Research Article

Long-Term Performance and Durability of Heat-Treated Alkali-Activated Slag Mortar Containing Silica Fume

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This study aimed to investigate the combined influence of heat curing and partial replacement of silica fume on the long-term behavior of alkali-activated slag (AAS) mortar. Silica fume replacement levels of 0%, 5%, and 10% were examined, with curing conducted at temperatures of 23 ± 2 and 80°C. The alkaline activator consisted of sodium hydroxide solution (4 M) mixed with sodium silicate in a 3 : 1 ratio by weight. The performance of the fabricated specimens was evaluated through compressive strength testing, mass change analysis, X-ray diffraction, and scanning electron microscopy. Heat treatment resulted in improved performance of AAS mortars, while silica fume replacement also positively influenced mortar behavior. Notably, the 5% replacement rate yielded the most favorable outcomes. However, it was observed that the long-term compressive strength of AAS mortar specimens decreased significantly. Potential adverse factors contributing to this decline were discussed. Furthermore, the durability of mortar samples exposed to adverse conditions was investigated. Results indicated that the combined use of heat curing and a 5% silica fume replacement level produced the best overall performance.

1. Introduction

The demand for infrastructure development has spurred an increasing utilization of construction materials. Ordinary Portland cement (OPC), the predominant choice, significantly contributes to CO₂ emissions, ranging from 5% to 8%, and ranks third in energy consumption, following steel and aluminum [1, 2]. In efforts to ameliorate the ecological footprint associated with concrete production, scholars have delved into the investigation of alternative binding agents, including ground granulated blast furnace slag (GGBFS), fly ash, rice husk ash, cenosphere, palm oil fuel ash, biochar, marble and granite dust, volcanic glass, and zeolite [3, 4]. GGBFS, classified as an alkali-activated material, exhibits heightened reactivity and strength owing to its high calcium content in comparison to low-calcium counterparts such as fly ash [5]. The utilization of alkali-activated slag (AAS) offers substantial environmental benefits, with potential reductions in CO₂ emissions ranging from 25% to 50% compared to OPC [6]. Furthermore, AAS-based concrete presents several advantageous properties, including high compressive strength, low permeability, significant fire resistance, remarkable durability against aggressive environmental conditions, and favorable electrical and self-sensing properties [7]. On the other hand, challenges associated with AAS-based materials have also been documented, including issues with low workability, rapid setting rate, significant drying shrinkage, efflorescence, conflicting findings regarding carbonation susceptibility, and inconsistent outcomes about alkali–silica reaction [2, 8–10].

Various factors influence AAS properties, including its chemical composition, particle size, amorphous structure of slag, type and concentration of alkaline activator, SiO₂/Na₂O ratio, water to binder ratio, treatment methods, and curing conditions [7, 8, 11].

The chemical composition of slag predominantly comprises four constituents: MgO, Al₂O₃, SiO₂, and CaO. This composition exhibits considerable variation contingent upon the mineral nature, the composition of molten iron ore, and the product type within the iron smelting facility [12].
Optimal precursors for alkali activation of GGBFS typically feature CaO/SiO₂ ratios ranging from 0.5 to 2.0 and Al₂O₃/SiO₂ ratios of 0.1–0.6. Calcium silicate hydrate (C–S–H) emerges as the principal hydration phase in AAS, resembling the hydration products of OPC [5]. The specific gravity of slag falls within the range of 1.3–3.5, while its bulk density ranges between 1,600 and 1,760 kg/m³. The particle size of slag significantly impacts its reactivity, with finer particles exhibiting increased surface area conducive to strength development and water demand. Nonetheless, particle fineness must be judiciously controlled to balance setting time and economic considerations [12]. Particles larger than 20 μm demonstrate slower reactivity, whereas those smaller than 2 μm achieve complete reaction within 24 hr [13]. The optimal fineness of GGBFS for alkali activation typically lies between 400 and 550 m²/kg. Concrete/mortar based on AAS displays enhanced cohesion and setting time in comparison to OPC. Workability and plasticizing admixtures formulated for OPC-based concrete may not exhibit optimal efficiency for AAS concrete. However, modifying the slag composition offers potential avenues for practical application. Hence, researchers possess a clear trajectory towards facilitating the utilization of AAS concrete/mortar [5].

