



Laval (Greater Montreal)

June 12 - 15, 2019

COMPARATIVE ANALYSIS OF REACTION KINETICS OF ONE AND TWO PARTS ALKALI ACTIVATED SLAG

W. Almakhadmeh¹ and A.M. Soliman²

1. PhD student, Department of Building, Civil and Environmental Engineering,

Concordia University, Montreal Quebec Canada

2. Assistant Professor, Department of Building, Civil and Environmental Engineering,

Concordia University, Montreal Quebec Canada

Abstract: Alkali activation technique for different industrial by-products has become a useful procedure showing a promising environmentally friendly alternative to cement-based binders. The activation kinetics depends on activator type, concentration and chemical composition. Hence, this study aims to compare the early-age reaction kinetics of alkali activated slag by solid sodium silicate (One part AAS) and the commonly AAS by waterglass. The influence silica modulus (M_s) on the isothermal calorimetric (i.e. heat of hydration) response were evaluated. Results showed that the calorimetric response of one and two part activated systems are different demonstrating single early-age reaction peaks and undetected induction period. In contrast with the calorimetric response of waterglass activated systems which similar show two main peaks and a marked induction period similar to those in OPC system. Moreover, the cumulative heat of hydration decreased by increasing the silica modulus for both systems.

1 INTRODUCTION

Alkali activation technology provides a sustainable alternative binder to Portland cement (OPC) by reducing carbon dioxide (CO_2) emissions and reusing industrial byproducts instead of sending to landfill. In two part AAM, concentrated aqueous alkali solutions are used to start the activation process of the solid aluminosilicate precursor form the binding material. However, these solutions are very corrosive, viscous, and not convenient for storage or transportation. Therefore, the development of one-par AAM “just add water” similar to OPC, increases its commercial viability and especially its in-situ applications.

Conventional (two-part) alkali activated materials are formed by using a concentrated aqueous solution of alkali hydroxide, silicate, carbonate, or sulfate as activator. Despite of the superior engineering properties and environmental benefits, two parts alkali activated materials are not commercially accepted. Some limitations associated with the nature of the chemical solutions which are corrosive, viscous, and not easy to handle, hinder its acceptance as a construction material (Hajimohammadi *et al.* 2011). Attention should be taken during working with such corrosive solution to avoid physical risks such as burns with contact or ingestion (Hartford, 2010). Usually, trained and skilled labors are essential to transport, handle, prepare and mix these concentrated solutions. Hence, there is a desire to develop dry one-part alkali activated materials that could be used similarly to cement.

In one-part alkali activated materials, a solid (powder) alkali-activator is mixed with a solid aluminosilicate precursor and then water is added (Duxson *et al.* 2008). Therefore, using powder activators gives an

opportunity to produce pre-packaged alkali activated binder systems, whereas the water is just added to start the reaction (Neupane, 2016).

2 MATERIALS AND EXPERIMENTAL WORK

Granulated blast furnace slag (GBFS) was used as the aluminosilicate precursor in all mixtures. Its specific gravity was 2920 kg/m³ and Blaine fineness was 515 m²/kg with an average particle size value was around 14.5 μm. The chemical compositions of GGBFS is shown in **Table 1**. The basicity coefficient [$K_b = (CaO + MgO)/(SiO_2 + Al_2O_3)$] and the hydration modulus [$[CaO + MgO + Al_2O_3]/SiO_2$] based on the chemical composition were 1.06 and 1.63, respectively. Natural sand with maximum size of 4 mm, water absorption of 3.5% and saturated surface dry density of 2.72 was used. For two-part mixtures, the alkaline activator was prepared by mixing sodium hydroxide (>98% purity) with distilled water and sodium silicatesilicate solution. The SiO₂/Na₂O molar ratio of the sodium silicate (SS) was 2.0 with chemical composition of 29.40 wt% SiO₂, 14.70 wt% Na₂O and 55.90 wt% H₂O. The activator solution was prepared 1 day before, and kept at laboratory temperature to allow cooling. Moreover, for one part mixtures, powder sodium silicate with a chemical composition of 65.4 wt% SiO₂, 32.7 wt% Na₂O (molar ratio of SiO₂/Na₂O =2) and reagent grade sodium hydroxide powder were used. All powder materials were milled before for 10 min before using. For both one and two part, mixtures compositions were adjusted to have 4% Na₂O and different silica modulus (i.e. 0.5) (Table. 2)

Table: Chemical compositions of slag.

