Sixth International Conference on the Durability of Concrete Structures **Paper Number 1256**
18-20 July 2018
University of Leeds, Leeds, West Yorkshire, LS2 9JT, United Kingdom

Effect of Mix Parameters on Strength of Geopolymer Mortars - Experimental Study

A. Naghizadeh, S.O. Ekolu
Department of Civil Engineering Science, University of the Johannesburg, Auckland Park Kingsway Campus, 2006, Johannesburg, South Africa. Corresponding author: honair@yahoo.com

ABSTRACT

In this article, an investigation is reported on development of strength in South African fly ash (FA) – based geopolymer mixtures. Locally available Class F, FA from one of the coal power stations was used in the investigation. The alkali-activator used consisted of sodium silicate (SS) and sodium hydroxide (SH) mixed in varied ratios of 1.0, 1.5, 2.0, 2.5 and 3.0 SS to SH. The SS of silicate modulus = 2.5 was used but the SH concentration in the activator was varied to 10, 12, 14M NaOH. Mortars of 2.25 aggregate/binder ratio were used to prepare 50 mm cubes. In preparing mortar mixtures, the liquid to solids (L/S) ratios were varied to L/S = 0.3, 0.4, 0.5 and 0.6. Mortar cubes were cast and cured at 80^{o}C for 7 days then tested for compressive strength. It was found that all three parameters consisting of SS/SH ratio of the activator, concentration of NaOH used in the activator and the L/S ratio, showed significant influence upon compressive strength development. The optimum strength of the geopolymer mortar mixtures was obtained at SS/SH = 2.0, 12M NaOH concentration and L/S = 0.5.

Keywords: Fly ash, geopolymer, compressive strength, alkali-activator, sodium silicate

INTRODUCTION

Geopolymers Cements (GPC) are produced by activating alumino-silica materials such as FA, Metakaolin (MK) and Volcanic Ash (VA), using alkaline compounds typically hydroxides, silicates (Skvara et al., 2007; Ekolu et al., 2006; Tchadjie and Ekolu, 2018.). Several studies demonstrated that the performance of GPCs may be equivalent or even superior to that of Ordinary Portland Cement (OPC) (Kupwade-Patil and Allouche, 2011; Ridtirud et al., 2011; McKenzie, 2014; Attwell, 2014; Tho-In et al., 2012). Durability problems such as delayed ettringite formation, alkali-silica reaction, chloride attack etc. which are endemic in OPC concretes (Ekolu, 2004) are anticipated to be diminished or non-existent in GPC concretes. Moreover, GPCs are known to be environmentally friendly binders due to their low energy consumption and lower CO_2 emissions compared to OPC. These advantages have made GPCs more desirable in recent years. FA is one of the most adequate aluminosilicate raw materials for use in geopolymerization. A number of experimental studies have been conducted on mechanical properties and durability of FA-based GPCs. Arioz et al. (2013) studied the effect of curing condition on mechanical properties of FA-based GPC. Their results showed that curing conditions significantly influenced the physical properties of geopolymer samples. Compressive strength increased, when the curing duration increased from six to 24 hours. Another study (Vora and Dave, 2013) also showed similar results, when the curing duration was increased. In a study by Ahmari et al. (2012), the effect of curing temperature on compressive strength of copper mine tailings-based geopolymer was investigated. The results showed that compressive strength increased with increase in curing temperature from 60 to 90°C. However, there was strength reduction on curing beyond 90°C. Moreover, it was noted that increasing the curing temperature had inflective effect, when higher NaOH concentration was used in activator. The drying shrinkage of GPC concretes is less than that of OPC concretes (Attwell, 2014). Findings of a study by Yusuf et al. (2014) reported the ratio SH/SS to significantly affect shrinkage of palm oil ash-based GPC paste and mortars. Shrinkage was found to decrease with increase in SH/SS.

