Porous Fire-Resistant Materials Made from Alkali-Activated Electric Arc Furnace Ladle Slag

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Abstract: The application of electric arc furnace ladle slag (EAF ladle slag) in cement products might be limited due to the volume expansion and volume instability created by late hydration. Proper control technique should be developed before the reuse of ladle slag (LS). With the addition of aluminum powder in alkali-activated slag pastes, porous materials were produced. By adjusting the activator modulus between 1.25 and 2.00, fine pores were produced in the foamed pastes, and the material densities were controlled between 594 and 1184 kg/m³. The compressive strengths increased from 0.95 to 9.04 MPa with the increase in density. Direct firing tests showed that the produced porous materials could resist fire damage. With low thermal conductivities range from 0.532 to 1.435 W/m·K, the temperatures in the back panel of the materials were below 100 °C, even under flames of 800 °C for 1 h, which were better than marketing rock wool. The alkali-activated technique was proven to be applicable for the manufacturing of porous fire-resistant materials from ladle slag in this research.

Keywords: fire resistance material; porous material; alkali-activation; EAF ladle slag

1. Introduction

Steel slag is a solid by-product in the steel-making industry. It has high potential for use as a substitute for cement and concrete in building construction [1–4] or as an aggregate in road construction [5,6]. Steel slag is often categorized into carbon steel slag and stainless steel slag. The application of stainless steel slag, especially electric arc furnace ladle slag (EAF ladle slag), in engineering is restricted, depending on the amount of free-CaO (f-CaO) and free-MgO (f-MgO) in the slag. A high content of f-CaO or f-MgO might result in volume expansion or instability [7–9] because f-CaO and f-MgO might react with water in the paste and lead to Ca(OH)₂ and Mg(OH)₂ transformation. Volume expansion due to transformation would cause volume instability in concrete, which might potentially reduce its strength [10,11].

To restrain the volume expansion caused by f-CaO hydration, various techniques, such as weathering, autoclave, alkali activation, etc., have been applied to stabilize ladle slag [12]. Polanco et al. [13] used weathering preprocessing to hydrate most of the f-CaO in their study into calcium hydroxide (CH). Nevertheless, the hydration rate in ambient conditions is low; it often takes days for the weathering process to complete. Shi and Hu [14] mixed silica flour and ladle slag in proper proportions; the mix was then autoclaved under high temperature and pressure. The f-CaO was consumed and transformed to stable calcium silicate hydrate (CSH) relatively quickly. This reaction only takes hours to complete; however, pursuing high f-CaO hydration reactivity under high temperature and pressure sometimes means high energy consumption and might not be economically efficient. In alkali activation, a
high reaction rate is achieved by blending slags with alkaline. In the system, ladle slag was either introduced as an exclusive binder [15] or in combination with other precursors, such as metakaolin, bottom ash, and fly ash [16–18]. The f-CaO reacts with alkali activators and silicates rapidly and transforms to highly stable CSH-based products. In addition, the solid products also possess high compressive strength [19,20]. Nevertheless, variation in the parameters of the alkali-activation technique affect the product properties [20,21]. Proper parameter settings should be investigated for controlling these properties.

Originally, alkali-activated materials were developed for high temperature utilization such as coating for fire protection on cruise ships, thermal protection for wooden structures, heat adhesives, etc. [22–24]. In ordinary portland cement (OPC) concrete, calcium silicate hydrate (CSH) and calcium hydroxide (CH) dehydrate under high-temperature heating, and the chemical structures are seriously affected. The chemical structure of alkali-activated material is little affected by the dehydration reactions. The strength loss in alkali-activated materials subject to high-temperature heating can be prevented by the careful selection of the binder and aggregates [22]. Alkali-activated materials, when adequately synthesized and cured, consist largely of amorphous material, with a small content of crystalline phases. Si–O–Si and Si–O–Al bondings in alkali-activated materials exhibit remarkable thermal stability, retaining their amorphous structure until the onset of melting. Since the chemical structures in alkali-activated materials are relatively stable during the heating process, these materials possess heat-resistance properties [24,25].