Items	Percentage (%)
SiO ₂	36.5%
Al ₂ O ₃	10.2%
CaO	37.6%
Fe ₂ O ₃	0.5%
SO ₃	3.1%
MgO	11.8%
K ₂ O	0.4%
Na ₂ O	0.3%
TiO ₂	1%
Manganese Oxide	0.4%

Table 2. Experimental work

Two part Mixture	One part Mixture	4% Na ₂ O (Ms values:)
T1	S1	0.5
T2	S2	1
T3	S3	1.5

2.1 Test Methods

The water/solid ratio was kept constant (0.4) and the needed water is calculated for each mixture. The solid weight includes the binder weight and the solid weight of activator (NaOH and sodium silicate). Alkali-activated slag mortars were prepared with sand to binder ratio of 2 for all mixtures. Mixture were prepared by using an electrically driven mechanical mixer conforming to the requirements of ASTM C305. Initially, slag and sand were in a dry state for a minute. The activator solution was dissolved in mixing water and gradually added while mixing continued for about 2 min. For one part mixes, slag, sand and the solid activator were in a dry state for a minute and the water is added gradually.

Compressive strength testing was conducted on 50 mm cubes at the ages 7, and 28 days. Specimens were kept in a humid environment at 22 ± 1 °C and 85 ± 3 % RH, until the testing age. The average of three tested cubes were taken at each age for each mixture. The evolution of hydration was determined in paste samples prepared with a water/solid ratio of 0.4. Measurements of heat of evolution were performed at a constant temperature of 23 C by using Isothermal Conduction Calorimeter. The samples were mixed externally and then loaded into the isothermal calorimeter and tests were run for 48h.

3 RESULTS AND DISCUSSION

3.1 Heat of hydration for two part mixtures

Fig. 1 shows heat evolution rates of AAS paste activated by 4% Na₂O with silica modulus of Ms = 0.5, Ms=1 and Ms =1.5 at 23C. As observed, for AAS samples T1, the accelerated peak is higher than those for T2 and T3. It means that by increasing Ms, the amount of initial and accelerated peaks decrease. Additionally, Ms has retardation effect on the reaction rate and lead to longer induction period, whereas the precipitation of C-S-H gel results in a very long induction period. Furthermore, the accelerated peaks were 9h, 34 and 37h for T1, T2 and T3 respectively. and the top of the accelerated hydration peak for L4, L5 and L6 is about 9h and 17 h, and 37h respectively. On another hand increasing Ms (lower alkalinity), decreases the released cumulative. Fig 2, presents the cumulative heat of hydration for the three mixtures. Mixture T1 with lowest Ms exhibits the highest released heat.

