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A Comparative Study on Deflection-Hardening Behavior of Ductile Alkali-Activated Composite

Shizhe Zhang(✉), Marija Nedeljković, Bahman Ghiassi,
and Guang Ye

Microlab, Section of Materials and Environment,
Department of Structural Engineering, Faculty of Civil Engineering
and Geosciences, Delft University of Technology, Delft, The Netherlands
`Shizhe.Zhang@tudelft.nl`

Abstract. Alkali-activation technology as an environmental friendly approach to produce construction materials can lead to a great CO_2 emission reduction and provide high potential for waste reutilization. Alkali activated materials (AAMs) generally exhibit better durability and comparable or superior mechanical performance in comparison to traditional cementitious materials. However, like cementitious binders, AAMs are inherently brittle (quasi brittle). When it comes to engineering practices, the size effect also contributes to a more severe brittleness, which limits the applications in large scale structures. Fiber reinforcement is one of the solutions, which can be used to control the brittleness of AAMs. This paper presents a comparative study on the deflection-hardening behavior of ductile alkali-activated slag/fly ash (DAASF) composites reinforced with three types of synthetic fibers. All the developed DAASFs exhibited a deflection hardening with multiple cracking behavior under four-point bending tests. The influence of different fiber types, different slag/fly ash ratio as well as liquid/solid (L/S) ratio were investigated. The obtained results are a first step contribution to understanding the feasibility of using different fiber types in AAMs and development of mixture design of DAASF composites.

Keywords: Deflection-hardening · Alkali-activated materials · Fly ash · Slag

1 Introduction

Fast growing world population and its related industrialization have raised concerns about waste handling and their environmental consequences. Development of a circular economy for mineral waste materials within the Netherlands and the European Union (EU) has been found to be highly potential and economically profitable. Within the construction sector, the potential waste materials are promoted to be recycled and re-used as supplementary cementitious materials to bring greenness to ordinary Portland cement (OPC) concrete. On the other hand, the alkaline activation technology has emerged as an effective tool to reuse different wastes and industrial by-products to cementitious materials. Compared with traditional cementitious materials, alkali

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activated materials (AAMs) derived by the reaction of an alkali metal source (solid or dissolved) with a solid silicate powder (Shi 2006) are environmental friendly and need only moderate energy to produce. As one of the best alternatives for Portland cement, these materials maintain comparable and even better performance to traditional cementitious binders, such as higher compressive strength, better carbonation resistance, better Cl^- resistance, etc. They also can provide added advantage on greenhouse gas emission reduction. An 80% or greater reduction of greenhouse gases compared with OPC is achieved through alkali activation (Duxson et al. 2007).

However, like OPC concrete, AAMs as construction materials are also inherently brittle. To modify the brittle behavior of cementitious materials, fiber reinforced cementitious composites (FRCC) using either continuous or short fibers have been developed in the last decades (Naaman 2007). However, studies on the performance and properties of fiber-reinforced alkali activated materials are currently scarce compared to conventional FRCC.

This research work presents an experimental study on development of ductile alkali activated slag/fly ash (DAASF) composites. DAASF composites reinforced with three different types of short fibers are produced and their mechanical properties are compared. The influence of different slag/fly ash ratio, liquid to solid mass ratio (L/S) as well as type of alkali activator are also investigated and discussed.

2 Materials

The solid precursors used in this study were blast furnace slag and Class F fly ash. The chemical compositions deduced from X-ray Fluorescence and some properties of precursors (LOI at 950 °C and fineness passing 45 μm) are shown in Table 1.

Table 1. Chemical compositions and properties of raw materials.

