Research Article

Pottery Sand as Fine Aggregate for Preparing Alkali-Activated Slag Mortar

Zhenzhen Jiao^{1,2,3} , Ying Wang^{1,2,3}, Wenzhong Zheng^{1,2,3} and Wenxuan Huang^{1,2,3}

¹Key Lab of Structures Dynamic Behavior and Control of the Ministry of Education, Harbin Institute of Technology, Harbin 150090, China

²Key Lab of Smart Prevention and Mitigation of Civil Engineering Disasters of the Ministry of Industry and Information Technology, Harbin Institute of Technology, Harbin 150090, China

³School of Civil Engineering, Harbin Institute of Technology, Harbin 150090, China

Correspondence should be addressed to Zhenzhen Jiao; jiaozhenzhen@hit.edu.cn

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Alkali-activated slag (AAS) mortars were prepared using pottery sand as a fine aggregate in a ratio of 1 : 1.75 using a blend of sodium silicate and NaOH as an alkaline activator at room temperature. The effects of sodium oxide content and silicate moduli on the setting time, fluidity, consistency, compressive strength, and drying shrinkage of different AAS mortars were determined. These results revealed that sodium oxide content and silicate modulus had little effect on the setting time and workability of the mortar; however, they did have a significant effect on their mechanical performance and drying shrinkage levels. All the AAS mortars exhibited faster setting times, better workability, and higher early and late compressive strength compared to traditional mortars. Optimum compressive strength was achieved at 93 and 123 MPa after 1 d and 28 d, respectively, using a silicate modulus of 1.2 and Na₂O content of 8%. The microstructures of mortars were characterized using scanning electron microscopy with energy dispersive spectrometric (SEM/EDS) and mercury intrusion porosimetry (MIP). These results reveal that AAS mortars containing pottery sand as a fine aggregate may represent a promising building material with improved properties for use in the construction industry.

1. Introduction

Blast furnace slag is a solid waste product from the steel making process that is produced by rapid cooling of molten slag at the furnace exit [1]. Therefore, its transformation into building materials that can be used by the construction industry represents an environmentally friendly and economically useful use for this waste material. Furthermore, blast furnace slag has often been used in the construction industry as an alternative mineral admix to cement for incorporation into concrete materials [2]. However, current processes only employ a small amount of the slag that is potentially available for construction applications, with its utility for building applications currently underexploited [2, 3]. Blending slag with an alkaline activator has previously been shown to afford cementitious materials with excellent potential for construction applications [4–6]. This is because the alkali activation process affords slag-derived

mortars with improved strength and decreased setting times when compared to more conventional mortars. Consequently, the use of AAS (alkali-activated slag) processes for the preparation of cementitious materials has gained in popularity, primarily due to lower CO₂ emissions in its manufacturing process and the improved performance properties of the mortars that it produces [7]. Other significant advantages that AAS mortars have over Portland cement include better mechanical properties, superior frost resistance, lower porosity, and lower permeability to water. However, AAS mortars also have some disadvantages, including faster setting properties and higher levels of mass loss and drying shrinkage during their curing phases.

A range of protocols for the preparation of AAS mortars have been reported by a number of different research groups. Their studies have shown that the type of aggregate employed for the preparation of AAS mortar plays a significant role in determining its physical and hardening

TABLE 1: Chemical composition (%) of slag and pottery sand.

	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	Na ₂ O	SO ₃	Others
Slag	32.83	17.19	36.69	0.38	0.37	8.20	0.65	1.94	1.75
Pottery sand	62.12	16.32	3.26	7.84	1.25	2.04	1.97	—	5.2

(a)

(b)

FIGURE 1: The cementitious material and fine aggregate. (a) Blast furnace slag. (b) Pottery sand.

