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A discussion on improving hydration activity of steel slag by altering its mineral compositions

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ABSTRACT

This study aims to investigate the ways to improve the cementitious properties of steel slag. The results show that the cementitious phase of steel slag is composed of silicate and aluminate, but the large particles of these phases make a very small contribution to the cementitious properties of steel slag. RO phase (CaO–FeO–MnO–MgO solid solution), Fe₃O₄, C₂F and *f*-CaO make no contribution to the cementitious properties of steel slag. A new kind of steel slag with more cementitious phase and less RO phase can be obtained by removing some large particles. This new steel slag possesses better cementitious properties than the original steel slag. The large particles can be used as fine aggregates for concrete. Adding regulating agent high in CaO and SiO₂ during manufacturing process of steel slag to increase the cementitious phase to inert phase ratio is another way to improve its cementitious properties. The regulating agent should be selected to adapt to the specific steel slag and the alkalinity should be increased as high as possible on the premise that the *f*-CaO content does not increase. The cooling rate should be enhanced to improve the hydration activity of the cementitious phase at the early ages and the grindability of steel slag.

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1. Introduction

Cement concrete is the most utilized among the building materials in civil engineering constructions around the world. Cement industry is a high energy consumption industry and its CO_2 emission accounts for about 7% of global CO_2 emissions. It is very advantageous to reduce the cement consumption in the concrete industry. Some materials, especially fly ash and ground granulated blast furnace slag, are widely used as partial substitutes for cement (called mineral admixture) in many concrete applications. These mineral admixtures have become rare in many countries especially in China whose cement production exceeds half of the global production. Developing new mineral admixtures is currently a very important aspect to research.

Steel slag is a by-product in the steel production, which makes up a portion of about 15% of steel output [1]. The main chemical compositions of steel slag include CaO, SiO₂, Al₂O₃, Fe₂O₃, MgO and FeO [2,3]. The common minerals in steel slag are olivine, merwinite, dicalcium silicate (C₂S), tricalcium silicate (C₃S), tetracalcium aluminoferrite (C₄AF), dicalcium ferrite (C₂F), RO phase (CaO–FeO–MnO–MgO solid solution) and free CaO [4,5]. It is well known that C_2S and C_3S are the main cementitious phase in cement, so steel slag is believed to have certain cementitious properties.

Some investigations were performed for using steel slag as aggregates in asphalt mixtures and concrete [1,6–8]. The results have confirmed that the steel slag aggregates show better properties than crushed limestone aggregates because steel slag has certain cementitious properties. Some researchers investigated the utilization of steel slag for cement clinker production, and revealed that the quality of the produced cement could get satisfactory quality with 10.5% [9] or 8% [10] addition of steel slag. When steel slag is used as a mineral admixture to replace part of cement in the concrete production, its addition amount has great influence on the properties of concrete. If the addition amount of steel slag is low (e.g. 10% of total cementitious materials by mass), only the early strength of concrete decreases; but if the addition amount of steel slag is high (e.g. 30% of total cementitious materials by mass), both the early and late strengths of concrete decrease sharply [11,12].

Though the utilization ratio of steel slag in some regions (e.g. America, Europe, and Canada) is high, most of the steel slag is used as aggregates for road bases, asphalt paving and concrete, but the utilization ratio as a mineral admixture in concrete is very low [5,13–15]. Around 300 million tons of steel slag has been wasted in China and around 60 million additional tons of steel slag is discharged every year. The steel slag dump occupies large areas of land and results in many serious environmental problems. At the same

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Table 1
Chemical composition of cement and fly ash (wt%).

Composition	SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	Na_2O_{eq}	Loss on ignition
Cement	21.86	4.25	2.66	63.59	2.19	0.55	1.75
Fly ash	48.67	30.95	5.62	2.44	1.15	0.78	7.65

Note: $Na_2O_{eq} = Na_2O + 0.658K_2O$.

time, the concrete industry urgently needs mineral admixtures. The purpose of this research was to discuss the ways to improve the cementitious properties of steel slag when it is used as a mineral admixture based on the analysis on the contributions of the mineral phases on the cementitious properties of steel slag.

