Review Article

Advances in Understanding the Alkali-Activated Metallurgical Slag

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Received 14 April 2021; Accepted 8 May 2021; Published 25 May 2021

Academic Editor: Tingting Zhang

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This paper summarized and reviewed the mechanism and macro-performance of alkali-activated metallurgical slag, including steel slag, copper slag, ferronickel slag, and lead-zinc slag. Better activated method and alkali-activator are still needed to be developed to improve the performance of the metallurgical slag with low reactivity. Besides, the chemical components' variation of these metallurgical slags from different regions will lead to unpredictable performance, which needs further study.

1. Introduction

It is widely accepted that alkali-activated material (AAM) is a potential alternative for ordinary Portland cement (OPC) [1]. These materials are commonly generated by aluminosilicate precursor, which can be obtained from solid industrial waste, such as granulated blast furnace slag (GBFS), fly ash, mineral processing tailings [2], catalyst residues, waste glass, waste ceramic, coal bottom ash [3], rice husk ash [4], palm oil fuel ash, etc. [5]. In 2016, approximately 1.45 Gt CO₂, which is about 8% of CO₂ total emissions from human activities, was released from the cement industry [6]. About 50%–60% CO₂ emission of cement industry comes from the calcination of limestone. Therefore, the replacement of OPC by AAM is recognized as one potential way to reduce carbon emission [7]. Life-cycle analysis (LCA) of alkali-activated materials has been thoroughly discussed by Habert. According to the statistics of different studies, LCA of AAM reduces approximately 40%–80% CO₂ emissions compared to an OPC baseline [1]. Need to add that the baselines of OPC are specified inconsistently among various reports because the mix design, local conditions (such as transport distances and cost of electricity generation) as well as industry and environmental policy significantly affect the baseline [1].

Compared to OPC, AAM could have superior properties through reasonable design. Better acids and sulfate resistance [8], higher strength [9], and higher temperature resistance [10] were reported in AAM. These advantages of AAM are mainly achieved by the presence of aluminosilicate gel such as hydrated calcium aluminosilicate gel (C-A-S-H) and hydrated sodium aluminosilicate gel (N-A-S-H). These two kinds of aluminosilicate gel are different from the main product, namely, C-S-H in cement. A highly crosslinked silicon structure of Q3 and Q4 with less bound water was found in aluminosilicate gel due to the substitution of aluminum for silicon [11], which contributed to the better properties of AAM. Furthermore, AAM shows a better performance in alkali-aggregated reactions (AAR) compared to OPC [12].

According to the different precursors activated, AAM is commonly divided into two different binder systems [5]. The primary type of alkali-activated binder is high-calcium alkali-activated binder mainly derived from alkali-activated blast furnace slag (AAS) [13]. The secondary-type binder system is the so-called “geopolymer” mainly derived from alkali-activated fly ash (AAF). The former system is dominated by C-A-S-H products with a tobermorite-like structure (mainly Q2 with smaller parts of Q1 and Q3) [14]. The main product in geopolymer is N-A-S-H with a zeolitic-like

structure (mainly Q4 with few Q3) [12]. Blast furnace slag is more reactive than fly ash, as a higher pH and temperature is needed to activate the fly ash [5]. This means AAS has a wider range of activators compared to AAF, such as sodium carbonate and sodium sulfate. Besides the different gels in these two systems, a wide range of secondary phases such as hydrotalcite and AFm-like crystal were observed in AAS [15].

Recently, various metallurgical slags are generated in the production of metal processing, such as steel slag, copper slag, ferronickel slag, and lead-zinc slag. Steel slag is a solid waste generated during the conversion of iron into steel, which is about 15% of the crude steel output [16, 17]. Copper slag is an industrial by-product produced by the copper-making process, whose yield is 2–3 times that of copper output [18, 19]. Ferronickel slag as an industrial waste comes from the process of nickel-iron alloy production [20, 21]. Approximately 12–14 tons of ferronickel slag per ton of nickel is produced [22, 23]. The main solid waste generated during lead and zinc production is lead-zinc slag. According to statistics, the extraction cost per 100 tons of lead and zinc is 71 tons of lead slag and 96 tons of zinc slag [24, 25]. It is estimated that the annual production of steel slag, copper slag, ferronickel slag, and lead-zinc slag each year worldwide is 200 million tons, 70 million tons, 150 million tons, and 25 million tons, respectively [17, 23]. Although the yield of these slags is high, the utilization rate is low. For example, until now, the steelmaking industry produced nearly 1.2 billion tons of steel slag in China, and only less than 30% slag is recycled and applied in some low value-added fields [26, 27]. Most of metallurgical slags are stockpiled in an open field. Dealing with a large amount of industrial waste is a severe challenge to global environmental governance. The best way to solve this problem is to transform metallurgical slags into new materials with high added value, which will also bring huge economic benefits to the society.

Most metallurgical slags are used as potential alternative materials in civil engineering. They are usually used as aggregates or fillers in place of other conventional sand and stone materials due to their low activity [28, 29]. Powdered slag has higher market value as compared to granulous slags for construction. A possible application for those solid wastes is to produce alkali-activated materials because they are high-quality aluminosilicate resources. With the further implementation of the concept of sustainable development, the research on cement with less clinker and no clinker has been paid more attention. Alkali-activated material as a kind of inorganic polymer material has great potential and is expected to be an alternative to cement and concrete. Meanwhile, in recent decades, with the development of mine cemented backfill technology in underground mine backfill, more and more mines use cementitious materials and alternative binders to replace conventional hydraulic backfilling at home and abroad [30]. Compared to building materials, the quantity of mine backfill material is large and the strength requirement is easily met. When alkali-activated materials are used to replace other conventional materials for mine backfill, it helps to effectively deal with the solid wastes, substantially preserve natural resources and energy,

and create the conditions for reducing potentially harmful waste disposal costs. Hence, it indicates that alkali-activated material is suitable for mine backfill.

Within this context, the purpose of this paper is to review steel slag, copper slag, ferronickel slag, and lead-zinc slag as a precursor in alkali-activated material. The challenges and opportunities of using slags in alkali-activated material are also discussed.