Several researchers have explored the use of heat treatment to improve AAS behavior in terms of compressive strength, durability, and shrinkage. They earned conflicting results regarding compressive strength. For instance, Bakharev et al. [14] conducted experiments involving heat curing of AAS at 70°C and observed a 30% reduction in 90-day compressive strength compared to room temperature curing. Aliabdo et al. [15] studied the compressive strength of AAS by treatment at 30, 60, and 90°C, noting a 9% and 16% reduction in 28-day compressive strength for samples treated at 60 and 90°C, respectively, compared to those cured at 30°C. Conversely, Aliques-Granero et al. [16] found that curing AAS samples at 35 and 80°C resulted in a 12% and 15% decrease in 28-day compressive strength, respectively, compared to ambient temperature treatment. Whereas, El-Feky et al. [17] reported a 17% improvement in AAS compressive strength at 28 days when subjected to thermal curing at 80°C compared to the ambient curing. Bilim et al. [18] used heat treatment at 65°C along with various additive ratios to reduce AAS shrinkage, with the heat-treated samples exhibiting higher compressive strength than those cured at ambient temperature. Additionally, Chi et al. [19] tested different ratios of alkaline activator solution to slag and sand to slag, with heat curing at temperatures of 65 and 85°C, and found that the 65°C curing temperature yielded the best outcomes in terms of 56-day compressive strength. It is important to note that the methods varied among these studies, including differences in heat curing procedures, slag composition, additives, type and concentration of alkaline activator, and activator to slag ratio. Consequently, drawing a general conclusion about the effect of heat treatment on the compressive strength of AAS-based concrete/mortar based solely on these results is challenging.

Silica fume (SF) serves as a supplementary material renowned for its pozzolanic reactivity, exerting a substantial influence on concrete properties. Incorporating SF into concrete initiates a pozzolanic reaction, resulting in the generation of additional C–S–H gel. Notably, SF particles act as nucleation sites for C–S–H crystallization. Moreover, the infiltration of SF particles into the interstitial spaces of the matrix structure enhances the physical characteristics of concrete [20].

Some researchers have explored the use of SF as a mineral replacement for slag to enhance the performance of AAS concrete. Rostami and Behfarnia [21] observed a decrease in the 90-day compressive strength of AAS concrete by 22.7%, 28.1%, and 32.4% with SF replacement levels of 5%, 10%, and 15%, respectively. They also noted a reduction in chloride ion penetration in AAS concrete with SF substitution, with the most effective replacement level being 15%. Zamanabadi et al. [22] found that using SF as a repair material in AAS paste, at a 10% substitution rate, led to an increase in compressive strength. Ramezanianpour and Moenia [23] investigated the effect of SF replacement levels (5%, 7.5%, and 10%) on the compressive strength of AAS coating mortar. They reported a 4% increase in 90-day compressive strength with a 5% replacement level, which yielded the best result. Additionally, they noted that SF replacement levels up to 5% improved AAS durability against chloride penetration. Sharifi et al. [24] incorporated a 10% partial substitution of SF with slag in AAS pastes. They varied the mixture designs in terms of sodium hydroxide concentration and sodium hydroxide to sodium silicate ratio, resulting in an increase in 28-day compressive strength ranging from 3.2% to 26.2%.

Few studies were found regarding the long-term performance of AAS. Bernal et al. [25] evaluated the performance of AAS over a span of 180 days and noted varying outcomes based on different SiO₂/Al₂O₃ ratios. Wardhono et al. [26] assessed the long-term compressive strength of AAS up to the age of 540 days and observed a decrease in the compressive strength after 90 days, which they attributed to a high SiO₂/Al₂O₃ ratio. Humad et al. [27] assessed the effect of heat treatment on the long-term performance of AAS and observed a decrease in compressive strength at later ages. Therefore, the present study aims to assess the long-term behavior of AAS mortar immersed in lime water (LW) solution. Given the above conflicting findings regarding heat treatment, this research was planned to investigate the effect of separate and combined effects of thermal curing and SF replacement on the long-term performance of alkali-activated GGBFS obtained from Esfahan Steel Company in Iran. In this regard, the compressive strength, mass change, X-ray diffraction (XRD) analysis, and scanning electron microscopy (SEM) tests were conducted. In several studies, the mass change test has been employed to examine the durability of AAS against sulfate and acid attacks [16, 26]. Consequently, the findings from this test appear to be valuable additions to the ongoing discussion.

Thermal curing and partial slag substitution with SF positively influenced the performance of AAS mortars. However, the long-term compressive strength of AAS mortar specimens significantly decreased regardless of curing temperature and SF substitution dosage. The reasons for this strength reduction were not discussed by the aforementioned authors; therefore, we address possible explanations in this study.
Table 1: Chemical composition of ordinary Portland cement (OPC), ground granulated blast furnace slag (GGBFS), and silica fume (SF).

