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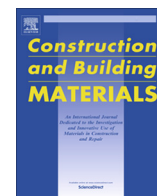
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Early-age properties of alkali-activated slag and glass wool paste

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HIGHLIGHTS

- Waste glass wool is utilized by means of alkali-activation.
- Early-age properties such as the workability, reaction rate, strength and shrinkage of alkali-activated slag and glass wool pastes are comprehensively studied.
- Glass wool is found to prolong the setting time and reduce the shrinkage of alkali-activated slag.

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ABSTRACT

In this study, glass wool waste was utilized by means of alkali-activation with blast furnace slag. Reaction kinetics, workability, mechanical properties and autogenous shrinkage of alkali-activated slag and glass wool were comprehensively studied. Results indicated an optimal modulus ($\text{SiO}_2/\text{Na}_2\text{O}$) of the activator related to a long enough setting time and a high reaction degree of alkali-activated slag paste. The incorporation of glass wool as partial slag replacement did not necessarily lead to degradation in the performance of the pastes. While the compressive strength was always lower when glass wool was incorporated in the mixture, the flexural strength and workability could be improved with proper glass wool dosages. Autogenous shrinkage of blended pastes was always lower compared to the mixture without glass wool. The results in this paper suggest that waste glass wool can be used as a precursor in slag-based alkali-activated system, resulting in improvements in the early-age properties of the paste such as a prolonged setting time and reduced shrinkage.

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1. Introduction

An important part of demolition wastes is mineral wool waste, originated from insulation materials in buildings. Nowadays, around 2.5 million tonnes of mineral wool waste are produced worldwide every year [1]. While by weight, the mineral wool waste accounts for only 0.2% of the total construction demolition wastes, it requires disproportionately large space to store due to the low bulk density. Traditionally, mineral wools, which are mainly stone wool and glass wool, are considered unrecyclable wastes due to their fibrous nature and their low density [2]. Some efforts have been made to recycle and valorise these materials, for example, as supplementary cementitious materials (SCMs) [3], but the current utilization amount is still rather limited.

In recent years, alkali-activated materials (AAMs) have emerged as promising alternatives to ordinary Portland cement (OPC) [4]. Considering the high alkalinity of the activator, mineral wools may show higher reactivity in AAMs than in OPC. Both stone wool and glass wool contain Si, which is an important element for the production of the binding phases (C-A-S-H type gel and/or N-A-S-H type gel) in AAMs. Therefore, alkali-activation might be a feasible way to reuse the waste mineral wool. Several studies have been conducted on alkali-activated mineral wool [2,5–7], showing that although the wool presents certain reactivity, the mechanical properties of alkali-activated stone wool (AASW) and alkali-activated glass wool (AAGW) are low [2].

One way to increase the mechanical properties of AASW and AAGW is to increase the reactivity of the precursor. During the production of mineral wool, organic resin, typically phenol-formaldehyde, is usually used as an additive to bind the wool together [5]. The organic resin partially covers the surface of the wool and hence impairs the exposure of the reactive phases to the alkali solution.

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Therefore, ignition of wool waste to remove the organic resin is a potential strategy to increase the reactivity of the wool. Another way to facilitate the development of mechanical properties is to use elevated temperature curing of the paste or concrete. In this way, the dissolution rate of wool and the reaction degree of the paste can both be improved [8]. However, both ignition of the raw materials and elevated-temperature curing impose high costs and energy consumption on the recycling process. Moreover, for cast-in-situ concrete, elevated temperature curing is not practical. Therefore, using stone wool or glass wool as the single precursor seems not a promising way to produce high strength AAMs.

Blast furnace slag is widely used as a high calcium precursor for AAMs. The strength of slag based AAMs is normally high [9]. Hence, co-activation with slag might be a better strategy to reuse waste mineral wools and to produce strong building materials. In particular, high-Si glass wool may have similar behaviour compared to waste glass powder, which has already shown good potential to be used together with slag upon alkali-activation [10–12]. However, very few studies can be found in the literature on the properties of alkali-activated slag blended with mineral wools.

The aim of this study, therefore, is to investigate the feasibility of co-activating waste glass wool with slag at ambient temperature. It should be noted that the utilization of glass wool as an addition/substitution of slag in AAMs can be considered promising only if the systems show comparable or even superior performance compared to that of pure AAS. A high strength is not the only criterion for building materials. In fact, due to the high reactivity of slag in alkali environments, a high strength of paste is always easy to realize irrespective of the presence of wastes. Besides strength, satisfactory workability, stiffness and volume stability are also required.

In this study, the early-age properties of alkali-activated slag and waste glass wool (AASG) were systematically investigated. To identify the optimal mixture design, various compositions of the activator and glass wool dosages were considered. Several criteria in aspects of reaction rate, flowability, setting time, compressive and flexural strengths and autogenous shrinkage were used to evaluate the performance of AASG mixtures. AAS systems were used as references. This study is expected to contribute also to the knowledge on mixture design of pure slag based AAMs.

2. Methodology

2.1. Raw materials

The precursors used in this study were blast furnace slag and waste glass wool powder supplied by Ecocem Benelux and Saint-Gobain Ecophon, respectively. The glass wool powder was supplied by Saint-Gobain factory through milling acoustic panels by an industrial ball-milling machine. The chemical compositions of the precursors determined by XRF are shown in Table 1. It can be seen that the glass wool contains a high amount of SiO_2 . The contents of CaO and Al_2O_3 in glass wool are much lower than in slag. The pH of the glass wool suspension in water was 7.8, as determined by titration method against HCl solution (0.1 M). A detailed procedure of the titration can be found in [13]. The pH value of the glass wool suspension indicates that a high content of Na detected in glass wool existed in the glass structure (probably incorporated with

fluxing agent during the production of glass wool [14]) rather than free Na_2O or NaOH. In other words, the change of dosage of glass wool in the precursor would not induce significant change in the alkalinity of the pore solution. This information is important for the determination of alkali content of activators and for the analyses of properties of various mixtures.

The colour of glass wool was light yellow (see Fig. 1a). A picture and two representative scanning electron microscopy (SEM) images of glass wool are shown in Fig. 1. The glass wool was fibre-like rather than powder-like. Therefore, laser diffractometer could not be used to determine the particle size of glass wool. From Fig. 1(b) and (c), it can be seen that the glass wool had a diameter of around 5–15 μm and a length of 20 – 200 μm . The spots on the surface of the wool were suspected to be organic resins from the coating of the wool.

2.2. Mixture design

Twenty AASG paste mixtures with different activator compositions and glass wool dosages were prepared in this study to study the behaviours of AASG thoroughly, as shown in Table 2.

Five glass wool dosages were considered from 0% to 100% of the binder, with increments of 25%. Since the dissolution of glass wool did not contribute to the alkalinity of the pore solution, a constant Na_2O content was used for pure slag pastes and glass wool incorporated pastes. According to [15], a Na_2O content lower than 4% of the binder would result in weak activation effect and consequently a low strength of the paste. Na_2O contents higher than 5% can increase the strength but can also increase the price and the corrosiveness of the solution, leading to less user friendly solutions. Hence, the alkali content of the paste was fixed at 5% of the binder (around 0.8 mol to 1000 g of binder). The modulus ($\text{SiO}_2/\text{Na}_2\text{O}$) of the activator has a major impact on the properties of the paste [16]. In this study, four moduli (0, 0.5, 1.0 and 1.5) were considered. The water content of the paste was kept constant, making modulus the only variable in the activator. As a result, different liquid contents were yielded due to the different SiO_2 contents.

All the activators were prepared by mixing commercial NaOH solution (solid NaOH content: 50%), commercial water glass solution (PQ Crystal 0112) and deionized water. The reasons why NaOH solution was used to prepare activator, instead of NaOH pellets, are the lower price of the solution, the easier mixing process and the lower heat generated during mixing, when NaOH solution are used. The solution was allowed to cool down for 24 h to room temperature before mixing with the precursors. A Hobart mixer was used for mixing. After premixing of the raw materials for 1 min, activator was added at low speed mixing. The mixing continued at low speed for 1 min and for another 2 mins at high speed. All the samples were cured in sealed condition at 20° C. Regarding the labelling of the mixtures, for example, S50-1.0 means the paste with a slag content of 50% of the binder and a modulus of 1.0 of the activator.

