Component Modification of Basic Oxygen Furnace Slag with C₄AF as Target Mineral and Application

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Abstract: In this paper, a new method of basic oxygen furnace (BOF) slag component modification with a regulator was studied. The main mineral was designed as C₄AF, C₂S and C₃S in modified BOF slag, and the batching method, mineral compositions, hydration rate, activation index and capability of resisting sulfate corrode also were studied. XRD, BEI and EDS were used to characterize the mineral formation, and SEM was used to study the morphology of hydration products. The results show that most inert phase in BOF slag can be converted into active minerals of C₄AF and C₃S through reasonable batching calculation and the amount of regulating agent. The formation of C₄AF and C₂S in modified BOF slag is better, and a small amount of MgO is embedded in the white intermediate phase, but C₃S is not detected. With the increase in the CaO/SiO₂ ratio in raw materials, the CaO/SiO₂ ratio of calcium silicate minerals in modified BOF slag increases, the contents of f-CaO are less than 1.0%, and the activity index improves. Compared with the BOF slag, the activity index and exothermic rate of modified BOF slag improved obviously, and the activity index of 90 days is close to 100%. With the increase in modified BOF slag B cement, the flexural strength decrease; however, the capability of resisting sulfate corrode is improved due to the constant formation of a short rod-like shape ettringite in Na₂SO₄ solution and the improvement of the structure densification of the hydration products.

Keywords: steel slag; component modification; C₄AF; resisting sulfate corrode

1. Introduction

The cement industry is a high energy consumption industry, and its CO₂ emission accounts for about 7% of global CO₂ emissions [1]. Manufacturing Portland cement clinker consumes large amounts of energy and resources, such as coal, limestone, clay, etc. (nearly 1.7 tons of raw materials produce 1 ton of clinker), and involves the emission of CO₂ (each ton of clinker emits about 850 kg of CO₂), SOx and NOx [2,3]. It is urgent to find alternative cementitious materials to replace conventional cement. Some industrial solid waste which chemical composition is similar to Portland cement with high hydration activity have been widely used as partial substitutes for cement (called mineral admixture) in the building industry, such as fly ash and ground granulated blast furnace slag, but these mineral admixtures have become scarce resources in many countries especially in China [1]. Therefore, searching for new mineral admixtures is very important to the construction industry.
Basic oxygen furnace (BOF) slag is a byproduct of the conversion of iron to steel in a BOF, which main chemical compositions are CaO 35–50%, SiO$_2$ 10–25%, Al$_2$O$_3$ 1–8%, Fe$_2$O$_3$ (FeO/Fe) 10–35%, MgO 1–10% and P$_2$O$_5$ 1–5% [4–7], and the common mineralogical composition include dicalcium silicate (C$_2$S), tricalcium silicate (C$_3$S), tetracalcium aluminoferrite (C$_4$AF), dicalcium ferrite(C$_2$F), merwinite (MgO-2SiO$_2$-3CaO), olivine ((Mg$_x$Fe$_{1-x}$)$_2$SiO$_4$), RO phase (FeO-MnO-CaO-MgO solid solution) and f-CaO [7–9]. However, the hydration activity of C$_2$S, C$_3$S and C$_4$AF in BOF slag is relatively lower than those in Portland clinker due to the dense structure and large crystal size formed at higher temperature (~1600 °C) [9,10], the phases of RO and C$_2$F are almost inert [10], and f-CaO may cause uncontrolled volume expansion [11], all of these limit the use of steel slag as mineral admixtures. At present, the BOF slag in China is mainly used as coarse aggregates for cement concrete, road base material and asphalt paving [12–15], and the total utilization ratio is under 30% [16]. China is a major producer of steel, 90% of steel is produced in BOF. In 2018, the total output of crude steel had reached 927 million tons (about 51.3% of world crude steel output) [17], steel slag output has exceeded 100 million tons, and the cumulative output has exceeded 300 million tons [16]. The disposal of such a large amount of steel slag leads to a serious environmental threat and occupies large numbers of agricultural land. Thus, it is necessary to explore a new method to improve the utilization of steel slag.