2. Alkali-Activated Steel Slag Material

2.1. Physical Properties and Chemical Composition. The type of modern steel determines the elimination of different impurities in the steelmaking process. In terms of carbon steel, it can be produced in a ladle furnace (LF), an electric arc furnace, and a basic oxygen furnace (BOF) in different countries [30–32]. Thus, depending on the type of furnace, steel slag can be broadly classified into three categories, i.e., BOF steel slag, EAF steel slag, and LF steel slag [33]. As for steel slag in alkali-activated material, it usually refers to BOF steel slag, which is also called converter steel slag [34]. Today, in China and the United States, BOF steel slag makes up approximately 70% and 40% of steelmaking, respectively [35, 36]. BOF steel slag is rock-like and dark. The density is 3–3.6 kg/m³, which is higher than the natural aggregate [37]. The water absorption rate of steel slag is 0.4%–3.5% [38, 39]. BOF steel slag is very hard and not easy to be ground due to its high Fe content, so BOF steel slag and its products have good abrasion resistance [37, 38].

Different chemical compositions are heavily affected by steel slag type. BOF slag has more FeO than EAF steel slag and less SiO₂ than LF steel slag [35, 37]. The main chemical compositions of BOF steel slag are presented in Figure 1. In general, BOF steel slag primarily consists of 35%–50% CaO, 15%–35% Fe₂O₃, 10%–20% SiO₂, 2%–10% MgO, 0%–5% MnO, 1%–7% Al₂O₃, 1%–3% P₂O₅, and 0%–2% TiO₂. It is worth noting that there is a great difference in Fe₂O₃ content. High Fe₂O₃ content in steel slag plays an important role in grinding and application quality of steel slag. However, with the improvement of magnetic separation technology of BOF slag, the Fe₂O₃ content in BOF slag has been effectively reduced [26, 37]. The chemical composition analysis on newly produced slag has showed that the total amount of Fe₂O₃ is less than 20%.

Steelmaking slag is usually air-cooled to ambient conditions, and so BOF steel slag is highly crystallized [38, 39]. Those oxides in BOF steel slag form different mineralogical compositions. Essential mineral phases in BOF steel slag are tricalcium silicate (C₃S), dicalcium silicate (C₂S), CaO-FeO-MnO-MgO solid solution (RO phase), dicalcium ferrite (C₂F), tetracalcium aluminoferrite (C₄AF), merwinite (Ca₃MgSi₂O₈), lime (free CaO), and periclase (free MgO) [16, 26, 37, 40]. The Fe mainly exists in forms such as RO phase, C₄AF, and C₂F, and these phases have no sufficient reactivity. During cooling, C₂S undergoes polymorphic transformations, where β -C₂S transforms γ -C₂S at approximately 500°C, resulting in volumetric expansion of 12% [37–39]. A small amount of C₃S and β -C₂S with dense structure and large crystal size have low reactivity, while

FIGURE 1: The chemical composition of BOF steel slag.

γ -C₂S is considered to have a negligible cementitious capability [41]. Therefore, BOF steel slag powder can result in poor hardening reaction after prolonged curing at room temperatures but show better cementitious properties under the action of chemical activator. The content of free CaO increases with the alkalinity of steel slag. And, even the content in BOF steel slag is up to 10%, which has a negative impact on the stability of steel slag products. Although the contents of free CaO and MgO in BOF steel slag have become very low with the improvement of heat and vapour process, the stability of steel slag should be considered when it is used as aggregate [37, 42, 43].

2.2. Reaction Mechanism of Alkali-Activated Steel Slag.

Compared to amorphous GBFS, fly ash, and metakaolin, the biggest challenge of steel slag as a precursor is its high crystallization. Therefore, it should have different inorganic polymerization mechanisms between alkali-activated steel slag materials and alkali-activated amorphous slag materials. Although few in number, some publications regarding the exploration of the reaction mechanism of steel slag in the production of alkali-activated materials do exist in literature.

The hydration sensitivity and even mechanical behavior of the material to activation depends on several factors, such as the phase compositions and fineness of the precursor, the curing conditions and alkaline conditions containing initial alkalinity, and the type and concentration of activator used [37]. Wang et al. [44] changed the pH value of NaOH solution in NaOH-activated steel slag and studied effects on kinds and morphologies of hydration products. They found that although increasing the initial alkalinity could promote the early hydration of active components like C₂S, C₃S, and C₁₂A₇, it had little effect on their late-age hydration degree [44]. As for inert components like Fe phases, the hydration degree of steel slag was still very low even under strong alkaline conditions [44]. They also found that changing the alkaline conditions did not change the type of hydration products [44].

The alkaline activator has a very important function. According to research findings, compared to sodium sulfate, sodium hydroxide, and sodium carbonate as activators, liquid sodium silicate (water glass) could activate steel slag more efficiently and is an appropriate activator for alkali-

activated steel slag materials [34, 37, 45, 46]. Sun et al. [47] had investigated the hydration properties and microstructure characteristics of alkali-activated steel slag binder [47]. According to their findings, both hydration processes and products between water glass-activated steel slag and Portland cement were similar: (i) five hydration stages including the rapid exothermic stage, the dormant stage, the acceleration stage, the deceleration stage, and the steady stage and (ii) C-(A)-S-H gel and crystalline Ca(OH)₂ as the main hydration products [47]. The increasing of the moduli of water glass solution from 0.5 to 2.0 lead to the finer pore structure and higher mechanical strength [48]. Meanwhile, additional silicate had a retarding effect on the development of hydration process and the formation of hydration products [48]. However, increasing modulus had a negligible impact on the type of products of alkali-activated steel slag [48]. In addition, they also conducted detailed comparisons between the alkali-activated steel slag binder and Portland cement with the same water/binder ratio of 0.45 due to similar reaction conditions [47]. They found that alkali-activated steel slag has a faster reaction, fewer hydration products, poorer crystallization of Ca(OH)₂, a lower Ca/Si ratio, and a similar Al/Si ratio of gels than Portland cement [47]. Meanwhile, in terms of microstructure, alkali-activated steel slag hardened paste had more pores and looser microstructure causing long-term adverse impact on strength development [47].