Oxide (wt%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI	Fineness, % passing 45 μm
Slag	32.91	11.84	0.46	40.96	9.23	1.60	–	0.33	1.15	95
Fly ash	52.90	26.96	6.60	4.36	1.50	0.73	0.17	–	3.37	81

The crytallline phases were measured using powder X-ray diffraction (XRD). The XRD patterns of slag and fly ash are shown in Fig. 1. The crystalline phases in fly ash are mainly quartz, mullite and hematite, while the blast furnace slag contains mainly amorphous phases. All precursors contain considerable amounts of amorphous phases as can be reflected from the hump in the XRD patterns (from 17° to 35° for fly ash and from 25° to 35° for slag).

The alkaline activator was prepared by dissolving NaOH pellets (analytical grade, purity $\geq 98\%$) and sodium silicate (Na_2O: 8.25 wt%, SiO_2: 27.50 wt%) in distilled water. The activator was cooled down to room temperature prior to mixture preparation.

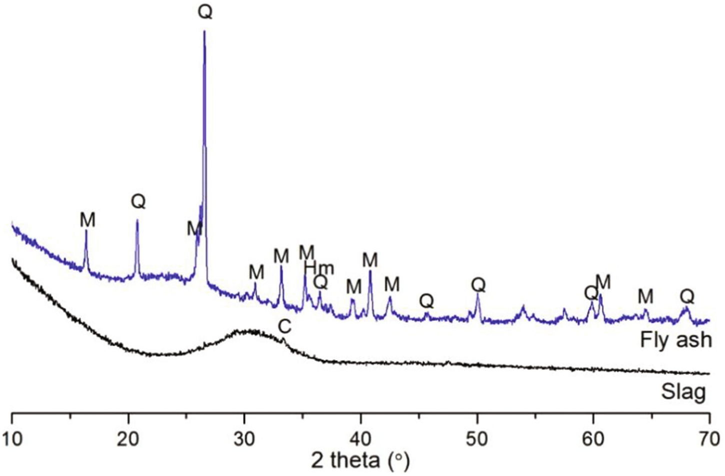


Fig. 1. XRD patterns of solid precursors.

Considering the high alkalinity of the alkali-activated slag/fly ash system, only three synthetic short cut fibers were used in this research, including polyvinyl alcohol (PVA), high tenacity polypropylene (HTPP) and high modulus polyethylene (HMPE). These fibers are all resistant to high alkaline conditions and can therefore be used in AAMs composites. Their properties given by supplier is presented in Table 2.

Table 2. Properties of selected fibers.

Fiber	Diameter (μm)	Density (g/cm^3)	Length (mm)	Strength (MPa)	Young's modulus (GPa)	Producer
PVA	39	1.30	8	1640	41.1	Kuraray
HTPP	10	1.00	6	819	12.7	Redco
HMPE	20	0.97	12	3400	113.0	DSM

3 Methods

3.1 Mix Design

Three slag/fly ash ratio (S10, S30 and S50), three liquid to solid mass (L/S) ratio (0.4, 0.42 and 0.46) and three types of fibers (PVA, HTPP and HMPE) were considered for preparation of trial mixes. The detailed mixture designs are shown in Table 3. The mixtures were named with three identifiers (XXX-Y-ZZZ). The first number represents the slag content of the solid precursors used, the second number represents the L/S ratio and the third represents the fiber type. For instance, S10-0.42-PVA means that from all the solid precursors used, the slag content is 10 wt% (and the rest is fly ash), the L/S ratio is 0.42 and the mixture is reinforced with 2% PVA fiber by volume. The fiber volume in all mixes was similar.

Table 3. Mixture design of ductile alkali-activated slag/fly ash.

Mixture	Slag (wt%)	Fly ash (wt%)	L/S ratio	Alkali activator	Fiber (Vol.%)
S10-0.42-PVA	10	90	0.42	NaOH + Water glass	2%
S30-0.42-PVA	30	70			
S50-0.42-PVA	50	50			
S30-0.42-HTPP	30	70			
S30-0.42-HMPE					
S30-0.4-PVA			0.4		
S30-0.46-PVA			0.46		

The raw materials were first dry mixed for 5 min using a HOBART® mixer at low speed. Activating solution was then gradually added to the raw materials and mixing continued for another 5 min at middle speed. Afterwards, the fibers were added and mixed for additional 5 min to achieve a homogeneous fiber distribution. The mixtures were cast in couple sample molds of 10 × 60 × 240 mm^3. The fresh mixtures were then put on vibration table to remove entrapped air. Afterwards, the molds were sealed with plastic foam for curing. The samples were demolded after 24 h and transferred to a climate room (20 °C and 95% RH) until the testing age of 28 days.

