Effect of Ethanol-diisopropanolamine on the Hydration and Mechanical Properties of Steel Slag-cement Composite System

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Abstract

Steel slag as a solid waste during steel-making has potential cementitious activity, and its use as supplementary cementitious material is an effective way to improve its resource utilization. Ethanol-diisopropanolamine (EDIPA), as an organic admixture, affects the hydration of the steel slag-cement composite system. In this paper, the effect of EDIPA on the hydration and mechanical properties of the steel slag-cement composite system was investigated by using mortar strength tests and combining with heat of hydration, X-ray diffraction (XRD), thermogravimetry/differential thermogravimetry (TG/DTG), scanning electron microscope (SEM) and other testing methods. The results show that EDIPA can improve both early and late compressive strength of steel slag-cement mortar at low dosing (0.02%). However, at high dosing (0.06%, 0.10%), EDIPA is unfavorable to early strength, but enhanced late strength more strongly than low dosing. Low dose of EDIPA can promote the hydration of steel slag-cement composite pastes, and can also improve the hydration degree in the early and late stages. At low dosing, EDIPA can promote the formation of ettringite (AFt) and facilitate the transformation from AFt to monosulfoaluminate (AFm). In addition, EDIPA affects the crystal structure and morphology of Ca(OH)₂ prepared by co-precipitation method. And the morphology of Ca(OH)₂ gradually changes from long hexagonal column to short hexagonal column and then to irregularly radiating lamellar with the increase of EDIPA dosing. This indicates that EDIPA inhibits the vertical growth of Ca(OH)₂ and has the reference significance for the change of Ca(OH)₂ morphology in the composite paste.

1. Introduction

Steel slag is a solid waste produced during the smelting of steel to remove impurities from steel. Generally, the amount of steel slag generated is 15-20% of crude steel production (Chen et al. 2022). In 2021, China’s crude steel production was about 1.033 billion tons, and the new production of steel slag was about 150 million tons, and the accumulated stockpile was over 1 billion tons, which caused a lot of land waste and environmental pollution (WSA 2021; Zhuang and Wang 2021). The use of steel slag as supplementary cementitious material is not only an effective way to scale up the treatment of steel slag, but also a carbon neutral support path for the cement and concrete industry. However, the current effective utilization rate of steel slag in China is less than 30%, mainly due to the low strength and other performance problems of steel slag cementitious materials (Jing et al. 2020).

To solve the problem of poor cementitious activity of steel slag, high temperature conservation, high temperature reconstruction, carbonization treatment, mechanical milling, inorganic chemical and organic chemical activation methods have been widely investigated (Cui et al. 2016; Duan et al. 2019; Han et al. 2015; Huo et al. 2020; Lu et al. 2019a; Singh et al. 2021; Yang et al. 2021). High-temperature conservation, high-temperature reconstruction, and carbonation treatment methods all contribute to the improvement in the cementitious properties of steel slag and volume stability, all play an important role in improving the utilization of steel slag, and generally require specific sites with complex operations/equipment that can be used for pretreatment of steel slag or for the production of steel slag products process (Cui et al. 2018; Huo et al. 2021; Morandeau et al. 2014). Mechanical grinding is a prerequisite for the use of steel slag as supplementary cementitious material, and grinding of steel slag can effectively reduce its stability problems, but it has limited improvement in the cementitious properties of steel slag, and milling alone does not change the status of the overall utilization of steel slag (Zhang et al. 2015). Inorganic chemical methods generally require larger dosing, have higher excitation costs, and have problems with unfavorable or
unclear effects on long-term performance (Qian et al. 2017).