<table>
<thead>
<tr>
<th>Materials</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>CaO</th>
<th>MgO</th>
<th>Na$_2$O</th>
<th>K$_2$O</th>
<th>SO$_3$</th>
<th>LOI</th>
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<tr>
<td>OPC (%)</td>
<td>22.28</td>
<td>4.71</td>
<td>2.85</td>
<td>65.65</td>
<td>1.95</td>
<td>0.155</td>
<td>0.484</td>
<td>—</td>
<td>1.41</td>
</tr>
<tr>
<td>GGBFS (%)</td>
<td>35.1</td>
<td>10.30</td>
<td>0.80</td>
<td>36.00</td>
<td>10.80</td>
<td>0.71</td>
<td>0.61</td>
<td>—</td>
<td>5.35</td>
</tr>
<tr>
<td>SF (%)</td>
<td>93.16</td>
<td>1.13</td>
<td>0.72</td>
<td>—</td>
<td>1.60</td>
<td>—</td>
<td>—</td>
<td>0.05</td>
<td>2.26</td>
</tr>
</tbody>
</table>

Table 2: Applied binder mix design and their curing temperature.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>OPC</th>
<th>SF0.23</th>
<th>SF0.80</th>
<th>Mix ID</th>
<th>SF5.23</th>
<th>SF5.80</th>
<th>SF10.23</th>
<th>SF10.80</th>
</tr>
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<tbody>
<tr>
<td>OPC (%)</td>
<td>100</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>95</td>
<td>95</td>
<td>90</td>
<td>90</td>
</tr>
<tr>
<td>GGBFS (%)</td>
<td>—</td>
<td>100</td>
<td>100</td>
<td>5</td>
<td>5</td>
<td>10</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>SF (%)</td>
<td>—</td>
<td>—</td>
<td>—</td>
<td>80</td>
<td>80</td>
<td>80</td>
<td>80</td>
<td></td>
</tr>
<tr>
<td>Curing temp. (°C)</td>
<td>23 ± 2</td>
<td>23 ± 2</td>
<td>80</td>
<td>23 ± 2</td>
<td>80</td>
<td>23 ± 2</td>
<td>80</td>
<td></td>
</tr>
</tbody>
</table>

OPC: ordinary Portland cement, GGBFS: ground granulated blast furnace slag, SF: silica fume, and (SFX.Y: X: silica fume replacement level, and Y: curing temperature).

2. Materials and Methods

2.1. Materials. The materials used in this study included sand, GGBFS (referred to as slag), SF, Portland cement type 2 (referred to as OPC), solid sodium hydroxide, and sodium silicate. The sand was in accordance with ASTM C778-17 [30] standard. The slag, obtained as a byproduct from Esfahan Steel Company in Iran, had a specific gravity of 2.85 g/cm$^3$ and a Blaine fineness of 4,500 cm$^2$/g. The SF was sourced from Iran Ferroalloy Industries Company. OPC, commonly produced in Iran, was used. Solid sodium hydroxide with a purity of 98.6% and sodium silicate with a purity of 44.5% and a silicate modulus of 2.07 were also utilized. Table 1 indicates the chemical composition of the cementitious materials utilized in the present research, determined using X-ray fluorescence (XRF) technique. The slag used in the study had a basicity coefficient ($K_b = (\text{CaO} + \text{MgO})/(\text{SiO}_2 + \text{Al}_2\text{O}_3)$) of 1.03 (>1) and a hydration modulus ($HM = (\text{CaO} + \text{MgO} + \text{Al}_2\text{O}_3)/(\text{SiO}_2)$) of 1.68 (>1.4). This indicated that the slag was nearly neutral in alkalinity and exhibited efficient hydration during the alkali activation process [31].

2.2. Mix Design. The mortar samples were cast with dimensions of 50 mm × 50 mm × 50 mm for the compressive strength test according to ASTM C109/C 109M-20b [32] standard. The water-to-cement ratio was applied as 0.485, as per the specified standard. For the AAS-based mortars, the ratio of alkaline activator solution to slag was 0.45. The concentration of sodium hydroxide in the alkaline activator solution was 4 M, with a sodium hydroxide to sodium silicate ratio of 3. It’s worth noting that the concentration of activator and the sodium hydroxide to sodium silicate ratio varied in similar studies [23, 25]. The hydration coefficient value for slag used in the study by Rostami and Behfarnia [23] ($HM = 1.68$) was comparable to that of the present research ($HM = 1.63$). Hence, we adopted the type and concentration of the alkaline activator, as well as the ratio of alkaline activator solution to slag, as outlined in their study.