2. Experimental

2.1. Raw materials

The XRD results and chemical compositions of steel slag used are given in Ref. [16]. C₂S, C₂S, RO phase, C₂F, Fe₃O₄, C₁₂A, Ca₂Al₂Si₃O₁₂ and *f*-CaO are the mineral phases of the steel slag used, of which C₂S, C₂S and RO phase are the main phases. The specific surface area of the steel slag used was 458 m²/kg. The cement used was Portland cement with the strength grade of 42.5 complying with the Chinese National Standard GB 175-1999. The specific surface area of cement was 312 m²/kg. The fly ash used was low calcium fly ash complying with the Chinese National Standard GB 1596-91. The specific surface area of fly ash was $354 \text{ m}^2/\text{kg}$ and it was the common specific surface area in engineering. The chemical compositions of cement and fly ash are shown in Table 1.

2.2. Test methods

Steel slag paste was prepared by mixing steel slag with water at the water-to-steel slag ratio of 0.30 (mass ratio), which was denoted by S. Cement paste was prepared by mixing cement with water at the water-to-cement ratio of 0.42 (mass ratio), which was denoted by C. Steel slag needs less water than cement for hydration, so the water-to-steel slag ratio was set smaller than the water-to-cement ratio. The pastes were cast into plastic centrifuge tubes immediately after being stirred uniformly and then cured at 20 ± 1 °C to testing ages. At the ages of 1, 3, 7, 28, 90, and 360 days, the samples were placed in acetone. The absorption of the acetone into the pores of paste can stop the hydration process by eliminating the remaining water. After removing the free water, the samples were dried in vacuum at room temperature.

The morphologies of steel slag and its hydration products were characterized using a FEI Quanta200F scanning electron microscope under a high vacuum condition (SEM). EDX was used to identify the element distributions of the mineral phases and hydration products.

The steel slag and its hydration products were mineralogically determined by X-ray diffraction. XRD measurements were conducted with a TTR III diffractometer using nickel-filtered Cu K α_1 radiation (=1.5405 Å), 50 kV voltage and 200 mA current.

The non-evaporable water content of paste was obtained as the difference in mass between the sample heated at 65 °C and 1000 °C normalized by the mass after heating 65 °C, and correcting for the loss on ignition of unhydrated samples.

Thermogravimetric analysis (TG-DTG) was carried out using a Setaram thermoanalyser at a heating rate of 10 °C/min up to 900 °C.

Apart from the original steel slag, two other kinds of steel slag were obtained by sieving the original steel slag into two portions. Steel slag-A represented the portion of particles smaller than



Steel slag

80 72.

60 Volume/ %

42.

40

32.

Cement

steel slag. Steel slag-B represented the other portion. Mortar bars of $40 \text{ mm} \times 40 \text{ mm} \times 160 \text{ mm}$ were prepared. The water-to-binder ratio of all mortars was 0.50. The sand-to-binder ratio of all mortars was 3.0. Mortars made by binders composed of 100% cement, 80% cement and 20% original steel slag, 80% cement and 20% fly ash, 80% cement and 20% steel slag-A, 60% cement and 40% original steel slag, 60% cement and 40% fly ash, and 60% cement and 40% steel slag-A were denoted by CM, SM1, FM1, SSM1, SM2, FM2, SSM2, respectively. Mortars were cured first at 20 ± 1 °C and 95% relative humidity for the first day, and then cured in water of $20 \pm 1^{\circ}$ C for the remaining ages. At the age of 3, 28 and 90 days, their compressive strengths were tested according to Chinese National Standards GB/T17671-1999.

3. Results and discussion

3.1. Characteristics of the mineral compositions of steel slag

Fig. 1 shows the particle size distributions of steel slag and cement. The steel slag contains 42.9% (volume fraction) particles with diameters smaller than 10 µm and 28.8% particles with diameters larger than 60 $\mu m.$ The cement contains 32.3% and 4.3% of particles with diameters smaller than $10\,\mu m$ and larger than 60 µm, respectively. Steel slag has a little higher proportion of small particles (<10 µm) and much higher proportion of large particles (>60 µm) than Portland cement, however, the proportion of moderate particles $(10-60 \,\mu\text{m})$ in steel slag is far less than that in Portland cement, so steel slag indicates a relative poor continuity in particle size distribution. The poor continuity is also reflected in Fig. 2, a morphological micrograph of steel slag grains at $1000 \times$ amplification.