In this study, the alkali-activated stabilization of ladle slag is discussed. To further utilize alkali-activated slag (AAS) as fire-resistant material, the thermal conductivities and stabilities of AAS under an 800 °C flame were investigated. Aluminum powder was also introduced as a foaming agent for the enhancement of the fire-resistance properties.

2. Materials and Methods
2.1. Materials

Ladle slags were sampled in an electric arc furnace steel-making factory in central Taiwan. EAF ladle slags size below 8/3 mesh (9.5 mm) were sampled and then ground to 100 mesh (0.14 mm); the ground slags were dehydrated and preserved in containers (ladle slag in this research). Ladle slags were acid-digested with aqua regia and hydrofluoroboric acid. In order to enhance the digestion efficiency, the digestion was performed in a microwave-assisted digestion oven. The digested solutions were analyzed with induced coupled plasma spectrometer (ICP-OES, Perkin-Elmer, Waltham, MA, USA, Optima 2000 DV) for major elemental analysis (Si, Al, Ca, Fe, Mn and Mg). Other, minor elements were analyzed with X-ray fluorene (XRF, Olympus, Tokyo, Japan, DP-4050). Leaching characteristics were analyzed according to toxicity characteristics leaching procedure (TCLP, USEPA Method 1311/NIEA R201.15C). Chemical composition in Table 1 shows that the ladle slag composites mainly contained Si, Al, Ca, Fe, Mn and Mg, which possesses the potential to be reused in cementitious material. TCLP test showed that concerned heavy metal concentrations in leachate are all below regulatory standards (Standards for Defining Hazardous Industrial Waste, Taiwan) provided in Table 1, and the slag could be considered as ordinary industrial waste. Nevertheless, the results also showed that Ca content in ladle slags is high, which might cause volume instability in the late hydration age. Hence, further evaluation, such as autoclave expansion test (ASTM C151/C151M), was employed to determine the volumetric stability of produced materials.
Table 1. Elemental and leaching characteristics of ladle slags.

| Elements   | Chemical Compositions | TCLP Leachate Concentrations (mg/L) | Regulatory Standards (mg/L) |
|------------|-----------------------|-------------------------------------|-----------------------------|
| Major Elements (%) |                       |                                     |                             |
| Si         | 2.26 ± 0.007          | –                                   | 1                           |
| Al         | 12.65 ± 0.634         | –                                   | 1                           |
| Ca         | 31.71 ± 0.927         | –                                   | 1                           |
| Fe         | 0.89 ± 0.029          | –                                   | 1                           |
| Mg         | 3.22 ± 0.034          | –                                   | 1                           |
| Mn         | 0.33 ± 0.001          | –                                   | 1                           |
| Minor Elements (mg/kg) |               |                                     |                             |
| Ag         | –                     | ND                                 | 2                           |
| Ba         | –                     | 0.99                               | 100.0                       |
| Cd         | <4.3                  | –                                   | 1                           |
| Cr         | 120.1 ± 7.2           | ND                                 | 2                           |
| Cu         | 9.7 ± 1.5             | ND                                 | 15.0                        |
| Hg         | –                     | ND                                 | 0.2                         |
| Ni         | 33.5 ± 1.9            | ND                                 | 2                           |
| Pb         | 3.3 ± 0.7             | ND                                 | 2                           |
| Se         | –                     | ND                                 | 5.0                         |
| Zn         | 4.9 ± 1.3             | ND                                 | 50.0                        |

1 Not available; 2 not detected (<0.1 mg/L).

Ordinary portland cement (OPC) and blast furnace slag (BF slag, as basic binder in alkali activation) were acquired from market and steel company in southern Taiwan, respectively. Chemical compositions provided by each company are listed in Table 2 for further calculation of addition ratio. Industrial-grade sodium hydroxide (45%, specific density 1.48) and water glass (SiO<sub>2</sub>/Na<sub>2</sub>O = 3.21, 34.33 mass% water solution) were also acquired from the market for use as activators in alkali activation. Chemical-grade aluminum powder (Merck) was used as foaming agent.