2.3. Experimental methods

2.3.1. Framework

The framework of the experiments conducted in this study is shown in Fig. 2. To identify mixtures with optimal early-age

Table 1
Chemical compositions of slag and glass wool.

Oxide (wt. %)	SiO_2	Al_2O_3	CaO	Na_2O	MgO	Fe_2O_3	SO_3	TiO_2	Other
Slag	31.8	13.3	40.5	–	9.3	0.5	1.5	1.0	2.1
Glass wool	61.8	5.8	10.4	15.8	2.1	0.5	0.2	0.9	2.5

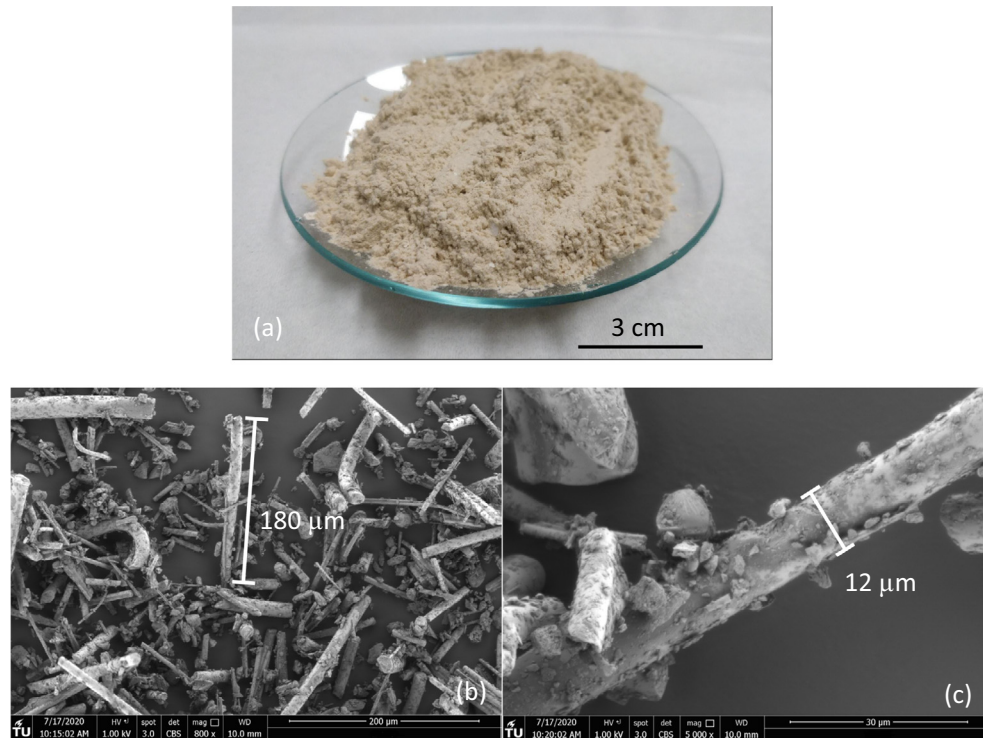


Fig. 1. A picture (a) and two SEM images ((b) and (c)) of the glass wool.

Table 2

Mixture design of AASG pastes (per 1000 g of the binder).

Mixture	Slag (g)	glass wool (g)	SiO ₂ /Na ₂ O	SiO ₂ (mol)	Na ₂ O (mol)	H ₂ O (g)	l/b
S100-1.5	1000	0	1.5	1.2	0.8	426	0.548
S75-1.5	750	250					
S50-1.5	500	500					
S25-1.5	250	750					
S0-1.5	0	1000					
S100-1.0	1000	0	1.0	0.8			0.524
S75-1.0	750	250					
S50-1.0	500	500					
S25-1.0	250	750					
S0-1.0	0	1000					
S100-0.5	1000	0	0.5	0.4			0.5
S75-0.5	750	250					
S50-0.5	500	500					
S25-0.5	250	750					
S0-0.5	0	1000					
S100-0	1000	0	0	0			0.476
S75-0	750	250					
S50-0	500	500					
S25-0	250	750					
S0-0	0	1000					

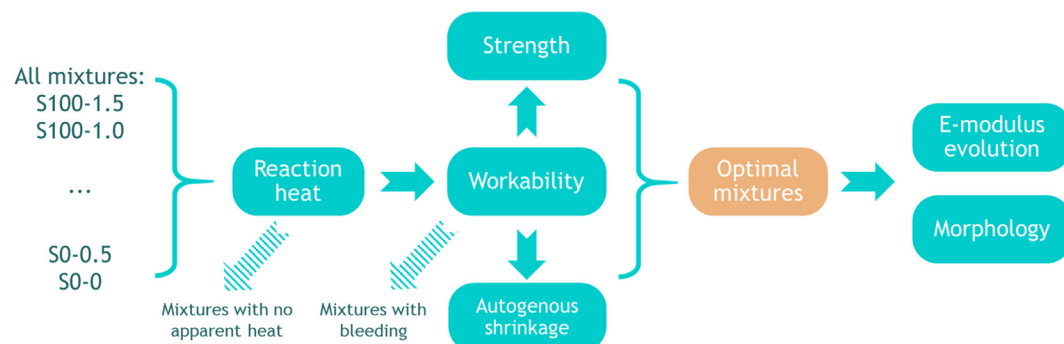


Fig. 2. Experimental approach of this study.

properties, isothermal calorimetry was first utilized to characterize the reaction heat of the mixtures. Only the mixtures that showed considerable reaction heat were considered for subsequent experiments. Attention was also paid to the consistency of the samples during hardening. Workability tests, including the mini-slump test and Vicat setting time test, were performed. The mixtures with serious bleeding were excluded from further tests. Flexural and compressive strength tests and autogenous shrinkage tests were then conducted. Based on the workability, strength and shrinkage results, the optimal mixture was identified. The evolution of the elastic modulus and the morphology of the optimal AASG mixture was evaluated, providing a further understanding of the behaviours of AASG.

The details of the experimental methods are given in the following sections.

2.3.2. Reaction heat

The reaction heat of AASG paste was measured by isothermal calorimetry using a TAM Air conduction calorimeter. Calibration was done at 20 °C before measurements. Heat flow was recorded for pastes for a period of 120 h. About 6 g of paste was prepared for each mixture [17]. The samples were loaded into the chamber immediately after mixing. The temperature of the chamber was controlled at 20 ± 0.02 °C during the experiment. Both the heat flow and cumulative heat were normalized by the mass of the binder.

2.3.3. Workability

The mini-slump of the paste was measured according to Tan et al. [18]. The freshly mixed paste was poured into a slump cone with a top inner diameter of 38 mm, a bottom inner diameter of 90 mm, and a height of 75 mm. The cone was placed on a smooth glass plate to minimize the friction between the plate and the paste. The cone was lifted up slowly after filled by the paste. The average spread of the mixture was measured along the two diagonal directions of the paste. Photos were taken from the top view.

The initial and final setting time of the paste were measured according to NEN 196-3 [19] by an automatically recording Vicat apparatus (Controls VICATMATIC 2). It was noted that the upper surface of the sample needed to be covered by plastic film to avoid moisture loss during the measurement. It was found that the needle could easily penetrate into the sample through the thin plastic film, and the penetration depths into the samples with or without plastic covering, during one penetration, were similar. However, if the sample was exposed during the entire measuring period, the surface part of the material was found to harden faster than the inside due to drying and/or carbonation, which would lead to underestimation of the setting time. Immersing the sample under water during the measurement as prescribed in NEN-EN 196-3 [19] for OPC was believed not to be suitable for AAMs, since the ions could leach out to the surrounding water and the reactions could be seriously affected.

2.3.4. Strength

The flexural strength and compressive strength of the paste were measured according to NEN 196-1 [20] at the age of 1 day, 3 days and 7 days. Three prisms with the size of $40 \times 40 \times 160$ mm³ were measured for flexural strength of each mixture at each age, while six halves for compressive strength. The samples were cured in sealed condition before subject to measurements.

2.3.5. Autogenous shrinkage

The autogenous shrinkage of the paste was measured by corrugated tube method according to ASTM C1968 [21]. Three replicates were measured for each mixture. The data was recorded by linear

variable differential transformers (LVDTs) from the final setting time of the paste until 7 days [22].