3.2 Test Methods

The compressive strength of the samples (at 1, 7, 28 and 90 days) was measured in accordance with EN-196-1 (EN 2005). Four-point bending tests were conducted on samples with dimensions of 10 × 30 × 120 mm^3 after 28 days curing. The support span was 110 mm and the load span was 30 mm as shown in Fig. 2. The deflection of the specimens was measured by two LVDTs located at the center on both sides. The tests were performed on displacement controlled condition with loading speed of 0.01 mm/s. Three specimens were tested for each mixture.

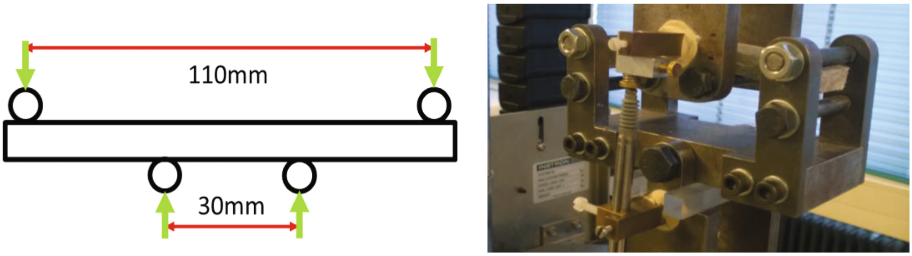


Fig. 2. Four-point bending test set-up.

4 Results and Discussions

4.1 Compressive Strength

The compressive strength development up to 90 days of plain S30 paste with L/S ratio 0.4, 0.42 and 0.5 is summarized in Fig. 3. The mixture with L/S ratio 0.4 shows the highest compressive strength at 90 d. On the other hand, excessive amount of alkali activator (L/S ratio 0.5) lead to lower 90 d compressive strength, which may be due to the effect of high liquid content which limits the development of a dense microstructure. Previous studies on alkali-activated fly ash system modified with various slag content shows that alkali activation of slag mainly contributes to the gel formation and compressive strength development under room temperature (Kumar et al. 2010). An optimum combination of alkali activator with suitable L/S ratio can result in an improved compressive strength. S30 paste with 0.42 L/S ratio exhibits the highest 7 d compressive strength, which is due to the optimization of the fresh state properties and the enhanced reaction kinetics due to the more available alkali and silica from the alkali activator. All mixtures show compressive strength higher than 49 MPa, which can fulfill the engineering requirements for most projects.

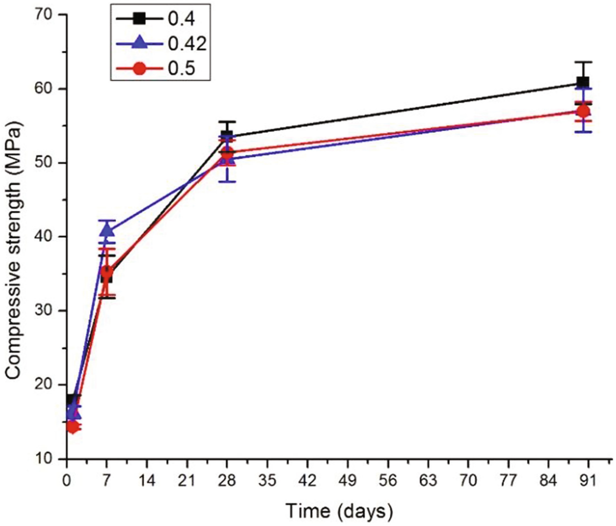


Fig. 3. Compressive strength development of alkali-activated slag/fly ash paste.