Table 2. Chemical compositions of cement and blast furnace slag.

| Element (as Oxides, %) | OPC | BF Slag |
|------------------------|-----|---------|
| SiO<sub>2</sub>        | 22.1| 32.7    |
| Al<sub>2</sub>O<sub>3</sub> | 5.1 | 14.5  |
| Fe<sub>2</sub>O<sub>3</sub> | 3.1 | 0.4   |
| CaO                    | 64.6| 41.1   |
| MgO                    | 1.4 | 4.7    |
| SO<sub>3</sub>         | –   | 0.4    |
| Loss on ignition       | 1.1 | 0.6    |

2.2. Methods
2.2.1. Alkali Activation

Alkali activation was performed by mixing BF slag (as binder), sodium hydroxide, water glass (both as alkaline activators) and water in proper proportion. The dosage of alkaline activators was determined according to following equations.

Activator modulus (Ms): mass ratio of SiO<sub>2</sub> to Na<sub>2</sub>O in an alkaline activator.

Activator Modulus (Ms) = \( \frac{\text{mass of SiO}_2}{\text{mass of Na}_2\text{O}} \)
Alkaline concentration (Na₂O%): concentration of Na₂O in the paste, which divides the total mass of Na₂O by the mass of binder.

\[
\text{Alkaline concentration (Na}_2\text{O\%)} = \frac{\text{mass of Na}_2\text{O}}{\text{mass of Binder}} \times 100\%
\]

Water-to-binder ratio (W/B): water added in the paste is adjusted according to ratio of the mass of water divided by the mass of binder.

2.2.2. Volume Stability

Volume stabilities of samples were tested according to standard test method for autoclave expansion of hydraulic cement (CNS 1258/ASTM C151/C151M-18) [26]. Pastes with standard consistency were molded (25 × 25 × 285 mm). Researchers reported that ladle slag might have the potential to be used as replacement for cement in cement-based concrete [12,27]. However, the specimens would encounter volume instability with replacement percentage around 20–30%. To evaluate the volume stability of the ladle slag application, 20% (by mass) of binders were replaced by ladle slag in the cement system (OPC) and alkali-activation slag system (AAS), which are denoted by OPC+20% and AAS+20%LS respectively. The volume stabilities of specimens were tested according to autoclave expansion test. Molded specimens were autoclaved in 215 °C, 2 MPa for 3 h, then naturally depressurized to 0.07 MPa, and the specimens were placed in hot water, at 90 °C. The container was cooled to room temperature by adding water slowly. Expansion of specimens was measured by digital length comparator for evaluation of volume stability.

2.2.3. Pore Size Distribution

Samples were horizontally cut into three equal portions. The pore distribution of the cross-sections was observed with digital microscope. The images of magnified cross-sections were converted to 8-bit B/W files. ImageJ software developed by National Institutes of Health was applied for the pore identification and pore size distribution calculation.

2.2.4. Thermal Conductivity

Thermal conductivity was tested according to Fourier’s law of heat conduction. The equation could be expressed as follows.

\[
k = \frac{Q \times L}{A \times \Delta T}
\]

k: Thermal conductivity (W/m·K)
Q: Heat transferred across test sample per unit time (W)
L: Thickness of test sample (m)
A: Cross-section area of test sample (m²)
ΔT: Temperature difference (K)

2.2.5. Fire Resistance Test

Fire resistance test was performed by direct flame firing test, as shown in Figure 1. Tests were carried out by exposing plates to 800 °C acetylene flame on the center of plates. Back panel temperatures were measured at the center of the other side of plates by infrared thermometer (Tecman Electronic Instrument, Hong Kong, TD1500) at 10-min intervals.
3. Results