properties. Many researchers [8–11] have investigated the role of recycled by-products as fine aggregates in AAS processes, including the use of granulated blast furnace slag, limestone aggregate, ladle slag, and waste glass. For example, Elibol and Sengul [12] investigated the use of natural sand and ferrochrome slag as a fine aggregate for the preparation of AAS mortars at 80°C for 28 d, which gave AAS mortars with highest compressive strengths of 51.3 and 43.5 MPa, respectively. However, this process is economically unfeasible for industrial processes due to the high temperatures and extended times required during their curing phase. Alternatively, Aydin and Baradan [3] reported that AAS mortars containing crushed limestone sand exhibited high compressive strengths of 22.6 MPa after 2 d and 99.0 MPa after 90 d. Similarly, Zuda et al. [13] employed recycled electrical porcelain as a fine aggregate in an attempt to improve the high-temperature performance of AAS mortars, which gave flexural and compressive strengths of only 3.3 MPa and 22.6 MPa at 25°C, respectively. Huiskes et al. [14] have also described that an ultra-lightweight concrete manufactured from recycled glass aggregate demonstrates good mechanical performance and durability. However, no studies describing the use of readily available pottery sand (easily prepared from fly ash and bauxite) as a fine aggregate for the preparation of AAS mortars had been described. Consequently, we now report our investigation into the use of pottery sand as an aggregate material for the preparation of AAS mortar materials and demonstrate that they exhibit excellent setting, shrinkage, and hardness properties that make these mortars a potential useful building material for applications in the construction industry.

2. Materials and Experimental Methods

2.1. Materials. Blast furnace slag was obtained from the Tangshan steel plant in China, and the slag was classified as S95 according to GB/T 18046-2008, with a specific surface area of 424 m²/kg. Pottery sand was prepared from fly ash

and bauxite calcining with the high temperature of 1200–1600°C and used as a fine aggregate with a particle size ≤ 1 mm, a specific gravity of 1.8, and a bulk density of 1.2 g/cm³. The chemical composition of the slag and pottery sand is shown in Table 1. Images of the blast furnace slag and pottery sand are shown in Figure 1. The alkaline activator used for the AAS mortar was prepared using water glass and sodium hydroxide. The water glass used in this study was supplied by the Julide Chemical Co., Langfang, China, which contained 10.3%, 32.1%, and 57.6% of Na₂O, SiO₂, and H₂O, respectively. NaOH pellets with a purity of 96% were supplied by Dalu Chemical Reagent Factory, Tianjin, China.

2.2. Mortar Preparation. Three different silicate moduli (1.2, 1.4, and 1.6) and Na₂O contents (6%, 8%, and 10%) were used to prepare the alkali activator. The Na₂O contents in this study refer to 6%, 8%, and 10% Na₂O with respect to the weight of slag. The water to slag ratio of the AAS mortar was 0.35 by mass, using slag to aggregate ratios of 1/1, 1/1.25, 1/1.5, 1/1.75, and 1/2 by weight. Table 2 shows the mixing proportions used for preparing AAS mortars. AAS mortars were prepared by mixing the slag and pottery sand together for 5 min, before the alkaline activator was added and the resultant slurry stirred for 1 min at a slow rate of 200 $\pm$ 5 rpm, followed by stirring for 1 min at a faster rate of 400 $\pm$ 10 rpm.

2.3. Methods. Fresh mortar, containing different mixtures of slag, silicate moduli, and Na₂O, were cast into cubic (70.7 mm $\times$ 70.7 mm $\times$ 70.7 mm) and prismatic (25 mm $\times$ 25 mm $\times$ 280 mm) moulds that were then subjected to compressive strength and drying shrinkage tests. Three duplicate samples were analyzed for each mixing proportion, with samples being stored in sealed containers covered with plastic foils to avoid water loss for 24 h at 20°C. The samples used for compressive strength tests were demoulded and cured in the standard room (20 $\pm$ 2°C and RH > 95%) and tested using an electrohydraulic machine at 1, 3, 7, 14,

TABLE 2: The mixing proportions used in this study.

Number of mix	Nomenclature ^{1,2,3,4}	Silicate modulus	Na ₂ O content (%)	Slag to aggregate ratio	Water to slag ratio
1	M1.2N6M	1.2	6	1/1.75	0.35
2	M1.2N8M (SA1/1.75M)	1.2	8	1/1.75	0.35
3	M1.2N10M	1.2	10	1/1.75	0.35
4	M1.4N6M	1.4	6	1/1.75	0.35
5	M1.4N8M	1.4	8	1/1.75	0.35
6	M1.4N10M	1.4	10	1/1.75	0.35
7	M1.6N6M	1.6	6	1/1.75	0.35
8	M1.6N8M	1.6	8	1/1.75	0.35
9	M1.6N10M	1.6	10	1/1.75	0.35
10	SA1/1M	1.2	8	1/1	0.35
11	SA1/1.25M	1.2	8	1/1.25	0.35
12	SA1/1.5M	1.2	8	1/1.5	0.35
13	SA1/2M	1.2	8	1/2	0.35

