



Article Investigating Mechanical Properties of Alkali-Activated Slag Cementitious Material for Load-Bearing Layer of Sandwich Panels

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Abstract: The research presented in this paper is about the mechanical properties of fiber-reinforced alkali-activated slag cementitious sandwich panels with different types and amounts of admixtures. The mechanical properties, drying shrinkage properties, and micro-morphology were used to determine the optimal ratio of the admixtures. The results show that the alkali-activated slag sandwich panels have the characteristics of light weight, high strength and excellent thermal insulation, and the factors such as magnesium oxide, expansion agent and solution temperature have significant influence on their mechanical properties and dry shrinkage. This paper provides a theoretical basis and experimental data for the preparation process and application of alkali-activated slag sandwich panels.

Keywords: fiber reinforcement; alkali-activated slag cementitious materials; sandwich panels; mechanical properties



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1. Introduction

Sandwich-type insulation wall panels have been extensively studied for their light weight and thermal insulation properties. They have been found to be widely used in exterior wall insulation systems worldwide. This thesis focuses on the load-bearing layer of sandwich panels, which is produced using alkali-activated slag cementitious material (AASCM) [1–3]. Originating from the work of Professor Glukhovsky in 1957, AASCM is formulated by activating industrial waste residues, such as slag, using alkaline activators [4]. These materials have achieved compressive strengths of up to 120 MPa and have demonstrated remarkable stability [5–7].

Sandwich-type insulation wall panels has been a subject of extensive research since the 1960s, particularly as external wall insulation systems. Recent advancements have highlighted the unique advantages of lightweight, high-strength, and superior thermal insulation properties. This has led to a surge of interest in fiber-reinforced AASCM sandwich panels [8,9]. Liu et al. [10] conducted double shear tests on concrete sandwich panel specimens reinforced with basalt fiber-reinforced composite (BFRP) connectors, deriving a bearing capacity formula. Tomlinson et al. [11] developed a numerical model for predicting the response of partially composite load-bearing concrete sandwich panels under arbitrary eccentric loads. Alchaar et al. [12] performed a finite element analysis, demonstrating that the composite action of the sandwich panel led to ductile failure rather than sudden fracture.

As a new environmentally friendly building material, the field of AASCM had been rigorously studied, with a focus on mechanical properties and durability. Mavroulidou et al. [13] delved into the role of different activators, comparing sodium carbonate-activated slag and sodium silicate-activated slag. Kiachehr et al. [14] explored the effects of curing time and temperatures on alkali-activated concrete, proposing a predictive model for residual

strength and mass loss. Mikhailova et al. [15] investigated the influence of shrinkagereducing agents like polyethylene glycol and polypropylene glycol, resulting in a model for estimating shrinkage. Aydın et al. [16] specifically studied the drying shrinkage of AASCM using steel fiber-reinforced alkali-activated slag/silica fume mortar and proposed a drying shrinkage stress model.

While AASCM has shown promising mechanical properties, its substantial drying shrinkage has limited its broader applications. This issue is particularly relevant when considering the load-bearing layers of sandwich panels. The existing literature lacks comprehensive studies on the effects of different admixtures and solution temperatures on AASCM's mechanical properties and drying shrinkage. Furthermore, the impact of additives on the microscopic morphology of AASCM is not well-understood, leaving room for further exploration.

This study focuses on a comprehensive investigation into the mechanical properties and drying shrinkage of AASCM, in order to address existing gaps in the literature. Specifically, the research will explore the effects of different admixtures and varying solution temperatures on these properties when AASCM is used in the load-bearing layer of sandwich panels. Additionally, the study will examine the impact of additives on the microscopic morphology of AASCM. By achieving these objectives, the research will contribute to a deeper understanding of how admixtures and solution temperatures can optimize the material for use in the load-bearing layers of sandwich panels, thereby expanding their potential applications.

2. Material Proportions and Experimental Design

2.1. Materials

AASCM is an early-strength material. To apply this material on a large scale, it is necessary to reduce the hydration heat and microcracks generated by the intense early hydration reaction. Therefore, aggregates, gypsum, fly ash, silica fume, etc., were added to the material to lower the early reaction degree of the AASCM. MgO and expansive agents were added to compensate for shrinkage and inhibit crack formation. Specifically, to optimize the proportion of AASCM for balanced mechanical and drying shrinkage properties, these different types of admixtures using different amounts were selected to be investigated in this study. The materials used in this study included cement, slag, potassium silicate, magnesium oxide, refractory fiber, and other reagents, and their treatment and mix design are detailed in this section.