All mixtures gained at least 88% compressive strength with respect to 90 d compressive strength, which is assumed to be quite similar to the mixture final strength. Therefore, the testing age of other mechanical properties such as flexural strength, deflection hardening capacity of the ductile alkali-activated slag/fly ash system is set at 28 d.

4.2 Deflection-Hardening Behavior

Under four-point bending load, all the mixtures exhibited multiple-cracking as shown in Fig. 4. A set of representative flexural stress-deflection curves of three DAASF reinforced with three different fibers are shown in Fig. 5, all of which indicate deflection-hardening behaviors. In the flexural stress-deflection curves, the maximum flexural stress is defined as the flexural strength, and the corresponding deflection is defined as the flexural deflection capacity (Fig. 5(a)). The flexural strength and deflection capacity were calculated taking the average results of the three measurements in the four-point bending tests.

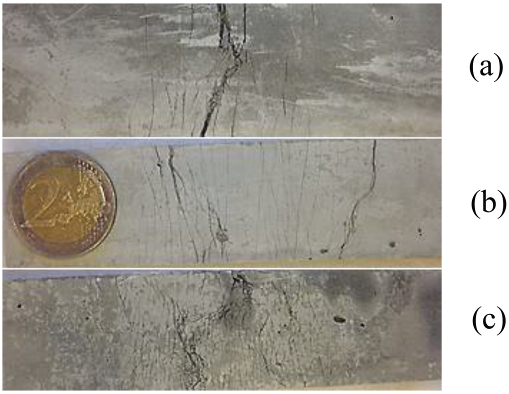


Fig. 4. Multiple cracking of the specimens under four-point bending load: (a) S30-0.42-PVA; (b) S30-0.42-HTPP and (c) S30-0.42-HMPE.

The influence of fiber type, slag/fly ash ratio and L/S ratio is studied and the calculated flexural strength and corresponding deflection capacity are compared in Fig. 6. DAASF reinforced with HMPE fiber shows the highest flexural strength and deflection capacity, followed by mixture with HTPP fiber and then mixture with PVA fiber. As the matrix and fiber volume were kept the same, the different flexural behavior of three mixtures should be due to the fiber and the fiber/matrix interface properties. Compared with PVA fiber, the HMPE fiber has higher tensile strength and Young's modulus which are beneficial for the deflection-hardening behavior. On the other hand, the calcite coating on the HTPP fiber along with its high tensile strength can be the reason for the observed increase in flexural strength and deflection capacity.

From Fig. 6(b), it can be observed that with lowering the slag content from 50% to 30% a higher potential for the strain-hardening behavior with even flexural strength and deflection capacity is obtained. The flexural strength largely decreases when the slag content is reduced to 10% (S10). At the same time, however, the deflection capacity is increasing. This may due to the decrease of the matrix fracture toughness which is beneficial for the ductile or even strain hardening behavior of the material (Nematollahi et al. 2014).

The L/S ratio also have a very large influence on the flexural strength. From Fig. 6(c), it can be observed that an L/S ratio larger than 0.42, leads to a large decrease in the