Many approaches have been applied to improve the hydration activity of BOF slag, such as fine (superfine) grinding [10], chemical and thermal activation [8,18] and higher temperature carbonation curing [15,19]. However, inert phases (RO, C$_2$F and Fe$_{1-x}$O) make no contribution to the cementitious properties of steel slag, and there is still uncontrolled volume expansion. A new method to improve the hydration activity of steel slag was proposed, which can modify the chemical and mineralogical compositions of steel slag by adding high content regulating agent high in CaO and SiO$_2$ during the steelmaking process, and accelerated the cooling rate to make the mineral crystal in small particle status [1]; however, no specific experimental results and analysis were given, and inert phases (RO, C$_2$F and Fe$_{1-x}$O) still existed. Some researchers used industrial waste as the regulating agent to modify the mineralogical compositions of steel slag, the f-CaO content in modified steel slag was reduced, and the cementitious properties were improved remarkably [4], which proved the feasibility of steel slag modification.

High-ferrite Portland cement has an outstanding performance in sulphate erosion [20]; however, the excessive introduction of the iron phase will reduce the production of kiln crust and C$_3$S, and the mechanical properties of cement are reduced. In this study, high iron and low calcium minerals can be prepared from BOF slag by adding regulating agent high in CaO or SiO$_2$ and Al$_2$O$_3$ during the steelmaking process and composite with different amounts of ordinary Portland cement according to different corrosion resistance requirements. It will greatly reduce the cost of cement preparation and realize the high added value utilization of BOF slag.

In this study, the batching method of steel slag modification was studied, regulating agents high in CaO (or SiO$_2$) and Al$_2$O$_3$ were introduced to convert merwinite, olivine, RO phase and f-CaO in BOF slag into more active minerals phase (C$_3$S, C$_2$S and C$_4$AF). The influence of regulating agents on the mineral compositions, mineral morphologies, hydration heat and activation index were investigated, respectively. The capability of resisting sulfate corrode of cement mixed with different modified BOF slags was studied.

2. Experimental
2.1. Materials
The BOF slag was obtained from Liuzhou Iron and Steel Co., Ltd. (Guangxi, Liuzhou, China). The lime was purchased from a factory in Guilin (Guangxi, Guilin, China). The sintered bauxite was received from Yinzhou bauxite Co., Ltd. (Shanxi, Yinzhou, China). The standard cement was Portland cement with a strength grade of 42.5, which was purchased
from Yufeng cement Co., Ltd. (Guangxi, Liuzhou, China). The main chemical compositions of materials are presented in Table 1.

Table 1. Main chemical compositions of raw materials.

| Materials       | Main Chemical Compositions (wt.%) | LOI * |
|-----------------|-----------------------------------|-------|
|                 | CaO     | Fe₂O₃ | Al₂O₃ | SiO₂ | MgO | BaO | SO₃ | Others |       |
| BOF slag        | 43.41   | 18.44 | 7.33  | 16.60 | 7.50 | -   | -   | 5.21   | 1.51  |
| Sintered bauxite| 1.15    | 1.90  | 79.00 | 12.00 | -    | -   | -   | 4.75   | 1.20  |
| Lime            | 97.25   | 0.49  | 1.03  | 0.15  | 0.25 | -   | -   | 0.63   | 0.20  |
| Cement          | 64.02   | 3.44  | 4.85  | 20.94 | 1.70 | -   | 1.88 | 1.29   | 1.88  |

* LOI: Loss on ignition, mass loss at 1000 °C. -: no test.

2.2. Experimental Methods

2.2.1. Batching Calculation

In BOF slag, as the content of CaO was lower than the amount required to form active minerals, regulating agents high in CaO and Al₂O₃ were introduced. Suppose that all Fe₂O₃ formed C₄AF and all SiO₂ generated C₂S and C₃S, the percent content of SiO₂ formed C₂S was x, and the rest of SiO₂ that generated C₃S was (100% − x). Equations (1) and (2) were used to calculate the adding quantity of Al₂O₃ and CaO in the BOF slag.

\[
Al₂O₃ * = 0.640 \times Fe₂O₃ - Al₂O₃
\]

\[
CaO * = (1.867 \times SiO₂ * x + 2.80 \times SiO₂ * (100% - x) + 1.41 \times Fe₂O₃) - CaO
\]