Liu et al. [49] investigated the early age evolution including microstructure and reaction degree of alkali-activated steel slag from multiple perspectives under high curing temperature. They used ground steel slag with a specific surface area of 440 m²/kg, SiO₂/Na₂O molar ratio equal to 2.42 in the activator, and curing temperature of 60°C [49]. The most important conclusion is that they demonstrated the type of gel product [49]. According to their findings, the nano-C-S-H and nano-C-A-S-H gel first condensed due to the dissolution Si and Al phases, and then the formation of C-A-S-H gel was continuously conducted at longer curing time because Si-O-Si bond translated into Si-O-Al [49]. Kang et al. [50] synthesized a novel CeO₂-loaded porous NaOH-activated steel slag-silica fume catalyst for photocatalytic water-splitting of hydrogen production, and they found that three-dimensional polymeric structure C-S-H gel (Ca_{1.5}SiO_{3.5}·xH₂O) was the main phase in the alkali-activated steel-slag-based material.

2.3. Properties of Alkali-Activated Steel Slag. The potential utilization of alkali-activated steel slag as an alternative binder has been drawing much attention recently. However, unfortunately, its strength is very low even under strong alkaline conditions. The reason is that less active components limit the amount of hydration products from steel slag, although the activation effect of alkaline condition on hydration of steel slag is obvious. Wang et al. [44] and Sun et al. [47] found that the strength of alkali-activated steel slag is far from the strength of cement. The compressive strength of alkali-activated steel slag is only 30%–40% of that of cement slurry [47]. But, adding 20% GBFS can increase the 28-day

compressive strength by 40% [51]. So steel slag as a solo precursor is not an ideal material for the production of alkali-activated materials. In most studies on alkali-activated steel slag materials, better cementitious property is achieved by blending with other materials like GBFS, fly ash, and metakaolin.

When blended with blast furnace slag, alkali-activated GBFS-steel slag material shows significant cementitious properties in the presence of alkaline activator. You et al. [52] systematically studied the effect of steel slag on properties of alkali-activated GBFS material at room temperature. The Na_2O content was 4% by total weight of precursors and the modulus of water glass was 1.5 in all the alkali-activated mortars [52]. The content of steel slag was 50% by mass in the precursor [52]. Hydration process, strength, autogenous and drying shrinkages, pore structure, water absorption, and chloride ion penetration resistance of mortars were investigated [52]. They found that adding steel slag could decrease the hydration heat but prolong the setting time and improve workability [52]. Furthermore, incorporating steel slag could increase water absorption, reduce autogenous and drying shrinkage, and chloride ion penetration resistance [52]. The reason was that the replacement of steel slag could significantly increase the total porosity of the matrix due to its lower activity and the consequent less products [52]. You et al. [53] also investigated corrosion behavior of low-carbon steel reinforcement in alkali-activated GBFS and alkali-activated GBFS-steel slag under simulated marine environment. They found that the corrosion products were hematite and goethite [53]. The addition of steel slag had a beneficial influence on corrosion resistance due to improved interface transition zone between reinforcements and mortars [53].

Several studies have been undertaken to understand the investigation effects of steel slag on hydration properties of alkali-activated fly ash materials. Song et al. [54] used steel slag with various replacement levels (0, 10%, 20%, 30%, 40% and 50% by mass) to replace fly ash for alkali-activated binary composite material. They evaluated the influence of steel slag on setting times, flowability, viscosity, strength, absorptivity, and microstructural properties at standard curing conditions [54]. Adding steel slag obviously increased the setting times and flowability but decreased the viscosity [54]. The optimum content of steel slag was found to be 20% due to the negligible 28-day compressive strength loss and best flexural strength, elasticity modulus, and absorptivity [54]. The reason for the development of the strength was the formation and coexistence of C-S-H gel and C-A-S-H gel exhibiting better bonding [54]. Guo and Yang [55] synthesized engineered cementitious composite by using fly ash-steel slag activated by water glass with the modulus of 1.5 and polyvinyl alcohol fibers. They also thought C-S-H gel and N-A-S-H gel as self-healing products had a positive effect on self-healing property [55]. However, Niklić et al. [56] had different conclusions about the type of reaction product and the development of compressive strength due to high curing temperature of 65°C at the early age. They thought that the main products were N-(C)-A-S-H gel along with N-A-S-H gel [56]. They found that steel slag up to 30% in the range of 0%–40% positively affects the strength

evolution [56]. The 28-day compressive strength of alkali-activated fly ash mortar containing 30% steel slag exceed 35 MPa, and the study by Guo X had come to the same conclusions [56–58]. Niklić et al. [56] also evaluated the thermal resistance of alkali-activated fly ash-steel slag materials. They found that steel slag had a negative effect on the thermal resistance, i.e., the mechanical and dimensional stability was above 600°C [56].

Bai et al. [59] and Furlani et al. [60] investigated the content and fineness of steel slag as a precursor on the properties of alkali-activated metakaolin material. In the study by Bai et al. [59], they set two curing conditions (exposed curing at room temperature, sealed curing, and moist curing) and four substitution rates (0, 10%, 20%, and 40%). Mechanical properties, acid and alkali erosion endurance, and microstructure were investigated [59]. They found that adding 10% steel slag could ensure the optimum properties and moist curing was the best curing method [59]. The highest compressive strength and bending strength could reach 70 MPa and 8 MPa, respectively [59]. Moreover, microstructure was enhanced due to beneficial physical and chemical reactions between the active components of steel slag and metakaolin [59]. According to the research of Furlani et al. [60], two steel slag maximum particle sizes (250 μm and 125 μm) were used to replace metakaolin (0%, 20%, 40%, 60%, 80%, and 100% by mass). They found that finer steel slag could play a better role, and 40% steel slag was the best dosage [60]. They thought that the increase of compressive strength was attributed to the formation of stronger mechanical bonds replacing part of the original N-A-S-H gel [60].