3.1. Evaluation of Volume Stability

Ladle slag (LS) contains high levels of f-CaO. When ladle slag is applied in cement and concrete, the f-CaO reacts with water and forms Ca(OH)$_2$, which has been reported to have 127.78% volume expansion in complete reaction situation [28]. The volume expansion of f-CaO transformation to Ca(OH)$_2$ led to slag pop-out from the specimen, and the volume of the specimen was unstable. The results in Figure 2 show that OPC + 20%LS cracked and, hence, was not stable. On the other hand, alkali activation transformed the reaction of f-CaO. Instead of reacting with water, the f-CaO reacted with alkaline and SiO$_2$ and formed C-S-H gel in the paste. Hence, the AAS + 20%LS specimen remained intact. Further length comparison results in Table 3 show that the replacement of the binder with LS (OPC + 20%LS) in the cement system cracked and no length comparison data are available. According to the CNS 1258 criteria, the autoclaved specimen should remain intact, and the length expansion percentage should be less than 0.8% to be deemed as volume-stable. AAS + 20%LS was intact; although it slightly expanded, the length expansion percentage of 0.1409% still met the criteria. The alkali-activation system was effective at controlling the volume stability of ladle slag during application.

![Figure 1. Layout of fire resistance test.](image1)

![Figure 2. Specimens for autoclave expansion tests (a) before autoclave and (b) after autoclave.](image2)

| Specimen         | Expansion Percentage (%) | Volume Stability (<0.8%) |
|------------------|--------------------------|--------------------------|
| OPC              | 0.1238                   | Stable                   |
| OPC + 20%LS      | ~ (cracked)              | Unstable                 |
| AAS              | 0.0976                   | Stable                   |
| AAS + 20%LS      | 0.1409                   | Stable                   |
3.2. Manufacturing Porous Material through Alkali Activation

3.2.1. Gas Generation

To manufacture porous material, two methods are typically used to introduce voids, i.e., mechanical foaming and chemical foaming. Aluminum-powder chemical foaming is used to generate gas in the alkali activation system according to the following reactions [29].

\[
2\text{Al} + 6\text{H}_2\text{O} + 2\text{NaOH} \rightarrow 2\text{NaAl(OH)}_4 + 3\text{H}_2
\]

\[
\text{NaAl(OH)}_4 \rightarrow \text{NaOH} + \text{Al(OH)}_3
\]

\[
2\text{Al} + 6\text{H}_2\text{O} \rightarrow 2\text{Al(OH)}_3 + 3\text{H}_2
\]

Although chemical foaming should be more effective at decreasing density, but it can result in the generation of large, disorderly voids that negatively affect the engineering properties [30]. The gas generated under different activator moduli was collected over water. The results in Table 4 show that by increasing the sodium hydroxide content (low Ms), the degree of aluminum reaction increased. With the decrease in Ms, gas was generated faster. For example, the time needed for generating 100 mL of gas volume significantly decreased, from 9.8 min to 3.2 min, with the decrease in Ms. Since the added Al powder amounts were fixed among these tests, the final gas volume calculated from theory should be the same. Nevertheless, the gas collected throughout the whole period significantly dropped, from 210 mL to 133 mL, with the increase in sodium silicate (high Ms). The reaction of aluminum powder was inhibited under high sodium silicate conditions. These results coincide with the phenomenon reported that aluminum reacts with alkaline in the presence of alkali accelerators and forms high amounts of aluminum hydroxide [31]. Furthermore, the aluminum hydroxide reacted with sodium silicate in the activator and formed a thin layer of amorphous aluminosilicate that covered the surface with aluminum powder and prevented gas formation. Hence, high sodium silicate amounts resulted in the formation of aluminosilicate species and low gas generation. With the addition of this amount of Al powder and binder, more gas bubbles are generated under low activator modulus.

