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The Influence of Metallurgical Slag on Properties of Magnesium Sulfoaluminate Cement-Based Coating Materials

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Abstract: Magnesium sulfoaluminate cement (MSC) can be used as an inorganic cement-based coating material on metal surfaces, which has the advantages of good durability compared with organic coating materials. The effects of metallurgical slag on the fluidity, setting time, strength, and stability of MSC were studied to further enhance the role of the coating material. The hydration products and microstructure of MSC were analyzed by combining X-ray diffraction (XRD) and scanning electron microscopy (SEM) testing techniques. The results showed that the slag reduced the fluidity of MSC and shortened their setting time. The incorporation of slag significantly increased the strength of MSC at 1 day of age while reducing the shrinkage rate of MSC, but the water resistance of MSC decreased with the increase of slag content. XRD and SEM analysis showed that the slag accelerated early hydration, generated abundant fine crystals and gels, and reduced structural porosity. This study provides a theoretical basis for subsequent research on inorganic cement-based coating materials for the MSC system.

Keywords: magnesium sulfoaluminate cement; cement-based coating; metallurgical slag; strength; stability

1. Introduction

Surface coating treatment technology for metal materials can enhance their corrosion resistance, wear resistance, and extend their service life. Organic coatings are widely used in metal surface coating treatment. They are simple to operate and cost-effective, making them an effective method for metal corrosion protection [1,2]. However, organic coatings exhibit poor weather resistance and are prone to ageing. The early strength development of the coating material is slow, with high shrinkage rates that make cracking likely. In contrast, inorganic cement-based coating materials have the advantage of excellent mechanical properties and stability.

In recent years, numerous scholars have conducted in-depth research on magnesium oxysulfide (MOS) cement formed by the ternary cement system of MgO-$MgSO_4$-H_2O, achieving significant progress [3-5]. In 2013, Professor Yu Hongfa and Dr. Wu Chengyou's team substantially enhanced the strength and thermal stability of MOS cement through modification techniques [6-10]. However, Zhou et al. observed that while modified MOS cement enhances compressive strength, it prolongs setting time [11]. Subsequently, Chen Meng's team introduced pozzolanic materials into the MOS cement system, preparing MSC through the synergistic action of alkaline activators and sulfate activators [12]. MSC exhibits superior physical-mechanical properties and chemical stability compared to MOS cement, offering new insights for MOS cement modification research. Due to its high-temperature resistance, strong corrosion resistance, and excellent weatherability, MSC can be used as a coating material for metal surfaces [13-15].

While the MSC system enhances cement strength, it still suffers from issues such as prolonged setting time and insufficient volume stability. As an inorganic cement-based metal coating material, it is prone to coating sagging and uneven thickness. Additionally, its poor resistance to external damage in the early stages makes the coating more susceptible to cracking and spalling. Therefore, investigating the impact of different materials on MSC performance has become a critical step in advancing metal coating materials.

Admixtures commonly used in ordinary cement are not suitable for the MSC system and cannot effectively improve its early-stage properties [16]. Powdered blast furnace slag, a primary byproduct of the steel industry, is one of the most widely used and effective slag types in alkali-reactive cement materials. Finely ground blast furnace slag predominantly exists in a glassy state, rich in calcium, silicon, aluminum, and magnesium-based compounds, often present as solid solutions. It possesses excellent potential gelation reactivity. Within the MSC system, the latent activity of this slag can be effectively stimulated, significantly enhancing the comprehensive properties of MSC [17]. This study aims to further enhance the performance of MSC as a coating material by incorporating metallurgical slag. It systematically investigates the effects of different slag dosages on MSC workability, setting time, strength, water resistance, and volume stability. XRD and SEM analyses are conducted to examine the formation of hydration products and the similarities and differences in microstructure. This research not only advances the development of the MSC system but also enhances certain properties of inorganic cement-based materials as metal coating materials. The findings provide theoretical support for inorganic cement-based coating technologies for corrosion and fire protection on metal surfaces.

2. Materials and Methods

2.1.Raw Materials

The magnesium oxide used in the current study was light-burnt MgO powder with a purity of 90 %, from Haicheng, Liaoning Province, China. The selected 325-mesh bauxite was produced by Henan Jiayuan Environmental Protection Materials Co., Ltd. The grade 105 slag used was produced by Tangshan Tielan Company. The chemical compositions of the light-burnt MgO, bauxite and slag in wt.% are presented in Table 1.