2.1.1. Slag

The S105-grade slag used in this study was an industrial byproduct generated during the iron reduction process at Tangshan Iron Orchid Company (Hebei, China). The slag had an alkalinity coefficient, $M_0 = (MgO\% + CaO\%)/(SiO_2\% + Al_2O_3\%)$, of 0.87, indicating that it was acidic slag. Its specific surface area was 550 m²/kg. The slag's chemical composition and XRD pattern are shown in Table 1 and Figure 1, respectively. As can be seen in Figure 1, the bread loaf-shaped peak represents amorphous SiO₂, with almost no distinct characteristic peaks, indicating that the slag primarily exists in an amorphous glassy state.

Table 1. Chemical composition of slag.

Component	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	SO ₂	Others	Loss on Ignition
Content (%)	36.9	15.66	37.57	9.3	0.36	-	0.57	-

2.1.2. Potassium Silicate

The potassium silicate used in the tests described in this paper was provided by Xingtai Da Yang Chemical Co., Ltd. (Xingtai, China). The XRD pattern shows that the main components of the dried potassium silicate powder are SiO_2 and K_2O in Figure 2. Combined with the production information, it can be learned that the chemical formula of

potassium silicate is $K_2O \cdot n \operatorname{SiO}_2 \cdot H_2O$, the initial modulus is 2.78, the group degree is 46.3, the content of silica is 27.49%, the content of potassium oxide is 15.50% and the density is 1.434 g/mL.



Figure 1. XRD pattern of slag.



Figure 2. XRD pattern of potassium silicate.

2.1.3. Magnesium Oxide

Lightly calcined magnesium oxide powder was produced by Liaoning Dashiqiao Tianyi Refractory Materials Co., Ltd. (Dashiqiao, China). It was processed from magnesite ore calcined at 950 °C. The chemical composition and XRD pattern of lightly calcined magnesium oxide are shown in Table 2 and Figure 3, respectively. The active content determined using the hydration method was 52.3%.

Table 2. The chemical composition of lightly burned magnesium oxide.

Component	SiO ₂	Al ₂ O ₃	CaO	MgO	Fe ₂ O ₃	SO ₂	Others	Loss on Ignition
Content (%)	7.32	0.13	2.02	88.36	0.28	-	0.01	1.88

2.1.4. Refractory Fiber

The refractory fiber was sourced from Shandong Luyang Energy Saving Materials Co., Ltd. (Zibo, China). It was a polycrystalline refractory fiber that included mullite fiber, alumina fiber, and zirconia fiber, with single filament diameters of 3 to 4 μ m.

2.1.5. Other Reagents

Expansive agent: The applied expansive agent was from Henan Mingzhu Chemical Co., Ltd. (Puyang, China), with the main component being calcium oxide (CaO) at a content of 70.8%.



Figure 3. XRD pattern of magnesium oxide when lightly burned.

Gypsum: Desulfurized gypsum was used in this study with its main component being calcium sulfate dihydrate (CaSO₄·2H₂O) at a content of \geq 93%. Processed desulfurized gypsum is a typical environmentally friendly material.

Fly ash: The fly ash used in this study was provided by Lingshou Chuangbo Mineral Products Co., Ltd. (Shijiazhuang, China). Fly ash consists of fine ash particles discharged during the combustion of fuel (mainly coal). Its particle size generally ranges from 1 to 100 μ m. Also known as pulverized coal ash or soot, it comprises fine solid particles from the smoke and ash generated by fuel combustion.

Silica fume: The silica fume used in this study was obtained from Gansu Lixin Source Micro Silicon Powder Co., Ltd. (Lanzhou, China). Silica fume is produced from the dust emitted with exhaust gases during the high-temperature smelting of industrial silicon and ferrosilicon in an electric furnace. It is collected and treated using specialized equipment. The SiO_2 content in the emitted dust accounts for approximately 90% of the total dust mass, and the particle size is exceptionally small, with an average size close to the nanometer scale.

Sodium hydroxide: Sodium hydroxide was supplied by Tianjin Tianli Chemical Reagent Co., Ltd. (Tianjin, China), with a mass fraction of \geq 96.0%.

Water: Tap water (H₂O) was used for the experiments in this study.

2.2. Mix Ratio Design

2.2.1. Adjusting the Modulus of Potassium Silicate to 1.0

According to previous studies, the optimal modulus of potassium silicate used for the preparation of AASCM was 1.0 [17]. The original potassium silicate had a modulus of 2.78, which meant that the molar ratio in the solution was $SiO_2:K_2O = 2.781$. When the modulus was adjusted to 1.0, the molar ratio in the solution was $SiO_2:(K_2O + NaOH) = 2.78:(1 + 1.78)$. Each gram of potassium silicate contained 0.155 g of K₂O, which was converted to a molar amount of 0.0017 mol. To adjust the modulus to 1.0, the molar amount of NaOH added to each gram of potassium silicate should have been 0.006052 mol, which, when converted to mass, was 0.24208 g.