On the contrary, if the content of CaO was excess the demand, regulating agents high in SiO₂ and Al₂O₃ were added. Suppose CaO generated C₂S and C₃S was 100%, the percent content of CaO which formed C₂S was y, and the rest of CaO that generated C₃S was (100% − y). Equations (1) and (3) were used to calculate the adding quantity of Al₂O₃ and SiO₂ in the BOF slag.

\[
SiO₂ * = (0.535 \times y + 0.357 \times (100% - y)) \times (CaO - 1.40 \times Fe₂O₃) - SiO₂
\]

The shortages of Al₂O₃ and CaO (or SiO₂) were supplemented by regulating agents. Five types of modified BOF slag were designed with different x, respectively. The rations of raw materials are shown in Table 2.

Table 2. Ratios of raw materials and sintering temperature.

| Sample | x * | Ratios of Raw Materials (wt.%) | Sintering Temperature (°C) |
|--------|-----|-------------------------------|---------------------------|
|        |     | BOF Slag | Sintered Bauxite | Lime |                   |
| A      | 100%| 83.48 | 4.73 | 11.79 | 1310 |
| B      | 75% | 80.80 | 4.57 | 14.63 | 1320 |
| C      | 50% | 78.28 | 4.43 | 17.29 | 1330 |
| D      | 25% | 75.91 | 4.30 | 19.79 | 1340 |
| E      | 0%  | 73.79 | 4.18 | 22.03 | 1345 |

* x *: The percent content of SiO₂ that formed C₃S in the BOF slag.

2.2.2. Preparation of Modified BOF Slag

The raw materials were dried at 105 °C for 6 h in an electric oven and ground in a ball mill to a size less than 80 μm, respectively. According to Table 2, the mixtures were homogenized in a ball mill and shaped into a circular sample of 50 mm diameter and 5–8 mm thickness by a manual tablet machine under a pressure of 3–5 MPa. Then the samples were sintered in a high-temperature box resistance furnace at a certain temperature...
for 40 min (heating rate of 10 °C/min below 900 °C and 5 °C/min above 900 °C), as shown in Table 2. The sample was cooled fast by a grate cooler.

2.2.3. Characterizations and Performance Tests

The mineralogical phases of modified BOF slag were identified using an X-ray diffractometer (XRD), scanning electron microscope (SEM) and energy dispersive spectrometer (EDS). The XRD tests were carried out with an "X’Pert PRO" (PANalytical B.V., Almelo, The Netherlands), which was operated at 40 kV voltage and 40 mA current with a CuKa radiation source. The phases of the clinkers were identified through Gemini SEM 300 (Zeiss, Germany), equipped with an EDS detector. The rate of heat evolution during the hydration of the sample was detected by I-Cal 8000 HPC (Calmetrix, Boston, MA, USA). The $f$-CaO of samples was tested with the method of rapid determination with ethanol-glycol. The specific surface of the sample was tested in accordance with Chinese National Standard GB/T 8074-2008. The setting time and soundness were tested in accordance with Chinese National Standard GB/T 1346-2011. The activity of modified BOF slags was evaluated in accordance with Chinese National Standard GB/T 12957-2005, the ratio of cement to modified steel slag admixture (10% gypsum and 90% modified steel slag) was 70:30. The capability of resisting sulfate corrode of cement mixed with different modified BOF slags was carried out according to the Chinese National Standard GB/T 749-2008.

