

Research Article

Influence of Mineral Admixtures on Corrosion Inhibition Effect of Nitrites

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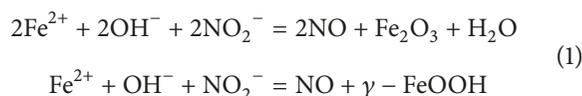
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Chemical quantitative analysis of effective anticorrosion component and micro-analysis of hydration products of fly ash and slag on the influence of the nitrites corrosion inhibition was studied by the free nitrite ion concentration and X-ray diffraction pattern. The free nitrite ion concentration was used to describe the corrosion inhibition effect of nitrites. And the X-ray diffraction patterns were used to analyze the adsorption properties. The research results show that fly ash and slag were beneficial for improving the corrosion inhibition effect of nitrites. Cement-based materials with slag at low content presented high free nitrite ion concentration, but the addition of low content of fly ash harmed the corrosion inhibition effect of nitrites. The specimens incorporated with both fly ash and slag can reach the highest free nitrite ion concentration when the compounding proportion was 1:1. It was concluded that the extent of mineral admixtures of the corrosion inhibition effect of nitrites was affected by its type and content.

1. Introduction

In civil engineering, reinforcement corrosion is considered as the leading cause of deterioration of concrete structures [1, 2]. Frequent repairs of reinforced concrete structures because of corrosion risk are costly and threaten the structures' durability and serviceability performance. To address the issue of reinforcement corrosion, the chemical compounds admixture known as corrosion inhibitor have been widely used which is considered an effective and economical method to avoid or delay the corrosion of reinforced concrete structures [3–6]. However, the most commonly used corrosion inhibitor is nitrite corrosion inhibitors, of which the effective anticorrosion component is the free nitrite ion existing in the concrete structures. The corrosion behavior of reinforced steel bars depends on the protective oxide film that forms on its surface under the alkaline environment. However, the free nitrite ion existing in the concrete structures can improve the corrosion resistance by repairing and reforming the passive film. The free nitrite ion reacts with ferrous ions on the surface of steel and oxidizes divalent iron ions into stable trivalent iron oxide, the composition of the passive film. The passive film is composed of an inner layer by Fe_2O_3 and an outer layer by FeOOH . Thus, the free nitrite ion content can be

considered as an important variable affecting the corrosion inhibiting and durability [7–16].



It is well known that cement-based materials combined with mineral admixtures can improve the performance of concrete. However, fly ash and slag are usually used in civil engineering, especially the coastal and offshore engineering such as sea crossing bridge and wind power plant and port. Due to the nitrites used as corrosion inhibitor, the mineral admixtures and nitrites are often applied in reinforced concrete structures at the same time [17, 18]. These cement-based materials combined with mineral admixtures and nitrites can not only improve concrete properties but also prolong the service life of structures. So it is of great significance to study the influence of mineral admixture on the content of effective anticorrosion component in cement slurry and its corrosion mechanism to improve the durability of reinforced concrete structures.

Therefore, four groups of cement pastes with water to cement ratio (W/C) of 0.4 were prepared by incorporating 2% $\text{Ca}(\text{NO}_2)_2$ by cement quality. The free nitrite ion

concentration and the X-ray diffraction patterns of cement-based materials with mineral admixtures have been studied. In addition, the free nitrite ion concentration is used to describe the corrosion inhibition effect of nitrites. The X-ray diffraction patterns are used to analyze the adsorption properties.

2. Experimental Program

2.1. Materials and Specimens. Ordinary Portland 42.5 cement, made by the Ningbo Conch Cement Co. Ltd., Zhejiang, was used as paste component. The fly ash and mineral with a density of $2.4\text{g}\cdot\text{cm}^{-3}$ and slag with a density of $2.87\text{g}\cdot\text{cm}^{-3}$ both sourced from the open market has a nominal (wt%) composition presented in Table 1.

NO_2^- was obtained from $\text{Ca}(\text{NO}_2)_2$ analytical reagent (A.R) and accounts for 2% of the cement quantity. The tested concretes were $40\text{ mm} \times 40\text{ mm} \times 160\text{ mm}$ rectangular specimens and the water to cement ratio (W/C) was 0.4. After demolding, the specimens were cured in a curing room for 28 days.

The cement paste specimens were separated into four groups representing different mixing method of mineral admixtures: add single fly ash (including A1-A3 group), add single slag (including B1-B3group), add both fly ash and slag (including C1-C3 group), and control group (including J group). More details of the experimental scenarios for these three groups of specimens are given in Table 2.