2.2.2. Treatment of Refractory Fibers

Untreated refractory fibers have a cotton-like appearance, and mixing them directly with AASCM may have resulted in uneven fiber distribution in the specimens. As shown in Figure 4, to better mix the fibers within the AASCM, the fibers were cut into 3–4 cm long pieces using scissors. The chopped fibers were soaked in a sodium potassium silicate solution for 30 min, followed by rapid stirring of the solution. Once the fibers were dispersed in the solution, the solution and fibers were poured into the slag and mixed together.



Figure 4. Refractory fiber treatment diagram.

2.2.3. Mix Ratio Design

In order to study the influence of different admixtures, 12 different mix designs were tested in this study, and the mix design numbers ranged from S-1 to S-12. The material combinations required per cubic meter of AASCMS are shown in Table 3. Previous studies had shown [18] that AASCM exhibits good performance when the modulus of potassium silicate is 1.0, the alkali content is 14% and the fiber content is 1% by mass. Thus, the fiber content in this study adopted 1% for all testing samples. The strength of the AASCM mortar is not only related to the properties of the binder material but also to the water-to-binder ratio and binder-to-sand ratio. Shen et al. [19] found through experiments that a larger water-to-binder ratio results in more capillary pores, while a smaller binder-to-sand ratio reduces cement usage and increases binder–sand interfaces, leading to more interface microcracks, capillary pores and microcracks. Therefore, this study selected a binder-to-sand ratio of 1:2 and a water-to-binder ratio of 0.5 to meet the casting requirements of sandwich panels.

Mix Design Number	Slag (kg)	Magnesium Oxide (kg)	Expanding Agent (kg)	Gypsum (kg)	Fly Ash (kg)	Cement (kg)	Silica Fume (kg)	Fiber (%)
S-1	1953	/	/	/	/	/	/	1
S-2	1758	195	/	/	/	/	/	1
S-3	1563	391	/	/	/	/	/	1
S-4	1367	586	/	/	/	/	/	1
S-5	1367	/	586	/	/	/	/	1
S-6	1367	195	391	/	/	/	/	1
S-7	1367	391	195	/	/	/	/	1
S-8	1133	586	/	234	/	/	/	1
S-9	1219	586	/	/	156	/	/	1
S-10	1719	/	/	234	/	/	/	1
S-11	1563	/	/	234	/	156	/	1
S-12	1758	/	/	/	156	/	39	1

Table 3. Mix ratio design.

2.3. Testing Methods

2.3.1. Compressive and Flexural Strength Tests

The specimens of the compressive and flexural strength testing were prepared following the following steps:

a. Add an appropriate amount of sodium hydroxide to the potassium silicate with a modulus of 2.79 and stir well to adjust the modulus to 1.0.

- b. Pour slag and MgO into the NJ-160A cement mortar mixer according to the proportion, and mix at low speed for 1 min at a speed of 140 r/min to achieve a uniform powder mixture.
- c. Pour the potassium silicate, water and refractory fibers into the mixer and mix at low speed for 2 min at a speed of 140 r/min, followed by high-speed mixing for 2 min at a speed of 285 r/min.
- d. Pour the mixed slurry into $40 \times 40 \times 160$ mm prismatic three-gang molds, ensuring the slurry covers the entire mold to prevent size deficiencies in the specimens due to slurry leakage during compaction, which may lead to incorrect flexural and compressive strength results.
- e. Compact the slurry on a vibrating table 120 times, and then, place the molds in a curing chamber with a standard temperature of 20 ± 2 °C and a relative humidity of 95% for curing. Demold the specimens after 1 day of curing.

The specimens were cured for 3, 7 and 28 days and tested using the HYE-300B cement flexural and compressive constant stress testing machine, as shown in Figure 5a, following testing standard JGJ/T70-2009 "Standard for test method of performance on building mortar" [20]. The flexural strength was tested by applying a load uniformly at a rate of 50 N/s until failure. After the flexural strength test was completed, the specimens were cut into two pieces. Then, the compressive strength of the intact specimens without any other damage was tested at a loading rate of 2400 N/s. The arithmetic mean of the measured values of three specimens was used as the compressive strength of the mortar cubic specimens for the test group.



Figure 5. Testing equipment used: (**a**) compressive strength testing machine, (**b**) comparator and (**c**) Vicat apparatus.

2.3.2. Drying Shrinkage Test

The drying shrinkage tests were performed using the BC-160-type comparator, as shown in Figure 5b. The comparator was placed on a flat table and adjusted to have the pointer aligned with the "0" on the scale. The clean specimen was placed in the comparator and tested for the shrinkage by observing the dial indicator. Due to mold factors, the length of the specimen could not be exactly 160 mm, so the initial length was determined by the length of the specimen when demolded at 1 day. The drying shrinkage could be estimated from the reading of the drying shrinkage tests as follows:

$$\varepsilon = \frac{L_1 - L_t}{L_t} \tag{1}$$

where ε is the shrinkage rate of the specimen at t (3, 7, 28) days; L_1 is the length of the specimen at 1 day, in mm; and L_t is the length of the specimen at t days, in mm.