3. Results and Discussion

The main mineralogical phase of the BOF slag was identified to be C$_2$S, C$_4$AF, melilite, RO phase, C$_2$F, Fe$_2$O$_3$ and MgO, as shown in Figure 1a. The XRD patterns of five modified BOF slags and BOF slag are presented in Figure 1b. It is seen that the characteristic peaks of dicalcium silicate (C$_2$S), tetracalcium aluminoferrite (C$_4$AF), C$_3$A and MgO are identified. Compared to the BOF slag, the peak intensity of C$_4$AF, C$_2$S and MgO increases as the lime content in the ingredients increases, the diffraction peak becomes sharp, and the diffraction peak curve bread gradually decreases, and the peak intensity of melilite, RO phase, C$_2$F and Fe$_2$O$_3$ decreases or disappears. All the above analyses show that the introduction of bauxite and lime can transform the inert phase (melilite, RO phase, C$_2$F and Fe$_2$O$_3$) in BOF slag convert into active mineral (C$_2$S, C$_4$AF and C$_3$A) gradually. When the lime content is relatively small, a large amount of Mg is solid dissolved in C$_2$S to form low-activity merwinte (CaMg(SiO)$_4$), the diffraction peak shifts to the right in the region between I and II of Figure 1c, and a large amount of Si is solid dissolved in the iron phase to form low-activity calcium magnesium iron silicate mineral (Ca$_2$Fe$_{1.2}$Mg$_{0.4}$Si$_{0.4}$O$_5$), and the diffraction peak shifts to the right in the region III of Figure 1c, which reduces the formation of active minerals. With the increase in lime content, the solution phenomenon decreased, and the peak value of active minerals increased. Compared with other samples, the formation of minerals in sample E is the best by sintering temperature. However, due to the low-sintering temperature and too much iron phase, the characteristic peaks of tricalcium silicate (C$_3$S) were impossible to find, even at the highest lime content in the ingredients. In the later experiment, the introduction of a mineralizer can be considered to reduce the formation temperature of C$_3$S.

Backscattered electron images (BEI) and the energy dispersive spectrometer (EDS) analysis area from the microanalysis of five modified BOF slags are presented in Figure 2. Meanwhile, the average percentage of main element atoms at each area in the figure is clearly shown in Table 3.
Figure 1. XRD analysis of (a) BOF slag and (b,c) modified BOF slags.

Figure 2. BEI of the polished section of (a–e) modified BOF slags A to E and EDS analysis in different grey areas.

Figure 3. Exothermic curve of (a) rate and (b) total during the hydration of cement samples.
Table 3. The percentage of the main element atoms at each area in Figure 3.

| Areas | Main Elements | Ca | Si | Al | Fe | O | Mg | Ti | P | Mn | Ca/Si |
|-------|---------------|----|----|----|----|---|----|----|---|---|-------|
| A-1   |               | 28.53 | 13.10 | 0.67 | 0.53 | 54.53 | 0.92 | 0.25 | 1.20 | 0 | 2.17  |
| B-1   |               | 28.18 | 13.06 | 0.63 | 0.52 | 55.51 | 0.75 | 0.21 | 1.14 | 0 | 2.15  |
| C-1   |               | 28.12 | 12.75 | 0.56 | 0.41 | 55.43 | 0.81 | 0.26 | 1.51 | 0 | 2.20  |
| D-1   |               | 28.08 | 11.87 | 1.25 | 0.76 | 55.73 | 0.61 | 0.23 | 1.47 | 0 | 2.36  |
| E-1   |               | 28.31 | 11.19 | 1.62 | 0.82 | 55.84 | 0.39 | 0.22 | 1.19 | 0 | 2.53  |

The BEI and EDS analysis indicate the formation of three phases in modified BOF slag, the dark grey areas (point 1) consist of O, Ca and Si in high levels, in which the atomic ratio is between the belite and the alite, and a small amount of P, Al, Fe, Mg and Ti are found as well. Combined with XRD analysis, it can be concluded that the atomic of P, Al, Fe, Mg and Ti probably enter the belite lattice, and the Ca/Si ratio and Al content in belite also increases as the lime content increase, but Mg content decrease. The light grey areas (point 2) are composed of O, Ca, Fe and Al in high levels, a little Mn, Mg, Si and Ti is detected, the atomic ratio is close to $C_4AF$. Combined with XRD analysis, it can be seen that the solid solution phenomenon is quite obvious, and the light grey areas might consist of several phases ($C_3A$ and $C_2$($A_xF_{(1-x)}$), and Mn, Mg, Si and Ti form a solid solution with iron phase. However, with the increase in the lime content in ingredients, the degree of the solid solution gradually weakened, the formation content of $C_4AF$ increased, and the Al/Fe ratio also had increased. The dark areas (point 3) contain high levels of O and Mg, the lower levels of Ca, Fe, Al and Ti, and the atomic ratio is close to MgO, the content of Ca, Fe, Al and Ti decrease with the increase in the lime content in the ingredients, which increase the content of active minerals. The above analysis further verifies the decomposition of RO in the BOF slag.