2.2. Specimen Sampling Method. (1) Determination of free nitrite ion in cement paste: The concentrations of free nitrite ion of the samples were measured according to the standard of "water quality determination of nitrite nitrogen-Spectrophotometric method." Firstly, the specimens were crushed and grounded into powder, then the standard square hole stone sieve and vibrating sieve machine were used to screen out the powder which has a particle diameter of less than 0.075mm , and the powder was put into a dry box and dried at $60\pm 5^\circ\text{C}$ for 24 h to attain constant weight. Subsequently, 1g powder was added to 100g distilled water in a conical flask. Put the conical flask into the water bath pot and keep the temperature at 60°C , stirring with a glass rod for 10 min. After this, place the conical flask in an air oscillator and centrifuge it still for 10 min. The solution was filtered and analyzed with the UV spectrophotometer. Finally, by comparing with the calibration curve, the concentrations of the free nitrite ion of the samples were determined, and the nitrite calibration curve is shown in Figure 1. The samples should determine whether those specimens were cured within 3d, 7d, 14d, and 28d. And in order to reduce the influence of the extract on the experiment results, the sample solution had an extraction temperature of 50°C , the vibration speed reached 220r/s, and the concussion time was 3 h.

(2) X-ray diffraction analysis: The core-drilling machine was used to drill the specimens which were cured in curing room for 90d, and the powder obtained should be passed through the standard square hole stone sieve of 0.3mm . Then the powder was put into a dry box and dried at $60\pm 5^\circ\text{C}$

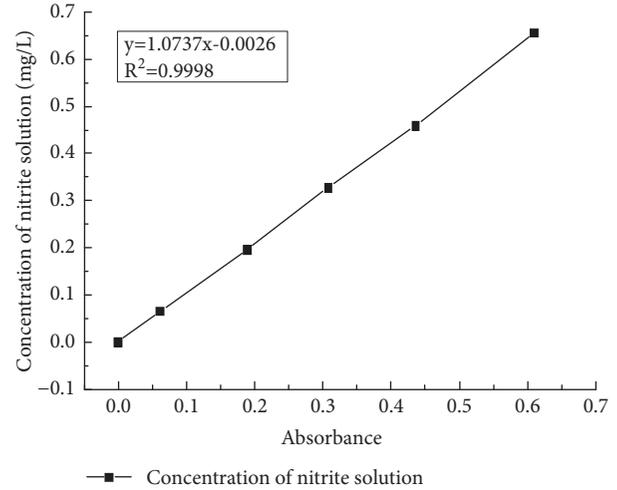


FIGURE 1: Calibration curve of absorbance and concentration of nitrite solution.

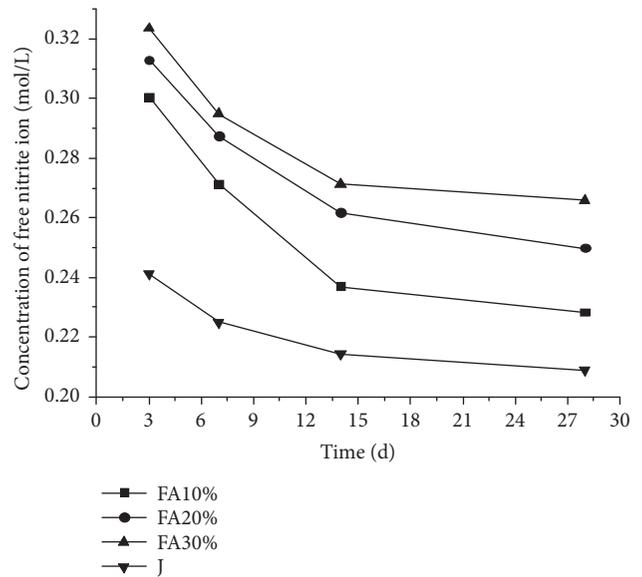


FIGURE 2: The free nitrite ion concentration curves of specimens A1-A3 and reference (J) group.

for 24 h to give off moisture. After cooling, the powder was placed in a plastic sample bag and sealed. Finally, use the X-ray diffractometer (D8) to scan the prepared sample powder to obtain a full X-ray diffraction pattern which can do both phase analysis and quantitative analysis.