2.3.3. Setting Time Test

To prepare the specimens for setting time tests, the well-mixed mortar was pulled into the round mold and placed in the curing chamber for 10 min. The specimens were then removed from the mold and tested using the Vicat apparatus, as shown in Figure 5c. The Vicat apparatus measured the initial setting time every 5 min. To obtain the setting time, the timer was started from the moment water was added and continued until the needle stopped sinking or, after releasing the needle for 30 s, the pointer reading was 4 mm \pm 1 mm from the bottom plate. This time was measured as the initial setting time of the mortar.

After determining the initial setting time, the round mold with an inner diameter of 65 mm was rotated immediately by 180° so that the top faced downward, and it was then placed in the curing chamber. As the final setting time approached, measurements were conducted every 15 min or at shorter intervals. The timer was started from the moment water was added and continued until the final setting needle, when approaching the specimen, did not leave a trace on the specimen after freely falling for 30 s. This was measured as the final setting time.

2.3.4. Microstructural Analysis Methods

- (1) X-ray Diffractometer (XRD): XRD tests were performed on the samples from the specimens using D8-Advance from Bruker Corporation (Billerica, MA, USA). To prepare the testing samples, the specimens were crushed and grinded into powder and then passed through a 0.075 mm sieve. The powder was dried in a beaker at 40 °C until a constant weight was achieved. The phases were scanned using an XRD instrument with a measurement accuracy of ≤ 0.010 and the scanning range of $10-70^{\circ}$.
- (2) Scanning Electron Microscope (SEM): SEM analyses were performed on the specimens using SUPERTM 55 from Carl Zeiss AG (Oberkochen, Germany). The testing samples were prepared as follows: (a) the broken center parts of the specimens with a curing age of 3 d and 28 d were placed in anhydrous ethanol to stop hydration; (b) after soaking for 7 days, the samples were dried at 60 °C in an oven until a constant weight was achieved; and (c) the samples were then attached to a tray and coated with gold. After gold coating, the SEM analysis was performed.

3. Results and Discussions

3.1. Effect of Admixture on Mechanical Properties of AASCM

3.1.1. MgO Content

The influences of MgO content ranging from 0% to 30% at an interval of 10%, as shown in Sample S1–S4 in Table 3, on the compressive and flexural strength of AASCM are shown in Figure 6a,b, respectively. It can be seen that as the MgO content increased, the compressive and flexural strength of the AASCM increased at all ages. MgO had a more significant effect on the flexural strength than on the compressive strength of AASCM, and it improved the low early flexural strength of AASCM. The MgO content improved the strength of AASCM because after hydration, MgO formed brucite (Ht) and magnesium silicate hydrate gel [21] (M-S-H), releasing OH-ions into the solution. The high concentration of OH⁻ ions entered the slag glass network, destroying the network structure and accelerating the dissolution of the slag glass. The rapid dissolution of slag glass led to a high concentration of Ca²⁺, Mg²⁺ and SiO₄⁴⁻ in the solution, accelerating the formation of calcium silicate hydrate gel (C-S-H), brucite and magnesium silicate hydrate gel.

In addition, a greater increase in the early curing stages was noticed when compared to the later curing stages. The smaller strength increase in the later curing stages may have been due to the decreasing ion concentration in the later curing stage, which slowed the reaction rate. Furthermore, there was a large amount of Mg²⁺ ions near the slag particles, which reacted with the SiO₄⁴⁻ ions dissociated from the potassium glass in water to form M-S-H, encasing unreacted slag particles and hindering the dissolution of slag [22].



Figure 6. Effect of magnesium oxide content on compressive and flexural resistance of materials.

The more pronounced effect of MgO on the flexural strength of AASCM may have been due to the fact that the density of both Ht and M-S-H generated by the hydration of MgO was smaller than that of C-S-H, which could more effectively fill the pores between materials [23], as shown in Figure 7. The filling of pores made AASCM more compact, and a smaller porosity led to a more significant increase in compressive strength.



Figure 7. Reaction mechanism diagram of magnesium oxide in AASCM.