Table 1. Chemical compositions of light burned magnesium oxide, bauxite and slag / %

Component		MgO	SiO_2	CaO	Al_2O_3	Fe_2O_3	Others
Content	Light - burned MgO	88.12	3.6	1.3	0.77	0.68	5.53
	Bauxite	0.35	39.48	0.92	53.33	1.75	3.23
	Slag	7.06	35	38.19	15.38	0.44	3.88

The magnesium oxysulfate ($MgSO_4{\cdot}7H_2O$) employed was a pure analytical reagent grade crystal obtained from Tianjin Biaozhunkiji Ltd., China. Aluminum sulfate is produced by an environmentally friendly building material manufacturer in Zhengzhou, Henan Province. An ultra pure analytical reagent grade citric acid ($C_6H_8O_7$) was selected as modifier additive for MOS cement.

The molar ratio of MgO: $MgSO_4$: H_2O in all the MSC cement mixtures was kept at 9:1:24. The specimens of MSC coatings with slag contents of 10 %, 20 % and 30 % in sequence were prepared in the experiment. The modifiers of citric acid and aluminum sulfate were 1 % and 2 % of the weight of light-burned MgO.

2.2. Test Methods

The test method for fluidity (mm: mm) of cement paste refers to the current Chinese National Standard GB / T 8077-2023 "Test Method for Uniformity of Concrete Admixtures".

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The determination of cement setting time refers to the current Chinese National Standard GB / T 1346-2024 "Test Method for Water Consumption, Setting Time, and Soundness of Cement Standard Consistency".

For each mixture, cubic specimens of 40 mm × 40 mm × 40 mm and 40 mm × 40 mm × 160 mm were prepared via vibration compaction. The strength development at 1, 3, 7, 14 and 28 days after air curing were recorded in a curing room at (20 ± 2) °C and (60 ± 5) % relative humidity. A concrete compression testing machine (TYE-300) was used and refers to current Chinese National Standard GB/T 17671-2021.

After 28 days of natural curing, the bottom pad was placed in still water to desolubilize the cement in unstable phase water. After soaking for a selected test duration, was taken out the test block and wipe off the surface moisture. Conducting compressive strength and flexural strength tests, the ratio of its strength to that of the test block cured under standard conditions for 28 days is the softening coefficient.

Pour the cement paste into a 40 mm × 40 mm × 160 mm mold and cure under natural conditions for 24 hours. After demolding the specimen, measure its length with an external micrometer and then place it in a curing chamber for further curing. Select specimens at the specified test age and measure their length again.

3. Results

3.1. Effect of slag on the flowability and setting time of MSC

As shown in Figure 1, the addition of slag reduces the fluidity of cement, and the fluidity decreases with the increase of slag content. At the incorporation amounts of 10 %, 20 %, and 30 %, the fluidity of the slurry is 73 mm, 69 mm, and 67 mm, respectively, which are 2.6 %, 8 %, and 10.6 % lower than that of the control sample.

The incorporation of slag leads to reduced slurry fluidity primarily due to two reasons. On the one hand, slag particles exhibit a flake-like structure with a large specific surface area and strong hydrophilicity, causing them to adsorb significant amounts of free water. Secondly, their high pozzolanic activity causes ion leaching when the system contains abundant OH^- and SO_4^{2-} ions. This leaching induces electrostatic interactions between particles, leading to flocculation and agglomeration of fine particles and increasing interparticle friction.