3. Results and Discussion

3.1. Effect of Fly Ash on the Concentration of Free Nitrite Ion. Figure 2 shows the changes of the free nitrite ion concentration in the specimens with curing ages of 3d, 7d, 14d, and 28d when adding different amounts of fly ash from 10% to 30%. It can be seen from the figure that the concentration of free nitrite ion in the specimens decreased significantly in the first 14 days. After 14 days, the free nitrite

TABLE 1: Oxide composition of the fly ash and slag.

Item	Fly ash	Slag
Composition (mass % as oxide)		
Calcium oxide (CaO)	7.66%	1.8%
Silica (SiO ₂)	32.95%	57.2%
Alumina (Al ₂ O ₃)	13.99%	20.0%
Iron Oxide (Fe ₂ O ₃)	2.91%	9.1%
Magnesium oxide (MgO)	34.5%	5.1%
Sulfur trioxide (SO ₃)		1.7%

TABLE 2: Mix proportions of cement paste specimens.

specimens number	Fly ash	Slag
A-1	10%	0
A-2	20%	0
A-3	30%	0
B-1	0	10%
B-2	0	20%
B-3	0	30%
C-1	10%	20%
C-2	15%	15%
C-3	20%	10%
J	0	0

ion concentration was still decreasing but the rate of decline began to flatten. Besides, the addition of fly ash can influence the corrosion inhibition effect of nitrites due to the changes of the free nitrite ion values in specimens. The cement-based materials with fly ash at low content present low free nitrite ion concentration. Compared with the reference (J) group, the free nitrite ion concentrations have been increased when cement-based materials combined with fly ash. Increase of the amount of effective anticorrosion component in specimens results in the formation of passivation film on the steel's surface, leading to lower corrosion rate. When the amount of fly ash added into the specimen is 10% by mass of cement, the free nitrite ion value is relatively lower at the identical age and the decline of the effective anticorrosion component value in the specimen is most obvious. After curing for 28 days, the free nitrite ion value in the specimen was only 0.22825mg/L, accounting for 0.0032% of the quality of cement. The corrosion inhibition effect of nitrites showed little efficiency to avoid the corrosion of reinforced concrete structures.

Figure 3 shows that the main hydration products of the reference (J) group are quartz, C-S-H gel, Ca (OH)₂ crystal, and ettringite. It can be found that the most abundant hydration product is Ca(OH)₂ crystal by analyzing the diffraction peak area and diffraction peak height. Its diffraction peak area is highest and its diffraction peak height is slightly lower than C-S-H gel. The diffraction peak area and diffraction peak height can reflect the quantity of phases. Larger diffraction peak area means higher quantity of the phases. Also higher the diffraction peak height means higher quantity of the phases too [17]. And also it is observed that

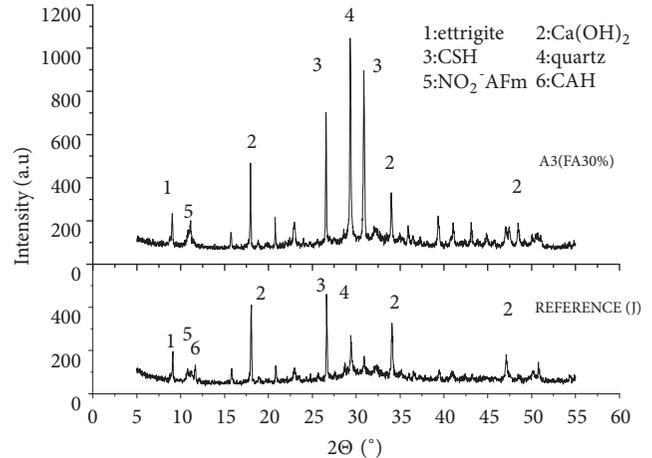


FIGURE 3: The X-ray diffraction pattern of the sample mixed with 30% single fly ash and the reference group.

CAH diffraction peak exists in reference (J) group. Compared with the X-ray diffraction pattern of the sample which adds fly ash to replace 30% quality of the cement, the CAH (3CaO · Al₂O₃ · nH₂O) diffraction peak disappeared and the NO₂⁻AFm diffraction peak is obviously enhanced. The diffraction peak height of the quartz is highest and that means that many SiO₂ do not participate in the hydration reaction. Additionally the most abundant hydration product is the C-S-H gel by analyzing the diffraction peak area and diffraction peak height. The diffraction peak of the CAH disappeared is possibly attributed to the presence of NO₂⁻, which can inhibit the chemical reaction of OH⁻ and C₃A. At the same time, the chemical reaction of NO₂⁻ and C₃A is promoted [18–21]. It is the reason for the enhancement of NO₂⁻AFm diffraction peak. And the production quantity of C-S-H gel is due to the predominant influence of the active substances in the composition of fly ash. They can react with the Ca (OH)₂ crystal produced during the cement hydration process and greatly promote the formation of gelatinous substances.