The type of alkali-activator is also one of the influential factors affecting mechanical properties of

GPCs. NaOH and Na_2SiO_3 are among the most commonly used alkaline activators in GPC. According to a study by Vora and Dave (2013), the compressive strength of geopolymer paste increased when NaOH concentration was raised from 8M to 14M. In another study by Kupaei et al. (2013), a similar trend was also observed. However, when the NaOH concentration increased above 14M, strength decreased. The results of another study by Tho-in (2012) showed that the GPCs made with activator 15M NaOH achieved the highest compressive strength. Torres-Carrasco and Puertas (2014) investigated the effect of activator type on compressive strength of FA-based GPC. Findings showed that the mixes made with activator containing 10M NaOH and waste glass gave the highest compressive strength, followed by mixes made with 10M NaOH and Na_2SiO_3. Ahmari et al. (2012) recommended using activator of Silicon Oxide (SiO_2) to Sodium Oxide (Na_2O) ratio of 1 to 1.25. Clearly, further studies need to be done to obtain comprehensive understanding on mixture proportioning for GPCs.

EXPERIMENTAL

Materials and mixtures used in the investigation are also described in (Naghizadeh and Ekolu, 2017) but relevant details are repeated here for convenience towards interpretation of results. The geopolymer used was Class F, fly ash (FA) obtained from Lethabo coal-powered electricity generating station belonging to ESKOM (pty) Ltd. This FA is widely used in South Africa as supplementary cementitious material or artificial pozzolan for blending with ordinary Portland cement. Its chemical composition is given in (Naghizadeh and Ekolu, 2017), indicating low CaO content, characteristic of Class F category. The alkali-activator employed in the study consisted of $NaSiO_3$ and NaOH mixtures. Both chemicals were supplied by Merck (pty) Ltd. The sodium silicate (SS) had silicate modulus of 3.2, 27% SiO_2 and 8.3% Na_2O while, the sodium hydroxide (SH) was of technical grade with 99.5% purity. To prepare the activator, the two chemicals were combined in varied ratios of 1.0, 1.5, 2.0, 2.5 and 3.0, SS to SH. For any given geopolymer mixture, the activator was prepared two hours prior to use. The greywacke fine aggregate obtained from the Cape Penisula was used in all mortar mixtures.

Mortars were prepared at aggregate /binder ratio of 2.25. Table 1 gives the mix proportions used to make the mortar mixtures for strength testing. Solid ingredients comprising aggregates and FA were measured in appropriate quantities then placed in a mortar mixer. The dry materials were mixed for one minute at low speed then the activator was added, and mixing continued for additional two minutes. After completion of mixing, the fresh GPC mortars were cast into 50 mm steel cube moulds and then sealed with plastic film, before placing in an 80°C oven, where the samples were stored for 7 days. At end of curing, the cubes were demoulded and tested for compressive strength.

Table 1. Mortar mix proportions

Mix No	Concentration of NaOH (M)	SS/SH	L/S
1	10	1	0.5
2	10	1.5	0.5
3	10	2	0.5
4	10	2.5	0.5
5	10	3	0.5
6	12	1	0.5
7	12	1.5	0.5
8	12	2	0.5
9	12	2.5	0.5
10	12	3	0.5
11	14	1	0.5
12	14	1.5	0.5
13	14	2	0.5
14	14	2.5	0.5
15	14	3	0.5
16	10	2	0.4
17	12	1.5	0.4
18	12	2	0.4
19	12	2.5	0.4
20	12	3	0.4
21	14	2	0.4
22	10	2	0.3
23	12	1.5	0.3
24	12	2	0.3
25	12	2.5	0.3
26	12	3	0.3
27	14	2	0.3
28	12	1.5	0.6
29	12	2	0.6
30	12	2.5	0.6
31	12	3	0.6

RESULTS AND DISCUSSION

Compressive strength test was carried out to evaluate strength development in mortar specimens. The samples were tested at the age of 7 days. Three specimens were tested for each mix. Fig. 1 shows the measured compressive strength of mortars, for varied SS/SH ratios and constant L/S ratio of 0.5. Overall, the results show that compressive strength increased with increase in the ratio of SS/SH from 1 to 1.5, followed by non-linear decrease in strength, as the SS/SH ratio increased further from 1.5 to 3.0. Regardless of the concentration of NaOH, the mixes with SS/SH ratio of 1.5 achieved the highest compressive strengths, which agrees with findings of another study by Ridrirud et al. (2011). However, it should be noted that the ratio of SS/SH, which provides the highest compressive strength depends on the properties of the activators and chemical composition of raw material in terms of the amount of SiO_2, Na_2O, and Al_2O_3 (Temuujin et al., 2009).

