

Article

Effect of Mineral-Generated Lithium Slag on the Properties of Magnesium Oxychloride Cement

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Abstract: A large amount of mineral-generated lithium slag will be produced after extracting lithium from spodumene. However, most lithium slag has not been well utilized. In this study, lithium slag was added to MOC, and its setting time, compressive strength, water resistance, phase composition, and microscopic morphology were studied. The results showed that although lithium slag prolonged the setting time and reduced the compressive strength of MOC, its water resistance improved. When the content of lithium slag was 20 wt%, the softening coefficient of the specimen reached a maximum value, and the softening coefficients at 3, 7, and 14 days were 1.47, 1.91, and 1.51 times that of MOC without lithium slag, respectively. A microscopic morphology analysis showed that the lithium slag transformed the MOC crystals from needle-like to column-like or block-like, the crystallization range expanded, and the binding force weakened. Magnesium oxychloride cement with excellent mechanical properties and good workability was combined with magnesium slag in this paper to provide the basis for the application of magnesium slag in building materials.

Keywords: magnesium oxychloride cement; lithium slag; setting time; compressive strength; water resistance



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1. Introduction

As an energy material, lithium plays an important role in mitigating problems caused by fossil fuels, and the use of lithium-ion batteries in electric vehicles can reduce environmental pollution caused by traditional vehicles [1,2]. Lithium is mainly produced in brine and hard rock mineral resources, among which spodumene is an important hard rock ore that contains lithium. At present, the industrial mainstream method of extracting lithium from spodumene consists of sulfuric acid roasting, and the main mineral compositions of the by-products of lithium slag are spodumene ($\text{LiAlSi}_2\text{O}_6$), gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), and quartz (SiO_2), as shown in Figure 1 and Table 1. The chemical composition mainly consists of CaO with a 3.6–12.1% content, SiO_2 with a 48.6–63.1% content, Al_2O_3 with a 14.0–20.7% content, SO_3 with a 4.5–9.3% content, Fe_2O_3 with a 1.0–1.8% content, MgO with a 0.2–0.8% content, and K_2O with a content of 0.1–5% [3]. The presence of sulfate ions in lithium slag makes its utilization in other industries difficult [4]. With the rapid development of the lithium salt industry, the discharge of lithium slag continues to increase, with the annual lithium slag emissions in China reaching 800,000 tons [5]. Except for a small portion of lithium slag that is used in construction projects, the remainder is stacked in the open air or is buried in roads [6], with a large amount of untreated lithium slag not only occupying farmlands but also causing serious environmental pollution [7].

Lithium slag has a high SiO_2 and Al_2O_3 content (Table 1); thus, the grinding powder of lithium slag can be reasonably applied to cement concrete as a mineral admixture [8–10]. Lithium slag used in the cement industry can be consumed on a large scale, with the nearby utilization of solid waste providing both environmental protection and economic benefits [11]. However, more SO_3 in lithium slag will not only affect the hydration of

ordinary Portland cement (OPC), but it will also affect the formation of ettringite in the later stage of hydration, resulting in cracking, strength loss, and other problems. As a result, the utilization rate of lithium slag in cement-based materials is low, especially in OPC concrete. To solve the problem of the mass utilization of lithium slag, scholars worldwide have conducted relevant studies on the application of lithium slag in cement. These studies have focused on the exploration of OPC as the goal, and the application of lithium slag in magnesium oxychloride cement materials has shown certain potential advantages [4,12–14].