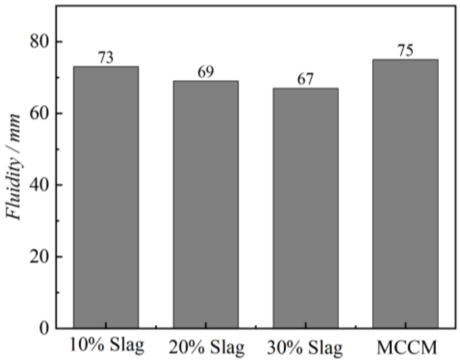

Figure 1. Effect of slag on flowability of MSC slurry

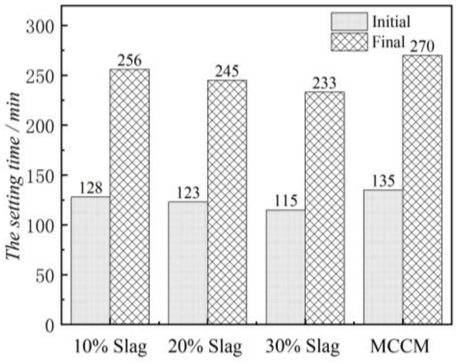

Figure 2. Effect of slag on the setting time of MSC slurry

Figure 2 shows that the setting time of MSC decreases with the increase of slag content. Compared with the control sample, the initial setting time of MSC with 10 % slag content was shortened by 7 minutes, and the final setting time was shortened by 14 minutes. At 30 % content, the initial setting time was shortened by 20 minutes, and the final setting time was shortened by 37 minutes.

The reason for the aforementioned phenomenon lies in the fact that ultrafine slag powder possesses a finer particle size distribution and larger specific surface area. This allows it to play a significant role in crystallization and nucleation, which promotes cement hydration. Additionally, slag glass with a high silicon-oxygen ratio exhibits high reactivity. Under the influence of OH^- and SO_4^{2-} ions, it undergoes depolymerization and condensation reactions, rapidly forming a large amount of M-A-S-H gel material. The increased gel content shortens setting time. The findings are consistent with the results of the study on the ability of adding ultrafine slag powder alone to reduce the setting time of aluminate cement [18].

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3.2. Effect of slag on the strength of MSC

Figure 3 shows that the early compressive strength increases with the increase of slag content. The compressive strength increases significantly at 1 day, the rising rate slows down at 3 days, and gradually becomes lower than that of the control sample in the later stage. When 10 %, 20 %, and 30 % slag is added, the compressive strength of MSC at 1 day is 67 %, 100 %, and 108 % higher than that of the control sample, respectively. At 3 days, the increases were 7 %, 11 %, and 18 %, respectively.

As shown in Figure 4, the slag also enhances the early flexural strength of the MSC. On the 1st day, each test group was 40 %, 15 %, and 28 % higher than the control sample, respectively. However, as the curing age increases, the flexural strength of the test groups begins to be lower than that of the control sample. On the 3rd day, the flexural strength of each test group decreased by 3 %, 21 %, and 6 %, respectively.

Ground granulated blast furnace slag possesses potential activity due to its glassy structure, and exhibits significant reaction characteristics in the MSC system under the synergistic effect of alkali and sulfate. In the early hydration stage, OH^- and SO_4^{2-} in the slurry promote the breaking of Ca-O bonds in the slag, releasing low bond energy Ca^{2+}. These Ca^{2+} destroy the network structure, and the Si-O and Al-O bonds break rapidly, dissociating the vitreous, dissolving the Al^{3+} and Si^{4+}, forming free unsaturated active bonds [19,20]. Subsequently, Ca^{2+} and Mg^{2+} in the system react with silica tetrahedrons and alumina tetrahedrons, hydrating to form gel products such as M-A-S-H and M-S-H. This series of processes promotes the improvement of slag activity and enhances the reactivity of the system. Meanwhile, XRD and SEM analyses show that the incorporation of slag has a certain filling effect, and the generated hydration products such as crystals and gels are intertwined with each other, which jointly improves the compactness of MSC and thus enhances its strength.

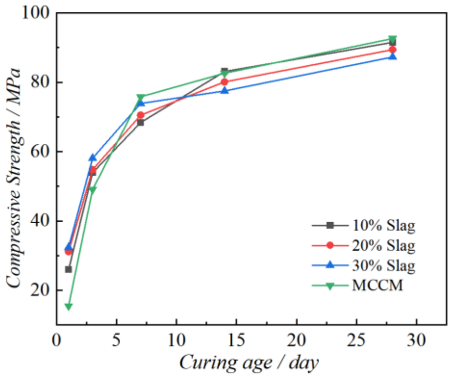


Figure 3. Effect of slag on compressive strength of MSC

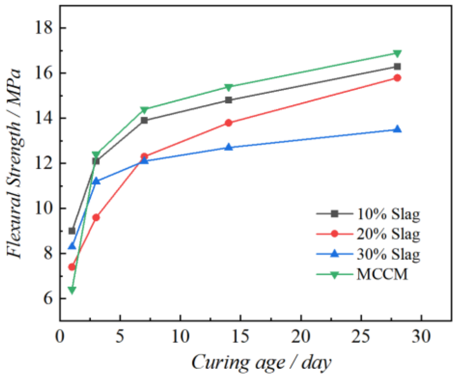


Figure 4. Effect of slag on flexural strength of MSC

3.3. Effect of slag on the stability of MSC

Figure 5 shows the softening coefficient of MSC decreases with increasing slag content. Adding 10 % slag reduces the softening coefficient compared to the control sample. At 20 % and 30 % slag content, the softening coefficient drops more significantly, at 28 days, the 30 % slag content MSC softening coefficient reaches a low level.