In XRD spectrum of steel slag, characteristic peaks of mineral phases concentrate at 30–45° [1,16–18]. Fig. 3 compares the XRD spectrums of the original steel slag, steel slag-A, and steel slag-B. It is derived from Fig. 3 that steel slag-B is lower at C₃S, C₂S, C₂Al₂Si₃O₁₂ and C₁₂A₇ but richer at RO phase than the original steel slag. Steel slag-A is richer at C₃S, C₂S, C₂Al₂Si₃O₁₂ and C₁₂A₇ but lower at RO phase than the original steel slag. Fig. 4 and Table 2 show the morphology and EDX results of the small particles in the steel slag, respectively. It is derived from EDX that these small particles are composed mainly by silicate and aluminate, and besides, a small amount of RO phase and Fe₃O₄.



Fig. 2. SEM morphologies of steel slag.



Fig. 3. XRD results of the original steel slag, steel slag-A, and steel slag-B.

3.2. Contribution of mineral to cementitious performance of steel slag

The results of Ref. [16] showed that most silicate and aluminate in steel slag have hydrated after 90 days of reaction, producing C–S–H gel, C–S–Al–H gel and Ca(OH)₂; but RO phase, Fe₃O₄ and C₂F barely participate in the hydration due to their low activity. XRD result of the hydration products of steel slag at 360 days is given in Fig. 5, in which gels are not reflected because of their amorphous structures. Fig. 5 indicates that RO phase, Fe₃O₄ and C₂F still show ultra low reaction extent even after having undergone hydration for 360 days. A micrograph of the hydration products of steel slag at 360

Table 2	
Chemical composition of particles in Fig. 4 (a	at.%).



Fig. 4. SEM morphologies of small steel slag particles.



Fig. 5. XRD results of hydration products of steel slag at the age of 360 days.

days is given in Fig. 6. The large unhydrated particles inlaid in the gel are diagnosed as RO phase by EDX analysis. RO phase not only contributes nothing to the cementitious properties of steel slag, but also forms a thin layer between the RO particle and the surrounding gel due to the relatively large particle size and smooth surface, so RO phase plays a negative role in the cementitious properties of steel slag to a certain extent.

	0	Mg	Al	Si	Р	Ca	Fe	Mn	Na	S	К
1	51.73	0.43	0.63	11.57	0.78	33.25	1.61	-	-	_	-
2	66.24	2.90	3.75	3.46	-	14.60	7.35	1.70	-	-	-
3	62.09	2.42	2.40	8.91	0.83	19.26	2.53	-	0.87	0.38	0.31
4	52.83	1.39	1.15	13.72	1.37	27.41	1.41	-	0.67	0.05	-
5	68.58	0.77	1.86	7.21	1.14	16.50	2.22	-	-	-	-
6	57.80	2.42	0.58	-	-	0.96	41.24	-	-	-	-
7	68.44	0.53	0.81	10.83	1.01	16.53	0.61	-	0.75	-	-
8	60.33	3.03	9.90	3.03	-	17.73	5.40	-	-	-	-



Fig. 6. SEM morphologies of the hydration products of steel slag at the age of 360 days.

Fig. 5 also shows that part of silicate and aluminate are still unhydrated even at 360 days. Generally speaking, hydration activity of silicate and aluminate is related with the particle size: the larger the size, the lower the activity. Some silicate and aluminate particles of steel slag are too large in size that only slight hydration occurs at the surfaces of these particles at 360 days and a very small amount of hydration product is generated (Fig. 7).

