Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 125 (2015) 650 – 656

Procedia Engineering

www.elsevier.com/locate/procedia

The 5th International Conference of Euro Asia Civil Engineering Forum (EACEF-5)

The strength of alkali-activated slag/fly ash mortar blends at ambient temperature

Arie Wardhono[a,]*, David W. Law[b], Anthony Strano[b]

[a]*Universitas Negeri Surabaya, Kampus UNESA Ketintang, Surabaya 60231, Indonesia*
[b]*RMIT University, RMIT City Campus, Melbourne VIC 3000, Australia*

Abstract

The implementation of sustainable development in civil engineering society has led to the use of new materials with low environmental impact. Ordinary Portland cement (OPC) is the primary material in the production of traditional concrete. However, the manufacturing of OPC has led to environmental concerns over the production of CO_2. The use of fly ash and slag, the most commonly used industrial by-products, as replacements for PC, has helped to reduce these CO_2 emission. Recent research has also shown that it is possible to use fly ash or slag as a sole binder in concrete by activating them with alkali components through a polymerization process. However, the main issue of the use of fly ash as a replacement material for cement is the need of heat curing regime to achieve structural integrity. While, the standard curing regime used for OPC concrete can be applied to the alkali-activated slag (AAS) due to the similar characteristic of the hydration product.

This paper reports the detail of the experimental work that has been undertaken to investigate the strength of AAS/fly ash (AASF) mortar blends. The AASF specimens were prepared using a mix of ground granulated blast-furnace slag (GGBS) and low calcium class F fly ash activated by high alkaline solution. The mix compositions of slag to fly ash were 1:0, 0.9:0.1, 0.8:0.2, 0.7:0.3, 0.6:0.4 and 0.5:0.5, respectively. The standard curing regime at ambient temperature was applied.

The results showed that the mix proportion of 0.5 slag : 0.5 fly ash produced the best strength results. The standard deviation values also reduced along with the increase of fly ash content indicating an improved stability of the specimens. It also suggested that 0.5 slag : 0.5 fly ash blend could provide a solution for the need of heat curing for fly ash-based geopolymer.

Peer-review under responsibility of organizing committee of The 5th International Conference of Euro Asia Civil Engineering Forum (EACEF-5)

Keywords: Alkali activated material; GGBS; fly ash; strength development; ambient temperature

---

* Corresponding author. Tel.: +62-81333371173; fax: +62-31-8299342.
E-mail address: ariewardhono@unesa.ac.id

Peer-review under responsibility of organizing committee of The 5th International Conference of Euro Asia Civil Engineering Forum (EACEF-5)
doi:10.1016/j.proeng.2015.11.095

1. Introduction

Concrete is the most commonly used construction material in society, which is conventionally produced by using the ordinary Portland Cement (OPC). As primary binder of concrete, the ratio of OPC in traditional concrete is approximately 10% – 15% by the mass of concrete. However, the production of OPC has resulted in the environmental problems over the production of CO_2 with approximately 1 ton of CO_2 produced per 1 ton OPC [1, 2]. This has led to the adoption of industrial waste materials, such as fly ash and ground granulated blast furnace slag (GGBS), as replacement materials for OPC due to their ability to enhance the physical, chemical and mechanical properties of cements and concretes [3,4].

Recent research has shown that it is possible to use fly ash or slag as a sole binder in concrete by activating them with alkali components through a polymerization process [5-8]. The activation of fly ash involves the activation of material containing primarily silicate and aluminates with a highly alkaline solution and forms an inorganic binder through a geopolymeric reaction [9,10]. While the activation of slag involves the activation by low to mild alkali of a material containing primarily silicate and calcium and produces calcium silicate hydrate gel (C-S-H gel) similar to that formed in OPC [11]. The products of geopolymeric reaction are different from other type of alkali activated materials (slag reaction) since the products are a polymer rather than C-S-H gel.

As cement replacement materials, both fly ash and slag have been observed to have a comparable compressive strength. However, the main issue of the use of fly ash as cement replacement materials is the need of heat curing regime to achieve structural integrity. While, the standard curing regime used for OPC concrete can be applied to the alkali activated slag (AAS) due to the similar characteristic of the hydration product (C-S-H gel), fly ash requires heat curing. This paper reports the results of experimental research on the development of alkali activated slag and fly ash (AASF) blends mortars cured at ambient temperature. The properties of AASF mortars were assessed in the form of compressive strength.