When slag content is at elevated levels, the system generates excessive gel products while the crystalline phase becomes relatively deficient. Under these conditions, moisture readily infiltrates the internal structure, causing low-reactivity MgO to react with water and form $Mg(OH)_2$. This process increases internal structural stress within the system, ultimately leading to a decrease in the softening coefficient of the MSC.

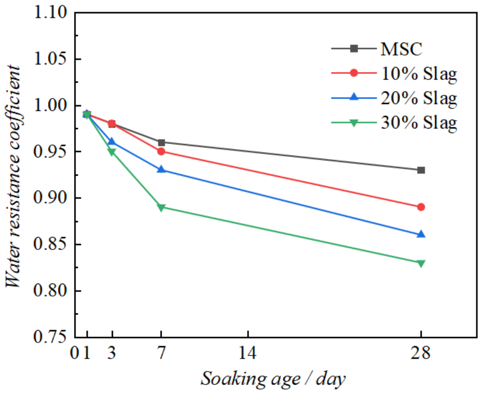


Figure 5. Effect of slag on softening coefficient of MSC

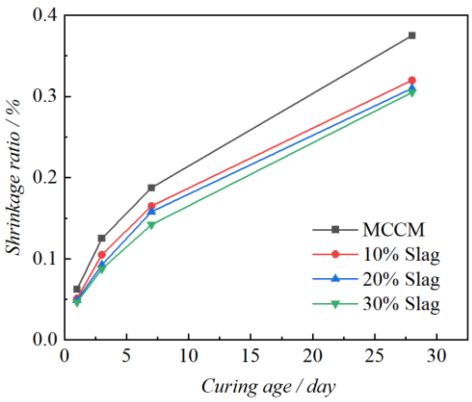


Figure 6. Effect of slag on shrinkage of MSC

It can be clearly observed from Figure 6 that the slag incorporation plays a role in reducing the shrinkage rate of MSC. And the shrinkage rate of MSC shows a gradual downward trend with the increase of slag content. The specific data showed that the shrinkage rates of 10 %, 20 %, and 30 % slag content were 18.4 %, 21.6 %, and 25.6 % lower than those of the control sample at 1 day of age. When the age period reached 3 days, the reduction of contraction rate in each proportional group was further expanded compared with that of the control sample, which was 19 %, 26 %, and 30 %, respectively. At 28 days of age, the contraction rate of each proportional group remained lower than that of the control sample, with a decrease of 14.6 %, 17.3 %, and 19.1 %, respectively.

The large specific surface area of slag confers strong water absorption capacity. This enhanced water retention slows evaporation, increases internal relative humidity, and ultimately reduces drying shrinkage [21]. Simultaneously, the filling effect of gel products on internal pores optimizes the microstructure of MSC, thereby reducing cement shrinkage [22]. XRD and SEM observations reveal that slag addition induces increased gel formation within the MSC matrix. These gel products fill and seal pores, reducing porosity connectivity and minimizing water loss. Furthermore, the interlocking arrangement of crystals formed during hydration with the gels further mitigates shrinkage in MSC.

3.4. XRD and SEM analysis

The XRD patterns of MSC and MSC mixed with 30 % slag at 1d and 28d are shown in Figure 7 and 8.