2.3.6. Elastic modulus

The elastic modulus evolution of the paste was measured by Elasticity Modulus Monitoring through Ambient Response Method (EMM-ARM) [23] from casting until 7 days, as shown in Fig. 3. The EMM-ARM method allowed continuous monitoring of the first resonant frequency of the paste, which was cast into a 450 mm span cantilevered acrylic tube. The tube had an outer diameter of 20 mm and an inner diameter of 16 mm. Based on the vibration frequency of the cantilevered tube excited by white noise, the elastic modulus of the paste can be calculated. The detailed procedure is given in [23]. Three replicates were tested for each mixture.

2.3.7. Morphology

The morphology of the optimal AASG paste was observed by a Philips-XL30-ESEM. The paste was cast into a PVC cylinder bottle with a diameter of 24 mm and a height of 50 mm. At the ages of 1 day and 7 days, the sample was taken out from the bottle and knocked into pieces with a size of around 1 cm³. These pieces were then immersed in liquid nitrogen to stop the reaction. Afterwards, the samples were put into a freeze dryer until a constant weight was obtained. The fracture surfaces of the samples were then imaged by SEM.

3. Results and discussion

3.1. Reaction heat

3.1.1. Heat flow

The heat flow of the 20 mixtures is shown in Fig. 4. For a pure slag-based paste, the heat flow curves show an initial peak immediately after mixing and a main peak that corresponded to the acceleration period of the reaction. The period between the two reaction peaks is called the dormant period or the induction period. With the decrease of the modulus of the activator, the main reaction peak of S100 appears earlier and is of higher intensity. For S100-0, the dormant period is so short that the main peak nearly overlapped with the initial peak. These findings are consistent with the results of [24]. A higher modulus of the activator means reduced pH of the solution [13], thus decreasing the dissolution rate of slag. Moreover, a higher content of silicate can facilitate the formation of early-age reaction products surrounding the slag particles, forming a barrier and hindering further diffusion of ions from the unreacted slag [25]. Therefore, S100 mixtures with a high modulus shows a longer dormant period, but the main peak tended to be wider due to the larger amounts of reactants in the pore solution.

With the incorporation of glass wool, the dormant period is significantly prolonged and the intensity of the main reaction peak is reduced. This is firstly because of the lower content of slag, thus

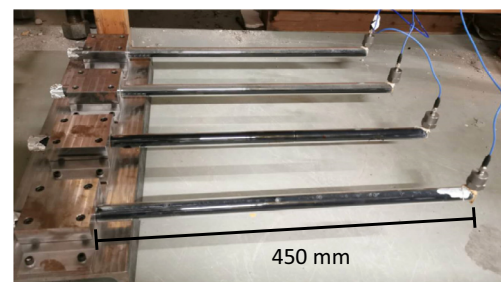


Fig. 3. EMM-ARM apparatus for the measurement of elastic modulus of the pastes.

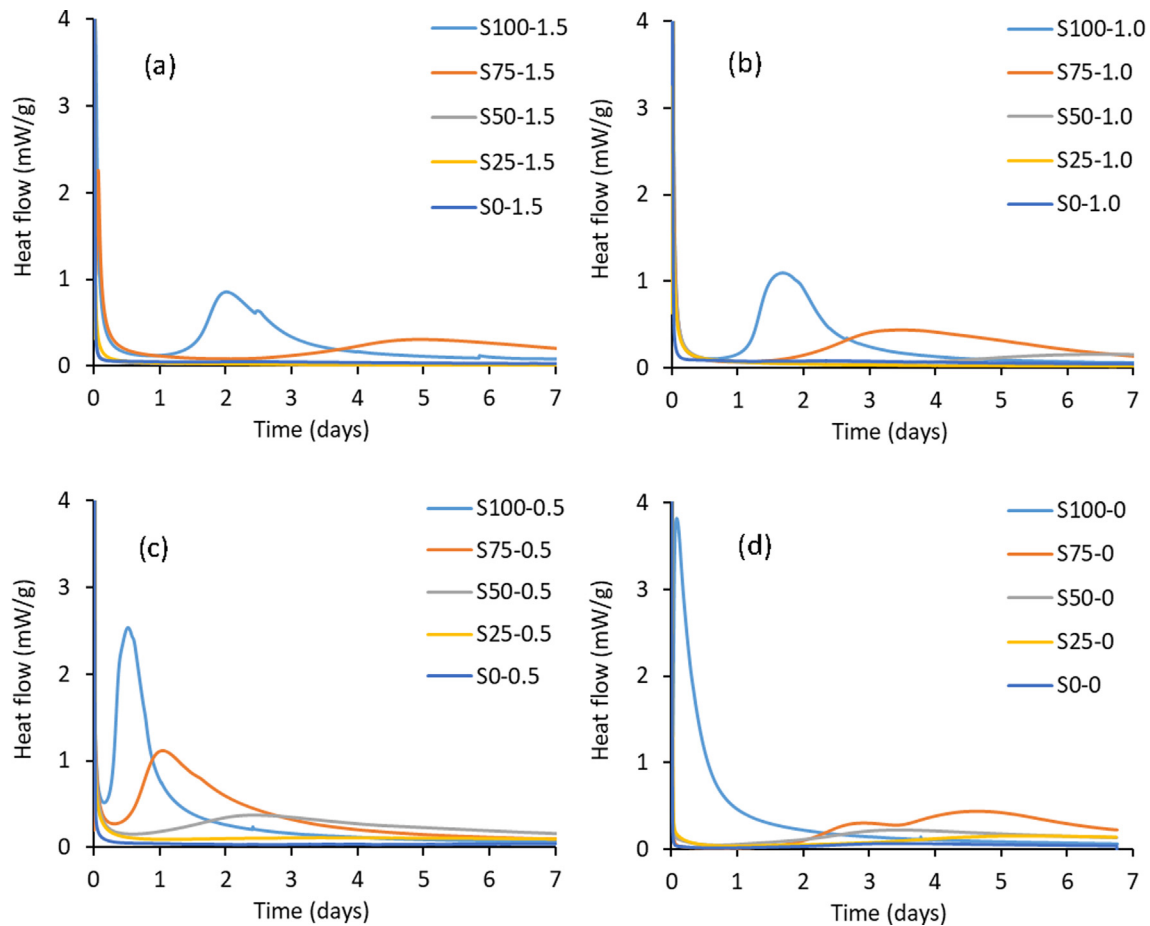


Fig. 4. Heat flow of AASG pastes. The results were normalized by the mass of the binder.

lower supply of Ca and Al, which are key to the formation of reaction products. Moreover, the incorporation of glass wool brings additional silicate (see Table 1). The effect of the additional silicate from glass wool is, to a certain extent, similar to that of a higher modulus of the activator, which can delay the occurrence of the acceleration period [17], although the composition of the glass wool is much more complex than pure silicate. For S75 activated by NaOH solution (S75-0), there are two adjacent main peaks, which cannot be explained yet based on the current knowledge and information from the literature.

When the content of glass wool exceeds 50% of the precursor, the intensity of the main heat flow peak become rather low (<0.15 mW/s) irrespective of the activator composition. This indicates a very limited reaction degree of the corresponding pastes, based on which a limited strength can be expected. Therefore, S25 and S0 series were not subject to further investigations.

3.1.2. Cumulative heat

The cumulative reaction heats of S100, S75 and S50 series are shown in Fig. 5. The reaction heat of most pastes shows two rapid developments in the first week, which correspond to the initial and the main heat flow peaks, respectively, as shown in Fig. 4. Despite the different lengths of dormant periods, the cumulative reaction heats of S100-1.5 and S100-1.0 are similar. S100-0.5 generates the most heat among the four S100 mixtures, indicating the highest reaction degree of slag in S100-0.5. These results show that neither too much nor too little silicate is beneficial for a high reaction degree of slag.

The substitution of slag by glass wool generally results in a lower cumulative heat of the paste, except for S75-0.5. The reaction heat of S75-0.5 is even higher than that of S100-0.5 after the 4th day, indicating the contribution of glass wool to the reaction. For S50 series, only S50-0.5 shows cumulative heat higher than 150 J/g in 7 days. The results in Fig. 5 indicate that the modulus of 0.5 is the optimal activator composition to spark the reactivity of not only slag, but also glass wool.

3.2. Workability

3.2.1. Flowability

The flowability of the paste can be indicated by the spread of the paste by mini-slump tests, as shown in Table 3 and Fig. 6.