2. Experimental procedures

2.1. Materials

The primary materials used for this study were a low calcium class F fly ash with high silicate (Si) content combined with ground granulated blast-furnace slag (GGBS), a construction grade slag type. The chemical composition breakdown of the fly ash and GGBS are shown in Table 1.

Table 1. Chemical compositions of fly ash and GGBS (mass %)

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	TiO_2	P_2O_5	Mn_2O_3	SO_3
GGBS*	36.87	14.23	0.32	36.0`	5.05	0.05	0.00	0.63	0.36	0.39	6.08
Fly ash	66.65	25.06	1.68	2.03	0.01	1.01	0.39	1.25	1.23	0.05	0.62

Note: * Wardhono, 2012 [12]

The fly ash used in this study was class F fly ash. It had a CaO of 2.03% (< 10%), SiO_2+Al_2O_3+Fe_2O_3 of 93.39% (>70%) and SO_3 composition of 0.62 as in accordance with ASTM C618 [13]. The GGBS used satisfies the requirement for an AAS. It had a CaO/SiO_2 ratio of 0.98 (between 0.5 and 2.0) and an Al_2O_3/SiO_2 ratio of 0.39 (between 0.1 and 0.6). The hydration modulus which was defined as the ratio of CaO+MgO+Al_2O_3 to SiO_2 was 1.5 (> 1.4) to ensure good hydration properties [8,14]. The fineness of the fly ash and GGBS materials were measured using a Malvern Particle Size Analyzer Instruments Mastersizer X and had a fineness of 67.22% and 90.90% (passing 45 μm), respectively. The fine aggregate used to make the specimens was blended concrete sand from Langwarrin, Victoria, Australia, with the fineness modulus of 2.0 and the coarse aggregates were from Mawson Lake Cooper quarry with the specific gravity of 2.9

The alkaline activator solutions were formulated by blending sodium silicate with sodium hydroxide (NaOH) to achieve a Na_2O dosage of 15% and activator modulus (Ms) of 1.25. A sodium silicate with alkali modulus (AM) of approximately 2.0 (Na_2O = 14.7% and SiO_2 = 29.4%) and a high concentration of sodium hydroxide, 15 M NaOH in liquid form were used.

2.2. Mix proportions

In accordance to ASTM C109, the sand to binder (a combination of slag and fly ash) ratio was 2.75 : 1 [15]. A water to binder ratio was 0.66 (the water from the alkali activator solutions was not included) was used to prepare the AASF mortars. The alkali activator present was 0.163 by the weight of mortar mix. The mix design of the AASF mortars was developed from the previous research [12]. The mix compositions of slag to fly ash were: 100% : 0%, 90% : 10%, 80% : 20%, 70% : 30%, 60% : 40% and 50% : 50%, respectively. Table 2 summarizes the mix design while Table 3 shows the summary of activator modulus of AASF mortar specimens.

Table 2. Mix design of AASF mortars

Mix	GGBS : FA Ratio	GGBS (kg)	Fly ash (kg)	Sodium silicate (kg)	NaOH (kg)	Water (kg)	Fine sand (kg)
M1	1.0 : 0.0	1.03	---	0.53	0.24	0.068	2.84
M2	0.9 : 0.1	0.93	0.10	0.53	0.24	0.068	2.84
M3	0.8 : 0.2	0.83	0.20	0.53	0.24	0.068	2.84
M4	0.7 : 0.3	0.72	0.31	0.53	0.24	0.068	2.84
M5	0.6 : 0.4	0.62	0.41	0.53	0.24	0.068	2.84
M6	0.5 : 0.5	0.52	0.51	0.53	0.24	0.068	2.84