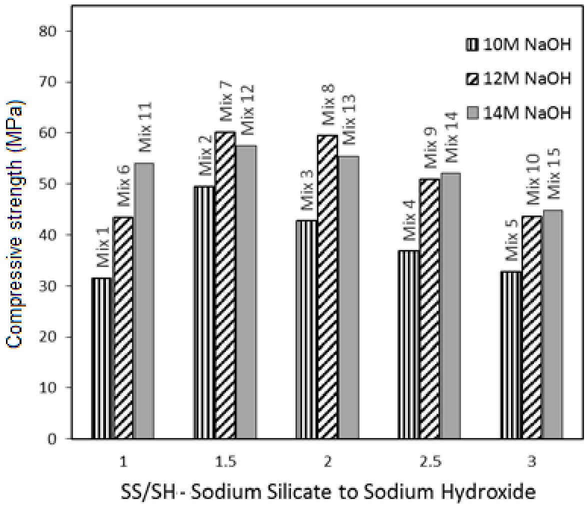

Fig. 1. Effect of SS/SH ratio on compressive strength of FA-based GPC mortars made at L/S ratio of 0.5: SS-Sodium Silicate, SH-Sodium Hydroxide, L/S-Liquid to Solid ratio

Fig. 2 shows the compressive strength of fly ash-based GPC mortars made at various L/S ratios. A constant concentration of 12M NaOH was used in the activator. It was observed that the mixes with L/S ratios of 0.5 gave the highest compressive strength. For the mixes with constant SS/SH ratio of 1.5, the compressive strength increased from 46 to 60 MPa, when L/S was raised from 0.3 to 0.5. However, it decreased to 33 MPa, when L/S ratio increased further to 0.6. This strength reduction may be attributed to excessive amount of H_2O in the GPC structure, which leads to higher permeability at higher L/S ratio. A similar trend was observed in the other mixes of SS/SH ratios 2.0, 2.5, and 3.0. Fig. 3 shows compressive strength results for GPC mortars made with activators containing various concentrations of NaOH and constant SS/SH ratio of 2.0. It can be seen that the compressive strength of GPC mortars of L/S ratio 0.3, increased from 33 to 47 MPa, when the concentration of NaOH increased from 10M to 14M. This gain in strength at higher NaOH concentration is related to greater geopolymerization achieved at higher pH value and high alkali content, which is required by these systems (Torres-Carrasco and Puertas, 2014). However, in mixes with L/S ratios of 0.4 and 0.5 strength decreased, when the NaOH concentration increased above 12M.

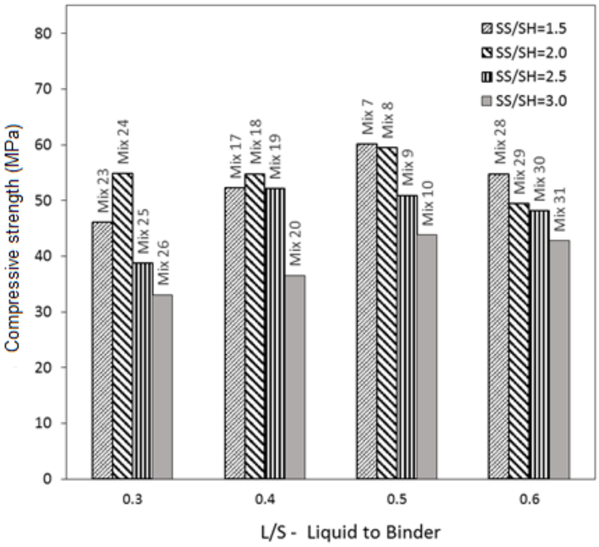

Fig. 2. Effect of L/S ratio on compressive strength of FA-based GPC mortars made with activator containing 12M NaOH: L/S-Liquid to Solid ratio.