3.1.2. Partial Replacement of MgO with CaO

The influences of the partial replacement of MgO with CaO on the compressive and flexural strength of AASCM are shown in Figure 8a,b. The highest compressive and flexural strength of the AASCM was noticed to be with a CaO content of 10%. After the addition of 10% CaO, the further increase in the CaO content decreased the compressive and flexural strength of the AASCM. Such an optimal ratio of CaO replacement ratio might have been induced by the fact that CaO hydrolyzes faster than MgO, so the added CaO could rapidly hydrolyze to form Ca(OH)₂. As Ca(OH)₂ continuously dissociated Ca²⁺ and OH⁻ into the solution, the high concentration of OH⁻ was beneficial for the dissolution of slag. When the CaO content was low, the Ca/Si molar ratio in the solution was low, and there was a large amount of free SiO_4^{4-} in the solution. The faster hydration rate of CaO resulted in a higher pH value in the early stage, which was beneficial for the hydration of Mg^{2+} to form Ht and M-S-H, thereby increasing the strength of the material. When the CaO content was higher than 10%, the Ca/Si molar ratio increased, and SiO_4^{4-} preferentially reacted with Ca²⁺ to form C-S-H, inhibiting the reaction between SiO_4^{4-} and Mg^{2+} and hindering the formation of Ht and M-S-H [24]. Moreover, as the CaO content increased, the concentration of Ca^{2+} increased, and there was not enough free SiO_4^{4-} in the solution to form M-S-H. Mg^{2+} existed in the form of $Mg(OH)_2$, which affected the strength of the material.



Figure 8. Effect of expansion agent content on compressive and flexural resistance of materials.

3.1.3. Other Admixtures

The related literature [25] shows that gypsum, fly ash, cement and silica fume have a beneficial effect on the mechanical properties of AASCM. Therefore, this study set the gypsum content at 12%, fly ash content at 8%, cement content at 8% and silica fume content at 2%. Data obtained from different combinations are shown in Figure 9a,b. It can be seen that when MgO was added alone, AASCM had the best compressive and flexural strength. When MgO and gypsum were added simultaneously, the strength was lower than when MgO and gypsum were added separately. Adding gypsum and fly ash will reduce the early strength of AASCM, while adding cement and MgO will increase the early strength of the AASCM. Since MgO has an activating effect on AASCM, the strength was higher than that in other groups. Gypsum and fly ash did not show as high reactivity as MgO in the material and are non-reactive or weakly reactive items, hindering the hydration of slag.



Figure 9. Effect diagram of other admixtures on compressive and flexural resistance of materials.

3.2. The Influence of Solution Temperature

When casting large components, the hydration heat accumulated inside the member is not easily dissipated, leading to potential for the internal hydration heat to reach 50 °C to 60 °C. The temperature stress causes expansion cracks in the components. Thus, measures are needed to reduce the hydration heat for AASCM when used on a large scale. Ma et al. [26] studied the strength development of alkali-activated slag mortar under ambient and high temperatures and found that heat-cured samples rapidly gained strength, but if given enough time, ambient temperature curing could produce comparable strength. Therefore, this study used the heat released by NaOH to dissolve in the solution and divide the solution temperature into four different temperature stages based on time, as shown in Table 4. Specimens were prepared at time t = 5 min, 30 min, 60 min and 1 day (1 d) to study the influence of NaOH dissolution time on the mechanical properties and workability of the material.

Table 4. Dissolution time selection.

Serial Number	S4-I	S4-II	S4-II S4-III	
NaOH Dissolution Time	5 min	30 min	60 min	1 d

To measure the temperature change for NaOH dissolution in water, the air temperature was measured using a thermometer, which was 18 °C, in addition to water temperature, which was 17 °C. Then, the weighed NaOH was poured into a potassium glass to adjust the modulus and the thermometer was placed in the NaOH solution to measure temperature stages with different NaOH dissolution times. The temperature was read and recorded at intervals, as shown in Figure 10. Figure 10 indicates that a large amount of heat was released when NaOH dissolved, and the solution temperature rose to 41 °C in 5 min. Afterward, the temperature began to decrease, with the early stage decreasing slower than the later stage as the NaOH in the solution was still dissolving. The solution temperature was the same as the room temperature after 1 day.



Figure 10. Temperature over time.

Figure 11 plots the relationship between the solution temperature and the initial and final setting times. Both the initial and final setting times increased with the increase in solution temperature; however, the setting time between the initial and final setting did not increase significantly. This indicates that the temperature of the solution has a significant impact on the setting time. The setting time decreased with the increase in temperature. The setting time and temperature showed a good quadratic polynomial fitting relationship, and the significance of the relationship can be determined by R^2 as:

$$T_1 = 70.59 - 0.188x - 0.022x^2 \quad R^2 = 0.996$$

$$T_2 = 86.82 + 0.67x - 0.034x^2 \quad R^2 = 0.987$$
(2)

in which T_1 is the initial setting time, in minutes; T_2 is the final setting time, in minutes; x is the temperature, in °C; and R^2 is the correlation coefficient, with values closer to 1 indicating a higher degree of fit.



Figure 11. Diagram of the relationship between solution temperature and initial and final setting time.

The increase in solution temperature accelerates ion movement, which in turn accelerates the decomposition of slag glass and magnesium oxide. The increases in Mg^{2+} , Ca^{2+} and SiO_3^{2-} concentrations in the solution accelerate the formation of C-S-H gel, Ht and M-S-H gel [27]. Therefore, the higher the temperature, the shorter the setting time of the material.