Besides the binary systems above, steel slag is commonly mixed with slag to form ternary and other composite systems. It is also expected to be an effective way to use steel slag. In alkali-activated fly ash-GBFS-steel slag ternary system by Song et al. [61], water glass with a modulus of 1.6 was used as activator, and composite additive of GBFS-steel slag varied from 10% to 50%. The optimum content of GBFS-steel slag was found to be 40% [61]. The setting time, initial flow, and early and later compressive strength of paste increased due to the presence of steel slag [61]. In addition, the brittleness decreased by adding steel slag [61]. More gel products formed by hydration of GBFS-steel slag refined the pore structure, which was the main reason for the improvement of strength [61]. In alkali-activated ultrafine palm oil fuel ash-steel slag composite system, Yusuf et al. [62] evaluated the contributions of steel slag on compressive strength and shrinkage of pastes and mortars. The dosage of steel slag varied from 0% to 80% for pastes and 0% to 60% for mortars [62]. They found that steel slags reduced shrinkage by refining pores, eliminating microcracks, and increasing the density and strength of microstructure [62].

3. Alkali-Activated Copper Slag

3.1. Properties of Copper Slag. Copper slag (CS) is a by-product generated from the refining of copper. About 2.2 tons of copper slag will be produced for each ton of copper

produced [63], and about 40 million tons of CS are produced annually in the world [64]. Depending on different cooling processes, CS can be divided into two different groups, namely, granulated water-cooled slag and air-cooled slag [64]. Granulated CS (GCS) contains an amorphous phase, which mainly consists of iron oxides, silicon dioxide, and calcium oxide [65]. Air-cooled slag with a slower cooling process mainly contains crystalline phases, which consist of similar chemical components [66]. Figure 2 shows the typical XRD patterns of GCS and air-cooled CS. Table 1 shows the chemical composition and mineral composition of copper slag cooled with different processes in other studies. Mineral composition of granulated water-cooled CS and air-cooled CS usually contains same mineral components, namely, magnetite (Fe_3O_4) and fayalite (Fe_2SiO_4) [67]. Figure 3 shows the statistical chemical content of copper slag from other studies.

The common utilization options of copper are recovering of the metal and producing value-added products, such as abrasive and cutting tools, tiles, glass, road-base construction, pavement, as well as cement and concrete [74]. Due to the amorphous nature of Granulated CS, the hydration properties of GCS are more active compared to air-cooled CS [73], which means granulated CS is more suitable for supplementary cementitious materials, while air-cooled CS is more suitable to be used as aggregate in concrete [75, 76].

Using granulated CS as supplementary cementitious material involves an optimal dosage of 5%–15% [77]; a higher dosage of GCS decreases the strength of the cementitious material [78]. Thus, this utilization method is not enough for utilizing GCS. Alkali-activated CS has been investigated by some researchers. CS can be used as a filling material activated by sodium hydroxide [70]. About 20–30 MPa compressive strength of the binder of alkali-activated CS was achieved [79–81], which shows that alkali-activated granulated CS is a potential environment-friendly material for replacing cement.

3.2. Mechanism of Alkali-Activated Copper Slag. The mechanism of alkali-activated granulated copper slag is different when different activators are used [82]. Compressive strength result shows that the activation effect of sodium silica (SS) is better than sodium hydroxide (SH) [82], as the binder compressive strength of sodium silica is 5–6 times higher than that of sodium hydroxide.

The mineralogical characterization of alkali-activated CS with XRD shows that different products were formed when different activators were used. In SS-activated GCS, a weak peak, which represents the poor crystallinity of C-S-H, was formed. In SH-activated GCS, a sharp peak occurs at the similar position of weak peak in SS, which represents the plombierite (tobermorite 14 Å) [82].

The reaction products in the pastes of CS activated with SS are mostly amorphous C-S-H gels with higher degrees of polymerization, which bond the matrix together with fewer pores. However, the products formed in the pastes of CS activated with SH contain some highly crystalline

plombierite, and the matrix is loose and porous [82]. Besides, quantitative XRD shows that the original crystal in GCS, especially the fayalite and monticellite, are reduced in SS-activated GCS. This can be interpreted as the original crystal in GCS dissolved and participated in the formation of product [82]. The reaction degree of alkali-activated CS of different activators is consistent with the compressive strength and XRD result. CS reaction degree of SH and SS are 37.8% and 47.8%, respectively.

The different mechanisms of SH and SS were determined by pH and $[\text{SiO}_4]^{4-}$ concentrations. CS surface will dissolve with the attack of OH^- , Ca^{2+} and $[\text{SiO}_4]^{4-}$ were dissolved into the solution to form the product. Compared to SH, although the pH of SS is lower, more $[\text{SiO}_4]^{4-}$, which dissolved slower than Ca^{2+} , is provided in SS. This can be explained that although the initial reaction rate of SS is slower than SH, the total heat release of SS is higher than SH [73]. The precipitation of the product might also be hindered when OH^- is excessive [82].

3.3. Performance of Alkali-Activated Copper Slag.

Alkali-activated GCS has the potential to be used as a construction material. The compressive strength of GCS varies when different activators are used. SS is more effective than SH, and a higher modulus of SS increases the compressive strength of alkali-activated CS mortar [75]. Twenty-eight-day compressive strength of alkali-activated CS can reach 20 MPa, and the strength can still develop before 90 days [73].

SH is not a suitable activator for GCS, and 28-day compressive strength of SH-activated GCS is lower than 5 MPa [73, 82]. The strength development of later stage (90 days) is not developed [73]. This might due to the products of SH-activated CS containing highly crystalline plombierite, which is small in specific surface area and thus loosens the matrix [82].

Shrinkage of alkali-activated CS was also investigated [81]; drying shrinkage of alkali-activated CS is higher than Portland cement due to refined pore structures of alkali-activated CS. Increasing of both alkali content and modulus will increase the shrinkage of alkali-activated CS. The porosity result shows that the increasing of alkali dosage refines the pore structure [81]. Similar with Portland cement, shrinkage was smaller after 14 days. Therefore, it is suggested that a lower alkali content and modulus is more effective for controlling the shrinkage of alkali-activated CS.

4. Alkali-Activated Ferronickel Slag

4.1. Raw Material Properties of Ferronickel Slag.

Ferronickel slag is an industrial waste obtained from ferronickel alloy production. The ferronickel industry uses two main smelting technologies: the electric furnace method and the blast furnace method. The electric furnace method is currently the main method of ferronickel alloy production, while the blast furnace method is only used in parts of eastern China. According to the differences of raw material and manufacturing technology, ferronickel slag can be

FIGURE 2: XRD patterns of the copper slag samples. (a) Air-cooled samples. (b) Water-cooled samples [67].