Table 3. Activator modulus of AASF mortars

Mix	Na_2O from sodium silicate	Na_2O from NaOH	Total Na_2O content	SiO_2 from sodium silicate	SiO_2 from NaOH	SiO_2 from GGBS	Total SiO_2 content	Activator Modulus (Ms) SiO_2/Na_2O
M1	0.077	0.046	12.32	0.155	0.000	0.345	0.500	4.06
M2	0.077	0.046	12.32	0.155	0.051	0.310	0.516	4.19
M3	0.077	0.046	12.32	0.155	0.102	0.276	0.533	4.32
M4	0.077	0.046	12.32	0.155	0.153	0.241	0.549	4.46
M5	0.077	0.046	12.32	0.155	0.204	0.207	0.566	4.59
M6	0.077	0.046	12.32	0.155	0.255	0.172	0.582	4.72

2.3. Specimen preparation and testing

The mixing for AASF mortar specimens was performed using a 5 liter Hobart mixer. The mixtures were cast in 50 × 50 × 50 mm^3 cubes moulds and vibrated for 1 minute to ensure no air/voids were present in the specimens. The mortar specimens were cured at room temperature for one day (Note: no heat curing treatment was applied to the AASF specimens) prior to being demoulded. The mortar specimens were then kept in water at a temperature of 20 ± 2°C until time of testing.

It should be noted that due to the high viscosity and fast setting rate of AASF mortar specimens, the workability of the AASF paste was low and it was difficult to ensure the mortar specimens was compact properly thus, the specimens were vibrated to ensure good quality mortar specimens. The mixing procedure was in accordance to the previous research [16] with a total mixing time of 9 minutes, with the casting process taking approximately 10 minutes. Thus, the relative setting time of AASF specimens was approximately 15-18 minutes. However, it was difficult to differentiate the relative setting time and workability of the mixtures for different slag/fly ash ratios.

Compressive strength measurement of 50 × 50 × 50 mm^3 cube mortars were carried out on a Universal Testing Machine, UH-F500 kNI Shimadzu, under a load control regime with a loading rate of 1 kN/s, within 0.9 to 1.8 kN/s as outlined in ASTM C109 [15]. Three mortar cubes were tested for each data point. The mortar specimens were tested at 3, 7, 14, and 28 day after casting. The test was completed until failure of the mortar specimens.

3. Results and discussions

Table 4 and Figure 1 give the compressive strength results reported for the AASF mortar specimens for all mixes. The AASF mortars demonstrates a good performance of strength at early age with all mixes producing compressive strength above 20 MPa during the first three days after casting. All mixes demonstrate a continual improvement on strength throughout 28 days, with the exception of AASF Mix 1 which lost strength between 14 and 28 days.

Table 4. Compressive strength test results of AASF (MPa)

Mix	Age of concrete			
	3	7	14	28
M1	32.92 ± 6.98	42.81 ± 2.95	43.86 ± 4.85	39.55 ± 8.42
M2	29.41 ± 0.52	31.08 ± 2.81	40.99 ± 1.84	48.17 ± 8.08
`M3	23.69 ± 5.05	33.60 ± 4.97	31.15 ± 0.57	53.28 ± 6.11
M4	23.25 ± 3.35	40.64 ± 3.15	41.94 ± 2.80	47.32 ± 4.64
M5	31.04 ± 1.47	32.04 ± 4.40	34.92 ± 2.42	55.63 ± 1.71
M6	28.65 ± 2.41	46.84 ± 0.55	48.76 ± 2.69	62.49 ± 0.77

Table 5. Strength development of AASF mortar specimens up to 28 days (%)

Mix	Age of concrete			
	3 to 28 days	7 to 28 days	14 to 28 days	28 days
M1	83.24%	108.24%	110.90%	100%
M2	61.05%	64.52%	85.09%	100%
`M3	44.46%	63.06%	58.46%	100%
M4	49.13%	85.88%	88.63%	100%
M5	55.80%	57.59%	62.77%	100%
M6	45.85%	74.96%	78.03%	100%

The strength development of AASF Mix 1 demonstrates the highest initial strength with 32.92 MPa at 3 days age (83.24% final strength) and slightly increased from 7 to 14 days age of 42.81 MPa and 43.86 MPa, respectively. However, beyond that, the compressive strength of AASF Mix 1 does not display any significant increase with time, but shows a slight reduction in strength at 28 days to 39.55 MPa. A similar finding was also found by other researchers [17,18]. According to Collins and Sanjayan [17], the deteriorating behavior of a specimen made by 100% slag material can be attributed to the growth of micro-cracking within the specimens. They found that the micro-cracking became progressively larger over time. In addition, Wardhono et al [18] also found a decrease of AAS concrete strength which is attributed to the growth of the micro-cracks with time. The authors found that the decreasing strength was coupled with an increase in the permeable voids ratio, as demonstrated by an increase of porosity and water absorption and decrease in ultrasonic pulse velocity.