The test results of the $f$-CaO content are presented in Table 4; it can be seen that the $f$-CaO content of five samples is less than 1.0%, even if the lime content in the ingredients reaches the highest. Combined with XRD analysis, backscattered electron images (BEI) and energy dispersive spectrometer (EDS) analysis, it can be concluded that excess CaO enters into $C_2S$ and $C_4AF$, and the CaO/SiO$_2$ ratios in calcium silicate and the CaO/(Fe$_2$O$_3$ + Al$_2$O$_3$) ratios in iron phase will increase.

Table 4. The $f$-CaO content in modified BOF slag.

| Sample | BOF Slag | A  | B  | C  | D  | E  | Cement |
|--------|----------|----|----|----|----|----|--------|
| $f$-CaO (%) | 6.50 | 0.15 | 0.21 | 0.34 | 0.63 | 0.97 | 0.63 |

The physical and cementitious properties of the modified BOF slags–cement system are given in Table 5, respectively. The soundness of all samples is qualified. As the lime content increases in the ingredients, the setting time of the modified BOF slag–cement system becomes shorter, the compressive strength and activity index increase gradually, the activity index obviously better than BOF slag, and the 90 days activity index of sample
E is close to 100%. Combined with the above analysis, it can be concluded that the compressive strength increase with the increase in the CaO/\(\text{SiO}_2\) ratio and CaO/(\(\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3\)) ratio in modified BOF slag, especially the later compressive strength, which are attributed to the decrease in solid solution phenomenon and the increase in the active mineral content in modified BOF slag.

Table 5. Physical and cementitious properties of the modified BOF slag–cement system.

| Sample     | Specific Surface (m\(^2\)/kg) | Setting Time (min) | Compressive Strength (MPa)  | Activity Index (%) |
|------------|-------------------------------|-------------------|-----------------------------|-------------------|
|            | Initial | Final |                  | 7d   | 28d | 90d | 7d   | 28d | 90d |
| A–C        | 403     | 156   | 252              | 22.3 | 36.7 | 49.6 | 68.2 | 84.3 | 88.7 |
| B–C        | 396     | 149   | 236              | 23.2 | 37.9 | 50.3 | 70.9 | 87.1 | 89.9 |
| C–C        | 389     | 146   | 229              | 23.9 | 39.0 | 50.7 | 73.1 | 89.6 | 90.7 |
| D–C        | 401     | 138   | 210              | 25.3 | 40.7 | 54.6 | 77.3 | 93.6 | 97.6 |
| E–C        | 383     | 127   | 196              | 26.5 | 42.3 | 55.3 | 81.0 | 97.2 | 98.9 |
| BOF slag–C | 396     | 161   | 260              | 21.6 | 33.8 | 45.0 | 66.0 | 77.7 | 80.5 |
| Cement     | 358     | 105   | 163              | 32.7 | 43.5 | 55.9 | 100   | 100   | 100   |

The exothermic curves of cement samples during the hydration are shown in Figure 3. The hydration processes of the three kinds of cement are very similar, an exothermic peak is formed soon after the cement is mixed with water, and the hydration heat release rate of cement with 30% modified BOF slag is obviously lower than that of standard cement and the cement with 30% BOF slag before 25 min; however, the hydration heat release rate increases significantly and exceed the other two samples after 30 min, and the cumulative heat release of 7 days is close to that of standard cement. From the above analysis, it can be seen that the increase in \(\text{C}_4\text{AF}\) retards the formation of ettringite, and the formation of ettringite overlaps with that of C-S-H.

The capability of resisting sulfate corrosion of cement at different curing ages are shown in Table 6, respectively. With the increase in modified BOF slag, the Flexural Strength. The \(\text{Na}_2\text{SO}_4\) solution also increases, the corrosion resistance coefficient also increases, and the corrosion resistance coefficient of 90 days is more obvious than that of 28 days. It can be concluded that the introduction of \(\text{C}_2\text{S}\) and \(\text{C}_4\text{AF}\) does improve the corrosion resistance of cement and make more contribution to the strength of 90 days.