TABLE 1: Chemical composition of copper slag (M = magnetite, F = fayalite, H = hematite, D: diopside, C: clinoferrrosilite).

Material	Fe_2O_3	SiO_2	CaO	Al_2O_3	MgO	Cooling process	Mineral composition	Ref.
1	59.78	25.18	3.75	3.51	0.69	Air-cooled	M, F, D	[67]
2	59.94	21.68	1.76	2.83	1.13	Air-cooled	F, H	[68]
3	44.21	35.29	1.82	2.7	1.14	Water-cooled	M, F	[67]
4	47.01	33.4	4.0	3.5	1.39	Water-cooled	F, H	[69]
5	30.48	39.14	13.41	7.76	2.09	—	M, F, C, D	[66]
6	53.11	28.70	3.87	5.8	1.56	—	F	[70]
7	65.27	27	2	3	0.29	—	—	[71]
8	57.8	30.53	1.6	2.8	1.49	—	—	[72]
9	49.6	34.17	4.29	4.67	0.71	Air-cooled	M, F	[73]
10	44.05	34.74	2.66	10.52	1.03	Water-cooled	M, F	[73]

FIGURE 3: Statistical chart of content of copper slag.

categorized as electric furnace ferronickel slag (EFFS) and blast furnace ferronickel slag (BFFS) with different chemical and mineralogical compositions. In addition, the cooling method of the molten slag has an important influence on its composition. The chemical compositions of ferronickel slag obtained from different sources are presented in Table 2. In general, BFFS are composed of SiO_2 , Al_2O_3 , and CaO , as BFFS has a large amount of amorphous phase [83, 84]. EFFS is mainly composed of SiO_2 , MgO , and Fe_2O_3 , and its mineral composition is mainly composed of crystalline phases, such as enstatite, forsterite, and dropsied. EFFS can be divided into air-cooled slag and water-cooled slag depending on different cooling methods. Generally, EFFS generated from laterite ore contains a high $\text{FeO}/\text{Fe}_2\text{O}_3$ and low MgO , whereas that from garnierite ore contains low Fe_2O_3 and high MgO [85–89]. The chemical composition of air-cooled EFFS differs only relatively little from that of water-cooled EFFS. However, the glassy phase content of water-cooled EFFS is higher than that of air-cooled EFFS. It can be seen that the nature of ferronickel slag depends on its source and treatment process.

4.2. Reaction Mechanism of Alkali-Activated Ferronickel Slag.

The reaction process of alkali-activated ferronickel slag is similar to that of alkali-activated slag/fly ash. The reaction of alkali-activated BFFS can be simplified as in Figure 4. After the addition of the activator solution, the structure of the BFFS is first attacked by the alkaline solution; then, BFFS is subsequently depolymerized to low polymer or silicate and aluminate tetrahedral units. Finally, the depolymerized substances polymerize to form the crystalline product strätlingite and amorphous product C-A-S-H, which is responsible for the high strength of alkali-activated BFFS [83]. BFFS tends to form strätlingite rather than hydrotalcite, mainly because of the high aluminum content of BFFS and the small amount of magnesium dissolved, which is mainly present in the form of spinel and forsterite with a stable structure, and hardly reacts in alkaline solutions. The Ca/Si and Al/Si ratios of C-A-S-H are 0.64 and 0.57–1.44, respectively. The difference in composition between EFFS and BFFS results in the generation of different hydration products. Hydroxysodalite was found in the reaction products of alkali-activated low-calcium and low-magnesium EFFS with kaolinite that exhibited higher strength [89]. Maragkos et al. [88] found that increasing the OH^- concentration could enhance the dissolution of silicon and aluminum in EFFS. And high $\text{SiO}_2/\text{Na}_2\text{O}$ ratio can promote the condensation reaction. The presence of alkali metal cations plays a catalytic role and has an important influence on gel hardening and crystallization. Compared to NaOH , KOH provides more inorganic polymer precursors, as the larger K^+ size contributes to the formation of larger silicate oligomers, to which $\text{Al}(\text{OH})_4^-$ tends to bind; thus, better solidification and higher compressive strength are obtained [95]. The main product of alkali-activated high-Mg EFFS/fly ash is N-A-M-S-H, where the magnesium dissolved in EFFS participates in the reaction and is incorporated into the N-A-S-H molecular structure [99, 100]. There are three

typical phases identified in water-quenched high Mg EFFS, namely, FNS I, II, and III phases, which are Mg-Si phase, Si-Ca-Al phase, and Gr-Fe phase, respectively [94]. The FNS I phase (Mg-Si phase) is more prone to dissolve and preferentially participate in the reaction process than the other two phases. The dissolved Mg from FNS is mainly involved in the formation of hydrotalcite and N-M-S-H gels. The heavy metals in ferronickel slag mainly include Mn, Cr, and Ni. Wang et al. [83] reported that the alkali-activated matrix has a good stabilization effect on heavy metals, which greatly reduces the risk of heavy metals leaching. Cao et al. [94] found that Cr exists in EFFS in the form of Gr-Fe phase, which remains stable under the activation of alkali. Cr could not be detected during the leaching process. According to Komnitsas et al. [101], alkali-activated EFFS encapsulated the heavy metals such as Pb, Cr, and Ni. Therefore, heavy metals could not leach out from the concrete and maintained the structural integrity. In summary, alkali-activated ferronickel slag is an environmentally friendly material, and there is no problem of heavy metal leaching.