Figure 12a,b show the influence of solution temperature on the compressive and flexural strength of AASCM. It can be seen that the compressive strength decreased with the increase in NaOH dissolution time, while the flexural strength showed the opposite trend. This phenomenon may be accounted for by the fact that at higher temperatures, more Mg²⁺ is hydrated to form Ht, which is particulate and can fill the pores between C-S-H gels, thereby improving its compressive strength. However, excessive Ht particles may cause gaps in the gel phase and reduce flexural strength. At lower temperatures, more Mg²⁺ is hydrated to form M-S-H gel, which has a smaller density than C-S-H gel [28]. The M-S-H gel formed covers the C-S-H gel, making the structure denser, thereby increasing flexural strength. However, due to the lack of Ht particles to bear pressure, the compressive strength decreases.



Figure 12. Effect of solution temperature on AASCM compressive and flexural strength.

3.3. The Influence of Admixtures and Solution Temperature on the Drying Shrinkage of AASCM

Figure 13 illustrates the effects of various admixtures on the drying shrinkage of AASCM. S-4 has less drying shrinkage than S-2, indicating that increasing the content of magnesium oxide is beneficial for reducing shrinkage. S-4 has less drying shrinkage than S-7, S-8 and S-9, indicating that magnesium oxide is more effective in reducing drying shrinkage than the expansive agents, gypsum and fly ash.



Figure 13. Effect of various admixtures on drying shrinkage of AASCM.

The early hydration of magnesium oxide forms $Mg(OH)_2$ has expansive properties to compensate for shrinkage. As the reaction proceeded, $Mg(OH)_2$ reacted with SiO_4^{4-} to form talc and hydrated magnesium silicate. The improvement in material drying shrinkage in the later stage was mainly attributed to the filling of voids by Ht [29]. Therefore, S-4 with 30% magnesium oxide content had less drying shrinkage than S-2 with 10% magnesium oxide content.

The improvement in material drying shrinkage via the expansive agent was mainly due to the formation of Ca(OH)₂ from some CaO, which acted as an expansive source to compensate for shrinkage [30]. The improvement in material drying shrinkage via fly ash was due to its filler effect, which reduced the content of reactants and reaction activity, resulting in a decrease in mechanical properties. The shrinkage reduction effect was more obvious when a larger amount of fly ash was added [31]. The improvement in material drying shrinkage via gypsum was due to the production of gypsum dihydrate, which can combine with a large amount of water to form a larger solid phase, and the formation of larger-sized dihydrate gypsum.

The influence of solution temperature on the drying shrinkage of AASCM is shown in Figure 14. The drying shrinkage was found to decrease with the decrease in solution temperature. This is because the higher the solution temperature, the higher the ion concentration in the early stage of the test, and the early hydration products Mg(OH)₂ and Ca(OH)₂ were more abundant. As the reaction proceeded, Mg(OH)₂ and Ca(OH)₂ were converted into denser C-S-H and M-S-H, resulting in volume shrinkage. Therefore, the S4-I group had larger drying shrinkage due to the formation of more gel phases.



Figure 14. Effect of solution temperature on drying shrinkage of AASCM.

3.4. Microscopic Analysis

3.4.1. X-ray Diffraction Analysis

The XRD patterns of S-4 and S-2, with 30% and 10% magnesium oxide content, respectively, are shown in Figure 15. From the XRD spectra, it can be seen that at the curing age of 28 days, the unreacted magnesium oxide in AASCM increased with the increase in magnesium oxide content. The Ht peak near 10° also increased with the increase in magnesium oxide content, indicating that magnesium oxide promoted the formation of Ht. The C-S-H peak near 30° became narrower with the increase in magnesium oxide content, and the possible reason for this is that M-S-H covered C-S-H, causing changes in the C-S-H structure.



Figure 15. XRD pattern of different magnesium oxide content levels.

3.4.2. SEM Analysis

Figure 16a–f show the SEM images of S-4 and S-2 at 3 d and 28 d, with 30% and 10% magnesium oxide content, respectively. From Figure 16a,c, it can be seen that at 3 days, there was still a large amount of unreacted slag and MgO in the system, forming a honeycomb-like porous structure. At 28 days, the hydration of the slurry was more sufficient, making the microstructure denser. From Figure 16b,d, it can be seen that the microstructure density of 10% MgO content was not as good as that of 30% MgO content, which is because the M-S-H formed by MgO could cover C-S-H and compensate for the structural pores [32]. From Figure 16e,f, it can be seen that the refractory fibers had good contact with the matrix, and the fibers were wrapped in dense hydration products. Based on the density and the mottled particles on the surface, it is possible that M-S-H binds C-S-H and Other hydration products together.



Figure 16. SEM pattern of different magnesium oxide content.