To a specific cementitious material, the amount of its hydration products is proportional to the non-evaporable water content of the products. The major cementitious components of both steel slag and Portland cement are C_3S and C_2S , and both with C–S–H gel and Ca(OH)₂ as major hydration products, so it is available to conduct a horizontal comparison to the non-evaporable water contents of the hydration products of steel slag and Portland cement. The non-evaporable water contents of the hydration products of steel slag and Portland cement are given in Table 3. It can be seen from Table 3 that at 360 days, the non-evaporable water content of the hydration products of steel slag is about 68.2% that of Portland cement. Meanwhile, the Ca(OH)₂ contents of the hydration products of steel slag and Portland cement determined by thermo-

Table 3

The non-evaporable water contents of the hydration products of cement and steel slag (/%).

Samples	Ages (days)								
	1	3	7	28	90	360			
С	9.32	12.73	14.27	15.34	16.77	17.30			
S	2.30	4.46	5.12	7.42	8.39	11.79			



Fig. 7. SEM morphologies of the hydration products of cementitious phase with large particles.

gravimetry are 5.82% and 20.08%, respectively. The Ca(OH)₂ content of the hydration products of steel slag is only 30.0% that of Portland cement. 1 mol C₃S generates about 1.5 mol Ca(OH)₂ while 1 mol C₂S generates about 0.5 mol Ca(OH)₂. Therefore, silicate in steel slag takes the form of C₂S mainly and C₃S captures a lower fraction, so the ratio of Ca(OH)₂ contents between the hydration products of steel slag and the hydration products of Portland cement is far lower than the ratio of the non-evaporable water contents.

The growth rates of hydration products for steel slag and Portland cement differ from each other: the hydration of cement is swift at early ages and a large amount of products is generated, then the hydration rate slows down gradually. By contrast, hydration products of steel slag increase slowly all the time. Ref. [16] concluded that the hydration activity of steel slag is much lower than that of Portland cement at the early ages by the determination of the hydration exothermic curve of steel slag. Other references also showed that the hydration rate of Portland cement-steel slag complex binder is lower than that of Portland cement, and the hydration rate of the complex binder slows further with the increase of steel slag [19,20].

f-CaO in steel slag differs from ordinary lime, as it has been "overburnt" during the steel making process. As a result, its hydration activity retards and the hydration may take place even long after the cement paste has hardened. The volume of $Ca(OH)_2$ expands at 1.98 times the reactant volume, causing cracking to hardened cement paste. It is a prominent factor giving ill stability to steel slag [10,18,21]. Therefore, *f*-CaO content in steel slag should be controlled.

1074	

Table 4			
Compressive	strength (of mortars	(/MPa).

Samples	Ages/d					
	3 days	28 days	90 days			
СМ	21.3 (0.20)	48.3(0.46)	64.7(0.51)			
SM1	15.4(0.24)	41.7(0.42)	58.9(0.42)			
FM1	16.9(0.22)	44.3(0.34)	61.7(0.61)			
SSM1	16.3(0.18)	44.8(0.47)	62.8(0.29)			
SM2	8.7(0.33)	28.1(0.51)	45.8(0.48)			
FM2	9.9(0.29)	30.4(0.37)	48.7(0.55)			
SSM2	9.4(0.12)	31.9(0.23)	52.3(0.62)			
SM1 FM1 SSM1 SM2 FM2 SSM2	15.4(0.24) 16.9(0.22) 16.3(0.18) 8.7(0.33) 9.9(0.29) 9.4(0.12)	41.7(0.42) 44.3(0.34) 44.8(0.47) 28.1(0.51) 30.4(0.37) 31.9(0.23)	58.9(0.42) 61.7(0.61) 62.8(0.29) 45.8(0.48) 48.7(0.55) 52.3(0.62)			

Note: the values in parentheses are the standard deviations.

Therefore, cementitious properties of steel slag comes from the cementitious phase which is composed by silicate (C_2S , C_2S) and aluminate ($C_{12}A$, $Ca_2Al_2Si_3O_{12}$) while the inert phase (mainly RO phase) and *f*-CaO make no contribution to the cementitious properties. It deserves attention that the hydration rate of the cementitious phase is much lower than that of Portland cement at the early ages, and the larger particles have ultra low hydration activity, contributing little to the cementitious properties of steel slag.