4.3. Properties of Alkali-Activated Ferronickel Slag.

Wang et al. [83] found that alkali-activated BFFS showed comparable compressive strength and lower 7-day autogenous shrinkage to the alkali-activated slag. And BFFS activated by $M_s = 0.5$ waterglass obtained the highest compressive strength (70 MPa) at 90 days. Xu et al. [92] investigated the type and content of solid activators on the compressive strength of alkali-activated BFFS. The results showed that alkali-activated BFFS with $\text{Na}_2\text{SiO}_3/\text{Na}_2\text{CO}_3$ activators have a denser microstructure, lower porosity, and smaller pore size than alkali-activated BFFS with Na_2SiO_3 or NaOH activators. The compressive strength of the $\text{Na}_2\text{SiO}_3/\text{Na}_2\text{CO}_3$ sample can reach 96 MPa when the Na_2O content is 0.107 mol. It had been shown that low-calcium EFFS can also be used to prepare alkali-activated materials with superior properties. According to Maragkos et al. [88], the properties of alkali-activated EFFS depends on the solid to liquid ratio (S/L). The optimum quantities of S/L and NaOH concentrations were 5.6 g/mL and 7 M, respectively. Under optimum conditions, the alkali-activated EFFS exhibited a very high compressive strength of 118 MPa and a very low water absorption of about 0.8%. Komnitsas et al. [89] investigated the performance of sodium-silicate-activated EFFS/kaolin-based materials. They found that only aging period had a very significant effect on the final compressive strength while heating time and the temperature had a negligible effect on strength development. The alkali-activated samples showed excellent resistance to freeze-thaw cycles. However, the strength declined in acidic environment due to the formation of halite, magnesium calcite, calcite, aragonite, and akermanite on the surface of the immersed samples. Sakkas et al. [87] evaluated the effect of fire exposure on the alkali-activated EFFS materials. The results showed that these samples had a low thermal conductivity and high fire resistance like commercial fire-resisting materials. It is even possible to prepare alkali-activated ferronickel slag concrete, which belongs to the category of ultrahigh performance concrete, with a

TABLE 2: Chemical composition of ferronickel slag.

Material	SiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MgO	CaO	MnO	Gr ₂ O ₃	NiO	Types of ores source	Ref.
BFFS	29.95	26.31		1.55	8.93	25.19	2.25	2.3	0.01	China	[83]
BFFS	33.15	21.94		2.15	12.54	22.5	3.36	2.08	0.02	China	[84]
BFFS	37.2	21.37		1.72	10.53	24.82				China	[90]
BFFS	36.7	18.11		1.83	11.63	28.77				China	[91]
BFFS	22.26	18.97		2.87	7.81	33.9	2.87			China	[92]
EFFS-W	46.1	4.46		12.25	27.12	6.75	0.79	1.5	0.19	China	[84]
EFFS-W	50.48	3.08		10.37	32.61	1.01	0.62	1.37	0.06	China	[84]
EFFS-W	44.9	4.94		14.36	23.29	8.24	0.98	2.47	0.13	China	[84]
EFFS-W	47.61	6.56		13.24	15.94	11.49		0.7		China	[93]
EFFS-W	45.23	5.91		9.74	24.17	8.93		1.14	0.29	China	[94]
EFFS-W	53.1	2.4		11.3	32.3	0.23				Soci'et'e Le Nickel (SLN), New Caledonia	[23]
EFFS-W	41.14	13.79	34.74		3.59	0.71		5.41	0.14	Laterites LARCO, Greece	[87]
EFFS-W	40.29	10.11	37.69		5.43	3.65		2.58	0.09	Laterites LARCO, Greece	[88]
EFFS-W	32.74	8.32	38.8		2.76	3.73		3.07	0.1	Laterites LARCO, Greece	[89]
EFFS-W	32.74	8.32		43.83	2.76	3.73		3.07		Laterites LARCO, Greece	[95]
EFFS-W	51.93	2.92		12.98	30.87	0.5				Garnierite ore, New Caledonia	[85]
EFFS-W	52.52	2.33		10.8	33.16	0.27				Garnierite ore, New Caledonia	[96]
EFFS-W	53.29	2.67		11.9	31.6	0.42		1.08	0.1	Garnierite ore, New Caledonia	[86]
EFFS-W	52.27	6.19		4.2	26.93	8.77			0.04	China	[96]
EFFS-W	58.1	2.29		11.1	26.5	0.29				SNNC, South Korea	[97]
EFFS-A	62.8	1.95		7.13	24.7	2.07				SNNC, South Korea	[97]
EFFS-A	51.23	3.66		8.06	31.91	1.52				China	[98]

EFFS-A: air-cooled EFFS slag; EFFS-W: water-cooled EFFS slag.

FIGURE 4: Reaction mechanism of alkali-activated blast furnace ferronickel slag [83].

strength of up to 120 MPa. Komnitsas et al. [101] reported that NO_3^- or SO_4^{2-} ions reduced the strength of alkali-activated EFFS due to the fact that they consumed most of the available moles of alkali activators, hindering the polymerization reaction and therefore producing a limited number of gels.

According to the literature, it was found that an EFFS with a high MgO content is not very well utilized due to the low reactivity and high magnesium oxide content, which may lead to bulk stability problems [8]. Cao et al. [102] used EFFS with high MgO content as a partial replacement for blast furnace slag to prepare alkali-activated cements (AACs). The results showed that the incorporation of less than 40% FNS has no significant effect on the setting time; however, the incorporation of 60% FNS will not only

prolong the setting time but also reduce the compressive strength. For Na_2SiO_3 -activated AACs, increasing EFFS content in the mixture will lead to the increase of autogenous shrinkage, drying shrinkage, and total porosity. For NaOH-activated AACs, the autogenous shrinkage and drying shrinkage are decreased with the addition of EFFS, while the total porosity is increased. Kuri et al. [103] reported that EFFS replacing part of the fly ash reduced workability and shortened setting time but increased compressive strength, with 75% EFFS being the optimum content. They found that Mg was involved in the formation of N-M-A-S-H and therefore did not cause bulk stability problems, whereas Komnitsas et al. [89, 95] argued that magnesium acted as chemically inert in alkali-activated EFFS materials. It also depends on the source of the FNS. It is closely related to the source of ferronickel slag. Yang et al. [100] further suggested that EFFS can improve the thermal stability of alkali-activated materials by replacing some of the fly ash, and can effectively reduce the shrinkage of alkali-activated materials below 600°C.

5. Alkali-Activated Lead-Zinc Slag

5.1. Characterization of Lead-Zinc Slag. Lead-zinc slag is a by-product of the lead and zinc production industry, generated from the ores during smelting [104, 105]. It is reported that the production of 1t of lead and zinc discharges 7,100 kg and 9,600 kg slag [24], respectively. These lead-zinc slags are generally landfilled, not only occupying large areas of arable land but also polluting the environment due to the leaching of heavy metals and the radiation of nuclides.

The chemical composition of lead-zinc slag changes depending on the ores, the fluxes, the smelting process, and

TABLE 3: Chemical compositions of lead-zinc slag.