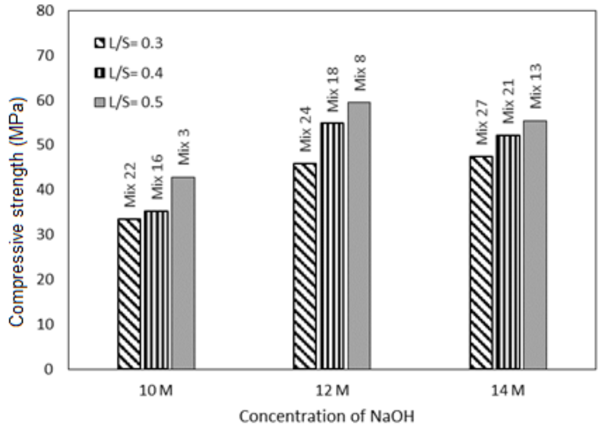

Fig. 3. Effect of NaOH concentration used in activator upon compressive strength of FA-based GPC mortars for SS/SH ratio of 2.0: SS- Sodium Silicate, SH- Sodium Hydroxide.

Similar results have been obtained in other studies (Skvara, et al. 2007, El-Dieb and Shehab, 2014, Khale and Chaudhary, 2007). These observations may be attributed to excess OH^- concentration, causing alumino-silicate gel precipitation at very early stage, in turn leading to lower strength (El-Dieb and Shehab, 2014). Data giving the highest strength from Figs.1 to 3 has been plotted as graphs shown in Fig.4. Evidently, each of the parameters gave peak strength within a specific values of SS/SH = 1.5, L/S = 0.5 and 12M NaOH.

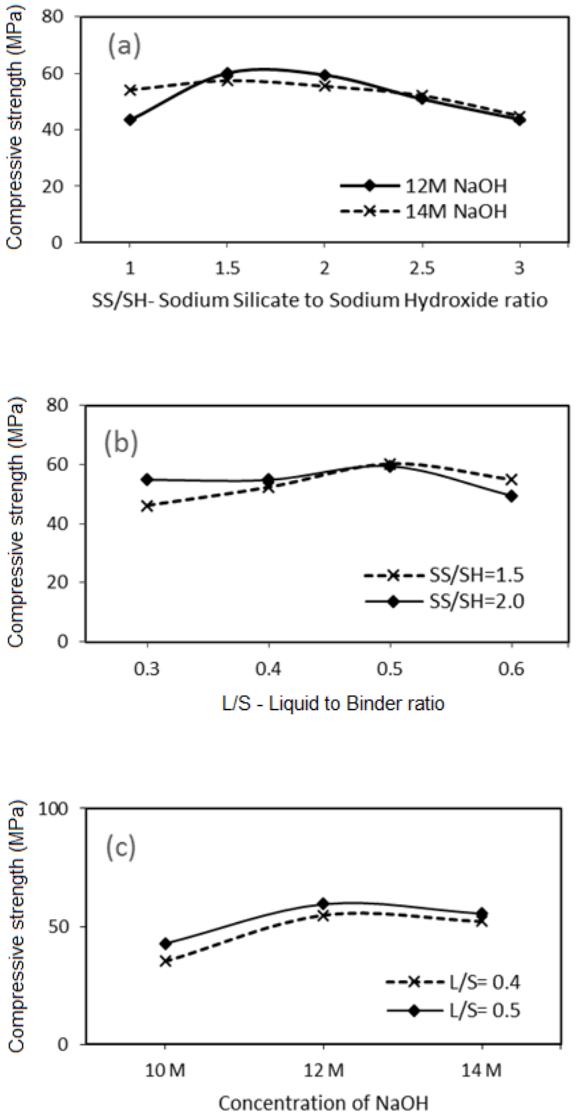

Fig. 4. Peak strength values at specific ranges of mix parameters.

CONCLUSION

The development of strength in Class F fly ash – based geopolymer mortars was investigated as described in the article. In the experiment, the sodium silicate (SS) to sodium hydroxide (SH) ratio, concentration of NaOH used in activator and the Liquid to Solids ratio (L/S) were varied. It was found that these parameters showed major influence on compressive strength of the geopolymer mortars. Optimum compressive strength was attained at SS/SH = 1.5, L/S = 0.5 and use of 12M NaOH in activator. Higher or lower values of these parameters, gave relatively lower strengths.