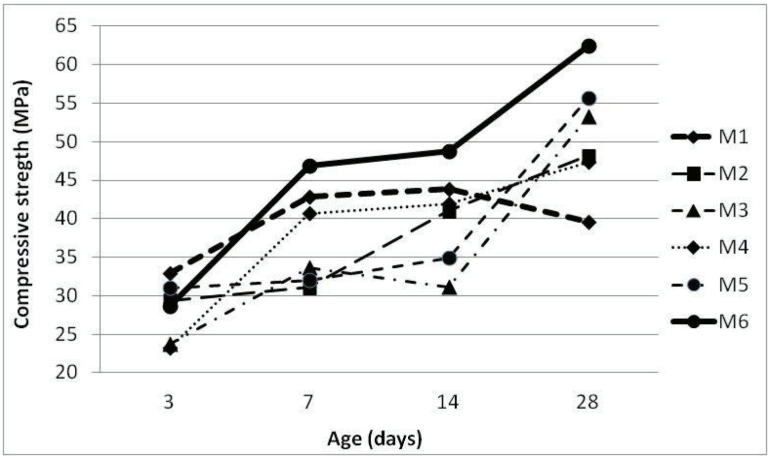

Fig. 1. Compressive strength of AASF mortars

AASF Mix 6, which is made by 50% slag and 50% fly ash, exhibits the highest compressive strength compared to other mixes with a strength of 62.49 MPa at 28 days. In contrast to AASF Mix 1, AASF Mix 6 displayed a low initial compressive strength with 28.65 MPa at 3 days, which only represents 45.85% of the final strength. However, it shows a significant increase of strength of 46.84 MPa (74.96% final strength) and 48.76 MPa (78.03% final strength) at 14 days and 21 days, respectively.

Overall the results show a general decrease in initial strength and increase in final strength as the fly ash content increases, though considerable variability is found within the results. This would suggest that the GGBS is the primary contributor to the initial strength with the fly ash contributing to the strength gain with time. This is consistent with the reported data for AAS materials, which have been observed to give a high initial strength, which generally gains little further strength before showing a reduction in strength with time. While 100% fly ash materials generally require heat curing to achieve high strength, often not achieving structural integrity at ambient temperatures.

This most likely suggests that the hydration reaction has two possible mechanisms: (1) the hydration reaction of slag and the polymerization of fly ash are occurring separately from one another, or (2) the two reactions are occurring simultaneously [19]. In the first case it is hypothesized that the GGBS reacts first to form a matrix around the fly ash and the fly ash then fills in the pores, to provide the increased strength. In the second mechanism the two reactions occur simultaneously with the GGBS reaction activating the fly ash at the ambient temperature.

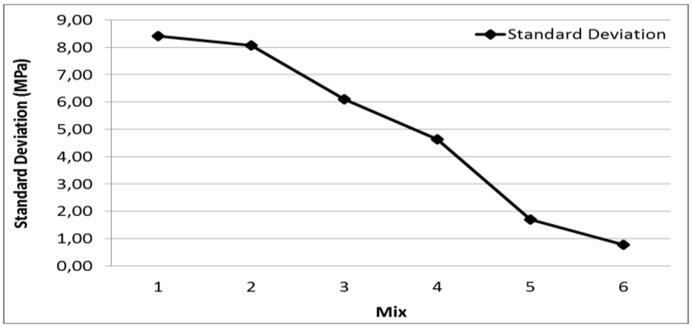

Fig. 2. Standard deviation developments of AASF mortars at 28 days age

Table 4 and Figure 2 show the standard deviation for each test of each mix design. This table shows that as the percentage of fly ash added to the mixture increases, the standard deviations for the tests reduce, and the consistency of the mix designs increase. The decreasing trend in the standard deviations as the mixture approaches 50:50 composition of slag and fly ash shows that the addition of fly ash resulted in a more stable product. Current research shows that AAS has an issue with long term durability due to the development of the micro-cracking [17]. It is possible that fly ash increases the stability of AAS because the polymerization process of the fly ash geopolymer fill the pores present in the AAS mortar. As a result the pores in the AAS mortar will have less water in them and in turn will shrink less and micro-cracking will be reduced. The variability in the results and the stabilizing effects of the fly ash suggest the second mechanism is the more probable.