As a kind of organic small molecule cement admixture, alkanolamines have been widely used in cementitious materials because of its significant advantages such as small admixture dosage (50-500 ppm) and low impact on long-term performance. In terms of mechanical properties, triethanolamine (TEA), triisopropanolamine (TIPA), diethanol-isopropanolamine (DEIPA), and ethanol-diisopropanolamine (EDIPA) can improve the early and late compressive strength of cementitious materials to different degrees (Qian et al. 2017; Wang et al. 2017b; Xu et al. 2017). In terms of hydration process, all four alkanolamines can promote the early hydration of cementitious materials (Lu et al. 2017; Yang et al. 2017). However, most of the current studies on alkanolamines are focused on Portland cement and slag, fly ash, limestone and other composite systems (He et al. 2021; Wang et al. 2021; Zhang et al. 2020). And alkanolamines, especially EDIPA, are less studied in the steel slag-cement system. Therefore, in this paper, the effect of EDIPA on the hydration and mechanical properties of the steel slag-cement composite system is investigated by using cement mortar strength tests and combining with heat of hydration, X-ray diffraction (XRD), thermogravimetry/differential thermogravimetry (TG/DTG), scanning electron microscope (SEM) and other testing methods.

2. Experimental

2.1 Materials

The steel slag used was milled from hot stewed steel slag of Shandong Luli Steel with a specific surface area of 420 m²/kg. The cement used was Portland cement with a specific surface area of 350 m²/kg. The chemical composition of the steel slag and cement determined by X-ray fluorescence spectroscopy is given in Table 1. The chemical composition of the steel slag and cement determined by X-ray fluorescence spectroscopy is given in Table 1. The X-ray diffraction diagrams of the steel slag and cement are given in Fig. 1. The molecular structure formula of EDIPA is given in Fig. 2. EDIPA is purchased from Nanjing HBL International Co., Ltd. And the purity of EDIPA is >85%, and other chemicals such as CaCl₂ and NaOH are analytically pure.

2.2 Test methods

2.2.1 Preparation of steel slag-cement mortar

In accordance with GB/T 20491-2017 "Steel slag powder used for cement and concrete" (SAC 1999) and GB/T 17671-1999 "Test method of cement mortar strength (ISO method)" (SAC 2017), the steel slag and cement were mixed well in advance in the ratio of 3:7. And EDIPA was prepared in advance as a solution with 0.02%, 0.06% and 0.10% of the mass of the composite system, according to GB/T 17671-1999 the operation and steps specified in the steel slag-cement mortar test block molding, maintenance and strength testing. The specific formulation is shown in Table 2.

![Fig. 1 XRD patterns of steel slag and cement.](image1)

![Fig. 2 Molecular formula of EDIPA (Li et al. 2015).](image2)
2.2.2 Heat of hydration test for steel slag-cement composite system
An eight-channel microcalorimeter (Thermometrics TAMair) was used to measure the heat of hydration of the composite pastes with a water-steel slag/cement ratio of 0.5 and EDIPA dosing of 0%, 0.02%, and 0.06% of the mass of the steel slag-cement (3:7) composite powder. The constant temperature was set at 25°C, the mixing method was internal mixing, and the test duration was 72 h.

2.2.3 The XRD testing of steel slag-cement composite system
The steel slag and cement were mixed well in advance in the ratio of 3:7, and the EDIPA dosing was selected as 0% and 0.02% of the composite powder mass, and the water-steel slag/cement ratio was selected as 0.5 to prepare the steel slag-cement pastes. The pastes of 3 d and 28 d hydration was smashed into small pieces less than 0.5 cm³ and soaked in isopropanol for 48 h to terminate the hydration. After the isopropanol was poured out and the small net paste pieces were baked in a vacuum oven at 40°C for 48 h. Some of the pieces were ground into powder and passed through a 200 mesh standard sieve for XRD testing. The XRD analysis was carried out with a D8 Advance X-ray diffractometer from Bruker, Germany, using a Cu target with a scan step of 0.02° and a scan range of 5 to 70°.

2.2.4 TG testing of steel slag-cement composite system
About 15 mg of the composite powder in Section 2.2.3 that had passed the standard sieve of 200 mesh was put into the crucible, and the TG test was performed with a NETZSCH STA 449F3 integrated thermal analyzer. The test temperature range was 40 to 1000°C, the temperature rise rate was 10°C/min, and the test atmosphere was nitrogen atmosphere.