For S100 systems, the lower modulus of the activator, the lower is spread of the paste. This is probably due to the higher pH of the activator with lower modulus, which enables a fast release of ions from slag at the very early age [13], thus increasing the viscosity of the paste. This point finds its evidence in [26], which shows that the shear stress of AAS paste increases with lower activator modulus. The flowability of S75 and S50 series follows similar trends against the activator modulus, except for S75-0, which shows a larger spread than S75-0.5.

Compared with the pure slag-based systems, the paste with 25% of glass wool shows better flowability irrespective of the activator used. The surfaces of S75 mixtures look smooth and shining. Flowability is worsened for binders that contained 50% of glass wool. Agglomerates of glass wool can be observed on the surface of S50 pastes, especially for those activated with lower moduli,

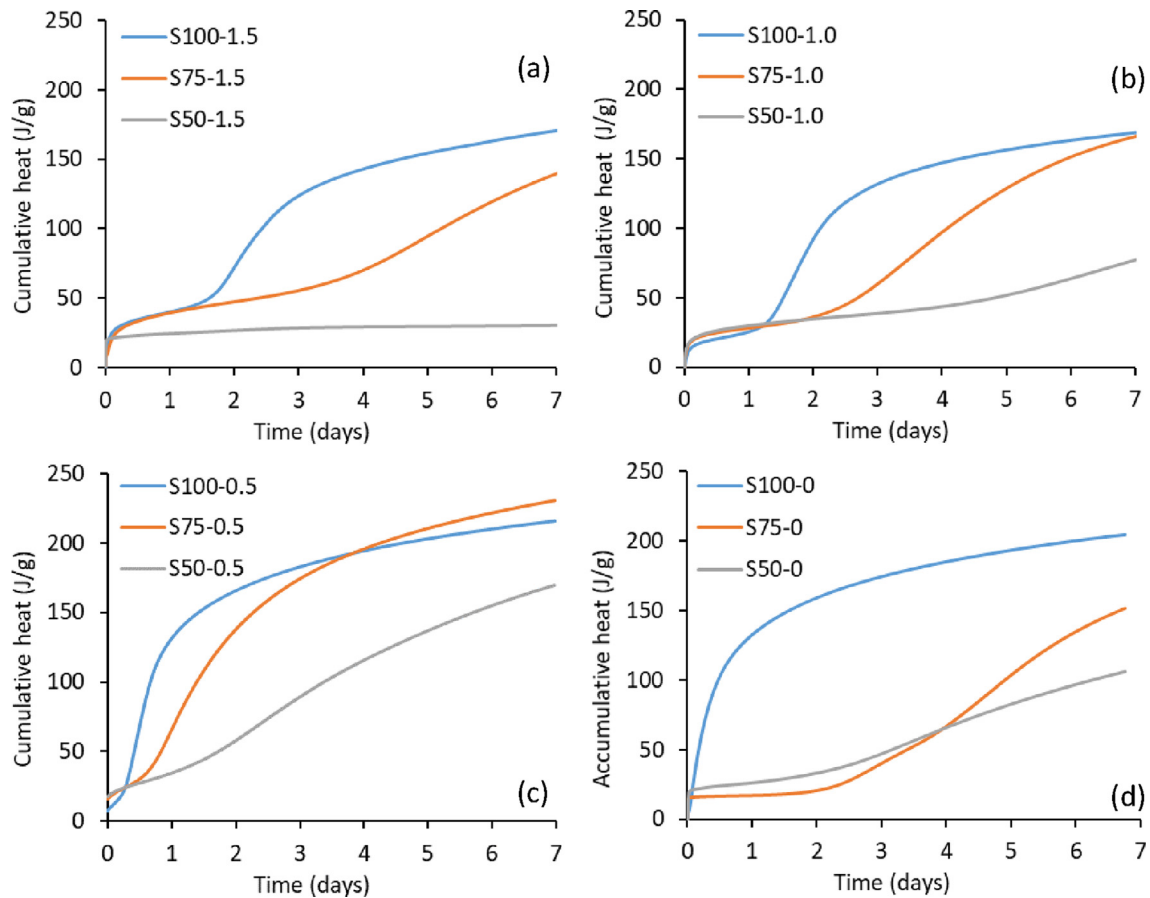


Fig. 5. Cumulative heat of AASG pastes. The results were normalized by the mass of the binder.

Table 3
Mini-slump spread of AASG pastes.

Mixture	Mini-slump spread (cm)
S100-1.5	28.5
S75-1.5	29
S50-1.5	24.5
S100-1.0	27
S75-1.0	28.5
S50-1.0	25.5
S100-0.5	18
S75-0.5	21.5
S50-0.5	17
S100-0	13.5
S75-0	28
S50-0	18.5

which are more viscous thus leading to worse dispersion of the wool. From the flowability point of view, it appears that 25% is the optimal dosage of the glass wool for all activator compositions.

3.2.2. Bleeding

Despite the improved flowability of S75-0 and S50-0 compared to S100-0, these two mixtures show bleeding after mixing for 2 h, as shown in Fig. 7. Large amounts of liquid can be observed on the surfaces of the samples. It should be noted that no additional liquid was added during the mixing of these pastes. As shown in Table 2, the liquid/binder ratio of S100-0, S75-0 and S50-0 is in fact the lowest among all the mixtures studied. For S100-0, the liquid/binder ratio of 0.476 is not even sufficient to ensure a good flowability, as shown in Fig. 6(j).

The key reason for the bleeding probably lies in the retarded reaction due to the incorporation of glass wool, in addition to the lack of silicates in the activator. For NaOH and Na_2SiO_3 activated slag systems, the silicates in the activator act not only as reactants but also nuclei for the formation of initial reaction products. Although the stiffness of this network in this stage is not sufficient to provide considerable strength [27], it can at least resist the subsidence due to gravity. For NaOH activated systems, by contrast, the initial products can form only based on the ions dissolved from the precursors. For S100-0, the dissolution of slag is fast and the acceleration period appears immediately after the initial reaction peak, as shown in Fig. 4(d). Therefore, S100 paste does not bleed even without the presence of silicate in the activator. When a large amount of slag is replaced by glass wool, the appearance of the acceleration period is greatly delayed and the intensity of the main reaction peak is reduced (see Fig. 4(d)). As a result, the initial network in S75-0 and S50-0 does not form in time to resist the deformation under gravity.

Another phenomenon observed is that the bleed liquid is not transparent or clear, but red. The colour of the bleed liquid of S50-0 is darker than that of S75-0. The non-transparency of the liquid is believed to be due to the organic impurities in the glass wool that are dissolved into the alkali activator. As stated in Section 2.1, the glass wool was obtained as industrial waste and contained organic resin as coating materials. While incorporating the waste glass wool into alkali-activated concrete may solidify the organic elements in the matrix, this can surely not be realized through a mixture that shows serious bleeding. Therefore, the mixtures S75-0 and S50-0 did not warrant further investigation from not only the paste consistency but also the leaching points of view.