Table 4. Gas generation under different activator moduli (Ms). (paste = 150 g, Na$_2$O% = 4%, W/B = 0.5, Al powder = 0.2%, LS replacement = 20%).

| Ms  | 1.25  | 1.50  | 1.75  | 2.00  |
|-----|-------|-------|-------|-------|
| $T_{50}$ (min) | 1.6   | 2.5   | 4     | 67    |
| $T_{100}$ (min) | 3.2   | 4.5   | 6.4   | 9.8   |
| $T_{\text{final}}$ (min) | 12.5  | 13.5  | 16.5  | 24.5  |
| $V_{\text{final}}$ (mL) | 210   | 190   | 168   | 133   |
| $V_{\text{theory}}$ (mL) |       |       |       | 260   |

3.2.2. Pore Observations

Further pore observations were made throughout the cross-sections of the materials. In Figure 3, the pores are large, interconnected, and in irregular shapes in Ms = 1.25. With the increase in Ms, the pore sizes decrease, the pores become disconnected, and the materials become denser. When the Ms increased to 2.00, the large pores almost vanished. From the cumulative size distribution, pores larger than 1.00 mm only contributed 0.68% with Ms = 2.00, which contributed far less than 28.72% with Ms = 1.25. Further statistics of the size distribution, presented in Table 5, showed that the average pore size dropped from 0.71 mm to 0.35 mm with the increase in Ms. Moreover, the ratios of D75/D25 decreased from 3.37 to 2.35 with the Ms increase from 1.25 to 2.00, which indicates that the pore size distribution was more concentrated in the high Ms.
Figure 3. Microscope images and pore size distribution of porous materials under different Ms.

Table 5. Pore size statistics under different activator moduli (paste = 150 g, Na$_2$O% = 4%, W/B = 0.5, Al powder = 0.2%, LS replacement = 20%).

| Pore Size (mm) | Ms 1.25 | Ms 1.50 | Ms 1.75 | Ms 2.00 |
|---------------|---------|---------|---------|---------|
| $D_{25}$      | 0.30    | 0.27    | 0.25    | 0.20    |
| $D_{50}$      | 0.56    | 0.46    | 0.41    | 0.32    |
| $D_{75}$      | 1.01    | 0.71    | 0.61    | 0.47    |
| $D_{average}$ | 0.71    | 0.51    | 0.45    | 0.35    |
The above results also coincide with the research reported by Hajimohammadi [29]. Lower Ms provide a higher proportion of alkaline, which leads to the faster dissolution of the binder and, consequently, samples that set quicker. At the same time, as mentioned above, lower Ms also accelerates aluminum reactions. With faster reaction and higher amounts of gas generated from Al powder, there would be a higher chance of creating larger gas bubbles. In combination with these two factors, larger bubbles are easier to form and capture; therefore, they form larger pores within materials. Controlling the Ms and the amount of Al powder could further control the pore structure and density of porous materials.

3.2.3. Density and Compressive Strength of Porous Materials

The density and compressive strength of porous materials made from different Ms is illustrated in Figure 4. Due to larger and interconnected pores, both the density and the compressive strength dropped with the decrease in Ms. When Ms = 2.0, the compressive strength was 9.04 MPa with density equal to 1184 kg/m$^3$. As the Ms decreased to 1.25, the compressive strength decreased to 0.95 MPa with density equal to 594 kg/m$^3$. These results are consistent with the results reported by Sanjayan [32]. By controlling the percentage of Al powder or activators, the density of porous materials could be controlled. Both the strength and the density behaved in the same manner and the strength of the materials decreased when the density was lower. Although it seems that the strength of the alkali-activated porous materials was well developed, it is not an acceptable material for construction engineering applications. Nevertheless, due to their low thermal conductivity, alkali-activated slag materials might be used as thermal barriers or in fire-resistant applications [33].

![Figure 4. Density and compressive strength of porous materials made from different Ms (Na$_2$O% = 4%, W/B = 0.5, Al powder = 0.2%, LS replacement = 20%).](image_url)

3.3. Fire-Resistance Applications of the Porous Material from Alkali-Activated Slag

Although fire-resistant materials could be produced based on the alkali activation of slags, to create materials with low thermal conductivity, low thermal transfer at high temperatures is required. Heat transfer at high temperatures is significantly affected by the efficiency of thermal radiation. Thermal radiation is a reciprocal function of the number of air–solid interfaces. Heat transfer/heat radiation could be reduced by creating as many air voids as possible in the materials. Hence, the foaming technique mentioned in the previous section by the addition of Al powder in alkaline condition was applied.