The free nitrite ions in cement paste can be solidified in two means. One is to react with cement clinker to form other chemical substances and the other is to adsorb on the surface of the gel substances through the adsorption of gel. Comparative observation of the free nitrite ion value and the hydration products shows that increase in the formation of C-S-H gel did not reduce the concentration of free nitrite ion

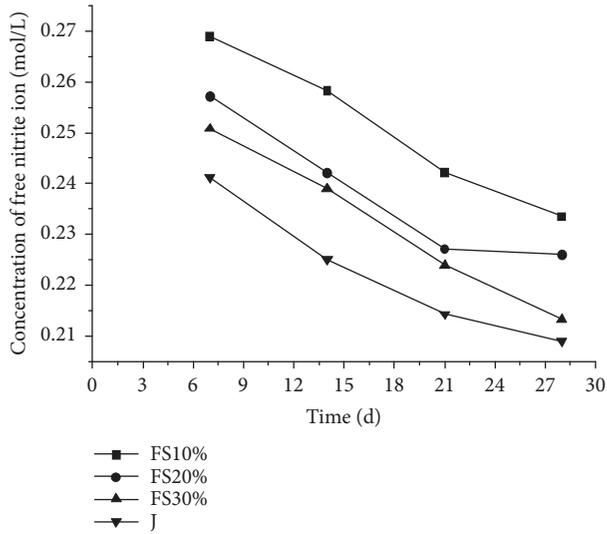


FIGURE 4: The free nitrite ion concentration curves of specimens B1-B3 and reference (J) group.

in specimens which is most likely due to the adsorption of C-S-H gel which was affected by the Ca/Si ratio of the sample [22, 23]. And the decrease of the physical adsorption capacity is possibly due to the C-S-H gel formatted become unstable. The composition of fly ash contains a large amount of active compounds known as SiO_2 , which not only consumes a lot of $\text{Ca}(\text{OH})_2$ crystal during hydration process but also increases the content of silicon and results in the decrease of Ca/Si ratio. Due to this, the free nitrite ions existing in the concrete are difficult to be adsorbed and improve the efficiency of the corrosion inhibition effect of nitrites.

3.2. Effect of Slag on the Concentration of Free Nitrite Ion.

Figure 4 shows the changes of the free nitrite ion concentration in specimens with curing ages of 3d, 7d, 14d, and 28d when adding different amounts of slag from 10% to 30%. It can be seen that contrary to the fly ash, as the amount of slag added to the specimens increased from 10% to 30%, the free nitrite ion values in specimens at the identical age are relatively higher and content of the effective anticorrosion component in specimens increased. Compared with the reference (J) group, the free nitrite ion concentrations have been increased when cement-based materials combined with slag. So for reinforced concrete structures, especially coastal and offshore concrete structures, which need to add both slag and nitrite corrosion inhibitors to improve concrete properties and durability, the amount of slag used should be strictly controlled. The results indicated that it is applicable to improve the corrosion inhibition effect of nitrites by using less slag as blending material of cement without affecting the properties.

Observing the X-ray diffraction pattern of the sample presented in Figure 5 which adds slag to replace 30% quality of the cement, the strongest diffraction peak is still the quartz; the second is the C-S-H gel. And the formation quantities of $\text{Ca}(\text{OH})_2$ crystal are significantly decreased.

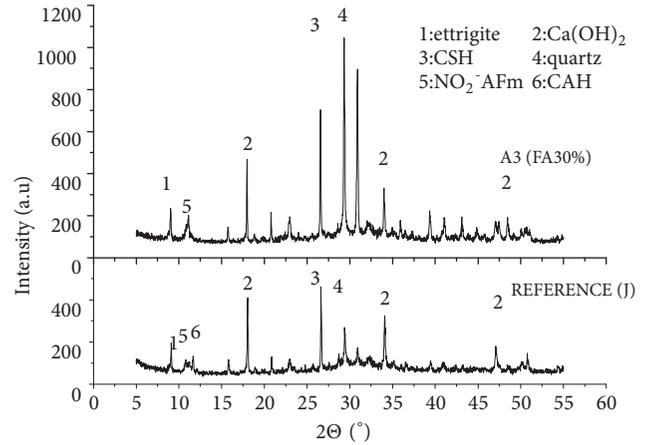


FIGURE 5: The X-ray diffraction pattern of the sample mixed with 30% slag and the reference group.