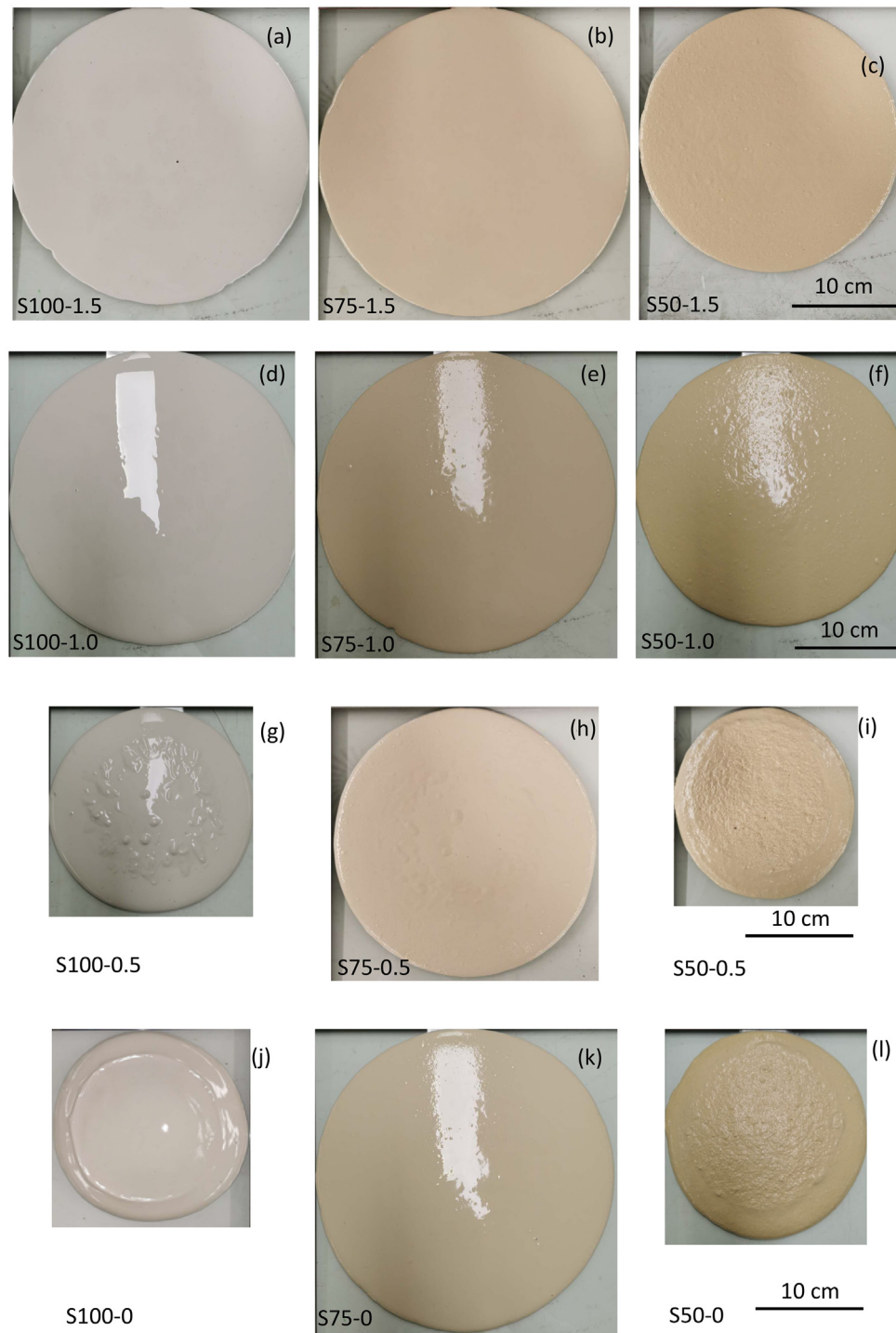


Fig. 6. Mini-slump spreads of AASG pastes.

Note that the evaluation of health and environmental aspects of the mineral wool wastes and their products is not within the scope of this study.

3.2.3. Setting time

The setting time of the mixtures are shown in Table 4. For NaOH and Na_2SiO_3 activated slag systems, a higher activator modulus results in a shorter setting time. As discussed in Section 3.2.2, the silicate that is directly available in the activator facilitated the formation of initial products in the interstitial space, leading to a

quick loss of flowability of the paste. That is the reason why $\text{Na}_2\text{-SiO}_3$ activated slag normally shows shorter setting time than OPC systems at similar reaction degrees [28]. The final setting time of S100-1.5, for example, is only 60 min.

The setting time of S100-0 is not as short as that of S100-1.5 and S100-1.0 due to the absence of silicate in the activator. However, since a high pH is provided by the activator, the dissolution of slag and the formation of reaction products are both fast, as indicated by the very early appearance of main reaction peak (see Fig. 4(d)). As a result, the setting of S100-0 is even faster than that of S100-0.5.

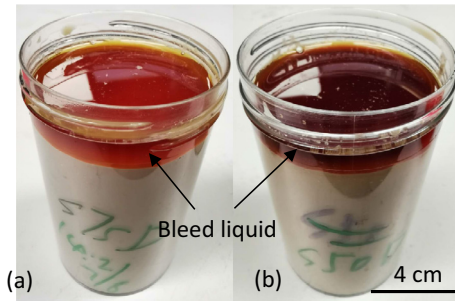


Fig. 7. Bleeding of S75-0 (a) and S50-0 (b) pastes.

Table 4
Setting time of AASG pastes.

Mixture	Initial setting time (min)	Final setting time (min)
S100-1.5	35	60
S75-1.5	42	71
S50-1.5	43	120
S100-1.0	43	93
S75-1.0	49	110
S50-1.0	55	144
S100-0.5	270	380
S75-0.5	360	480
S50-0.5	> 600	> 1d
S100-0	70	87
S75-0	–	–
S50-0	–	–

The setting time of paste becomes longer when more glass wool is used in the binder, irrespective of the composition of the activator. This is because slag is the main source of Ca, which is the key element for the formation of C-A-S-H type gels [29]. With less slag, the formation of initial products would become slower, resulting in a shorter setting time. For S75-0 and S50-0, the setting time results are no longer valid due to the bleeding as discussed in the previous section.

For engineering practice, very fast setting is normally not desired. For example, BS EN 197-1 [30] requires a minimum time of 60 min for cements with strengths of 42.5 MPa. ASTM C 150-09 [31] prescribes a minimum time for the initial setting of 45 min. Limits on the final setting time are not common in the European or American standards [32]. Chinese standard GB 175-2007 [33] requires an initial setting time longer than 45 min and a final setting time shorter than 510 min. With reference to these standards, the initial set of mixtures S100-1.5, S75-1.5, S50-1.5 and S100-1.0 seems too fast. S50-0.5 is not preferable since its setting was too slow. S75-1.0, S50-1.0 and S75-0.5 are the mixtures that show both satisfactory flowability and setting time.

3.3. Strength

3.3.1. Compressive strength

The compressive strength of the pastes is shown in Fig. 8 (pure NaOH activated AASG mixtures were excluded based on the workability results). As expected [34], alkali-activated slag pastes show high compressive strength. The compressive strength of S100-1.5 exceeds 60 MPa within the first day of curing. Afterwards, the

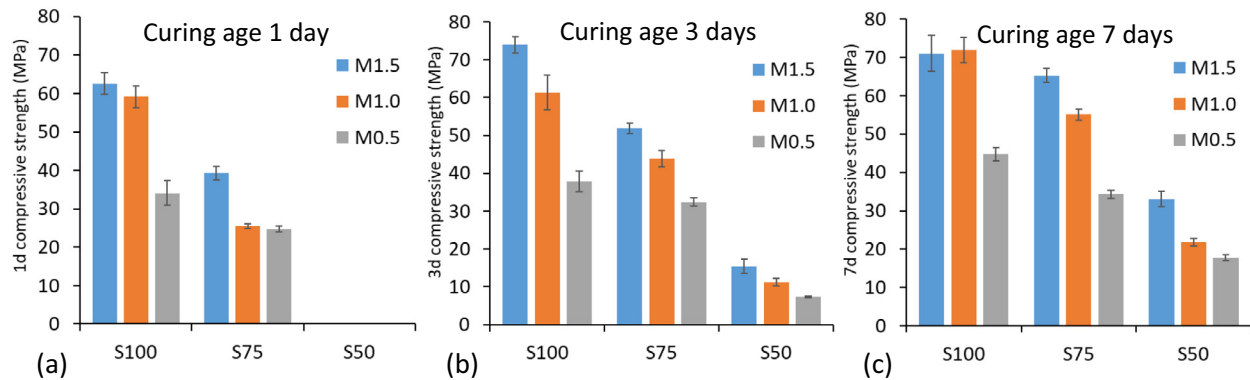


Fig. 8. Compressive strength of AASG pastes, cured for 1 day (a), 3 days (b) and 7 days (c). The label “M1.5” represents an activator modulus of 1.5.