Composition	SiO ₂	FeO _x	CaO	Al ₂ O ₃	ZnO	Cr ₂ O ₃	PbO	MgO	SO ₃	MnO	Ref.
1	31.34	32.26	18.34	4.26	8.20	—	2.69	1.72	1.19	—	[104]
2	18.89	39.15	13.92	8.52	13.95	0.19	1.37	2.05	3.77	1.12	[105]
3	30.67	29.73	12.48	7.27	6.39	—	—	3.27	3.06	2.97	[24]
4	34.92	24.98	20.50	5.07	3.63	—	1.12	2.36	1.11	—	[106]
5	24.33	33.70	22.10	2.46	11.11	—	3.63	2.71	0.39	—	[107]
6	43.09	14.99	23.05	6.22	4.01	—	—	1.58	—	—	[108]
7	14.68	41.96	4.50	4.7	2.82	—	10.34	1.43	6.51	—	[109]
8	35.50	46.37	11.53	3.85	6.02	—	4.03	4.65	0.24	—	[109]
9	21.39	28.10	23.11	3.56	9.47	—	4.06	5.44	0.37	—	[110, 111]
10	24.88	31.38	22.14	2.46	10.77	—	3.74	2.71	—	—	[112]
11	21.56	31.57	3.05	1.73	6.18	—	12.28	0.15	8.01	—	[113]
12	18.08	34.28	17.91	8.17	9.21	—	1.22	1.93	1.41	1.33	[114]
13	18.30	26.10	15.60	5.5	14.00	—	3.60	—	—	—	[115]
14	11.30	52.90	3.30	5.5	9.80	—	1.40	—	—	—	[115]
15	32.50	16.25	27.50	9.25	5.00	1.30	—	2.13	—	—	[116]
16	27.1	35.30	17.95	7.65	1.30	—	0.03	4.70	—	—	[117]
17	21.90	33.70	18.10	10.4	6.92	—	0.69	—	1.58	—	[118]
18	23.00	33.40	20.00	1.89	11.20	—	3.50	—	0.7	—	[118]
19	27.50	33.80	19.40	7.4	—	—	—	2.10	—	—	[119]
20	21.40	28.10	23.10	3.6	—	—	—	5.44	—	—	[119]
21	25.70	33.95	18.90	5.41	5.01	—	2.47	1.41	—	0.656	[120]
22	29.79	54.19	3.87	0.01	5.82	0.03	0.11	2.91	0.02	3.22	[121]
23	29.66	55.57	2.77	0.02	4.34	0.01	—	2.28	0.02	4.42	[121]
24	30.03	51.13	8.08	0.74	6.28	0.01	0.11	0.85	—	2.13	[121]
25	32.89	27.67	27.48	0.49	2.32	—	0.02	5.33	0.11	3.61	[121]
26	31.15	41.23	15.38	0.26	1.98	—	0.06	3.68	0.07	5.91	[121]
27	30.52	47.52	14.49	0.06	3.80	—	0.18	1.68	8.00	1.48	[121]
28	41.87	26.14	21.71	6.12	2.71	—	—	0.65	—	0.92	[121]
29	39.87	22.67	21.49	10.19	2.82	—	—	0.19	—	0.62	[121]
30	40.07	18.91	22.62	11.2	1.23	—	—	2.41	—	1.40	[121]
31	43.30	23.50	20.59	4.73	3.12	0.43	—	1.50	—	1.80	[121]
32	43.34	20.68	22.29	8.06	1.65	0.23	—	2.06	—	1.39	[121]
33	30.76	30.09	11.80	7.28	—	—	—	3.32	2.41	—	[122]
34	17.01	34.33	13.14	6.84	12.20	0.13	0.82	0.85	2.5	1.12	[123]

the impurities in the coke and the iron. The chemical compositions of lead-zinc slag from different research papers [24, 104–123] are summarized in Table 3. It could be found in Table 3 that the major composition of lead-zinc slag is FeO_x, SiO₂, CaO, Al₂O₃, and ZnO. As shown in Figure 5, the major constituents of the lead-zinc slag, in decreasing order of wt%, were the following: FeO_x (34%, ranging from 27% to 37%), SiO₂ (28%, ranging from 21% to 33%), CaO (17%, ranging from 14% to 23%), ZnO (8%, ranging from 3% to 10%), and Al₂O₃ (5%, ranging from 2% to 8%). Figure 5 also shows that the contents of the major compositions of lead-zinc slag are significantly different from various areas and the smelting factory. Some heavy and toxic elements, such as Pb, Zn, Cd, Cr, Mn, Cu, etc., could be found in lead-zinc slag, which limits its utilization due to the large leaching risk. The density of lead-zinc slag ranges from 3.6 g/cm³ to 3.9 g/cm³ [105, 118, 124], which is larger than that of traditional aluminosilicate waste due to high iron oxide content.

The phase composition of lead-zinc slag greatly changes depending on the ores, the fluxes, the smelting process, and the cooling method. The lead-zinc slag was reported to be mostly composed of an iron-silica-lime glass matrix and the content of the glass phase in lead-zinc slag is generally larger

FIGURE 5: Major compositions of lead-zinc slag (data from Table 1).

than 80% [24, 115, 117]. The kinds of the crystal phases in lead-zinc slag are also dependent on the ores and fluxes. Figure 6 provides an example of the XRD pattern of lead-zinc slag, which proves that most components are amorphous. As shown in Figure 6, the crystal phases in lead-zinc slag are ZnS, FeS, FeO, Fe₃O₄, and Pb metal, which is consistent with the results reported by Weeks [105]. Xia et al.

a: ZnS
b: Pb
c: FeS
d: Fe₃O₄
e: FeO

FIGURE 6: XRD pattern of lead-zinc slag.

(a)

(b)

FIGURE 7: XRD of lead-zinc slag with high percentage of crystal phases. (a) Lead-zinc slag from Belgium [120]. (b) Lead-zinc slag from Brazil [110].

FIGURE 8: Morphology of fly ash-zinc composite geopolymer. (a) 100% fly ash. (b) 60% fly ash and 40% zinc slag. (c) 20% fly ash and 80% zinc slag. (d) 100% zinc slag. Data from [117].