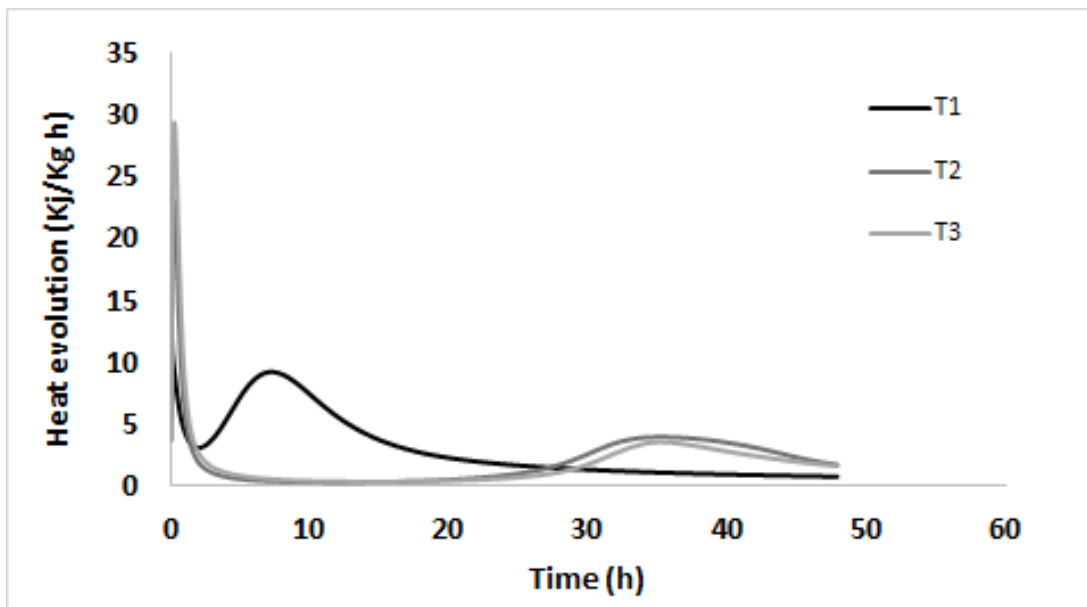


Figure 1: Effect of waterglass modulus on heat evolution of AAS.

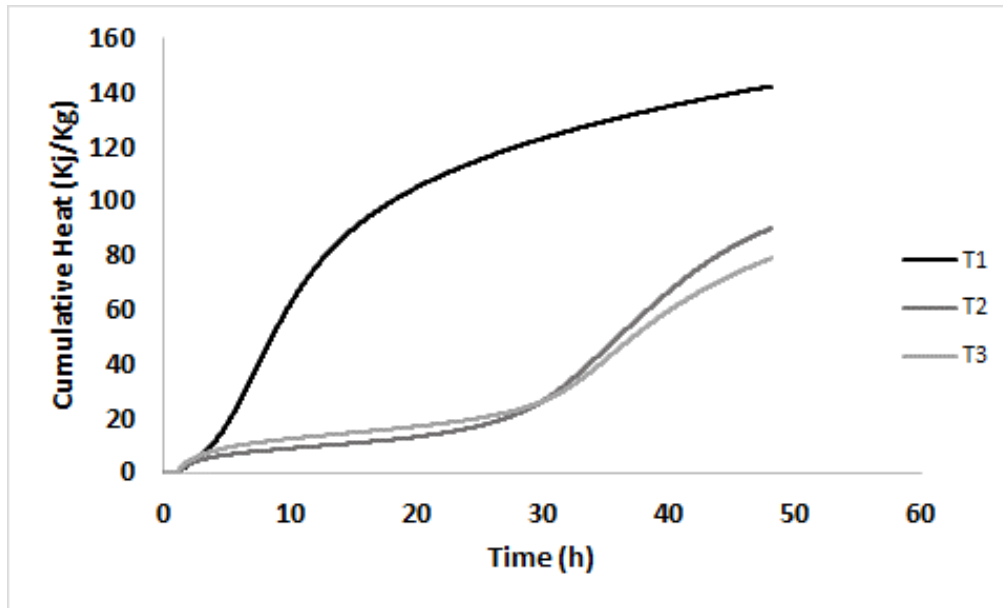


Figure 2: Effect of waterglass modulus on cumulative heat of hydration of AAS.

3.2 Heat of hydration for one part mixtures

Fig. 3 shows the heat evolution curves as a function of time for the powder activated slag pastes proportioned using activator Ms of 0.5, 1 and 1.5. Heat evolution of slag pastes activated using powder sodium silicate activator and NaOH required to lower the Ms. Based on the results, there is only one main peak. The initial heat release peak corresponds to the combination of the initial and acceleration peak. The induction period which is typical in the hydration of waterglass AAS is undetected in hydration of solid activated AAS. This may attribute to the quick precipitation of reaction products on the surface of slag. Mixture S1 peak is the highest, due to higher alkalinity than S2 and S3. Furthermore, increasing Ms, decreases the cumulative heat that released. Fig 2, presents the cumulative heat of hydration for the three mixtures. Mixture S3 with highest Ms exhibits the lowest released heat