Most researchers have suggested an optimal SF replacement level of 5%–10% in AAS-based concrete or mortar as the optimal rate [24, 25, 33, 34]. Therefore, we investigated substitution rates of 0%, 5%, and 10% in this study. Considering previous findings [14, 15, 17, 35], a heat-curing temperature of 80°C was selected. The binder mix designs used for the samples and their curing temperatures are presented in Table 2. The calculated SiO$_2$/Al$_2$O$_3$ ratio of the mix designs with the SF replacement levels of 0%, 5%, and 10% was 3.47, 3.84, and 4.2, respectively.

3. Sample Preparation and Test Method

3.1. Compressive Strength and Mass Change. Following molding, the mortar cubic specimens were placed in a sealed plastic bag and left at room temperature for 24 hr [36, 37]. Subsequently, the samples were demoulded and those intended for room temperature curing were immersed in LW, while the samples designated for heat treatment (Figure 1) were sealed in plastic bags and kept in an oven at a temperature of 80°C for 24 hr. After removal from the oven and reaching ambient temperature, the samples were submerged in LW. According to the research timeline, the compressive strength of the cubic specimens was measured and recorded at ages 7, 28, 90, 180, and 360 days. It’s worth noting that two parallel specimens were made for each mixing design at every age stage. The specimens designated for short- and medium-term testing were examined to investigate the trend of strength gain. Additionally, changes in the mass of the cubic specimens were measured at predetermined ages.

3.2. X-Ray Diffraction Analysis (XRD). The XRD test was applied to recognize the products formed in the hardened paste of mortar specimens. Following the compressive strength test at the ages of 7, 28, 90, 180, and 360 days and crushing the cubic specimens, their outer segments up to a depth of 10 mm were selected. The hardened paste powder was then separated by passing through a standard sieve No. 200. The powder specimens were placed in the oven at a temperature of 50°C for 14 days to give out their moisture completely. Thereupon, the specimens were placed in sealed
plastic bags immediately after removing the samples from the oven. This test was performed by Unisantis XMD300. To identify the products formed in the hardened pastes, based on the earned results from the XRD test, an analysis was performed applying HighScore Plus software.

3.3. SEM Imaging and EDS Analysis. Selected fragments of the crushed cubic specimens, measuring approximately 10–20 mm in size, were subjected to a temperature of 50°C for 14 days to ensure complete moisture removal from their pores. In the concrete water absorption test as per the ASTM C642-21 standard [38], to facilitate moisture evaporation from the specimens’ pores, they must be cured at temperatures ranging from 100 to 110°C for a minimum duration of 24 hr. However, in this study, mortar samples were subjected to a temperature of 50°C for a period of 14 days to mitigate the risk of thermal shock from abrupt exposure to high temperatures, subsequently inducing cracks on the paste surface. Throughout this process, the mass of the samples was monitored daily until it stabilized. Subsequently, they were promptly placed in sealed plastic bags until the time of SEM analysis. Prior to SEM imaging, the surfaces of the samples were coated with a thin layer of gold. The application of a gold layer served the following purposes: firstly, to prevent surface charging, thereby facilitating the emission of secondary electrons and ensuring even conductivity across the specimen. Additionally, it was employed to furnish a homogeneous surface conducive to analysis and imaging [39].

4. Test Results and Discussion

4.1. Mass Change. Figure 2 displays the progression of mass change in the samples over time. As observed, all samples
exhibited an increase in mass over time. This trend could be attributed to the formation of hydration products from slag and their replacement with the alkaline solution within the system [40, 41]. Another contributing factor may be the formation of pozzolanic reaction products in the SF-blended samples. Additionally, some researchers have reported an increase in mass due to carbonation [42, 43], while others have noted a decrease in mass due to calcium leaching into the surrounding solution [8, 44]. These various influencing factors make it challenging to compare sample durability based on mass change results. For instance, in the case of ambient treatment (Figure 2(a)), the mass gain of SF-free specimens was higher than that of SF-containing samples. It’s important to note that a higher mass gain in the SF-free sample does not necessarily indicate a higher specific gravity compared to specimens containing SF. This observation can be attributed to differences in the products resulting from the alkali activation of slag and the pozzolanic reactions of SF. Specifically, replacing SF leads to a decrease in the CaO/SiO₂ ratio, subsequently resulting in lower specific gravity in the reaction products [45]. Additionally, the presence of remaining unreacted SF particles can contribute to a reduction in mass gain. However, in the case of heat-treated samples, an opposite trend was observed. This discrepancy might be linked to the majority of pozzolanic reactions occurring at early ages due to the heat treatment. Nonetheless, it’s essential to acknowledge that while the results of the mass change test provide insights into other tests, discussing them separately may not significantly contribute to the primary objectives of the study [16].