But compared with the reference (J) group, the formation quantity and diffraction peak of the $\text{NO}_2^- \text{AFm}$ and CAH are both improved. One of the possible reasons is that the slag substituting cement equally leads to the content of Al_2O_3 being increased markedly in specimens, thereby increasing the content of C_3A in cement clinker. This promoted more OH^- and NO_2^- reaction with C_3A causing the improvement of the formation quantity of the CAH and $\text{NO}_2^- \text{AFm}$. In addition, the concentration of SO_4^{2-} in the slag is also lower than that of cement. Part of the cement substituted by slag reduces the concentration of SO_4^{2-} of samples, weakened the competitiveness between SO_4^{2-} and NO_2^- , and also promoted the reaction of C_3A and NO_2^- . The strongest diffraction peak of quartz is possibly due to the composition of slag contains 57.2% SiO_2 . Although a large amount of SiO_2 is participating in the chemical reaction, there are still many SiO_2 remaining. This is also the reason for the decreasing of $\text{Ca}(\text{OH})_2$ crystal.

Comparative observation of the free nitrite ion value and the hydration products shows that increase in the formation of C-S-H gel caused a decrease in the concentration of free nitrite ion. This may be due to the volcanic ash effect of slag which promoted the C-S-H gel change from crystalline to amorphous. Improvement of the adsorption capacity of C-S-H gel caused more free nitrite ions being adsorbed on the surface of the gelatinous substance [24]. Therefore, increase in the mix amount of slag results in the decrease of corrosion inhibition effect of nitrites because of the increase formation quantity of C-S-H gel and its stable physical adsorption capacity.

3.3. Effect of Slag on the Concentration of Free Nitrite Ion.

For this research, three kinds of compounding proportions of fly ash and slag were designed, and Figure 6 shows the changes of the free nitrite ion concentration in the specimens. It can be seen from the figure that the content of free nitrite ions in specimen is the highest when the compounding proportion of fly ash and ore powder is 1:1 and the second is the specimen whose compounding proportion of fly ash and slag is 1:2.

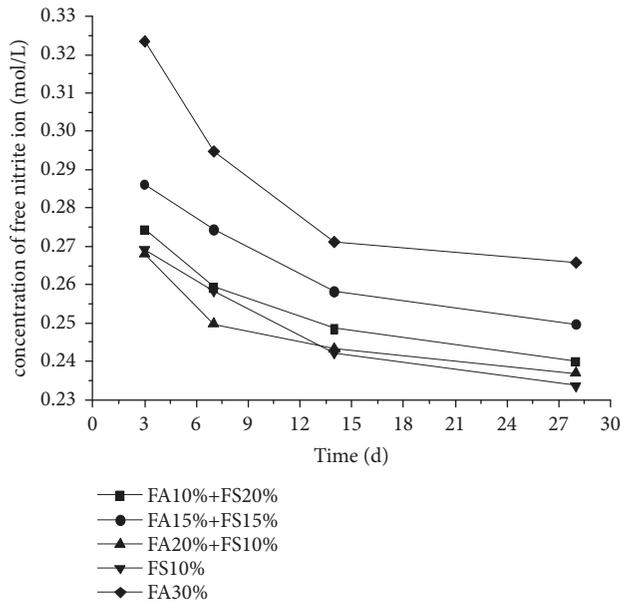


FIGURE 6: The free nitrite ion concentration of specimens A3, B1, and C1-C3.

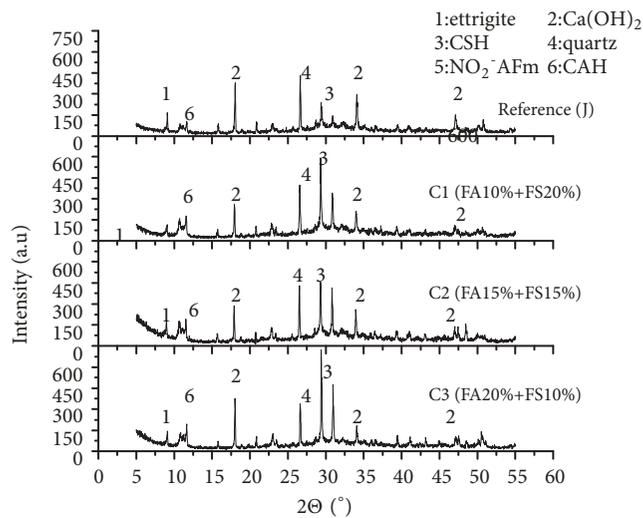


FIGURE 7: The X-ray diffraction pattern of the samples compounded different proportion fly ash and slag and reference group.