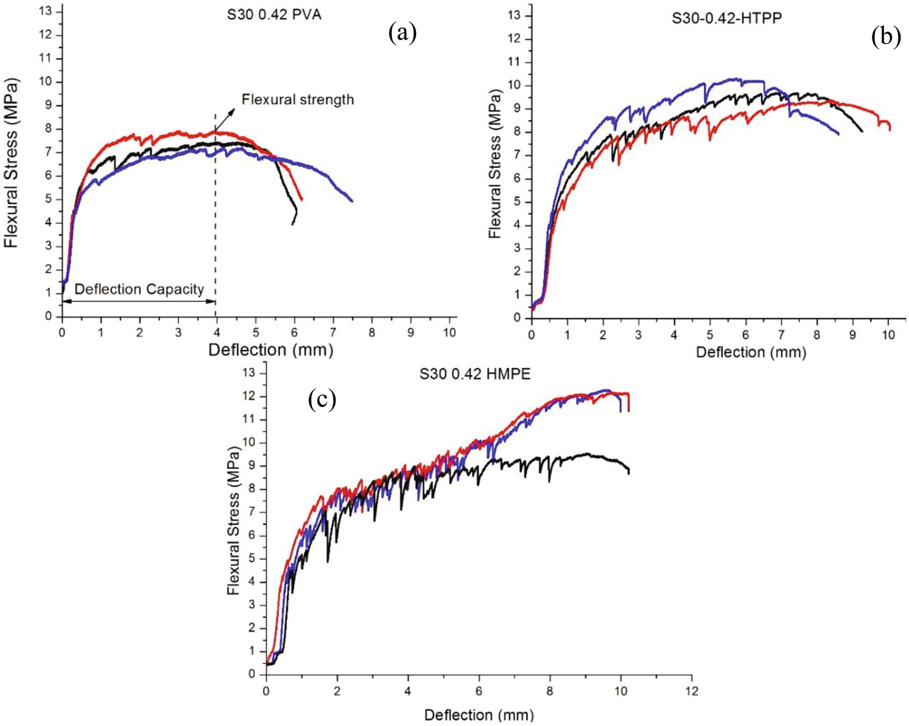
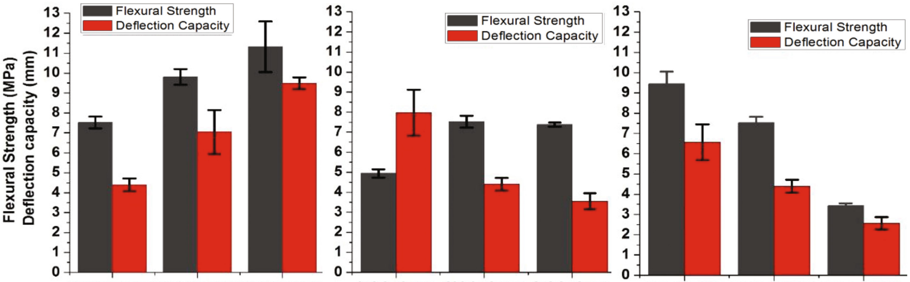


Fig. 5. Flexural stress-deflection curves of ductile alkali-activated slag/fly ash system (a) S30-0.42-PVA; (b) S30-0.42-HTPP and (c) S30-0.42-HMPE.



(a) **Fiber type** (b) **Slag:Fly ash** (c) **L/S ratio**

Fig. 6. Flexural strength and deflection capacity of ductile alkali-activated slag/fly ash considering: (a) fiber type (b) slag/Fly ash ratio and (c) L/S ratio.

flexural strength and deflection capacity. This decrement of flexural strength is in agreement with the compressive strength values at 28 d. It is believed that the change in L/S ratio largely influences the developed microstructure which can affect the bonding between fiber and matrix. Excessive L/S ratio leads to a porous microstructure and will decrease the bonding energy of the PVA fiber to the matrix, which negatively impacts the flexural behavior of the mixture.

5 Conclusions

All DAASF composites reinforced with synthetic PVA, HTPP and HMPE fibers developed in this study exhibited a deflection hardening with multiple cracking behavior under four-point bending tests. Based on the experimental results, DAASF with HMPE fiber in S30 matrix has the highest flexural strength and deflection capacity. With 0.42 L/S ratio, S30 DAASF shows higher flexural strength and deflection capacity compared with S10 and S50 DAASF. With S30 DAASF, L/S ratio larger than 0.42 leads to a large decrease in the flexural strength and deflection capacity. These findings are a first step contributing to understanding the feasibility of using different fibers in AAMs as well as the mixture design of DAASF.

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