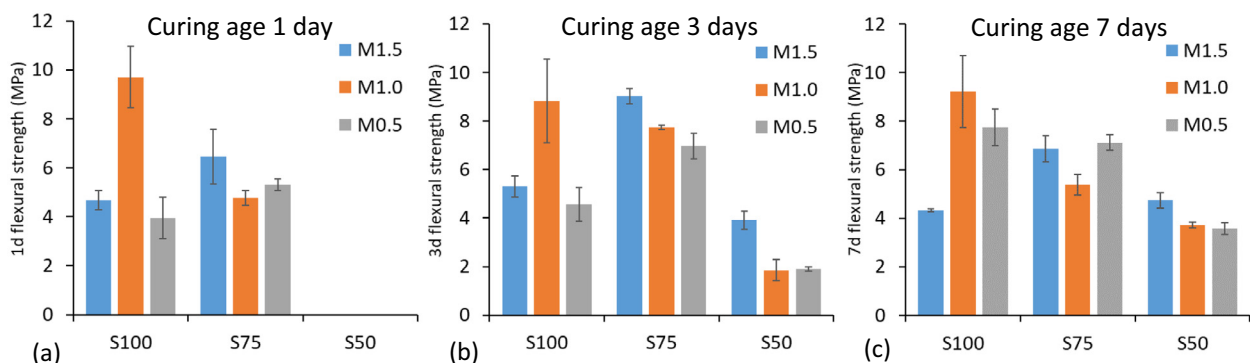


Fig. 9. Flexural strength of AASG pastes, cured for 1 day (a), 3 days (b) and 7 days (c). The code “M1.5” represents an activator modulus of 1.5.

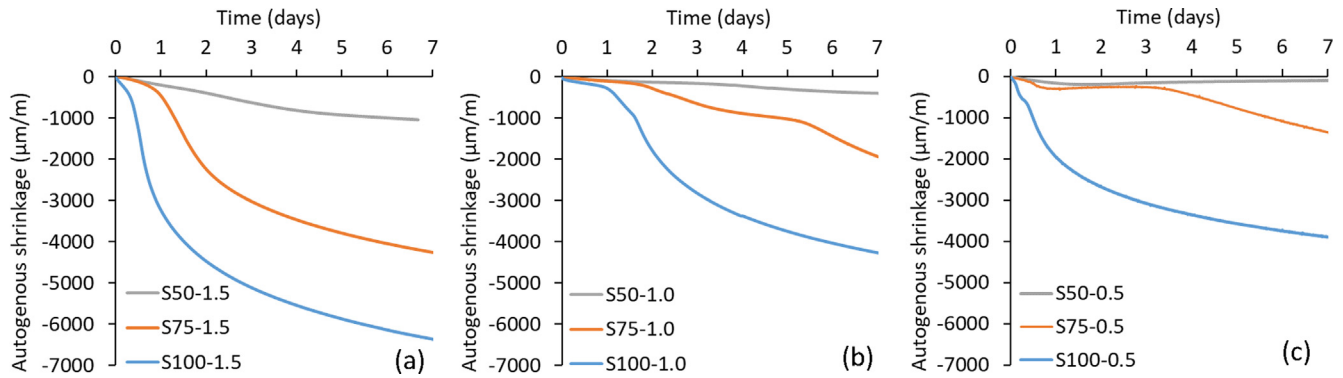


Fig. 10. Autogenous shrinkage of AASG pastes.

increment of compressive strength with time is limited. In general, lowering the modulus of the activator and increasing the content of glass wool in the binder both induce reductions in the compressive strength, irrespective of the curing age. This phenomenon is similar to what has been for alkali-activated slag blended with waste glass [11]. The lower compressive strength with the incorporation of glass wool is generally in line with the lower cumulative heat as shown in Fig. 7.

At the age of 1 day, S50 series of pastes remain weak and no strength is detected. With elapse of time, increments in compressive strength can be observed for S50 pastes. From engineering point of view, a minimum 1-day strength is normally required to ensure a smooth and timely demoulding. Therefore, a dosage of 50% and higher of glass wool is not recommended for AASG systems cured under ambient temperature.

3.3.2. Flexural strength

The flexural strength of the pastes is shown in Fig. 9. It can be observed that the mixtures that have higher compressive strength do not necessarily have higher flexural strength. For example, the flexural strength of S100-1.5, whose compressive strength is the highest among all mixtures, is lower than that of S100-1.0 in all the ages studied. S75-1.0 shows lower flexural strength than S75-0.5 at the age of 7 days (at 1 day, the flexural strength of S75-1.0 is also lower than that of S75-0.5, but the difference is of similar range to the error bar thus not considered). Moreover, several mixtures, such as S100-1.5, S75-1.5 and S75-1.0, even show decreasing flexural strength with time.

In fact, this is not the first time that a low or even decreasing flexural strength is reported for slag based systems activated by high-modulus activators [15,17,35]. Due to the presence of a large amount of silicate in the activator, a higher shrinkage is always generated by AAS paste [36,37]. During the curing under sealed

condition and the strength measurement afterwards under ambient relative humidity, autogenous shrinkage and drying shrinkage can develop, respectively. As a result of the drying shrinkage that occurred when the samples were exposed, samples probably displayed moisture gradient leading to development of eigenstresses, which could have influenced the flexural strength, although the exposure time was controlled to as short as possible. For the autogenous shrinkage that developed during curing, no moisture gradient would form in the sample, but the unreacted particles could act as local restraints of the shrinkage, leading to possible microcracking [38], which harms the flexural strength much more seriously than the compressive strength [39]. This hypothesis will be verified by the autogenous shrinkage results shown in the next section. For M0.5 series of mixture, by contrast, decreasing flexural strength is not observed.

According to the strength results, a modulus as high as 1.5 is not recommended for both pure slag and slag/glass wool blended systems, since it does not significantly improve the compressive strength but does reduce the flexural strength. In addition, the high price of Na_2SiO_3 solution also suggests the use of low-modulus activators. In fact, the early-age strengths of S100-0.5 and S75-0.5 are already sufficient for most structural uses as specified, for example, in the standard ACI 318 [40].

3.4. Autogenous shrinkage

The autogenous shrinkage of the pastes is shown in Fig. 10. S100-1.5 shows the highest autogenous shrinkage among the mix-

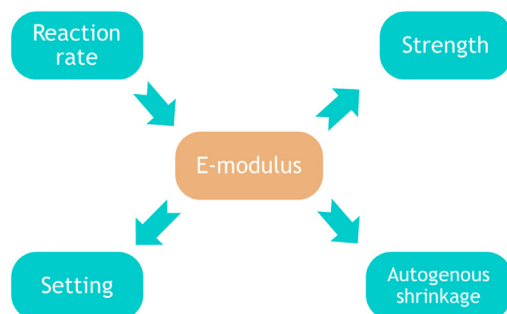


Fig. 11. Schematic demonstration of the relationships between elastic modulus and other properties.

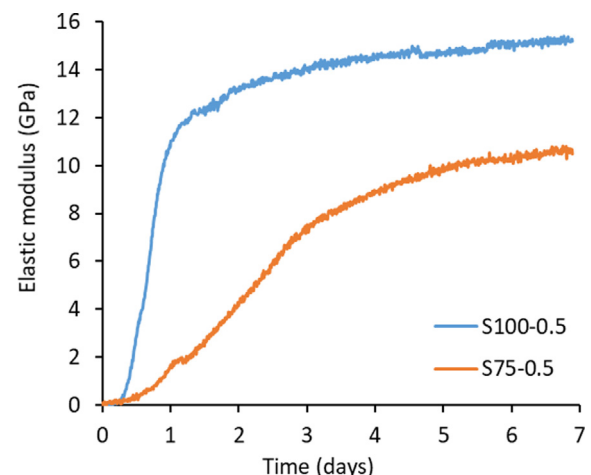


Fig. 12. Elastic modulus evolution of S100-0.5 and S75-0.5 paste in 7 days measured by EMM-ARM method.