4. Conclusions

This study provides a theoretical basis and experimental data for the preparation process and application of AASCM sandwich panels. The study also suggests some ways to optimize the mix ratio and curing conditions of AASCM to achieve better performance. The study's conclusions are summarized as follows:

Magnesium oxide is the most effective admixture for improving the mechanical properties and reducing the drying shrinkage of AASCM. The optimal content of magnesium oxide is 30% by mass of slag.

Solution temperature has a significant impact on the setting time, strength and shrinkage of AASCM. A lower solution temperature leads to a longer setting time, lower compressive strength, higher flexural strength and lower shrinkage.

The microstructure of AASCM is affected by the admixtures and solution temperature. Magnesium oxide promotes the formation of magnesium silicate hydrate (M-S-H) gel and brucite (Ht), which cover calcium silicate hydrate (C-S-H) gel and fill the pores between materials. A higher solution temperature accelerates the hydration of slag and magnesium oxide, resulting in more gel phases and volume shrinkage.

AASCM with the best mix ratio will be used in the research of the sandwich panels, and it will be the main material of the load-bearing layer of sandwich panels in the future.

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References

- 1. Huang, J.; Xu, Y.; Huang, H.; Dai, J. Structural behavior of FRP connector enabled precast geopolymer concrete sandwich panels subjected to one-side fire exposure. *Fire Saf. J.* **2022**, *128*, 103524. [CrossRef]
- Gaibor, N.; Mateus, R.; Leitao, D.; Cristelo, N.; Miranda, T.; Pereira, E.N.; Cunha, V.M. Sustainability assessment of half-sandwich panels based on alkali-activated ceramic/slag wastes cement versus conventional building solutions. *J. Clean. Prod.* 2023, 389, 136108. [CrossRef]
- Kumar, S.; Das, C.S.; Lao, J.; Alrefaei, Y.; Dai, J.G. Effect of sand content on bond performance of engineered geopolymer composites (EGC) repair material. *Constr. Build. Mater.* 2022, 328, 127080. [CrossRef]
- Glukhovsky, V.D.; Rostovskaja, G.S.; Rumyna, G.V. High Strength Slag-Alkaline Cements. In Proceedings of the 7th International Congress on the Chemistry of Cement, Paris, France, 3–6 September 1980.
- 5. Ranjbar, N.; Zhang, M. Fiber-reinforced geopolymer composites: A review. Cem. Concr. Compos. 2020, 107, 103498. [CrossRef]
- Choi, J.I.; Park, S.E.; Kim, Y.Y.; Lee, B.Y. Flexural behavior of composite beams of Kagome truss and fiber-reinforced cementitious composites. *Constr. Build. Mater.* 2022, 361, 129653. [CrossRef]
- Cui, Y.; Hao, H.; Li, J.; Chen, W.; Zhang, X. Structural behavior and vibration characteristics of geopolymer composite lightweight sandwich panels for prefabricated buildings. J. Build. Eng. 2022, 57, 104872. [CrossRef]
- 8. Mavroulidou, M.; Sanam, I.; Mengasini, L. Mechanical and durability performance of alkali-activated slag cement concretes with carbonate and silicate activators. *Sustain. Chem. Pharm.* **2023**, *31*, 100896. [CrossRef]
- Manalo, A.C. Recent developments on fibre composite sandwich structures in civil infrastructure. In Proceedings of the 6th International Composites Conference (ACUN 6), Melbourne, Australia, 14–16 November 2012.
- Liu, X.; Wang, X.; Yang, T.; Wu, Z. The Shear Behavior of Insulated Precast Concrete Sandwich Panels Reinforced with BFRP. Buildings 2022, 12, 1326. [CrossRef]
- 11. Tomlinson, D.; Fam, A. Analysis and parametric study of partially composite precast concrete sandwich panels under axial loads. *J. Build. Eng.* **2016**, *142*, 04016086. [CrossRef]