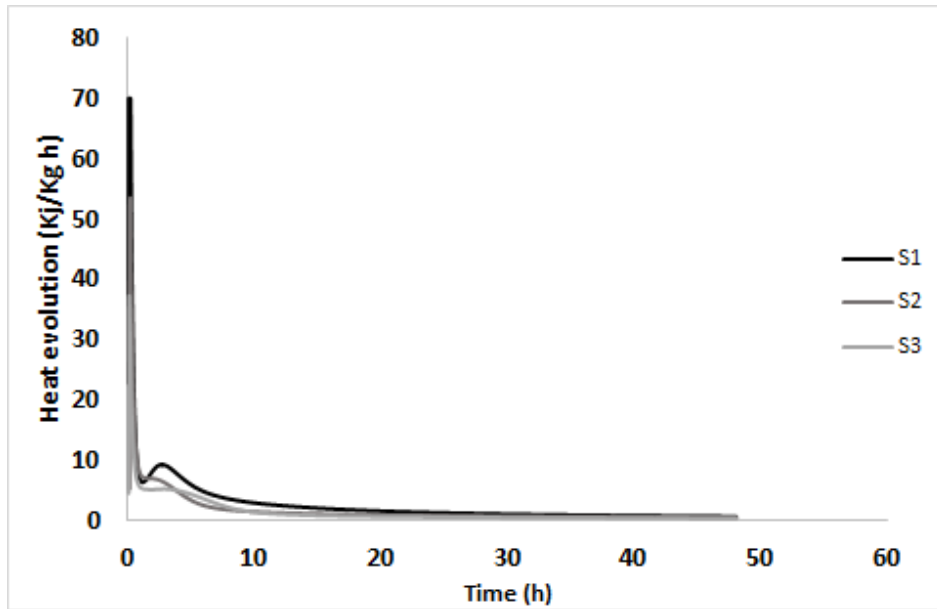


Figure 3: Heat evolution of slag pastes activated powder sodium silicate activator.

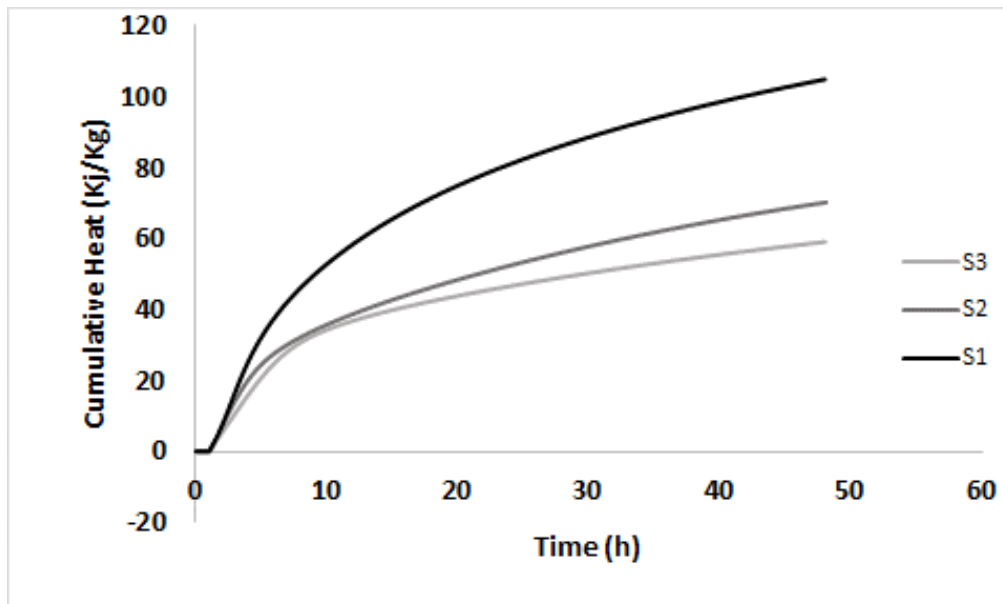


Figure 4: Effect of waterglass modulus on cumulative heat of hydration of AAS.