The first M = silicate modulus; N = Na₂O content; the last M = mortar; SA = slag to aggregate ratio.

and 28 days, respectively. The samples used for drying shrinkage/mass loss tests were stored in an isothermal chamber ($20 \pm 2^\circ\text{C}$ and $\text{RH} = (50 \pm 5)\%$), with drying shrinkage levels determined using a micrometer over 28 days. Mass loss throughout the curing process was monitored using an electronic balance in accordance with JGJ/T 70-2009. The cross-sectional area of the stainless-steel needle of the measuring instrument used to determine mortar setting times was 30 mm^2 . Setting times were specified as the time taken from addition of the activator to the raw starting materials to the point where penetration resistance of the mortar reached 15 N. Fluidity tests were performed using a jump table according to GB/T 2419-2005. Fresh mixtures were poured into truncated conical moulds that were then lifted into a vertical position, with the instrument then run through 25 cycles. The fluidity of each mortar was determined by measuring the average diameters of each sample along two perpendicular directions, with consistency measurements determined according to JGJ/T 70-2009. The average values of two tests were determined, with consistency and error limits between repeat samples found to be $<10 \text{ mm}$. The morphologies of the microstructures of samples were determined using SEM-EDS using a ZEISS MERLIN Compact FE-SEM, Germany. The pore structures and porosities of each specimen were determined using Autopore IV 9500, Micromeritics Instrument Ltd., America.

3. Results

The aim of this study was to identify an optimal process for incorporating pottery sand into AAS mortars that would display good fluidity, consistency, compressive strength, and drying shrinkage properties, with the microstructures of promising mortars then being explored using SEM-EDS and MIP analysis.

3.1. Effect of Slag to Aggregate Ratios on Composition of Mortars. We initially investigated the effect of slag to aggregate ratios on the setting time, fluidity, and consistency of alkali-activated slag-derived mortars. Mortars were prepared using a water-to-slag ratio of 0.35, using an alkali activator

FIGURE 2: The effect of slag to aggregate ratios on the setting time of AAS mortars.

with a silicate modulus of 1.2 and a Na₂O content of 8%. The setting times observed for the AAS mortars ranged from 12 to 20 min, with incorporation of larger amounts of pottery sand resulting in decreased mortar setting times (Figure 2). The fluidity and consistency values of the AAS mortars were also found to increase as the slag to aggregate ratio increased (Figure 3). For example, when the slag to aggregate ratio was increased from 1/2 to 1/1, the fluidity and consistency values of the mortar increased from 4.8 to 51.0% and 6.4 to 50.0%, respectively. These values suggest that the use of relatively high slag to aggregate ratios can be used to improve the workability of these AAS mortars. These studies enabled us to determine that a slag-to-aggregate ratio of 1/1.75 was the optimum value for preparing mortar for construction applications.

3.2. Setting Times of AAS Mortars. The setting times of AAS mortars increased as their Na₂O content increased, with setting times of 12.6, 15.5, and 24.7 min for 6%, 8%, and 10% Na₂O content, respectively (Figure 4). Moreover, the setting

FIGURE 3: The effect of slag to aggregate ratios on the fluidity and consistency of AAS mortars.

FIGURE 4: The effect of Na_2O contents on the setting time of AAS mortars.

times of mortar were found to only increase slightly with increasing silicate modulus (Figure 5), indicating that this parameter was less influential than Na_2O content. Gao et al. [15] indicated that the short setting time was due to the higher reactivity of slag in the high pH environment. In effect, more Ca, Si, and Al were dissolved by the slag which can effectively promote the formation of the hydration products; therefore, the condensation process was very fast.

3.3. Fluidity and Consistency of AAS Mortars. The fluidity and consistency of AAS mortars was found to increase as the silicate modulus increased (Figure 6). The slightly higher fluidity and consistency values were obtained using a Na_2O content of 8%, with silicate modulus playing an important role in determining the fluidity and consistency. Therefore, the fluidity of the mortar was found to increase

FIGURE 5: The effect of the silicate moduli on the setting time of AAS mortars.