2.2.5 Observation of the morphology of steel slag-cement composite system
The small pastes blocker dried in Section 2.2.3 was taken as the core part, smashed into pieces, the bottom surface was ground flat and glued to the sample table that had been attached with conductive adhesive, and after 120 s of gold spraying, observation was made with Hitachi S-3400N scanning electron microscope.

2.2.6 Synthesis and morphological observation of Ca(OH)₂ by co-precipitation
The CaCl₂ of 0.04 mol and NaOH of 0.08 mol were dissolved in 100 ml of ultrapure water, and the two solutions were added dropwise to 300 ml of ultrapure water solution containing different concentrations of EDIPA using a peristaltic pump in a water bath at 60°C. The stirring method is magnetic stirring, set the same drop addition time, stirred the reaction for 30 min from the end of the drop addition, cooled for 30 min, filtered and baked in a vacuum oven at 60°C for 24 h. Among them, the EDIPA concentrations were: 0 g/L, 0.5 g/L and 8 g/L. A small amount of the synthesized Ca(OH)₂ powder was taken and glued to a sample table with conductive adhesive, sprayed with gold for 120 s. After that, Hitachi S-3400N scanning electron microscope was used for observation.

3. Results and discussion

3.1 Analysis of compressive strength of steel slag-cement mortar
The compressive strength of steel slag-cement mortar at 3 d, 7 d and 28 d for different EDIPA admixtures is given in Fig. 3. From Fig. 3, it can be seen that at lower dosing (0.02%), EDIPA has a promoting effect on the compressive strength of both early and late steel slag-cement mortar, while at relatively high dosing levels (0.06% and 0.10%), the addition of EDIPA has a negative effect on the early compressive strength, but has a stronger promoting effect on the late compressive strength than the low dosing. This is in general agreement with the study of EDIPA in Portland cement by Xu et al. (2017). Similar to other alkanolamines, EDIPA has a promoting effect on the hydration of steel slag-cement, but it also has an air-entraining effect due to the presence of methyl groups (Zou et al. 2018). The former promotion of hydration is beneficial for strength, while the latter air-entraining effect has a negative impact on strength. Since the air-entraining effect occurs only at the early stage of hydration, as hydration proceeds, the hydration products gradually fill the pores, thus improving the compressive strength of the steel slag-cement system at late stage. The above shows the effect of EDIPA on the compressive strength of steel slag-cement from the macroscopic properties of the composite system, and its effect on the hydration process needs to be further explored by more characterization means.

3.2 Heat of hydration analysis
The effect of mixing with and without EDIPA on the

![Fig. 3 Effect of EDIPA on the compressive strength of steel slag-cement mortar.](image-url)
The exothermic hydration of the steel slag-cement composite system is shown in Fig. 4. As can be seen from Fig. 4(a), the hydration of the steel slag-cement system is similar to that of Portland cement and still contains mainly three peaks. The first peak is usually considered to be formed by the dissolution of the main mineral phases in steel slag and cement and the generation of ettringite (AFT) (Lu et al. 2019b). The second peak mainly originates from the generation of hydration products such as hydrated calcium silicate gels (C-S-H) and Ca(OH)₂ (Wang et al. 2017a). When gypsum dihydrate is depleted during the hydration process, a "shoulder peak" appears on the hydration curve, which is usually considered to be caused by the transformation of AFT to monosulfoaluminate (AFm), resulting in the formation of a third exothermic peak (Xu et al. 2021).