4 COMPRESSIVE STRENGTH

Compressive strength of the AAS mortars were tested at ages of 1, 3, 7, and 28 days. Fig. 5 illustrates the compressive strength of the one part AAS. Based on the result, the mixtures with lower Ms (i.g. S1) exhibit

the higher early age strength. However, the later age compressive strength is higher by increasing Ms (i.g. S3).

Fig. 6(c) presents the compressive strength of two part AAS with different Ms and 4% Na₂O. As can be seen the strength are significantly higher by using the activator as a solution rather than solid form. This is ascribed to the less amount of silica dissolution from powder activator.

The 28 - compressive strength ratios of solid to solution activator form are given in fig. 7. This figure shows, that one part AAS mixtures gained strength about half than those of two part AAS mixtures. Due to higher rate of C-S-H gel formation in activation of slag by alkaline solution

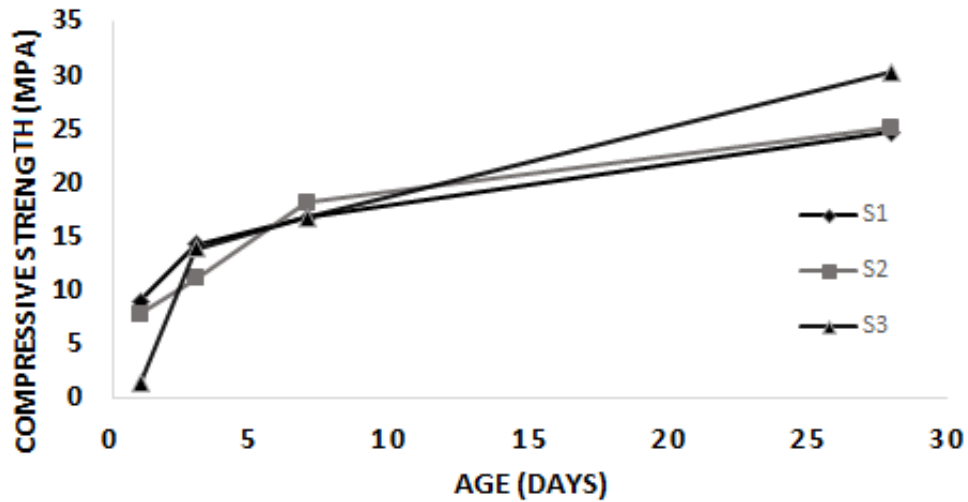


Figure 5: Compressive strength development AAS by using powder sodium silicate and 4% Na₂O