And when the ratio of the compounding proportion of fly ash and slag is 2:1, the free nitrite ion content in specimen is the least. However, compared with the specimens' added single fly ash or single slag, the free nitrite ion value in specimen is the highest when using fly ash to replace 30% quality of the cement, and the least is the specimen that used slag to replace 10% quality of the cement. The results indicated that substituting cement equally by slag has greater influence on the content of free nitrite ion in specimens than that of fly ash. And it is easier to consume the effective anticorrosion component of nitrite corrosion inhibitors and then have an influence on the formation of passivation film and corrosion inhibition effect. Therefore, when only

considering the corrosion effect of nitrites in engineering, it is preliminary to use fly ash as the mineral admixture in reinforced concrete structures and reduce or avoid use the slag. For the costal and offshore constructions which need adding both fly ash and slag, the ratio of compounding proportion of them is better to be 1:1.

Figure 7 shows the X-ray diffraction pattern of the specimens adding both fly ash and slag to replace part of the cement. Regardless of the compounding proportion of fly ash and slag, the main hydration products are quartz, C-S-H gel and $\text{Ca}(\text{OH})_2$ crystal, among which the diffraction peak of the C-S-H gel is the strongest. Ettringite, $\text{NO}_2^- \text{AFm}$, and CAH only account for a small portion. Compared with the reference (J) group, the diffraction peak of C-S-H gel is enhanced when the samples added both fly ash and slag, but the diffraction peak of $\text{Ca}(\text{OH})_2$ crystal is obviously weakened. The diffraction peak of ettringite and CAH barely change. The leading cause of this phenomenon is the incorporation of fly ash and slag promoting the hydration of cement clinker such as C_3A and C_2S , and a large amount of hydration product $\text{Ca}(\text{OH})_2$ crystal is consumed. The formation quantity of C-S-H gel is increased. Observing the figure, it can be seen that the diffraction peak of C-S-H gel and $\text{Ca}(\text{OH})_2$ crystal of the sample that the compounding proportion of fly ash and slag is 2:1; both are the strongest than the others, indicating that fly ash has a greater impact on the secondary hydration of the cement clinker and promotes the formation of C-S-H gel and $\text{Ca}(\text{OH})_2$ crystal [25]. In spite of a large amount of the $\text{Ca}(\text{OH})_2$ crystal generated in hydration process, the formation quantity of $\text{Ca}(\text{OH})_2$ crystal is still less than that consumed.

Comparative observation of the free nitrite ion value and the hydration products shows that when fly ash and slag are added into the reinforced concrete structures at the same time, the change of the free nitrite ion concentration in structures is influenced by the oxidation and the adsorption of the gelatinous substances. Both methods of solidifying the free nitrite ion have a great influence on the concentration of the effective anticorrosion component. The formation quantity of C-S-H gel is not the only factor to determine the free nitrite ion content. It is necessary to consider the stability of C-S-H gel and the chemical reaction consumption during hydration process, and the adsorption mechanism is relatively complicated.

4. Conclusions

For this research, the influence of fly ash and slag on the corrosion inhibition effect of nitrites was investigated using chemical quantitative analysis and micro-analysis method. The following conclusions were reached:

(1) Fly ash and slag are beneficial for improving the corrosion inhibition effect of nitrites. Cement-based materials with slag at low content present high free nitrite ion concentration, but the addition of low content of fly ash harms the corrosion inhibition effect of nitrites. The specimens incorporated with both fly ash and slag can reach the highest free nitrite ion concentration when the compounding proportion is 1:1.

(2) The activation and gelation of fly ash and slag are both brought into play, which can promote the hydration of cement and increase the formation quantity of C-S-H gel, changing the phase of concrete. And the slag has an even greater impact on the corrosion inhibition effect of nitrites than fly ash.

(3) The corrosion inhibition effects of nitrites are due to predominant influence of the formation quantity of C-S-H gel during hydration process and its stability. They determine the surface physical adsorption capacity of gelatinous materials.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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