From Fig. 4(a), it can be seen that the first exothermic peak gradually becomes higher with the increase of EDIPA dosing, which indicates that EDIPA can promote the hydration of aluminate phase in the steel slag-cement system, and then promote the generation of AFT. After the incorporation of EDIPA, the second exothermic peak gradually increased and the exothermic rate became faster, which indicated that EDIPA could improve the instantaneous exothermic rate of C₃S. In the blank sample without EDIPA, the third exothermic peak did not appear on the hydration curve. But when EDIPA was added, the third exothermic peak appeared and increased with the increase of the doping, which indicated that EDIPA could promote the depletion of gypsum by promoting the formation of AFT, thus promoting the transformation of AFT to AFm. From Fig. 4(b), it can be seen that the incorporation of EDIPA can increase the total exothermic heat of hydration of the steel slag-cement system at 72 h. Among them, the hydration of this composite system can be promoted more at low dosage (0.02%), which is one of the reasons for the increase of 3 d compressive strength at low dosage.

### 3.3 XRD analysis

Figure 5 shows the XRD patterns of the steel slag-cement composite pastes without and with 0.02% EDIPA at 3 d and 28 d of hydration and the XRD patterns of AFT, AFm, and Ca(OH)₂ characteristic peaks. From Fig. 5(e), it can be seen that the diffraction peak of AFT appeared significantly at 3 d of hydration, and when 0.02% EDIPA was added, the intensity of the diffraction peak of AFT decreased, while the diffraction peak of AFm appeared significantly. This indicates that the addition of EDIPA promotes the transition from AFT to AFm, which coincides with the appearance of the third exothermic peak on the heat of hydration curve. When gypsum is depleted, continued hydration of the aluminate phase will cause the transformation of AFT to AFm. In other words, the addition of EDIPA can promote the depletion of gypsum and thus accelerate the formation of AFm. In particular, the steel slag contains a certain amount of tricalcium aluminate (C₃A) and mayerite (C₁₂A₇), and the lack of gypsum, the promotion of hydration of the aluminate phase by EDIPA eventually leads to the acceleration of the AFT to AFm transition. Especially when the hydration reaches 28 d, the diffraction peak of AFT of the composite pastes doped with 0.02% EDIPA basically disappears, while the diffraction peak of AFm is more obvious, and the acceleration of AFT to AFm transition is further confirmed. From Figs. 5(e) to 5(f), it can be seen that the addition of EDIPA slightly reduces the intensity of the Ca(OH)₂ diffraction peaks, and there are two possible reasons for this. One is due to the inhibition of silicate hydration by EDIPA reducing the amount of Ca(OH)₂ production. The other is due to the amorphization of EDIPA reduces the amount of crystallized Ca(OH)₂. (Kong et al. 2013; Lu et al. 2020). However, the specific reason due to which one needs to be further analyzed.

### 3.4 TG/DTG analysis

The TG analysis could evaluate the degree of hydration of the steel slag-cement pastes. From the TG and DTG curves in Figs. 6(a) and 6(b), it can be seen that there are three main mass loss peaks in the tested temperature range. The first mass loss peak includes those caused by
AFt and C-S-H dehydration at around 110°C and those caused by AFm dehydration at 160°C (Lu et al. 2019b). The second mass loss peak is mainly located in the range from 400 to 500°C, which corresponds to the decomposition of Ca(OH)₂ (Zhang et al. 2020). The third mass loss peak is mainly located in the range of 500-800°C and corresponds to the decarbonation of CaCO₃ (Wang et al. 2019). In the first mass loss peak, it is obvious that the percentage mass loss of AFt and C-S-H and AFm increased significantly after the incorporation of 0.02% EDIPA, and in particular, the mass loss peak of AFm appeared significantly after the incorporation of EDIPA, and the mass loss peak of AFm was more obvious at 28 d. This indicates that the incorporation of EDIPA can promote the formation of AFm, namely, it can promote the transition from AFt to AFm which is basically consistent with the changes of heat of hydration and XRD.