3.3. Utilizing steel slag by classification

Raising the fineness of steel slag is beneficial for its hydration activity [16]. However, the RO phase of steel slag exhibits rather poor grindability. Grinding steel slag into the status with specific area more than 450 m²/kg demands too much energy and besides, grinding RO phase further finer would not help to improve the cementitious properties. So, when steel slag is used as a mineral admixture, it should be ground into the status with specific area of about 450 m²/kg. As mentioned above, in ground steel slag, large particles mainly take the form of RO phase while small particles mainly take the form of silicate and aluminate. Through "classification" to steel slag, large particles (steel slag-B) can be used as fine aggregates while the remains (steels slag-A) can be used as mineral admixtures. Theoretically, compared to the original steel slag, steel slag-A possesses better cementitious properties as RO phase is reduced and cementitious phase is increased.

Table 4 lists compressive strengths of cement mortar and complex binder mortars. At the same age, compressive strength FM1 > SM1, FM2 > SM2, indicating that property of the original steel slag is poorer than that of fly ash. At the ages of 3, 28 and 90 days, the strength corresponding to SSM1 is 5.8%, 7.4% and 6.6% more than that corresponding to SM1; strength corresponding to SSM2 is 8.0%, 13.5% and 14.2% more than that corresponding to SM2. At 90 days, strengths corresponding to SSM1 and SSM2 have already reached 97.1% and 80.8% of the strength of CM, respectively. Testing results indicate that steel slag-A is better than the original steel slag. Comparing steel slag-A with fly ash: at 3 days, compressive strengths of SSM1 and SSM2 are slightly lower than the strengths of FM1 and FM2, respectively, but at 28 days and 90 days, compressive strengths of SSM1 and SSM2 are higher than the strengths of FM1 and FM2, respectively. In other words, property of steel slag-A is inferior to fly ash's property at the early ages but better than fly Table 5

Chemical compositions of steel slag and Portland cement [5,22].

	CaO	SiO ₂	Al_2O_3	F_2O_3	MgO	FeO	P_2O_5
Cement	62–68	20–24	4–7	2.5–6.5	1–2	_	0–0.5
Steel slag	30–55	8–20	1–6	3–9	3–13	7–20	1–4

ash's property at the late ages. In the meanwhile, the large particles of steel slag contain more RO phase than the small particles. The possible long term deleterious behaviour of large particles of steel slag is much more serious than that of small particles. So removing part of large particles of steel slag is meaningful to its properties. In summary, "classification" to steel slag is an effective way to improve its cementitious properties.

3.4. Reconstruction of steel slag

Reconstruction of steel slag means adding regulating compositions to alter the mineral compositions of steel slag during its manufacturing process. Obviously, according to the relation between mineral phases and cementitious properties, the larger the cementitious phase (silicate and aluminate) to inert phase (mainly RO phase) ratio, the higher the activity of steel slag. Enhancing this ratio is a fundamental strategy for improving activity of steel slag. Chemical compositions of steel slag and Portland cement are given in Table 5 [5,22]. Contents of CaO and SiO₂ in steel slag are lower than in Portland cement, so in the reconstruction process of steel slag, increasing CaO and SiO₂ contents is an important path to enhance the cementitious phase to inert phase ratio. The contents of CaO and SiO₂ in steel slag show great fluctuation. Therefore, to different kinds of steel slag, the "reconstruction" results with the same "reconstruction" agent are different. For example, Ref. [23] used electric furnace slag (with 53.31% CaO and 22.97% SiO₂) as reconstruction agent to reconstruct a type of converter steel slag (with 35.99% CaO and 16.11% SiO₂), and the 28 d strength of the reconstructed steel slag cement was 4.1-18.0% higher than that of the original steel slag cement. It can be deduced that if the electric furnace slag mentioned above is used to reconstruct a type of steel slag higher in both CaO and SiO $_2$ (for example with nearly 55% CaO and 20% SiO₂), the effect will be much poorer.