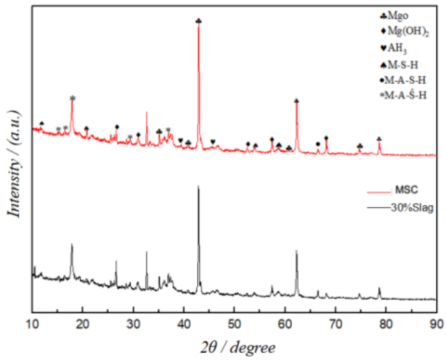


Figure 7. XRD patterns at the curing age of 1d

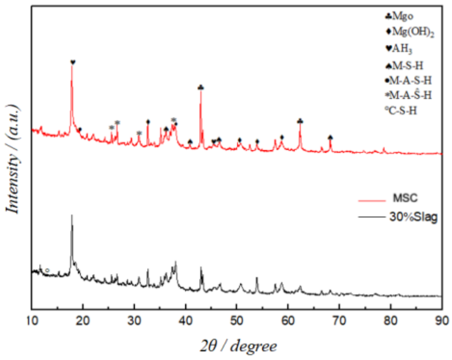
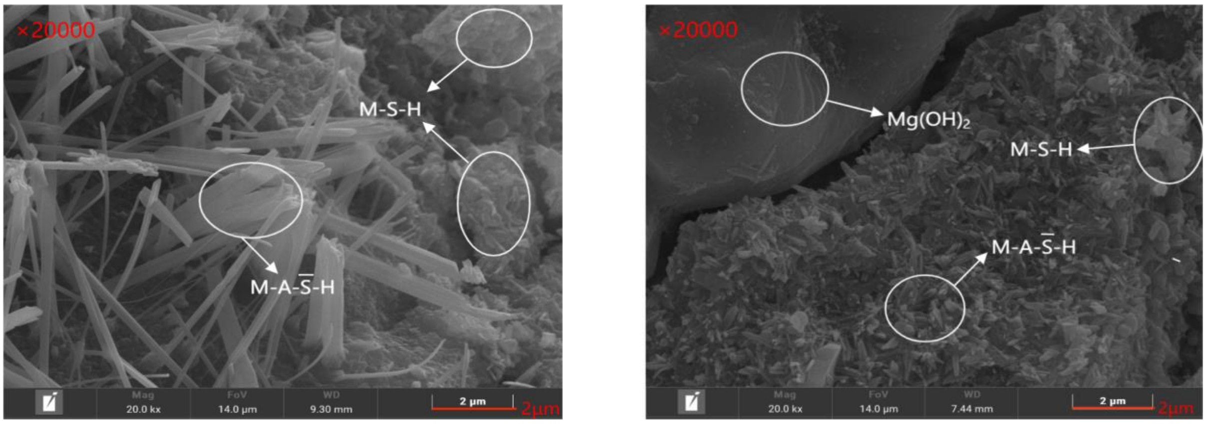


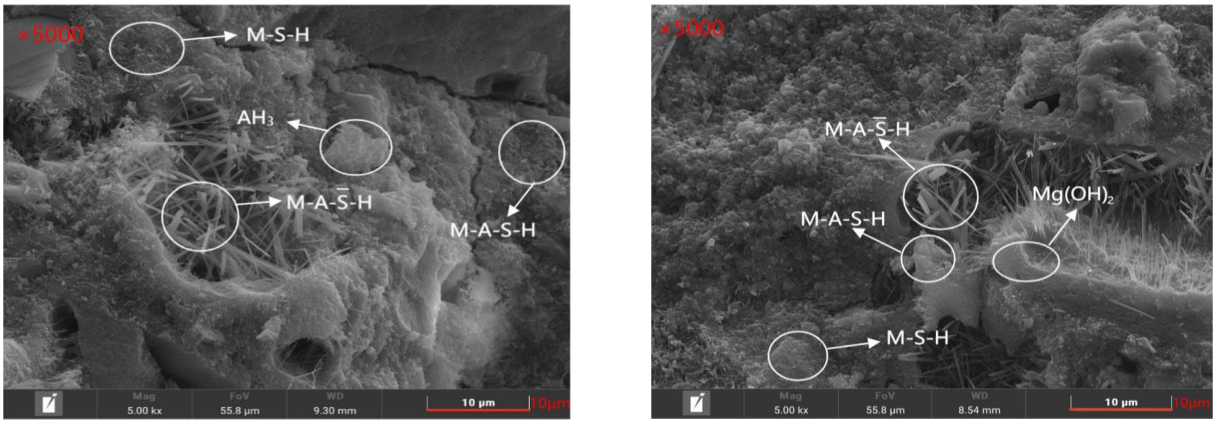
Figure 8. XRD patterns at the curing age of 28d

As can be seen from Figure 7, the main hydration products generated by MSC are M-A-S-H, M-A-$\overline{\text{S}}$-H, M-S-H, AH_3, and so on. The incorporation of slag accelerates the early hydration rate of MSC, increases the generation of M-A-$\overline{\text{S}}$-H and M-S-H hydration products, and reduces the generation of $Mg(OH)_2$. As the degree of hydration in the control sample increases, Figure 8 shows that the newly formed C-S-H exhibits poor crystallinity with a flattened peak shape. The final crystallinity of M-A-S-H is lower than that of the control sample, while the generation amount of $Mg(OH)_2$ is higher than that of the control sample.