Despite this, it should be noted that the mass change of the heat-treated samples (Figure 2(b)) was lower than that of the ambient-cured samples (Figure 2(a)). This can be explained that heat treatment accelerates the formation of slag hydration products. Therefore, a significant part of these products is formed at early ages. This reduces the trend of mass gain of the samples at higher ages.

4.2. X-Ray Diffraction Test. Figure 3 presents the XRD diffraction pattern diagrams of the samples. Notably, a high-intensity peak at 2θ = 26.7° was observed in the AAS diagrams, accompanied by the presence of vaterite, quartz, and C—S—H phases in the pattern list generated by HighScore Plus software. The predominance of calcite and the emergence of vaterite among the identified phases suggest the occurrence of carbonation [46, 47]. Vaterite phase was absent in the diffraction pattern of OPC specimens, indicating OPC’s superior resistance to carbonation compared to AAS [42]. The products formed in the XRD diffraction patterns in this study need to be compared with those reported in previous research on the long-term performance of AAS concrete/mortar. However, Bernal et al. [27] and Wardono et al. [28] did not employ XRD testing, and in the study by Humad et al. [29], XRD testing was conducted only at 7 and 28 days of age. In contrast, XRD testing in our study was carried out at longer term ages, making direct comparison challenging. Nonetheless, Ghahramani et al. [48] investigated the effect of carbonation on AAS and observed the formation of calcite and vaterite phases in the XRD diffraction pattern of AAS specimens. This observation supports the occurrence of carbonation in the AAS mortar samples in our study.

The peak intensity at 2θ = 26.7° and 2θ = 29.4° exhibited significant changes in the diffraction pattern of AAS specimens between ages 180 and 360 days. Vaterite is one of the low stability forms of calcium carbonate, which transforms to calcite after a while [47]. This transformation could be a contributing factor to the variation in peak intensity.
Another potential factor is the leaching of calcium ions from C─S─H into the LW solution. Additionally, the ongoing hydration of slag and pozzolanic reactions leading to the formation of C─S─H phase may play a role in this variation. Furthermore, the formation of different phases of calcium carbonate due to C─S─H calcification should also be considered. It is noteworthy that the quartz peaks forming in the diffraction patterns are due to the sand used in the mixture of the mortar samples. The amount of this phase may accidentally alter in various specimens causing alteration in the peaks’ intensity.

4.3. SEM Images. Figures 4–6 depicts the SEM images of the samples at the ages of 180 and 360 days. As can be seen, microcracks were observed in the AAS samples. This phenomenon was displayed in a study performed by Wardhono et al. [28]. It may be related to chemical shrinkage caused by hydration [49], rapid alkali reaction of slag at early ages [50], and carbonation [51]. Another notable observation was the presence of porosity in most AAS mortar samples, which appeared to increase over time from 180 to 360 days of age, as evidenced in the images shown in Figures 4 and 5. This increase in porosity could indicate carbonation of calcium–silicate–hydrate (C─S─H) gel [51] and/or related to the leaching of calcium ions from C─S─H toward the surrounding solution [52]. In contrast, porosity was not observed in the SEM images of the OPC specimens (Figure 6), suggesting greater resistance of OPC compared to AAS mortar in terms of carbonation and leaching. This resistance is attributed to the reaction of CO₂ with portlandite formed in OPC mortar, forming a protective coating (calcium carbonate) on C─S─H [53]. Similarly, Humad et al. [29] observed a porous texture in SEM images of carbonated AAS specimens. The presence of the alkaline activator in the pores of the system may increase the leaching tendency of AAS mortars [2]. Furthermore, as observed in Figures 4and 5, the heat-treated samples exhibited higher porosity compared to those cured at ambient temperature. The presence of a microcrack network in the heat-treated samples may accelerate carbonation and leaching trends, leading to increased porosity in the sample microstructure. The cracks in the SEM images of the ambient-cured specimens appeared to increase with the curing age, whereas the opposite trend was observed in the heat-treated specimens. This phenomenon may amplify the effects of
FIGURE 5: SEM images of the heat-cured AAS samples: (a) SF0.80.180 days, (b) SF0.80.360 days, (c) SF5.80.180 days, (d) SF5.80.360 days, (e) SF10.80.180 days, and (f) SF10.80.360 days.