- 12. Alchaar, A.; Abed, F. Finite element analysis of a thin-shell concrete sandwich panel under eccentric loading. *J. Build. Eng.* **2020**, 32, 101804. [CrossRef]
- Korniejenko, K.; Figiela, B.; Miernik, K.; Ziejewska, C.; Marczyk, J.; Hebda, M.; Lin, W.T. Mechanical and Fracture Properties of Long Fiber Reinforced Geopolymer Composites. *Materials* 2021, 14, 5183. [CrossRef]
- 14. Behfarnia, K.; Shahbaz, M. The effect of elevated temperature on the residual tensile strength and physical properties of the alkali-activated slag concrete. *J. Build. Eng.* **2018**, *20*, 442–454. [CrossRef]
- 15. Mikhailova, O.; Šimonová, H.; Topolář, L.; Rovnaník, P. Influence of Polymer Additives on Mechanical Fracture Properties and on Shrinkage of Alkali Activated Slag Mortars. *Key Eng. Mater.* **2018**, *761*, 39–44. [CrossRef]
- 16. Aydın, S. A ternary optimisation of mineral additives of alkali activated cement mortars. *Constr. Build. Mater.* **2013**, *43*, 131–138. [CrossRef]
- 17. Zhu, J.; Song, L.; Qu, Z.; Wang, X.; Wen, Z.; Liu, X.; Wang, H. Mechanical Strengths of Alkali-Activated Blast Furnace Slag Powder with Different Alkali Activators and Plant Fibers. *Coatings* **2023**, *13*, 664. [CrossRef]
- Feng, S.; Zhu, J.; Wang, R.; Qu, Z.; Song, L.; Wang, H. The Influence of CaO and MgO on the Mechanical Properties of Alkali-Activated Blast Furnace Slag Powder. *Materials* 2022, 15, 6128. [CrossRef] [PubMed]
- 19. Shen, C.; Fang, X.; Wang, H. Study on the influence of suction, moisture content, and dry density on the shear strength of remolded unsaturated soil. *Rock Soil Mech.* **2009**, *30*, 1347–1351.
- 20. JGJ/T70-2009; Standard for Test Method of Performance on Building Mortar. Industry Standard—Construction Industry: Beijing, China, 2009.
- 21. Wu, H.; Jin, F.; Zhou, A.; Du, Y. The engineering properties and reaction mechanism of MgO-activated slag cement-clayey sand-bentonite (MSB) cutoff wall backfills. *Constr. Build. Mater.* **2021**, 271, 121890. [CrossRef]
- 22. Zhu, J.; Liu, S.; Song, L.; Qu, Z.; Wang, H. Influence of Carbon Dioxide Curing on the Corrosion Resistance of Reinforced Cement Mortar under the External Erosion of NaCl Freeze–Thaw Cycle. *Appl. Sci.* **2022**, *12*, 5061. [CrossRef]
- 23. Ma, Y.; Zhang, B.; Wang, B.; Lin, X.; Zhu, J.; Huang, P.; Ji, T. Fluidity, mechanical properties, shrinkage of alkali-activated slag/stainless steel slag mortars with composite activators. *J. Build. Eng.* **2023**, *75*, 106877. [CrossRef]
- He, J.; Zheng, W.; Bai, W.; Hu, T.; He, J.; Song, X. Effect of reactive MgO on hydration and properties of alkali-activated slag pastes with different activators. *Constr. Build. Mater.* 2021, 271, 121608. [CrossRef]
- Coppola, L.; Coffetti, D.; Crotti, E.; Candamano, S.; Crea, F.; Gazzaniga, G.; Pastore, T. The combined use of admixtures for shrinkage reduction in one-part alkali activated slag-based mortars and pastes. *Constr. Build. Mater.* 2020, 248, 118682. [CrossRef]
- 26. Ma, H.; Zhang, S.; Feng, J. Early hydration properties and microstructure evolutions of MgO-activated slag materials at different curing temperatures. *Ceram. Int.* **2022**, *48*, 17104–17115. [CrossRef]
- Zheng, W.; Zhang, S.; Guo, H.; Chen, X.; Huang, Z.; Jiang, S.; Li, M. Multi-omics analysis of tumor angiogenesis characteristics and potential epigenetic regulation mechanisms in renal clear cell carcinoma. *Cell Commun. Signal.* 2021, 19, 39. [CrossRef] [PubMed]
- Zheng, W.; He, J.; Tong, Y.; He, J.; Song, X.; Sang, G. Investigation of effects of reactive MgO on autogenous and drying shrinkage of near-neutral salt activated slag cement. *Ceram. Int.* 2022, 48, 5518–5526. [CrossRef]
- 29. Yuan, X.; Chen, W.; Lu, Z.; Chen, H. Shrinkage compensation of alkali-activated slag concrete and microstructural analysis. *Constr. Build. Mater.* **2014**, *66*, 422–428. [CrossRef]
- Abdollahnejad, Z.; Mastali, M.; Woof, B.; Illikainen, M. High strength fiber reinforced one-part alkali activated slag/fly ash binders with ceramic aggregates: Microscopic analysis, mechanical properties, drying shrinkage, and freeze-thaw resistance. *Constr. Build. Mater.* 2020, 241, 118129. [CrossRef]
- 31. Dung, N.T.; Hooper, T.J.N.; Unluer, C. Enhancing the performance of MgO-activated slag-fly ash mixes by accelerated carbonation. *J. CO2 Util.* **2020**, *42*, 101356. [CrossRef]
- 32. Singh, R.J.; Raut, A.; Murmu, A.L.; Jameel, M. Influence of glass powder incorporated foamed geopolymer blocks on thermal and energy analysis of building envelope. *J. Build. Eng.* **2021**, *43*, 102520. [CrossRef]

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