Figure 3 displays the strength development of AASF mortar specimens with slag addition at 28 days age. The result shows a similar performance to other researcher with slag addition up to 30% [20]. The AASF specimen demonstrates a higher strength compared to Nath et al specimens at 10% and 20% of slag addition. However, Nath et al specimen shows a better strength performance at 30% addition. The AASF specimen achieves a comparable strength performance with Nath et al specimen at 50% slag addition. The slow rate strength development of AASF specimen might attributed to the high molarity of NaOH (15M) solution. According to Nath et al, increasing the alkaline solution cause reduction of strength while increasing setting time. This is because of higher water to solid ratio of mixture having higher liquid content. Excess alkali solution causes an increase in the amount of water in the system which hinders polymerization. This promotes an increase of poorly polymerized reaction products [20].

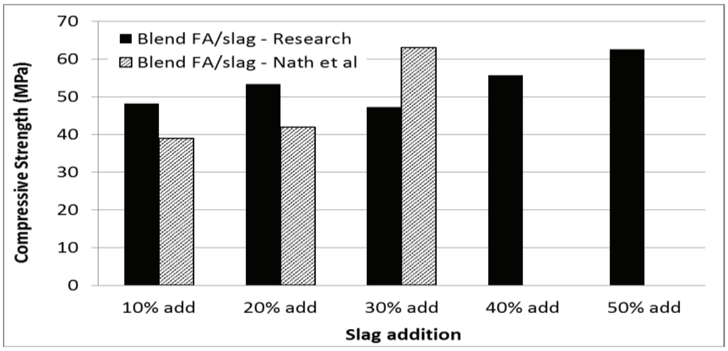

Fig. 3. Strength development of AASF mortars at 28 days age compred to Nath et al. [20]

All mixes were cured in ambient conditions and were able to be successfully removed from the casts after 24 hours. This was particularly interesting for AASF Mix 5 and 6 which contained higher percentages of fly ash (40% and 50%, respectively) which were expected to require heat curing to rapidly gain strength. Current research generally recommends heat curing for fly ash geopolymer specimen for the first 24 hours, with the initial 24 hours of heat curing resulting in rapid strength gain [16, 21]. Beyond this however, the gain in strength is only moderate and not necessary for practical applications of the concrete. These results do show promising signs for improving the durability of cement less concrete, however the mix designs researched in this paper cannot provide direct evidence to support this idea due to time constraints.

4. Conclusions

The following conclusions may be drawn based on this study:

1) The ability of slag and fly ash to replace ordinary Portland cement in concrete can potentially reduce the environmental impact over the production of CO_2.
2) AASF Mix 1 (made by 100% slag) demonstrates the highest initial compressive strength, however, it shows a reduction in strength over periods of time and reaches the lowest compressive strength at 28 days.
3) Although it demonstrates the low initial strength, AASF mix 6 (made by 50% slag and 50% fly ash) exhibits the highest compressive strength at 28 days.
4) AASF specimen test results suggest that the hydration reaction of slag and the polymerization reaction of fly ash could occur separately or simultaneously. The results suggest that the simultaneous reaction is the most likely with the GGBS reation activating the fly ash, enabling it to react at room temperature.
5) The addition of fly ash to the mixes resulting in lower standard deviations which means improved stability of AASF mortar specimens.

6) AASF mixes containing higher percentages of fly ash are still able to be successfully cured under ambient condition. This suggests that the blending of slag and fly ash could provide a solution for the need for heat in the curing of fly ash based-geopolymer concrete.

