, in this manner, been made to identify alternative fastener which has great fire safety at elevated temperature and spalling resistance.

Fly ash is a main source material of alkali-activated cement, thus the term alkali-activated fly ash-based cement. Geopolymer is a term used to portray inorganic polymers focused around aluminosilicates and can be delivered by blending Pozzolanic mixes or alumino silicates source materials with alkaline solution. Due to their ceramic nature, Geopolymer are accepted to have great fire safety (Cheng and Chiu 2003). In this study Class F fly ash is used, the energy consumption is calculated to be approximately 60% less than that required by OPC (Li, Ding and Zhang 2004).

The main objective of this study is to determine the compressive strength of Alkali activated fly ash based (geopolymer) paste and mortar after exposed to high heat up to 800^{o}C. An experimental program was designed based on Taguchi's method and the experimental results were analyzed using signal-to-noise (S/N) ratio concept. Further, ANOVA was carried out to determine the contribution of each test factor in the compressive strength of the tested geopolymer samples.

2 EXPERIMENTAL PROCEDURES

Class F fly ash was used to prepare alkali activated fly ash based geopolymer paste and mortar. Other materials used were silica fume, fine aggregate (sand), super plasticizer and alkaline solution. Aggregates were made to achieve saturated surface dry (SSD) condition before mixing them together with other materials in the mix.

Alkaline solution is the mixture of sodium hydroxide and sodium silicate. Grade D sodium silicate solution (Na2SiO3) having a specific gravity of 1.53 and a modulus ratio (Ms) equal to 2 (where Ms = SiO_2/ Na_2O, Na_2O = 14.7% and SiO_2 = 29.4%) was used. Ten molar of sodiumhydroxide with flakes of 100% purity and distilled water was prepared. The ratio of sodium silicate to sodium hydroxide was kept 2.5. Sodium silicate and sodium hydroxide were mixed 24 hours prior to the mixing of all the materials in the mixer. First of all fly ash was mixed with the alkaline solution in the mixer for three minutes, after three minutes mixer was stopped and sand, silica fume and super plasticizer were added one by one when the mixer running and mixing these materials. When all the materials were added the mixer was stopped and any fly-ash that was sticking to the sides of the pan was scraped with a plastic scraper. After this, the mix was mixed for another 3 minutes and was ready to be poured in the 50 mm cubic molds. During curing, cubic molds were wrapped with a plastic sheet and placed in a humidity chamber for 24 hours, with a 60˚C and 70% humidity condition, in order to increase the polymerization process. After 24 hours samples were removed from the cubic molds and were placed in the ambient curing conditions for further curing.

Samples upon curing in ambient condition for 28 days were placed in oven to be heated at elevated temperatures of 200˚C, 400˚C 600˚C and 800˚C. Samples were heated for 2 hours before they were allowed to cool naturally to room temperature inside the furnace. Upon cooling, compressive strength test was carried out on each sample and results were recorded.

3 TEST PARAMETERS AND ANALYSIS

The target is to choose the best proportions of control parameters so that the procedure is most vigorous with deference to noise factors (Unal and Dean 1991). The Taguchi system uses orthogonal array from the designed experimental hypothesis to study a substantial number of variables with a little number of analyses. Utilizing orthogonal array significantly decreases the quantity of experimental designs to be concentrated on. Besides, the conclusions drawn from little scale investigations are legitimate over the whole test area crossed by the control elements or factors and their settings. In this project following factors are used in the mix proportions.

- Alkaline solution to solid materials ratio (Liquid/Solid)
- Sand to binder ratio (sand/binder)

- Silica fume percentage (SF %)
- Superplasticizer percentage (SP %)

The four factors used and their variation levels and the 9 mix proportions according to the L9 orthogonal array are as shown in Table 1. The numbers in the mix proportions table are the ratio to a kilogram unit of fly ash.

Table 1. Factors and mix proportions considered.

Levels	**Liquid/Solid ratio**	**Sand/Binder ratio**	**Superplasticiser %**	**Silica fume %**
1	0.6	0	0	0
2	0.65	0.25	2	2
3	0.7	0.5	4	4

Mix	**Sand**	**Liquid**	**Superplasticiser**	**Silica Fume**
1	0	0.6	0	0
2	0.25	0.65	0.02	0
3	0.5	0.7	0.04	0
4	0	0.6	0.04	0.02
5	0.25	0.65	0	0.02
6	0.5	0.7	0.02	0.02
7	0	0.6	0.02	0.04
8	0.25	0.65	0.04	0.04
9	0.5	0.7	0	0.04

In total almost 400 samples were tested, four samples from each mix, and two batches were prepared for each mix for the accuracy. Compressive strength tests were conducted on the samples at ambient and after exposed to 200^{o}C, 400^{o}C, 600^{o}C and 800^{o}C. The results were taken from the average values of the tested results at each temperature range from the two batches.

4 RESULTS AND DISCUSSION

The compressive strengths of geopolymer paste and mortar from the 9 suggested experimental mixes upon 28 days of curing were tested. The results are as presented in Figure 1. The highest compressive strength achieved is related to Mix 5 (TM5) at ambient, and Mix 9(TM9) at elevated temperature of 800˚C. It can be seen that the strength decreased after heating the samples up to 200˚C and decreased further after exposed to 400˚C. This decrease in the compressive strength could be due to the thermal incompatibilities between the aggregates and the alkali activated geopolymer binder in the samples. Aggregates inside the geopolymer matrix have a higher rate of expansion at elevated temperature which creates stresses.