[122] reported that the small amount of crystal phases is ringwoodite and Fe metal. Spinel [24, 104, 107, 110, 112, 125, 126], calcium iron silicate [117], magnesioferrite [117], fayalite [125], Olivine [104, 110], kirschsteinite [125], hedenbergite [104, 125], willemite [110, 112], melillite [1, 8, 10, 22], and franklinite [126] were also found in lead-zinc slag from various areas. Due to the difference in smelting processes, two kinds of lead-zinc slag were reported to be composed mainly of crystal phases and their XRD patterns are provided in Figure 7.

5.2. Reaction Mechanism of the Alkali-Activated Lead-Zinc Slag. Similar to the blast furnace slag [127], fly ash [128], and rice husk ash [129], most of the lead-zinc slag is composed by amorphous aluminosilicate phases. So the reaction process of alkali-activated lead-zinc slag is similar to the traditional alkali-activated materials: dissolution and dispersion of raw materials, rearrangement and exchange of dissolved species, gelation and solidification, and continuous gel evolution toward crystallization [130, 131].

The leaching risk of the heavy metals in lead-zinc slag is the key factor that limits its application as construction materials. Alkali-activated material is an effective system for the solidification of heavy metals. But, high Si content and low Al content in lead-zinc slag make it difficult to form a

rigid Al-Si network structure [117]. The lack of AlO_4^- unit tend to decrease the capacity of alkali-activated materials for element immobilization since the heavy metals mainly bonded with alumina tetrahedron [132]. So, some studies were conducted by mixing the lead-zinc slag with fly ash and alkalis to form a geopolymer [113, 117, 119]. The morphology of the fly ash-zinc slag composite geopolymer was reported by Nath [117]. It was found that the reaction products were refined with the increasing of the zinc slag content (see Figure 8). Zhang [24] has also studied the self-cementation of lead-zinc slag through alkali-activated materials and found that the solidification efficiency was larger than 90% for most of the heavy metals. The physical encapsulation was found to be the main mechanism of the solidification of the heavy metals in alkali-activated materials.

5.3. Properties of the Alkali-Activated Lead-Zinc Slag Material.

The performance of the alkali-activated materials containing lead-zinc slag is also dependent on the properties of the raw materials. Xia et al. [122] found that the compressive strength of hardened alkali-activated materials decreases with the increasing of the lead-zinc slag content. It means that lead-zinc slag has a negative effect on the performance of the alkali-activated materials. It was supposed that the

high iron content in lead-zinc slag tend to be oxidized and the oxidation might increase the porosity and volume of solidified body, which ultimately resulted in decrease of compressive strength. But, Nath [117] found that the relatively high CaO content in lead-zinc slag results in the formation of Ca-rich dense gel and the development of compact microstructure. The 28-day compressive strength of alkali-activated zinc slag even reached 71 MPa. And the highest compressive strength even reaches 96 MPa. It means that the dispersion of the chemical composition of raw materials significantly affects the mechanical performance of the alkali-activated lead-zinc slag.

6. Conclusion and Outlook

The main mineral phases in steel slag are tricalcium silicate, dicalcium silicate, RO phase, tetracalcium aluminoferrite, etc. The phase composition of steel slag is similar to that of cement and thus steel slag has the potential cementitious property. But, the low activity of steel slag is the biggest obstacle and challenge to the proper utilization of alkali-activated steel slag materials. Moreover, there is great uncertainty about the hydration mechanism of the steel slag as a solo precursor.

Copper slag is mainly composed of crystal phases, i.e., magnetite (Fe_3O_4) and fayalite (Fe_2SiO_4). The utilization of copper slag as an alkali-activated material depends on the cooling process of the copper slag. Granulated water-cooled copper slag with a relatively higher amorphous phase is more suitable for alkali activation. In terms of activator, sodium hydroxide is less effective than water glass due to the formation of the high crystalized product. Although many studies have evaluated the mechanical properties of alkali-activated copper slag, heavy metal leaching assessment should be considered in the future study as copper usually contains heavy metals.

BFFS has good reactivity due to the large amount of amorphous phase; therefore, alkali-activated BFFS has superior mechanical properties. The reaction products are strätlingite and C-A-S-H. The reactivity of EFFS is closely related to its source and treatment process. EFFS generated from laterite ore contains a low MgO, whereas that from garnierite ore contains a high MgO. The amorphous phase content of EFFS with low Mg content is high, while the amorphous phase content of EFFS with high Mg content is low, and there are a large number of Mg-containing crystals for the main mineral phases in EFFS, namely, forsterite (Mg_2SiO_4), enstatite (MgSiO_3), and clinoenstatite (MgSiO_3). Good properties can be obtained in fly ash-based alkali-activated materials by incorporating EFFS, and the generation of the amorphous phase N-M-A-S-H leads to a denser microstructure. There is no negative impact on the environment due to the utilization of ferronickel slag in the alkali-activated materials.

The chemical composition of lead-zinc slag significantly changes depending on the ores, the fluxes, the smelting process, and the impurities in the coke and the iron. Thus, the phase composition and the reaction activity of lead-zinc slag and the mechanical performance of alkali-activated lead-zinc slag reported in different papers are quite

inconsistent. Generally, the lead-zinc slag is composed of iron-silica-lime amorphous phases. In order to improve the mechanical property and the solidification efficiency for heavy metals, lead-zinc slag is usually mixed with fly ash or granulated blast furnace slag to form a geopolymer.

There are few studies to fully understand the properties of alkali-activated metallurgical slag composite system, and in-depth research on durability is still a new topic. Better activated methods and alkali-activators are still needed to improve the performance of the metallurgical slag with low reactivity. It is expected that the acid-activated method may be an alternative method due to the high contents of iron, calcium, and magnesium in metallurgical slag. The chemical composition of the metallurgical slag is closely dependent on the ores, the fluxes, and the smelting process. Thus, the relationship between the chemical composition and the reactivity of metallurgical slags needs to be constructed. If these problems are solved, they will bring great environmental benefits to slag yards and enormous economic benefits to steel industries.

Data Availability

Previously reported data were used to support this study. These prior studies are cited at relevant places within the text as references.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

The authors are grateful for the financial support from the National Natural Science Foundation of China (Grant No. 52008229).

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