Fig. 5 XRD patterns of steel slag-cement pastes with and without EDIPA, and characteristic peaks of AFt, AFm and Ca(OH)₂. The figures on the left hand side [i.e., (a), (c) and (e)] are the patterns of the pastes hydrated for 3 d, while those on the right hand side [i.e., (b), (d) and (f)] are the patterns of the pastes hydrated for 28 d.
From the TG curves of Figs. 6 (a) and 6(b), the chemically bound water and Ca(OH)$_2$ content can be obtained as shown in Fig. 7. It can be seen from Figs. 6 and 7(a) that the content of chemically bound water in the early and late stages of the pastes doped with 0.02% EDIPA is higher than that of the blank group in the test range of 40 to 1000°C. This indicates that the amount of hydration products, i.e., the degree of hydration, of the pastes doped with 0.02% EDIPA is higher than that of the blank group. According to the mass loss curve, using the tangent method (Feng and Sun 2020), it can be calculated that the Ca(OH)$_2$ content per 100 g of steel slag-cement hydrated pastes blank group at 3 d hydration is 17.58 g, and the Ca(OH)$_2$ content of the pastes doped with 0.02% EDIPA is higher than that of the blank group. According to the mass loss curve, using the tangent method (Feng and Sun 2020), it can be calculated that the Ca(OH)$_2$ content per 100 g of steel slag-cement hydrated pastes blank group at 3 d hydration is 17.58 g, and the Ca(OH)$_2$ content of the pastes doped with 0.02% EDIPA is 17.47 g. The Ca(OH)$_2$ content of the pastes doped with 0.02% EDIPA was 20.19 g. Since the difference in Ca(OH)$_2$ content between the two ages was less than 1 g, and Ca(OH)$_2$ was mainly generated by the hydration of the silicate phase, it can be concluded that the incorporation of EDIPA had no significant effect on the hydration of the silicate phase (Shi et al. 2011).

### 3.5 SEM analysis

Figure 8 shows the SEM of the steel slag-cement pastes without EDIPA and with 0.02% EDIPA at 28 d of hydration. From Fig. 8(a), it can be seen that in the steel slag-cement composite pastes without EDIPA, there are hydration products such as AFt in the form of needles, Ca(OH)$_2$ in the form of hexagonal plates, and C-S-H in the form of spheres (Ma et al. 2019). From Fig. 8(b), it can be seen that in the steel slag-cement composite pastes mixed with 0.02% EDIPA, there is a large amount of thin plates AFm phase and spherical C-S-H, while no obvious needle-like AFt and hexagonal plates Ca(OH)$_2$ are observed. Combining XRD and TG, it can be found that when the hydration age reaches 28 d, a large amount of AFt is transformed into AFm, which can be directly observed in Fig. 8. In addition, it can also be found that certain fibrous material appears in Fig. 8(b) and the hexagonal plates Ca(OH)$_2$ is reduced. From the overall structure, the structure of the pastes without EDIPA is loose and has some pores, while the overall structure becomes dense after mixing with 0.02% EDIPA, and the transformation of AFt to AFm fills some of the pores. These changes of hydration products may be the reason for the growth of compressive strength of steel slag-cement mastic at the later stage.

Since Ca(OH)$_2$ is one of the main products of silicate phase hydration, as the weak link of the pastes, its morphology has an important influence on the mechanical properties. In order to further reveal the effect of EDIPA on its morphology, Ca(OH)$_2$ was prepared by co-precipitation method in aqueous phase environment with different concentrations of EDIPA, and the SEM images of its morphology are shown in Fig. 9. From Figs. 9(a) to 9(c), it can be seen that the morphology of Ca(OH)$_2$ synthesized in the aqueous phase environment of pure water mainly appeared as hexagonal columns. And when the concentration of EDIPA was 0.5 g/L, the morphology of Ca(OH)$_2$ was slightly affected, the column length became shorter, and Ca(OH)$_2$ changed from...
long hexagonal columns to short hexagonal columns. And when the concentration of EDIPA directly increased to 8 g/L, the morphology of Ca(OH)\(_2\) changed dramatically to irregularly radiating lamellar, indicating that EDIPA inhibited the vertical growth of Ca(OH)\(_2\) (Wang et al. 2020).