Acknowledgements

This paper is based on the PhD study of A. Naghizadeh, conducted through financial support from the National Research Foundation (NRF). The authors wish to thank NRF for the study scholarship.

References

Ahmari, S., Zhang, L., Zhang, J., 2012. Effects of activator type/concentration and curing temperature on alkali-activated binder based on copper mine tailings. Journal of Materials Science, 47:5933–5945.

Arioz, E., Arioz, Ö., M. Koçkar, Parksan, Ö., 2013. The effect of curing conditions on the properties of geopolymer samples. Int. Journal of Chemical Engineering and Applications, 4:423-426.

Attwell, C., 2014. Geopolymer concrete: a practical approach. Int. Conf. Construction Materials and Structures, Johannesburg, 466-466.

Ekolu S.O., 2004. Role of heat curing in concrete durability: effects of lithium salts and chloride ingress on delayed ettringite formation. PhD Thesis, Deparment of Civil Engineering, University of Toronto, Canada, 217p.

Ekolu S.O, Thomas M.D.A, Hooton R.D, 2006. Studies on Ugandan volcanic ash and tuff. Proc. 1st Int. Conf. on Advances in Engineering and Technology, Entebbe, Uganda, July 2006, 75-83.

El-Dieb, A.S. and Shehab, E.E., 2014. Cementless concrete using ceramic waste Powder. Int. Conf. Construction Materials and Structures, Johannesburg, 487-494.

Khale, D., Chaudhary, R., 2007. Mechanism of geopolymerization and factors influencing its development: a review, J. Mater. Sci. 42:729–746.

Kupaei, H.R., Alengaram J.U., Bin Jumaat, M.Z., Nikraz, H., 2013. Mix design for fly ash based oil palm shell geopolymer lightweight concrete. Construction and Building Materials, 43;490-496.

Kupwade-Patil, K. and Allouche, E., 2011. Effect of Alkali Silica Reaction (ASR) in geopolymer concrete. World of coal ash (WOCA) Conf, Denver, Co, USA. http://www.flyash.info/

McKenzie, W., 2014. Use of self-compacting geopolymer concrete in a precast environment - a case study. Int. Conf. Construction Materials and Structures, Johannesburg, 518-525.

Naghizadeh, A., and Ekolu, S., 2017. Investigation of mixture factors influencing alkali-silica reaction in fly ash-based geopolymer mortars. Intern. Conf.

Advances in Construction Materials and Systems, 3:395-400.

Ridtirud, C., Chindaprasirt, P., Pimraksa, K., 2011. Factors affecting the shrinkage of fly ash geopolymers. Int. Journal of Minerals, Metallurgy and Materials, 18:100-104.

Skvara, F., Dolezal, J., Svoboda, P., Kopecky, L., Pawlasova, S., Lucuk, M., Dvoracek, K., Beksa, M., Myskova, L., Sulc, R., 2007. Concrete based on fly ash geopolymer. Research project. www.geopolymery.eu,185-197.

Tchadjie, L.N., Ekolu, S.O., 2018. Enhancing the reactivity of aluminosilicate materials toward geopolymer synthesis. Journal of Material Science 53:4709-4733

Temuujin, J., Williams, R.P., Van Riessen A., 2009. Effect of mechanical activation of fly ash on the properties of geopolymer cured at ambient temperature. Journal of Materials Processing Technology, 209:5276-5280.

Tho-In, T., Sata, V., Chindaprasirt, P., Jaturapitakkul, C., 2012. Pervious high-calcium fly ash geopolymer concrete. Construction and Building Materials, 30:366–371.

Torres-Carrasco, M., Puertas, F., 2014. Waste glass in the geopolymer preparation, mechanical and microstructural characterisation. Journal of Cleaner Production, 1-12.

Vora, P.R., Dave, U.V., 2013. Parametric studies on compressive strength of geopolymer concrete. Procedia engineering, 51:210 – 219.

Yusuf, M., Johari, M.A.M., Ahmad, Z.A., Maslehuddin, M., 2014. Steel-slag and activators ratio impacts on the shrinkage of alkaline activated ultrafine palm oil ash-steel slag paste/mortar. Int. Conf. Construction Materials and Structures, Johannesburg, 511-517.