Plates (20 cm $\times$ 20 cm $\times$ 5 cm) with various densities, e.g., 600, 800 1000, and 2000 kg/m$^3$, were produced separately. Firing tests were carried out by exposing the plates to 800 °C acetylene flame. The back panel temperatures of the plates were recorded. The results in Figure 5 show that the OPC plates cracked under the flame. Fire would pass
directly through the crack and unreasonably high temperatures would appear; hence, no back-panel temperature was recorded. OPC-based concrete is not structurally stable when exposed to high temperatures. The main reason OPC-based materials fail after firing is that CSH-gel and other crystalline hydrates dehydrate and are destroyed during heating [34]. The back-panel temperature of the marketing rock wool is merely 156 °C, which makes it a good fire-resistant material. Nevertheless, since the surface of rock wool melts within 1 h, surface stability under longer firing times should be taken into consideration. Unlike rock wool, the surfaces of the AAS series plates only showed burnt marks due to accumulation of carbon black from the flame. The back-panel temperatures of the alkali-activated slag samples (AAS series) were below 100 °C, which is significantly lower than that of rock wool. With the decrease in density, the back-panel temperature dropped, which indicates that the fire resistance was better. The thermal conductivities in Table 6 also support this result. With lower density, the materials contained more pores; hence, thermal conductivities were lower. The thermal conductivity of porous AAS with a density of 600 kg/m³ (AAS-D600) was 0.532 W/m·K, which was almost one-third of the conductivity of the non-porous AAS (AAS) or OPC. Alkali-activated porous materials from EAF ladle slag are good fire-resistant materials.

![Figure 5. Back panel temperature of alkali-activated porous plates under 800 °C flame (Na₂O% = 4%, W/B = 0.5, Al powder = 0.2%, LS replacement = 20%).](image)

| Rock Wool | OPC |
|-----------|-----|
| 156       | cracked |
| 0.795     | melt  |

| Fire-Resistant Material Density (kg/m³) |
|---------------------------------------|
| 600  | 800  | 1000 | 2000 |
| 156  | cracked |
| 63   | 67    | 72    | 94    |
| 0.795 | melt |
| 0.532 | burnt mark |
| 0.602 | burnt mark |
| 0.908 | burnt mark |
| 1.435 | burnt mark |

4. Conclusions

It may be stated from the above results that alkali-activated porous materials from EAF ladle slag are good fire-resistant materials; thus, alkali activation and pore generation control are the essential factors in this reuse of ladle slag. The key results can be summarized as follows:

1. The alkali activation system is effective at controlling the volume stability of ladle slag during application. Unlike the OPC + 20%LS specimen, which cracked in the
autoclave expansion test, the AAS + 20%LS was intact; although it slightly expanded, the length expansion percentage still met the CNS 1258 criteria.

2. With the amount of Al powder and binder addition fixed, the low activator modulus generated more gas bubbles. A thin layer of amorphous aluminosilicate might have been formed from the reaction of aluminum hydroxide and sodium silicate and might have covered the surface of aluminum powder and inhibited gas formation.

3. Controlling the Ms could further control the pore structure and density of the porous materials. The lower Ms provided a higher proportion of alkaline, which led to faster dissolution of the binder, and the samples set quicker. At the same time, the lower Ms also accelerated the aluminum reaction. Controlling the Ms allows control of the size of the bubbles and setting time, and, hence, the pore structure and density of materials.

4. Both the strength and density of the porous materials behaved in the same manner. The strength of the materials decreased when the density was lower. The strength of the porous materials might not be acceptable for construction purposes; nevertheless, the porous material might be used as a thermal barrier or in fire-resistant applications.

5. The thermal conductivity of porous AAS with density 600 kg/m$^3$ was 0.532 W/m·K, which was almost one-third of the conductivity of the non-porous AAS (AAS) or OPC, and is comparable to 0.795 W/m·K of rock wool. The surfaces of the AAS series plates only showed burnt marks due to the accumulation of carbon black under 800 °C flame. Alkali-activated porous materials from EAF ladle slag are good fire-resistant materials.

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