FIGURE 6: The effects of silicate moduli and Na_2O contents on the fluidity and consistency of AAS mortars.

from 15.9 to 29.1% as the silicate modulus increased from 1.2 to 1.6.

3.4. Compressive Strength. It was found that AAS mortars containing a slag-to-aggregate ratio of 1/1.75 containing 8% Na_2O exhibited maximal compressive strength (Figure 7), with mortars containing silicate modulus of 1.2, 1.4, and 1.6 exhibiting strengths of 123.2 MPa, 113.2 MPa, and 118.2 MPa after 28 d, respectively. Mortars containing an optimum silicate modulus of 1.2 afforded comparatively high compressive strength values of 81.4–93.2 MPa after only 1 d at room temperature. We propose that this high early compressive strength is mainly due to rapid formation of the C–S–H gel phase that exhibits a more condensed structure in the early stages of curing [3, 16].

FIGURE 7: The effects of silicate moduli and Na_2O contents on the compressive strength of AAS mortars.

FIGURE 8: The effects of silicate moduli and Na_2O contents on the drying shrinkage of AAS mortars.

3.5. Drying Shrinkage and Mass Loss of AAS Mortars during the Drying Process. Shrinkage of mortar in the drying process occurs because water molecules continuously evaporate from their surfaces, or through their pore structures [17]. The structure of the AAS mortars was observed over a period of 28 d, with visual inspection revealing no obvious cracking of their external surfaces (Figure 8). It was found that drying shrinkage levels increased as both the Na_2O content and the silicate moduli levels increased. For example, increasing the Na_2O content from 6% to 10%, using a silicate modulus of 1.6 resulted in an increase in drying shrinkage of the AAS mortars from 0.32% to 0.39% at

FIGURE 9: The effect of silicate moduli and Na_2O contents on the mass loss of AAS mortars.

28 d. They are also in agreement with work reported by Aydin and Baradan [3], Duran Atiş et al. [18], and others [18, 19] who also reported increased drying shrinkage levels when the amount of Na_2O present in their mortars was increased. Mass losses from mortars were increased with decreasing Na_2O content and increasing silicate modulus levels (Figure 9). These results are consistent with the results reported by Thomas et al. [20], who also found that the rate of water loss from mortars increases as their Na_2O content decreased.

3.6. Characterization of Mortars Using Scanning Electron Microscopy with Energy Dispersive Spectrometry (SEM/EDS) and Mercury Intrusion Porosimetry (MIP). SEM/EDS images of AAS mortars containing silicate modulus of 1.2 and Na_2O content of 8% at 1, 3, 7, and 28 d are shown in Figure 10. These images reveal significant changes in the interfacial transition zone of the hardened AAS mortars over time, with their microstructures becoming gradually denser as curing times increase. Additionally, mortars displaying more microcracks in their interfacial transition zones are visible after 1 d, with fewer microcracks in their paste and interfacial transition zones being present after longer curing periods. These changes in structural morphology were mirrored by an increase in the compressive strength of AAS mortars over time (*vide supra*), which is consistent with strong interactions between the aggregate and paste playing an important role in providing compressive strength to the mortars [21]. It is better than the results by Aydin and Baradan who have also reported that microcracks were present in the interfacial aggregate-paste zones of their AAS mortars after 28 d [3].

The AAS mortars showed a clear demarcation at their aggregate and paste interface before 3 d, with no distinction being visible after 7 d. EDS analysis revealed that the

FIGURE 10: SEM/EDS of AAS mortars incorporated 8% Na_2O and $M_s = 1.2$ at different curing times: (a) 1 d; (b) 3 d; (c) 7 d; (d) 28 d.

main elements present in the AAS mortars were sodium, aluminum, silica, and calcium, with a Ca/Si ratio of 0.60, 0.97, 0.89, and 0.68 after 1, 3, 7, and 28 d, respectively. EDS analyses also revealed that the C-S-H gel phase contained

elemental Na, with the Na/Si ratio found to be in a range from 0.63 to 1.90, depending on the age of the sample. The Ca/Si ratio of the AAS mortars was lower than those reported previously by Aydin and Baradan [3] for their

FIGURE 11: The pore size distribution of AAS mortars with different Na₂O contents: (a) cumulative intrusion curves; (b) differential intrusion curves; (c) porosity.

mortars, whilst both the Ca/Si and the Na/Si ratio of the mortars were lower than those for mortars prepared by Gebregziabihier et al. [22]. They obtained that higher Ca/Si and Na/Si ratios represent the calcium and sodium-rich products on the surface of slag grains and the formation was denser.