FIGURE 6: SEM images of the OPC samples: (a) OPC.180 days and (b) OPC.360 days.
carbonation and leaching on the mortar sample, consequently leading to the formation of additional surface porosity. The heightened porosity in these samples could potentially obscure the observation of microcracks.

4.4. Compressive Strength. Figure 7 show the trend of compressive strength gain of the samples. As delineated in Section 3.1, two parallel specimens were designated for each compressive strength test. The results presented in the aforementioned figure encompass the average compressive strength of the two specimens ($\mu$) alongside their coefficient of variation ($1s\%$). According to ASTM C109/C109M-20b standard, the minimum acceptable values of the coefficient of variation are 3.6 and 3.4 for the ages of 7 and 28 days, respectively. The coefficient of variation observed in the results meets the acceptable criterion outlined above.

This strength decrease was not sighted in the OPC mortar results. The strength reduction of the ambient-treated AAS mortars with the SF replacement levels of 0%, 5%, and 10% from the age of 180–360 days was 37.8%, 23.4%, and 23.1%, respectively. While the increase in compressive strength of OPC mortar in the same period was 1.8%. The superiority of OPC mortars over AAS may be attributed to their carbonation resistance advantage, which was mentioned in the XRD test results discussion.

As shown in Figure 7, the compressive strength of the SF-free and ambient-cured AAS mortars (SF0.23) in the short-term (up to the age of 28 days) was lower than that of OPC. The sluggish trend of strength development in AAS mortars at early ages could be attributed to the low SiO$_2$/Al$_2$O$_3$ ratio in the mixture, approximately equal to 3.5. This finding is consistent with observations made Bernal et al. [27]. The inclusion of SF increases the SiO$_2$/Al$_2$O$_3$ ratio in the mixture. As can be seen in Figure 7, the compressive strength of the AAS mortars containing 5% and 10% SF surpassed that of the OPC after the age of 28 days. This tendency confirms the effect of the SiO$_2$/Al$_2$O$_3$ ratio on the strength development of the AAS mortar. The compressive strength of the AAS mortars increased until the medium-term ages, after which a decline in strength occurred. This reduction in strength was not observed in the OPC mortar results. The strength decrease of ambient-treated AAS mortars with SF replacement levels of 0%, 5%, and 10% from the age of 180–360 days was 37.8%, 23.4%, and 23.1%, respectively. In contrast, the increase in compressive strength of OPC mortar during the same period was 1.8%. The superiority of OPC mortars over AAS may be attributed to their advantage in carbonation resistance, as mentioned in the XRD test results discussion.

The compressive strength of the AAS mortars exhibited improvement due to heat treatment. At 360 days of age, the compressive strength of heat-treated AAS mortar samples increased by 81.1%, 30.5%, and 18.6% compared to those cured at ambient temperature for SF-replacement levels of 0%, 5%, and 10%, respectively. As mentioned in Section 1, previous research on AAS thermal curing has yielded
conflicting results. One reason for this inconsistency could be attributed to the sealing of the samples during the heat treatment process. Inadequate sealing may lead to moisture escaping from the system’s pores, thereby halting the enhancement in strength. Some of the research mentioned above did not provide details on sample sealing. Furthermore, variations in factors such as slag composition, type/concentration of alkaline activator, and curing regime among these studies make it difficult to compare them regarding the effect of heat treatment on compressive strength. The increase in compressive strength resulting from heat treatment can be explained as follows: The release of hydroxide and silicate ions in the formed gel facilitates the generation of reaction products. These products enhance polymer chains and promote the dissolution rate of aluminosilicate materials, leading to a reduction in unreacted particles and a decrease in porosity [54].

The compressive strength of AAS mortar specimens demonstrated an increase with the partial replacement of slag with SF. At age 360 days, this increase was 91.8% and 104.7% for the ambient-cured samples with SF replacement levels of 5% and 10%, respectively. Similarly, these increases were 38.1% and 34.1% for the heat-cured samples. The previous studies mentioned in Section 1 regarding the substitution of SF were limited to evaluating the effectiveness of AAS for up to 90 days. Therefore, the results of the present study were not directly comparable with those studies. However, the increase in compressive strength of AAS due to SF replacement was a collective outcome of these studies. The pozzolanic property of SF leads to the formation of C─S─H gel through its reaction with Ca(OH)₂ produced from slag activation. These reaction products fill the existing voids, thereby enhancing the compressive strength of the mortars. Additionally, some SF particles that do not participate in the pozzolanic reaction contribute to the strength increase by filling the pores [23].