The Ca(OH)\(_2\) belongs to a trigonal crystal system with a space group of \(\text{P3}\text{ml}\), and the Ca(OH)\(_2\) formed is predominantly hexagonal prismatic (Shen et al. 2016). When EDIPA is present in an aqueous phase environment, EDIPA will chelate the calcium ions in the aqueous phase. The literature by Zhang et al. (2016) shows that the hydroxyl group on EDIPA chelates with Ca\(^{2+}\). In the study by Yilmaz et al. (1993), the N atom also chelates with Ca\(^{2+}\) due to the presence of lone electron pairs. Therefore, both the O and N atoms on EDIPA have the ability to chelate Ca\(^{2+}\). The effect of chelation of EDIPA with Ca\(^{2+}\) on the crystal structure of Ca(OH)\(_2\) is shown in Fig. 10, which is a schematic diagram of the crystalline structure of the (010) crystal face of Ca(OH)\(_2\). From Fig. 10, it can be noticed that a Ca-rich layer with repeated arrangement of OH\(^-\)/Ca\(^{2+}\)/OH\(^-\) will be present in the direction perpendicular to this crystal face (Harutyunyan 2012). When EDIPA is present, here taken as an extreme case, EDIPA will chelate with the Ca-rich layer, which is equivalent to the fact that a layer of EDIPA with a very large molecular structure will be added to the crystal structure of Ca(OH)\(_2\), thus hindering the vertical growth of Ca(OH)\(_2\) (Wang et al. 2020). And when the EDIPA concentration is very high, the reason

![Fig. 8 SEM of steel slag-cement pastes with and without EDIPA at 28 d.](image)

(a) without EDIPA, 28 d  
(b) with 0.02% EDIPA, 28 d

![Fig. 9 SEM of Ca(OH)\(_2\) synthesized in aqueous phase environment with different concentrations of EDIPA.](image)

(a) 0 g/L EDIPA  
(b) 0.5 g/L EDIPA  
(c) 8 g/L EDIPA
that makes the Ca(OH)\(_2\) morphology no longer behave as a hexagonal may be the chelation of EDIPA with marginal Ca\(^{2+}\). During the hydration of the composite pastes, Ca(OH)\(_2\) is generated when the concentration of Ca\(^{2+}\) reaches supersaturation, but because the supersaturation of Ca(OH)\(_2\) in the composite pastes is much lower than normal, inhibiting Ca(OH)\(_2\) three-dimensional growth, this may be one of the reasons why Ca(OH)\(_2\) leads to a direct hexagonal flake/plate morphology in the composite pastes (Guan et al. 2021; Shen et al. 2016). Therefore, the synthesis of Ca(OH)\(_2\) in an aqueous phase environment with different concentrations of EDIPA using co-precipitation method is of reference significance for the formation of Ca(OH)\(_2\) in composite pastes.

4. Conclusions

(1) At low dosing (0.02%), EDIPA can improve both early and late compressive strength of steel slag-cement mortar. While at high dosing (0.06% and 0.10%), EDIPA has a negative effect on the early compressive strength of steel-cement mortar, but the enhancement effect on the later compressive strength is stronger than that of the low dosing (0.02%).

(2) During the hydration process, the low dose of EDIPA can promote the formation of AFt, and when the gypsum is depleted, it will promote the transformation of AFt to AFm. And EDIPA does not have a significant effect on the hydration of the silicate phase in the composite system.

(3) EDIPA has an effect on the structure and morphology of Ca(OH)\(_2\) prepared by co-precipitation method. EDIPA can inhibit the vertical growth of Ca(OH)\(_2\), and the morphology of Ca(OH)\(_2\) changes from long hexagonal column to short hexagonal column to irregular radial lamellar with the increase of EDIPA dosing. This is potentially helpful for the change of Ca(OH)\(_2\) morphology in steel slag-cement composite pastes by EDIPA.

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