Mason [24] proposed a method to assess activity of steel slag by alkalinity (denoted as M), which is defined as $M = w(CaO)/[w(SiO_2) + w(P_2O_5)]$. According to Tang's research [21], the major mineral compositions of steel slag depend on its alkalinity, as illustrated in Table 6. Hence certain extent alkalinity should be maintained during the "reconstruction" process of steel slag. If the alkalinity is too low, the cementitious phase will decrease instead of increase. For example, when "reconstructing" a type of steel slag high in CaO but low in SiO₂, the "reconstruction" agent high in SiO₂ should be considered, however, excessive mixture of this "reconstruction" agent will lead to too low in alkalinity and increase the proportion of low-activity components (olivine and merwinite).

As mentioned above, C_3S in steel slag is insufficient, but C_3S is more active than C_2S , so enhancing C_3S content is beneficial to steel slag's activity. During the steel manufacturing process, along with adding CaO, alkalinity of steel slag increases and mineral com-

Table 6

Hydration activity, alkalinity and mineral phases of steel slag [21].

Hydration activity	Types of steel slag	Alkalinity w(CaO)/[w(SiO ₂)+w(P ₂ O ₅)]	Major mineral phases
low	Olivine	0.9–1.4	Olivine, RO phase, and merwinite
LOW	Merwinite	1.4–1.6	Merwinite, C ₂ S, and RO phase
Medium	Dicalcium silicate	1.6–2.4	C ₂ S, and RO phase
High	Tricalcium silicate	>2.4	C_2S , C_3S , C_4AF , C_2F , and RO phase

positions change gradually. According to Table 6, appropriately increasing the alkalinity of steel slag is beneficial to the reconstruction, but the content of *f*-CaO should be prevented from being too high.

Only increasing cementitious phase to inert phase ratio is not enough, because the cementitious phase is far inactive at the early ages (compared with Portland cement). This phenomenon is mainly caused by the cooling methods: in industry, Portland cement is controlled, while as a waste, steel slag is cooled naturally. C₃S is stable only above 1250 °C, and it will decompose when subjected to natural cooling under this temperature. However, the decomposition rate is minor and negligible under quench, so C₃S is rich in cement and remains in a metastable state. During the natural cooling process of steel slag, most C₃S is decomposed and C₃S in the metastable state is less. Besides insufficiency in C₃S, the other major cementitious component, C₂S, is also inactive in steel slag. This is because β -C₂S turns to γ -C₂S when the temperature drops below 500 °C during cooling crystallization. As Portland cement takes a sharp cooling manner-quench, there is no time for lattice rearrangement, so C₂S in cement exists in a metastable state. The cooling process of steel slag is long enough to allow the lattice rearrangement, so considerable C₂S takes the form of γ -C₂S. The activity of γ -C₂S is much lower than that of β -C₂S. Thus silicate in steel slag is much more inactive than that in Portland cement. Therefore, after the steel slag is reconstructed, quench should be adopted to accelerate the cooling rate.

Accelerating the cooling rate can not only enhance the activity of cementitious phase in steel slag at the early ages, but also make the mineral crystals in cementitious phase stay in small particle status by depriving time for grain growth to reduce the proportion of large particles in cementitious phase. Meanwhile, during quench, relatively large crystal stress is generated in mineral particles and more microcracks are nucleated, endowing the steel slag better grindability.

4. Conclusions

Silicate and aluminate are the main cementitious phases in steel slag and they mainly exist in the small particles. The cementitious phase in steel slag is less active at the early ages and large particles contribute little to the cementitious properties of steel slag. RO phase, Fe₃O₄ and C₂F make no contribution to the cementitious properties of steel slag and RO phase is the main inert phase and also with large particle size. Based on the relation between mineral phases and cementitious properties, two schemes for enhancing activity of steel slag are proposed: one is to classify steel slag into two parts. The part with large particles is used as fine aggregates and the remaining part is used as a mineral admixture. The second scheme is to reconstruct steel slag, adding regulating agents to increase the cementitious phase to inert phase ratio to improve the cementitious properties of steel slag. It is important to be noticed: in the reconstruction process, the regulating agent should be selected to adapt to the specific steel slag and the alkalinity should be increased as high as possible. If the cooling rate is able to be enhanced, the hydration activity of the cementitious phase at the early ages can be improved and the steel slag is more easily to be ground.

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