Table 6. Capability of resisting sulfate corrosion of cement at different curing ages.

| Sample | 28 Days | 90 Days |
|--------|---------|---------|
|        | Flexural Strength (MPa) | Corrosion Resistance Coefficient | Flexural Strength (MPa) | Corrosion Resistance Coefficient |
|        | Water | 5% \(\text{Na}_2\text{SO}_4\) Solution | Water | 5% \(\text{Na}_2\text{SO}_4\) Solution | Water | 5% \(\text{Na}_2\text{SO}_4\) Solution |
| Cement | 100 | 0 | 10.81 | 10.37 | 0.96 | 11.98 | 11.02 | 0.92 |
|        | 90  | 10 | 10.56 | 10.876 | 1.03 | 11.65 | 12.11 | 1.04 |
|        | 85  | 15 | 10.50 | 11.23 | 1.07 | 11.56 | 12.76 | 1.11 |
|        | 80  | 20 | 10.27 | 10.58 | 1.08 | 11.26 | 12.83 | 1.14 |
|        | 75  | 25 | 10.01 | 11.31 | 1.11 | 11.02 | 12.56 | 1.14 |
|        | 70  | 30 | 9.73  | 11.57 | 1.13 | 10.78 | 12.50 | 1.16 |

Modified BOF slag *: 90% modified BOF slag B + 10% natural gypsum. Bold is to emphasize this content.

SEM images for hydration products with 25% modified BOF slag content in water and in 5% \(\text{Na}_2\text{SO}_4\) solution for 90 days are given in Figure 4; there is a visible difference that can be observed in the morphology of the hydration products. Figure 4a,b displays the SEM image of hydration products in water for 90 days, there is a large amount of calcium silicate hydrate (C-S-H) in the form of network and sheet, and a small amount of long needle-like ettringite (AFT) and pompon-like \(\text{Al(OH)}_3\) and \(\text{Fe(OH)}_3\), however, the morphology of
ettringite and calcium silicate hydrate transformed from the long needle-like shape and network shape into short rod-like shape and radial pattern respectively, and ettringite intersects with calcium silicate hydrate in 5% Na$_2$SO$_4$ solution for 90 days, as shown in Figure 4c. Morphology changes of ettringite in 5% Na$_2$SO$_4$ solution are consistent with that of Zhang [21]. Therefore, it can be speculated that Na$_2$SO$_4$ promotes the dissolution of Ca$^{2+}$ from hydrated calcium silicate to form radial pattern calcium silicate hydrate with a low Ca/Si ratio. Meanwhile, ettringite is formed and grows up continuously in the presence of calcium hydroxide, SO$_4^{2-}$, Al(OH)$_3$ and Fe(OH)$_3$, and transformed into a short rod-like shape, which increases the compactness of the structure and improves the flexural resistance of the sample. Otherwise, according to the report of Zhang [21], the hydrate rate of C$_4$AF is significantly slower than C$_3$A, and the slow formation of ettringite does not cause swelling damage. The above analysis explains the reason for the increase in C4AF content in cement and the improvement of corrosion resistance.

![Figure 4](image_url)

**Figure 4.** SEM images for (a, b) in water of sample and (c) in 5% Na$_2$SO$_4$ solution for 90 days.

### 4. Conclusions

In this study, a component modification method of BOF slag with C$_4$AF and C$_2$S as the target mineral was studied, and the batching method, mineral compositions, hydration rate, activation index and capability of resisting sulfate corrosion were investigated. It can be concluded as follows:

1. Most inert phase in BOF slag can be converted into active minerals phase of C$_4$AF and C$_2$S by reasonable batching calculation and a reasonable amount of the regulating agent. However, the tricalcium silicate (C$_3$S) cannot be detected, even with the highest lime content in the ingredients, due to the low sintering temperature.
2. The activity index of modified BOF slag increases with the increase in lime content in the ingredients, excess CaO enters into C$_2$S and C$_4$AF, and the $f$-CaO content of the samples is less than 1.0%.
3. The flexural strength of samples decreases with the increase in the content of modified BOF slag B, but the capability of resisting sulfate corrode is improved, especially the structure of the hydrated product compact and 90-day sulfate corrosion resistance due to the constant formation of short rod-like shape ettringite in Na$_2$SO$_4$ solution.
4. Low-cost sulfate-resistant cement can be prepared by mixing Portland cement with modified BOF slag which the main mineral phases are C$_2$S and C$_4$AF.

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