Previous studies have reported that the size of the pore structure of the alkali-activated cement plays a critical role in determining the physical properties of AAS mortars [23, 24]. The effects of Na₂O content and silicate modulus on the pore structure of AAS mortars were studied (Figures 11 and 12). Analysis of the MIP results revealed the presence of peaks corresponding to pore size of 200 nm diameter, whose

intensities were similar for all mortars tested. However, peaks corresponding to the presence of pores with a diameter between 500 nm and 3000 nm were significantly increased in mortars containing high silicate modulus levels. These MIP data also indicate that increasing Na₂O content gave an increase in their mesoporous volume (pore size diameter <50 nm), with their overall pore volume remaining essentially constant (Figures 11(a) and 11(c)). Moreover, the total porosities of AAS mortars increased as the silicate modulus increased (Figure 12). Additionally, the mesopore volume increased with an increase in silicate modulus, which also can explain the drying shrinkage increase as the silicate modulus increased (mentioned above in Section 3.5).

FIGURE 12: The pore size distribution of AAS mortars with different silicate moduli: (a) cumulative intrusion curves; (b) differential intrusion curves; (c) porosity.

4. Discussion

The composition of blast furnace slag has a significant effect on the physical properties of AAS mortars; therefore, it was necessary to optimize the alkaline activator conditions used for their preparation. A setting time of less than 30 minutes was targeted to match the specification required for quick repair cement [3], with setting times of 12–25 min observed for all pottery sand-containing AAS mortars tested in this study. JC 860-2008 specifications require that the consistency requirements for masonry mortar are between 50 and 80 mm, with the AAS mortars that were prepared using a silicate modulus of 1.2 fully meeting these requirements.

There are differences in the fine aggregate types that have been incorporated into AAS mortars, and the

aggregate type can thus affect the compressive strength. Optimization of the silicate modulus (1.2) and Na_2O (8%) content gave AAS mortars with pottery sand that exhibited impressive maximal compressive strength levels of 93 and 123.2 MPa after 1 d and 28 d, respectively. These compressive strength values compare favorably with those reported previously for mortars prepared from limestone and river sand aggregates, both of which gave lower values of ≤ 85 MPa at 28 d [3, 25].

The relatively high drying shrinkage levels observed for the mortars were also impressive, which is likely to be due to the fine capillary nature of their mesoporous microstructures [26]. There was the strong relationship between drying shrinkage and mesopore volume of the AAS mortars. The higher volumes of mesopores can lead to the larger drying

shrinkage magnitudes due to the higher contracting stress derived from the finer capillary pores [26].

The interfacial transition zone between the aggregate and paste plays a key role in the development of compressive strength in the mortars [27]. However, the SEM images of the AAS mortars with pottery sand exhibited that the pottery sand was found to participate in the alkali reaction as an increasing of curing time, the cause of which is currently under investigation.

5. Conclusions

This study has shown that the setting time and fluidity/consistency of AAS mortar derived from pottery sand increase as the slag to aggregate ratio increases, with an optimal ratio of 1/1.75 being the best proportion for producing materials for construction applications. AAS mortars generally exhibited good setting properties (12–25 min), with their setting rates dependent on the proportion of Na_2O present and their fluidity and consistency dependent on their silicate modulus content. Mortars containing 8% Na_2O exhibited good workability properties, with excellent compressive strength values being obtained after both 1 and 28 d. Higher drying shrinkage levels were found for AAS mortars containing higher Na_2O content and silicate modulus, with overall levels of mortar shrinkage dependent on the microporosity of their structure. The interfacial transition zone of these AAS mortars exhibited stronger bonding between gel products and the aggregate as curing times increased, with more microcracks present at the interfacial transition zone in the initial phase of curing than were present after more lengthy curing periods. In conclusion, these performance levels suggest that pottery sand-derived AAS mortars may be potentially useful as self-repairing materials for engineering or construction applications.

Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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