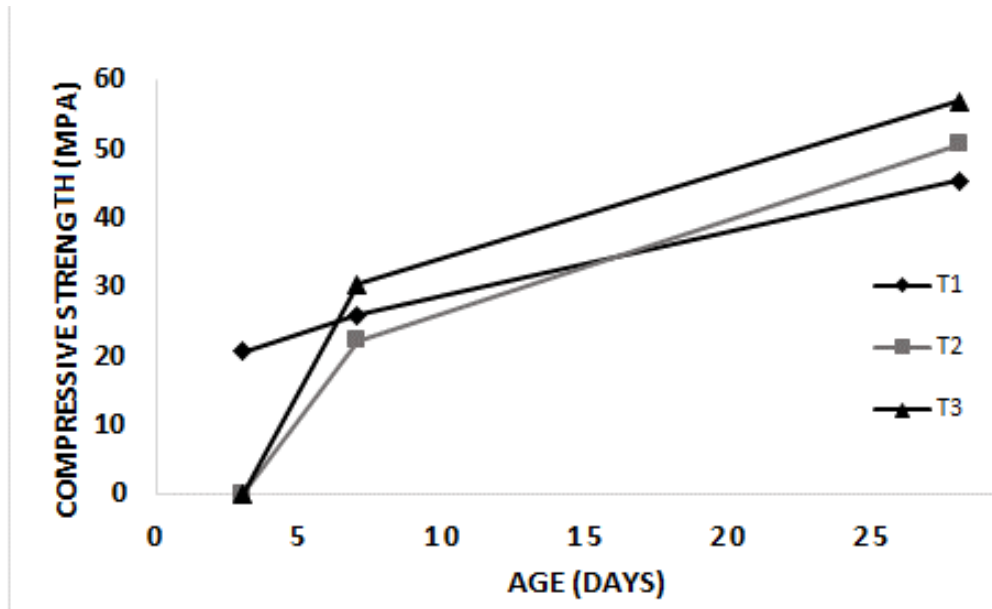


Figure 6: Compressive strength development AAS by using waterglass with 4% Na₂O

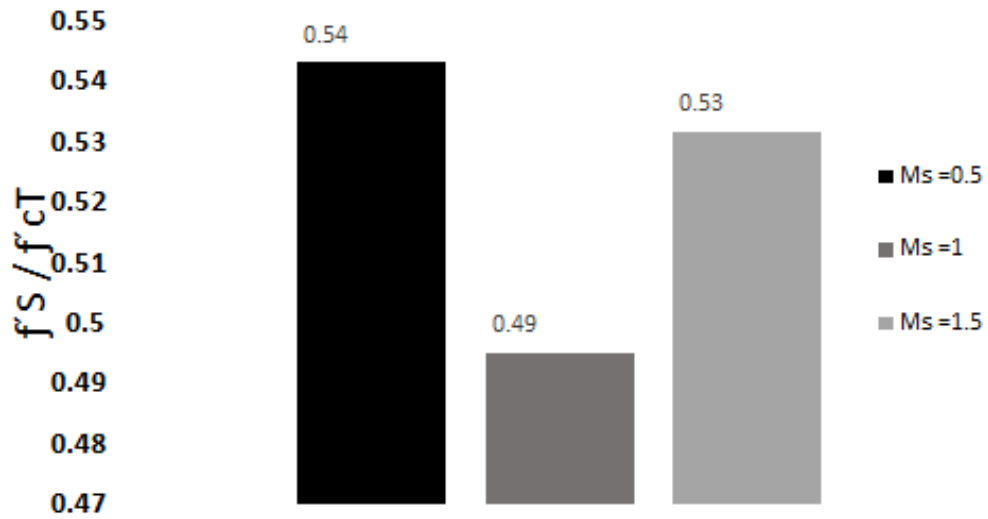


Figure 7: Compressive strength ratio between one and two part (s) AAS mortars at age of 28 days.

5 CONCLUSION

By comparing the reaction kinetics in one and two part(s) AAS systems, activated by sodium silicate with different modulus (Ms) and constant Na₂O-to-slag ratios (4%). The response of isothermal calorimetry has been presented in this paper. For the solid sodium silicate activated slag, one major heat flow peak was detected and appears at very early ages (order of 1 h). However, this observation indicates that the silica did not dissolve in the solution while using sodium silicate as a powder. On the other hand, the AAS mixtures activated by sodium silicate solution (liquid form) showed two main peaks. Moreover, the cumulative released heat decreased by increasing silica modulus value (Ms).

The 28-day compressive strength of one part mixtures were in the range of 25–30 MPa, which are suitable for general purpose concretes. Furthermore, the compressive strength values for two part mixtures were about twice the values of one part mixtures. This is attributed to the higher rate of C–S–H gel formation in a slag system activated by alkaline solution (liquid form). Nevertheless, producing one part AAS “just add water”, increases the commercial viability especially in-situ applications which is similar to OPC.

6 REFERENCES

- Duxson, P., and J. L. Provis. 2008. "Designing Precursors for Geopolymer Cements." *Journal of the American Ceramic Society* 91 (12):3864-3869. doi: 10.1111/j.1551-2916.2008.02787.x.
- Hajimohammadi, A., J. L. Provis, and J. S. J. van Deventer. 2011. "The effect of silica availability on the mechanism of geopolymerisation." *Cement and Concrete Research* 41 (3):210-216. doi: 10.1016/j.cemconres.2011.02.001
- Neupane, K. 2016. "'Fly ash and GGBFS based powder-activated geopolymer binders: A viable sustainable alternative of portland cement in concrete industry'." *Mechanics of Materials* no. 103:110-122. doi: 10.1016/j.mechmat.2016.09.012.