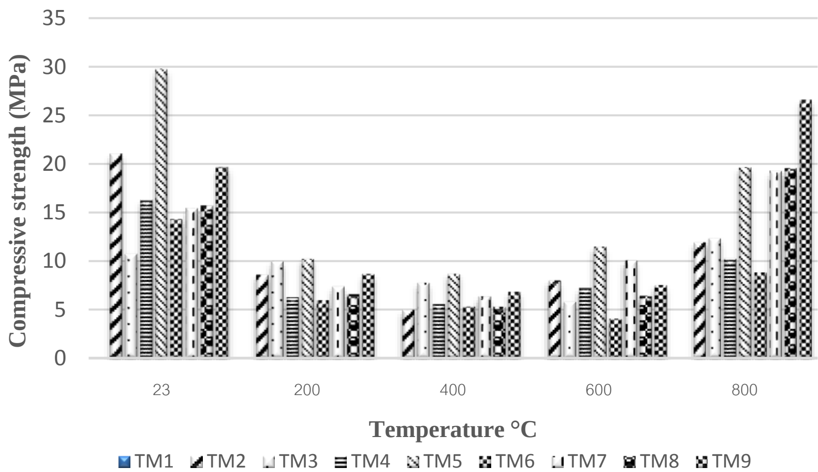

Figure 1. Compressive strength results.

These stresses create tension in the mixtures matrix, it is a known fact that geopolymer is weaker in tension and thus leads to the compressive strength reduction and formation of micro cracks. These micro cracks were also noticed by (Pan, Sanjayan and Rangan 2009) but they did not notice a significant decrease in the strength.

After exposed to 600˚C, a slight increase in strength was noticed in some mixes, which is contrary to the loss in strength after exposed to 200˚C. This increase in strength could be attributed to the general hardening of the geopolymer gel, mainly due to the evaporation of the moisture content with in the samples. After exposed to 800˚C, a further increase in the compressive strength was significant. This increase in strength was possibly due to the sintering of the geopolymer, which happens around the 700˚C (Pan and Sanjayan 2010). Sintering is the nuclear dissemination of molecules at below liquefying temperatures making the alkali activated Geopolymer interlocked.

Further analysis of the compressive strength results of the samples exposed to 800˚C was carried out. Signal to noise ratio of the results helped indicate the most appropriate mixing proportion for the four factors that were used to achieve the best compressive strength at elevated temperature of 800˚C. Table 2shows the appropriate mixing proportion of the four factors that were used in the experimental program with the highest achievable compressive strength of 60 MPa under the specified mixing proportions.

ANOVA was used to determine the contribution of each factor on the compressive strength. Figure 2 shows the ANOVA results of the compressive strength after exposed to 800^{o}C. Liquid to solid ratio, sand to binder ratio and silica fume have almost equal contribution to the strength, while superplasticiser has the least contribution.

Table 2. Best response prediction.

	Liquid/Solid ratio	Sand/Binder ratio	Superplasticiser %	Silica fume %
Level	2	3	2	3
S/N		36		
Log ($1/Y_i^2$)		-4		
$1/Y_i^2$		0		
Y_i		60 MPa		
Best response for Means	0.65	0.5	2	4

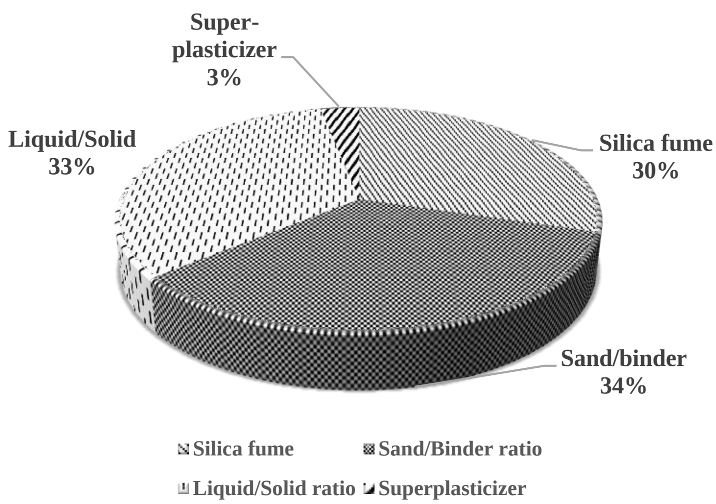

Figure 2. ANOVA of 28-day compressive strength results at 800°C.

5 CONCLUSIONS

The tested geopolymer paste and mortar had a slight loss in compressive strength after exposed to 400°C compared to the ambient strength. There was an increase in strength after exposed to 600°C, which could be caused by the general stiffening of the Geopolymer gel as a result of the increase in the surface forces between gel particles due to the removal of absorbed moisture. Further gain in strength was achieved after exposed to 800°C.

Based on the four factors and three variation levels considered, signal to noise ratio was used to determine the best possible mix to obtain the highest compressive strength under ambient and elevated temperature conditions. ANOVA was used to determine the contribution of each factor on the compressive strength. It should be noted that the number of samples tested in this research was quite limited. More tests are required to create a larger database for the statistical analysis. In addition, geopolymer properties are highly dependent of the raw materials and curing condition, a more controlled testing condition will be necessary to ensure the consistency and reliability of the results.

Geopolymer concrete potentially has superior strength at elevated temperatures and can be further developed for application in high fire risk areas, as permanent structures or fire protection materials.

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