As seen in Figure 7, the optimal SF replacement level changed with time. Consequently, direct comparisons and determination of the best SF replacement level were not feasible. Therefore, a durability prediction was conducted considering potential aggressive factors such as carbonation and leaching. The 28-day compressive strength of OPC mortar served as the baseline for this prediction. By using the return to this value as a criterion, the durability of the mortar samples could be estimated through linear extrapolation based on the functions depicted in Figure 8. The fitting of the curve on the points related to all ages did not result in a favorable match. Therefore, the results of 6 and 12 months were used for the fitting. Figure 9 illustrates the predicted durability of the AAS mortar specimens. Notably, heat
SiO₂ content has been supported by the replacement levels, respectively. The influence of this phenomenon could be attributed to chemical shrinkage resulting from hydration, rapid alkali reaction of slag at early ages, and carbonation. Similarly, the heat-cured specimens showed declines of 24.4%, 30.1%, and 31%, respectively. As mentioned in Section 1, Bernal et al. [27], Wardhono et al. [28], and Humad et al. [29] documented a decline in compressive strength over the long-term. The reported strength reductions from 180 to 360 days of age by these authors, comparable with the present study’s results, are depicted in Figure 10. In this context, the SF-free samples of this study were compared with those of the referenced studies. These studies differed in various aspects such as the physical characteristics and chemical composition of slag, the type and concentration of activator, and curing conditions. For instance, while the specimens in the present research were submerged in LW during curing, Humad et al. [29] and Wardhono et al. [28] applied nonsubmerged conditions with sealed and nonsealed status, respectively.

The various factors that can explain this trend are outlined below.

(i) Measurement error was one of the potential factors affecting the results. Hence, apart from regularly calibrating the measuring device, a thorough investigation was conducted to ensure its accuracy. Furthermore, the coefficient of variation for all seven mixture combinations was below 3%, which meets the requirements of ASTM C109/C 109M-20b [32] standard. Hence, the influence of this factor appears to be negligible.

(ii) Larrard and Bostvironnois [55] and Liew et al. [33] attributed the decrease in strength to the high SF content in the system. However, as indicated in Figure 7, the rate of strength reduction in the ambient-cured AAS mortar specimens without SF replacement (SF0.23) was higher than that of the SF-blended samples (SF5.23 and SF10.23). This suggests that this factor may have a low likelihood of effectiveness in the present study.

(iii) Heat curing may contribute to the long-term strength reduction. Accelerating the reaction rate of slag grains compared to the diffusion rate of reaction products can lead to their accumulation around the slag particles, resulting in structural discontinuity and porosity [14]. This porosity can subsequently decrease compressive strength. The presence of porosity in the SEM images of heat-treated samples in Figure 5 supports the influence of this phenomenon.

(iv) An extension in the interconnected microcracks observed in the SEM images of samples may contribute to the decline in strength. As discussed in sub-section 4.3, this could be attributed to chemical shrinkage resulting from hydration, rapid alkali reaction of slag at early ages, and carbonation.

(v) The detachment of calcium from the C—S—H gel as a result of carbonation can be considered a significant factor in this context. Exposure of mortar/concrete to water, which serves as a source of
CO₂, accelerates the process of carbonation [56]. Several findings in the current study support the role of carbonation in the long-term reduction of strength. The presence of calcite and vaterite phases in the XRD diagrams of slag-based samples, as depicted in Figure 3, provides evidence of carbonation [57]. Additionally, the porous structure and microcracks observed in the SEM images of the AAS specimens, as shown in Figures 4 and 5, further indicate the occurrence of natural carbonation [57]. Moreover, previous studies by Jun et al. [58] and Humad et al. [29] have reported a decrease in compressive strength attributed to carbonation. Mohamed [59] also highlighted a higher degree of strength reduction due to carbonation in AAS systems activated through the hybrid utilization of Na(ΟH)₂ and Na₂SiΟ₄. These findings collectively support the impact of carbonation on AAS concrete/mortar strength reduction.