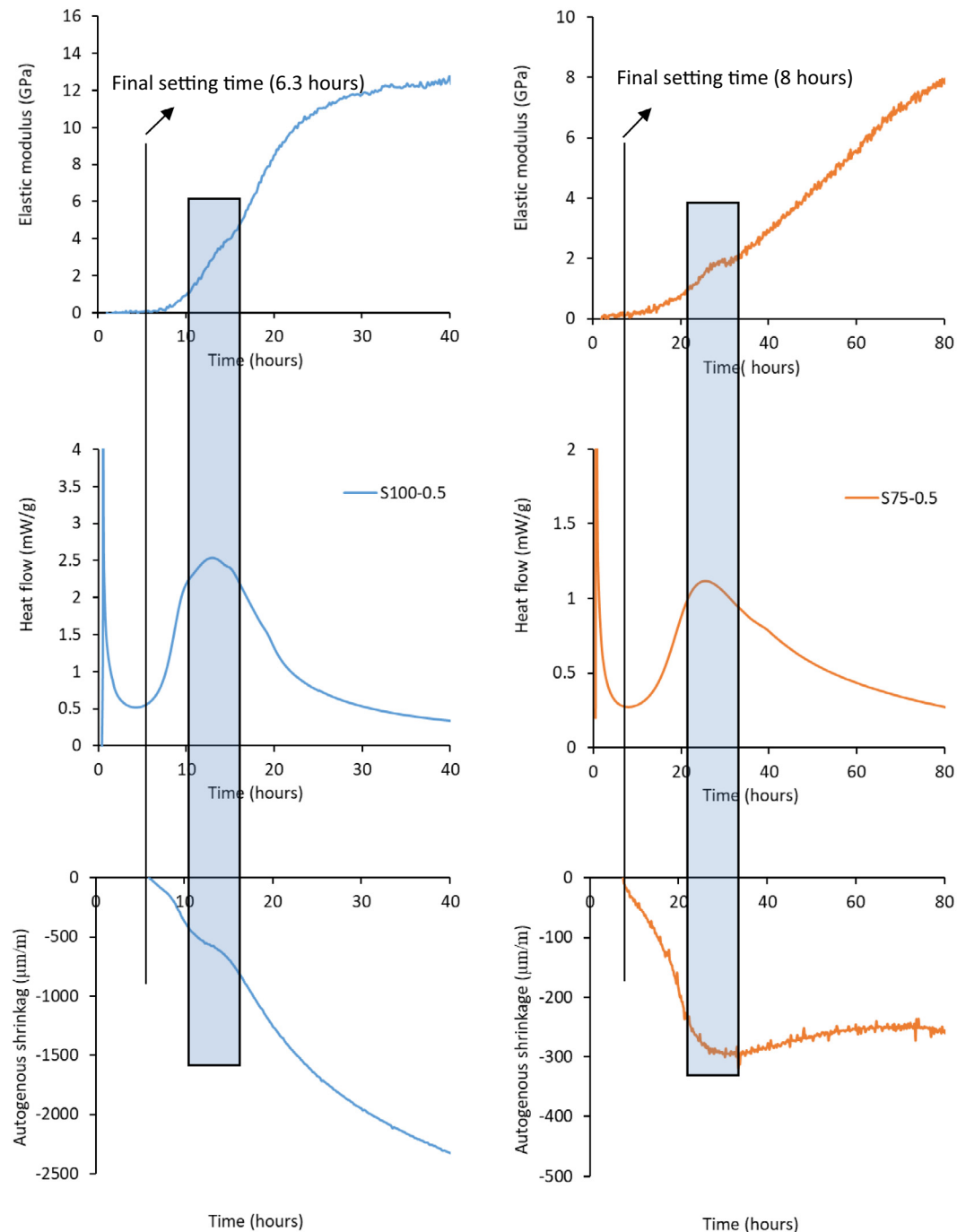


Fig. 13. Elastic modulus plotted together with final setting time, heat flow and autogenous shrinkage of S100-0.5 (left) and S75-0.5 (right) paste. The zero time of all curves was the starting time of casting. For the purpose of clarity, different scales were used for the plots of S100-0.5 and S75-0.5.

tures, which reaches 6500 $\mu\text{m/m}$ in 7 days. The autogenous shrinkage decreases with the decrease of modulus of the activator. This is consistent with results from literature [41,42]. The autogenous shrinkage of S100 series is much higher than that of OPC paste with or without SCMs [43,44].

It is also observed that the incorporation of glass wool led to a significant lower autogenous shrinkage of the paste.

For S75 mixtures, the autogenous shrinkage of S75-1.5 is 30% lower than that of S100-1.5 at the age of 7 days. This reduction can be partially due to the “diluting” of slag since the slag proportion is reduced with the presence of glass wool. However, for mod-

ulus 1.0 and 0.5 series, the autogenous shrinkage is substantially reduced, which can surely not be explained by just the “diluting” effect. Samples S75-1.0 and S75-0.5 even show a certain expansion at the age of 4 days and 2 days, respectively. Due to the expansion, the autogenous shrinkage of S75-0.5 remains lower than 300 $\mu\text{m/m}$ in the first 3 days. At later age, the shrinkage starts to develop rapidly. The expansion may be due to the formation of some crystalline phases, which is not evident in pure slag-based systems.

For S50 mixtures, the autogenous shrinkage is further reduced. For example, S50-0.5 shows a deformation close to zero in the first

week of curing. This is not because the reactions in S50-0.5 are retarded. As shown in Figs. 5, 8 and 9, S50-0.5 generate considerable reaction heat and show a certain strength after the first day. The limited deformations of S50-1.0 and S50-0.5 probably result from the competition between shrinkage and expansion. Future studies are needed to elucidate the mechanisms behind the mitigating effect of glass wool on the autogenous shrinkage of AASG pastes.

Based on the autogenous shrinkage results, moduli lower than 1.5 are suggested for AASG pastes. Pure slag systems are not preferred due to the high autogenous shrinkage.

In summary of the results shown above, pure slag-based systems show fast setting, low flexural strength and high autogenous shrinkage. Substitution of 50% slag by glass wool leads to poor flowability and limited early-age strength, irrespective of the activator composition. Hence, S100 and S50 series are not recommended for engineering applications. For S75 series, the moduli of 1.0 and 0.5 both yielded satisfactory setting time, workability, strength and autogenous shrinkage. Considering the comparable or even lower flexural strength, slightly higher autogenous shrinkage and potentially higher cost of S75-1.0, it appears that S75-0.5 is the optimal mixture among all the mixture studied.

In the next section, the elastic modulus evolution of S75-0.5 is measured for a further understanding of the early-age behaviours of this mixture. S100-0.5 is used as a reference.

3.5. Elastic modulus

Elastic modulus is not usually used as a criterion to evaluate the early-age performance of a paste, partially because the elastic modulus is difficult to measure. In fact, elastic modulus is an important parameter, whose evolution closely relates to other properties like reaction rate, setting, strength and shrinkage, as demonstrated in Fig. 11.

The evolutions of the elastic moduli of S100-0.5 and S75-0.5, measured by EMM-ARM method, are shown in Fig. 12. The elastic modulus of S100-0.5 develops faster than that of S75-0.5 in the first day. From 2 days to 7 days, the elastic modulus of S100-0.5 more or less stabilizes, while the one of S75-0.5 keeps increasing. Nonetheless, S100-0.5 has higher elastic modulus than S75-0.5 in the whole period considered. The magnitude of the elastic modulus of S100-0.5 is in similar range to those of the AAS pastes studied previously [45].

In Fig. 13, the evolution of elastic modulus in the very early age is plotted together with final setting time, reaction heat and autogenous shrinkage. It can be seen that the final setting time is approximately the moment when the elastic modulus of the paste start to increase for both mixtures. Besides, the final setting also roughly corresponds to the start of the acceleration period indicated by the heat flow. It should be noted that, for AAS systems with high Na_2SiO_3 content in the activator, the final setting time is normally faster (see Table 4) and can occur much before the end of the dormant period [27]. This is because the large amount of silicates available in the activator can immediately react with the ions dissolved from the precursors, precipitating metastable nuclei of new phases in the interstitial space, thus leading to a quick loss of flowability of the paste even if the majority of reaction products have not been formed and equilibrium within the pore solution has not reached yet [28]. For the mixtures considered here, S100-0.5 and S75-0.5, the modulus of the activator is low; therefore, fast setting is not shown.

Together with the increase of the heat release rate, the elastic modulus starts to develop rapidly. However, the developing rate of elastic modulus is not constant. A small hump (as marked by the rectangles) can be observed in the elastic modulus curves of S100-0.5 and S75-0.5 in the stage when the reaction heat reached

its peak, as shown in Fig. 13. This might indicate the formation of additional reaction products or some products that have high elastic modulus in this period. Meanwhile, the rate of autogenous shrinkage of the paste experiences a decrease. This decrease is certainly not due to slowdown of the reaction, as indicated by the high heat release rate in this period. The most probable reason can be the formation of some expansive phases (as discussed in Section 3.4), which compensates the shrinkage and affected the elastic modulus evolution. Future research is needed to characterize the reaction products formed in the acceleration period in these mixtures.