Figure 9 shows scanning electron microscope images of MSC mixed with 30 % slag magnified 20,000 times on day 1 and magnified 5,000 times on day 28. The M-A-$\overline{S}$-H crystal whiskers formed in Figure 9a are more elongated than those in the control sample (Figure 9b), exhibiting an interlaced stacking pattern. The M-S-H flocculent gel formed between crystals is denser than in the control sample, resulting in a more compact system. Figure 9c shows that after 28 days, the system mixed with 30 % slag exhibited abundant flocculated M-S-H gel, AH_3 lamellar gel, and layered $Mg(OH)_2$. In Figure 9d, the control sample generated abundant needle-like M-A-S-H crystals and flocculated gel. This indicates that slag accelerates the early hydration rate of MSC. The large number of fine crystals and gels formed within one day enhance the early performance of MSC through a dense structure and pores. However, excessive addition is detrimental to the formation of long-term hydration products in MSC.



a) MSC with 30 % slag at 1d **b)** Control sample of MSC at 1d



c) MSC with 30 % slag at 28d **d)** Control sample of MSC at 28d

Figure 9. SEM images of MSC

4. Discussion

This study systematically investigated the regulatory effects of slag content on the multidimensional properties of MSC coating materials. Comparing the experimental results with existing research reveals that as slag content increases, the flowability and setting time of MSC continuously decrease. At a 30 % content, MSC flowability decreased by 10.6 %, with initial and final setting times decreasing by 14.8 % and 13.7 %, respectively consistent with trends observed in literature studies. Appropriate setting acceleration prevents sagging on inclined metal surfaces, reduces interface contamination between the coating and metal substrate, and lowers protective costs. Regarding MSC mechanical properties and shrinkage, slag addition enhances early compressive and flexural strengths, particularly at 1 day when MSC strength significantly increases. MSC shrinkage progressively decreases with higher slag content. Improved shrinkage and early strength enhance coating-to-metal adhesion, boosting protective capability and stability while reducing shrinkage-induced cracking and delamination. Characterization via XRD and SEM revealed that slag activity is stimulated within the MSC system, accelerating early-stage hydration. This promotes the formation of numerous interlocking crystals and gels that fill and seal pores, reducing porosity and improving certain MSC properties. However, as the curing period extends, slag addition impedes hydration reactions, resulting in poorly crystallized strength phases. Unreacted low-reactivity magnesium oxide reacts with water to form $Mg(OH)_2$, causing insufficient late-stage strength in MSC. Simultaneously, the softening coefficient continuously decreases with increasing slag content. At 28 days, the softening coefficient of MSC with 30 % slag content drops below 0.85, significantly reducing water resistance. This allows continuous water penetration into the coating interior, accelerating structural loosening and further exacerbating late-stage strength decay. Consequently, the coating becomes prone to cracking and peeling under minor external impacts, friction, or temperature fluctuations. This directly exposes the metal substrate, allowing corrosive media like water and salt spray to contact the base material and induce rusting, completely nullifying the protective function and creating a vicious cycle of performance degradation.

This study effectively addresses issues such as insufficient early strength, high shrinkage, and susceptibility to cracking in the MSC system. Consequently, it significantly enhances the overall performance of inorganic cement in coatings. It establishes a solid theoretical foundation for subsequent research on MSC coating materials. Nevertheless, MSC still holds potential for improvement in late-stage strength and water resistance. Future research may further explore the interaction mechanisms between slag and other components, as well as their influence on the material's late-stage strength and water resistance characteristics.

5. Conclusions

The results showed that with the increase of slag content, the fluidity of MSC decreased, and the coagulation time of MSC was shortened. The incorporation of slag significantly increased the early strength of MSC while reducing the shrinkage rate of MSC, while the water resistance of MSC decreased with the increase of slag content. XRD and SEM analyses showed that the slag accelerated the early hydration reaction, generated abundant fine crystals and gels in the early stage, reduced the structural porosity, but hindered the hydration reaction as the age increased. This study lays a solid theoretical foundation for subsequent research on the MSC system and coating materials.

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