Acknowledgements

The authors would like to acknowledge the facilities, and the scientific and technical assistance, of the Australian Microscopy & Microanalysis Research Facility at the RMIT Microscopy & Microanalysis Facility, at RMIT University, Melbourne, Australia. Materials support from the Independence Cement Pty. Ltd. Australia and PQ Australia for carrying out this research project is gratefully acknowledged.

References

[1] Davidovits, J., Global warming impact on the cement and aggregates industries, World Resource Review, 6(2) (1994) 263-278.
[2] Berry, M., D. Cross, and J. Stephens, Changing the environment: An alternative "green" concrete produced without Portland cement in World of Coal Ash (WOCA), Lexington, KY, USA, 2009.
[3] Law, D.W., et al., Durability assessment of alkali activated slag (AAS) concrete, Materials and Structures, 45 (2012) 1425-1437.
[4] Neville, A.M., Properties of Concrete, 5th ed, Harlow, England; New York: Pearson, 2011.
[5] Davidovits, J., Geopolymers. New Inorganic polymeric new materials, Journal of Thermal Analysis, 37 (1991) 1633-1656.
[6] Davidovits, J., Properties of geopolymer cements in First International Conference on Alkaline Cements and Concretes, Scientific Research, Kiev, Ukraine, 1994.
[7] Bakharev, T., J.G. Sanjayan, and Y.B. Cheng, Alkali activation of Australian slag cements, Cement and Concrete Research, 29 (1999) 113-120.
[8] Taling, B. and J. Brandstetr, Present state and future of alkali activated slag concretes, American Concrete Institute, 114 (1989) 1519-1546.
[9] Barbosa, V.F.F., K.J.D. MacKenzie, and C. Thaumaturgo, Synthesis and characterisation of materials based on inorganic polymers of alumina and silica: sodium polysialate polymers, International Journal of Inorganic Materials, 2 (2000) 309–317.
[10] Xu, H. and J.S.J.v. Deventer, The geopolymerisation of alumino-silicate minerals, International Journal of Mineral Processing, 59 (2000) 247-266.
[11] Brough, A.R. and A. Atkinson, Sodium silicate-based, alkali-activated slag mortars. Part I. Strength, hydration and microstructure, Cement and Concrete Research, 32 (2002) 865-879.
[12] Wardhono, A., D.W. Law, and T.C.K. Molyneaux, Strength of alkali activated slag and fly ash-based geopolymer mortar, in Microdurability 2012 “The 2nd International Conference on Microstructural-related Durability of Cementitious Composites”, Amsterdam, Netherlands: RILEM Publications, 2012.
[13] ASTM C618-03, Standard specification for coal fly ash and raw or calcined natural pozzolan for use in concrete, ASTM International: USA, 2003.
[14] Chang, J.J., A study on the setting characteristics of sodium silicate-activated slag pastes, Cement and Concrete Research, 33 (2003) 1005-1011.
[15] ASTM C109/C109M-07, Standard test method for compressive strength of hydraulic cement mortars, ASTM International: USA, 2008.
[16] Adam, A.A., Strength and durability properties of alkali-activated slag and fly ash-based geopolymer concrete, in School of Civil, Environment and Chemical Engineering, RMIT University: Melbourne, Australia, 2009.
[17] Collins, F. and J.G. Sanjayan, Microcracking and strength development of alkali activated slag concrete, cement and Concrete Composites, 23 (2001) 345-352.
[18] Wardhono, A., D.W. Law, and T.C.K. Molyneaux, Long term performance of alkali activated slag concrete, Journal of Advanced Concrete Technology, 13 (2015) 187-192.
[19] Wardhono, A., D.W. Law, and T.C.K. Molyneaux, The mechanical properties of fly ash geopolymer in long term performance in The CIC 2014 “Concrete Innovation Conference", Oslo, Norway, 2014.
[20] Nath, P. and P.K. Sarker, Effect of GGBFS on setting, workability and early strength properties of fly ash geopolymer concrete cured in ambient condition, Construction and Building Materials, 66 (2014) 163-171.
[21] Bakharev, T., Geopolymeric materials prepared using Class F f ly ash and elevated temperature curing, Cement and Concrete Research, 35 (2005) 1224-132.