After the main reaction peak, the developing rate of elastic moduli of the mixtures decreases.

It is interesting to compare the elastic modulus/compressive strength (E/f_c) ratios of the two mixtures. As shown in Fig. 14, the E/f_c ratios of S100-0.5 fluctuate around 0.35 GPa/MPa in the period studied. By contrast, the E/f_c ratio of S75-0.5 is only 0.06 GPa/MPa at 1 day and increases with time. These results highlight the difference made by incorporation of glass wool on the mechanical behaviours of AAS. Nonetheless, the E/f_c ratios at 7 days of these two mixtures are similar.

3.6. Morphology

The morphology of the fracture surface of the optimal mixture S75-0.5 is shown in Fig. 15. It can be seen that the glass wool is well distributed in the matrix. However, there appears a certain extent of orientation of the wool. For example, most of the wool fibres in Fig. 15(a) are parallel to the broken surface while those in Fig. 15(c) are vertical. This is probably due to the large length of the wool as also presented in Fig. 1 and it was orientated during mixing. The micro- to macro cracks observed in the fracture surface are probably due to the freezing of the sample in nitrogen and the drying afterwards.

By comparing Figs. 15 and 1, it is found that the surface of the wool became smoother or cleaner after activation. This indicates that the organic resin on the wool is partly dissolvable under alkaline environment. However, it is seen that the wool fibres are not severely eroded, indicating a low reactivity of the wool. Reaction products can be observed forming onto the surface of the wool in Fig. 15(b) and (d), which contribute to the bonding between the wool and the matrix. Some holes can be observed on the surface due to the pulling out of the fibres during sample preparation, which indicates a reinforcing effect of the wool contributing to a higher early-age flexural strength of S75-0.5 than that of S100-

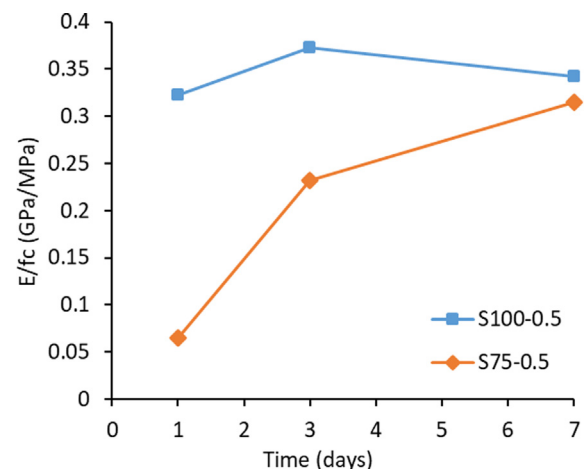


Fig. 14. Elastic modulus/compressive strength (E/f_c) ratios of S100-0.5 and S75-0.5.

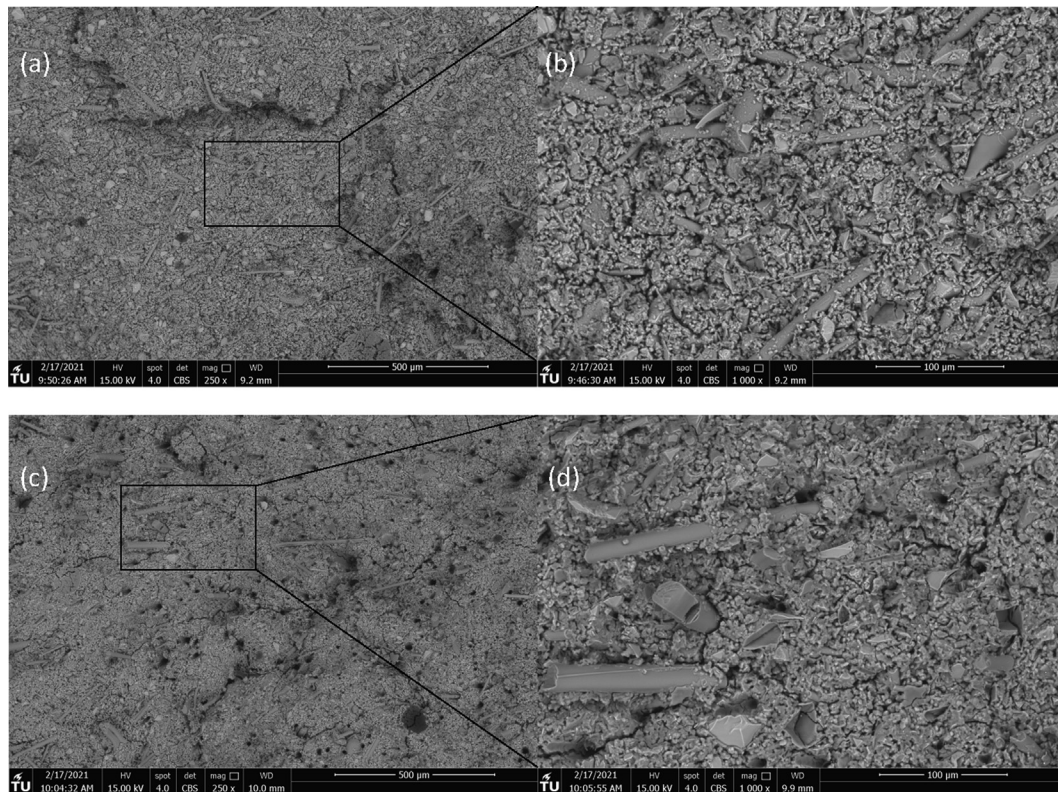


Fig. 15. SEM images of S75-0.5 cured for 1 day (a and b) and 7 days (c and d).

0.5 (see Fig. 9). Fig. 15(b) shows a dense microstructure of S75-0.5 paste with small capillary porosity. This is consistent with the high 1-day compressive strength (>20 MPa) of the paste as shown in Fig. 8. At 7 days, the morphology of S75-0.5 paste is not significantly different from that at 1 day nor from that of AAS paste without wool [15].

In this paper, the properties of alkali-activated slag and glass wool pastes are measured and discussed. The insights obtained on the role of glass wool in AAMs will be useful for the research and industry communities to recycle the glass wool waste. An optimal mixture is identified according to general engineering requirements, such as long enough setting time, high early-age strength and low autogenous shrinkage. For a specific application, other mixtures might be optimal. Tailored mixture design of both AAS and AASG can also be conducted based on the data provided in this paper.

4. Conclusions

In this study, the early-age properties of alkali-activated slag blended with waste glass wool are investigated. Mixtures with various activator compositions and glass wool dosages are studied. The main conclusions of this study can be drawn as follows:

1. The main reaction peak of AAS appears earlier and the intensity became higher with the decrease of modulus of the activator. A modulus of 0.5 is beneficial to reach a long enough setting time and a high reaction degree of alkali-activated slag paste. The incorporation of glass wool leads to a longer dormant period of the paste and a lower intensity of the main reaction peak.
2. Substitution of 25% slag by glass wool improves the flowability of the paste irrespective of the activator composition. However, the flowability decreases if higher amount of glass wool is

incorporated. NaOH-activated AASG pastes show bleeding. The setting time of paste increases with the content of glass wool in the binder.

3. Lowering the modulus of the activator and increasing the content of glass wool in the binder both induce reductions in the compressive strength, regardless of the curing age. However, a modulus as high as 1.5 impairs the flexural strength of the paste.
4. The autogenous shrinkage of AASG paste decreases when lower-modulus activator is used. The incorporation of glass wool has a significant mitigating effect on the autogenous shrinkage.
5. Considering all the early-age properties, S75-0.5 appears to be the optimal mixture, which shows satisfactory workability, high strength and low autogenous shrinkage. The elastic modulus of S75-0.5 is lower than that of S100-0.5. The elastic moduli of these mixtures start to develop at approximately the final setting time and the beginning time of the acceleration reaction period.

The results in this paper suggest that using waste glass wool, as a blend with slag into AAMs, is feasible and even recommendable, for not only recycling industrial waste, but also for improvements in early-age properties, such as prolonged setting time and reduced shrinkage.

CRediT authorship contribution statement

Zhenming Li: Conceptualization, Methodology, Investigation, Writing - original draft. **Irving Alfredo Flores Beltran:** Investigation. **Yun Chen:** Investigation. **Branko Šavija:** Writing - review & editing. **Guang Ye:** Project administration, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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