(vi) Some authors have suggested that the leaking of alkali ions through the mortar pores into the surrounding solution could contribute to the reduction in strength [2, 60]. While this phenomenon typically coincides with a decrease in mass [8, 44], in our study, all samples exhibited a gain in mass over time. However, due to other contributing factors leading to mass gain, the impact of this leaking tendency was not ignored. Moreover, as illustrated in Figure 10, the rate of strength reduction observed in the studies by Humad et al. [29] and Wardhono et al. [28] was notably lower compared to our findings. Considering the nonsubmerged curing conditions in those studies, the substantial disparity in strength reduction observed in our research could be attributed to the occurrence of leaking phenomena associated with the immersion condition. Therefore, it is plausible that leaking plays a significant role in the observed strength reduction.

(vii) The leaking of calcium ions from C−S−H gel through the pores into the surrounding solution may also be a significant factor. Similar to the leaking of alkali ions, this process typically results in a loss of mass from the samples [8, 44]. If this phenomenon is indeed occurring, its rate of mass loss would likely be overshadowed by the mass gain associated with other contributing factors. The arguments presented regarding the leaking of alkali ions support the possibility of calcium ions leaking as well.

(viii) Some researchers have highlighted the influence of a high SiΟ₂/AlΟ₃ ratio on the long-term compressive strength [28, 61]. Excess SiΟ₂ content can slow down the reaction rate of slag particles, resulting in unreacted alkaline activator solution remaining in the pores, which increases the leaking potential of this solution. Wardhono et al. [28] observed a 2.01% decrease in the compressive strength with a SiΟ₂/AlΟ₃ ratio of 2.59 from 180 to 360 days of age. However, in our study, the strength reduction for samples with similar ratios was 37.8% (SF0.23 with a SiΟ₂/AlΟ₃ ratio of 3.47). Furthermore, the slope of the compressive strength diagrams shown in Figure 8, indicates the rate of strength reduction of the mortar samples. The substitution of SF for the ambient-cured AAS mortars resulted in a decrease in their slopes. These inconsistent findings suggest a minor effect of this factor.

The aforementioned factors exhibit overlapping symptoms and often occur simultaneously. Consequently, isolating their individual contributions is challenging. However, the presence of calcite and vaterite phases in the XRD diffraction pattern of the AAS samples suggests carbonation. Additionally, the observed strength reduction rates in our study closely align with findings by Ghahramani et al. [48] concerning carbonation. Thus, carbonation and leaching emerge as primary contributing factors. Nevertheless, a high SiΟ₂/AlΟ₃ ratio, heat curing, and the proliferation of interconnected cracks may exacerbate the strength reduction.

In contrast to the AAS mortar, OPC samples did not exhibit a reduction in long-term compressive strength. This resilience may stem from OPC mortar’s superior resistance to carbonation. The absence of the vaterite phase in the XRD diffraction pattern and the non-porous morphology observed in the SEM images of OPC samples corroborate this advantage. OPC paste replaces CaCO₃ with Ca(ΟH)₂ arising from the carbonation reaction in OPC paste [62]. Moreover, due to not using any alkaline activator in OPC mortar, its performance in terms of alkaline ion leaching is better than AAS.

5. Conclusion

This study aimed to evaluate the effect of curing at temperatures of 23 ± 2 and 80°C, as well as the substitution of SF at levels of 0%, 5%, and 10% on the long-term performance of AAS mortar submerged in LW solution. The results of this research can be summarized as follows:

(i) The replacement level of 5% SF yielded the most favorable outcomes. At the age of 360 days, for curing temperatures of 23 ± 2 and 80°C, this SF substitution level led to a 91.8% and 38.1% increase in compressive strength of the AAS mortar samples, respectively. Correspondingly, the predicted durability increased by 134% and 16.4%, respectively.

(ii) Elevated temperature curing positively influenced the performance of AAS mortars. Heat-cured AAS mortars exhibited a predicted durability increase of 118.5%, 8.7%, and 7.1% compared to ambient-cured ones for SF substitution levels of 0%, 5%, and 10%, respectively.

(iii) The combined use of heat treatment and SF substitution enhanced the durability of AAS mortars in
LW solution. The most durable specimen was the heat-cured one with a 5% SF replacement rate.

(iv) A significant reduction was observed in the long-term compressive strength of AAS mortar specimens, contrasting with the performance of OPC mortars, which showed an increasing trend in compressive strength over time.

Data Availability
The data used to support the findings of this study are included within the article.

Conflicts of Interest
The authors declare that there is no conflict of interest regarding the publication of this article.

References