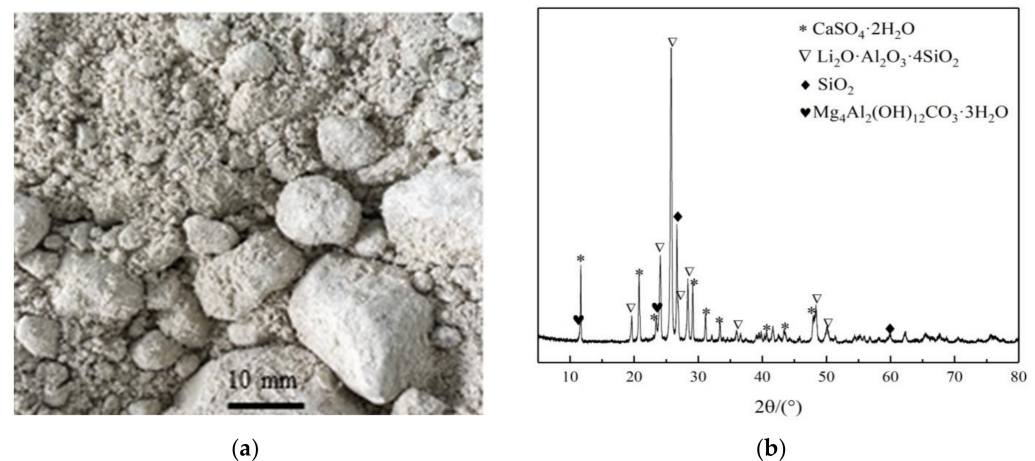
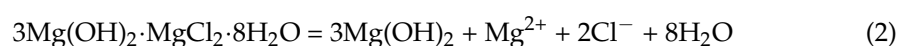
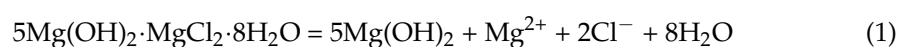


Figure 1. The appearance morphology (a) and XRD diffraction pattern (b) of the lithium slag.

Table 1. Chemical composition of the lithium slag.

Component	SiO ₂	Al ₂ O ₃	SO ₃	CaO	Fe ₂ O ₃	P ₂ O ₅	MgO	K ₂ O	Loss on Ignition
Content (%)	53.83	21.31	12.82	9.56	1.12	0.37	0.23	0.35	0.41

Magnesium oxychloride cement (MOC), which was successfully discovered by French scientist Sorrel in 1867, is a type of gas-hardening cement material formed by mixing active magnesium oxide powder with magnesium chloride solution [15]. Compared with OPC, MOC has excellent properties, such as lightweight, high strength, fast hardening, low thermal conductivity, good fire resistance, good wear resistance, and good compatibility with various materials [16–19]. In recent years, MOC has been widely used in the construction field [19–22], $5\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (abbreviated as P5) and $3\text{Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (abbreviated as P3) consisting of the two main hydrated phases of MOC at room temperature [4,23]. These two products will play a major role in the mechanical strength of MOC [24,25]. However, as metastable structures, the structures of these two phases are not very stable and are, prone to hydration reactions, generating $\text{Mg}(\text{OH})_2$, Mg^{2+} and Cl^- ions in the MOC system, resulting in a decrease in strength, a significant increase in porosity, and moisture absorption. This problem can seriously hinder the application of MOC. To solve this problem, many scholars worldwide have conducted numerous studies to modify MOC [21–32]. By adding various modifiers to MOC, the effects on the basic properties of MOC can be analyzed, as P5 and P3 can be easily dissolved in water into $\text{Mg}(\text{OH})_2$, Mg^{2+} , and Cl^- ions [33], with Formulas 1 and 2 showing the dissolution reactions [34,35].



As mentioned above, MOC has a high workability, strength, and other advantages. The mixing of waste lithium slag produced by spodumene lithium extraction into MOC will form building materials with economic value. However, there are few reports available

regarding this aspect. In addition, high contents of SiO_2 and Al_2O_3 in lithium slag mainly exist in amorphous form, with the potential of hydraulic properties. When lithium slag is added to MOC as a mineral admixture, the high contents of SiO_2 and Al_2O_3 in the lithium slag may react to form an Al–Si gel material, with the generated Al–Si gel material exhibiting certain hydraulic properties, and potentially improving the water resistance of MOC to a certain extent.

Based on this, in this work, lithium slag was added to MOC, and the influence of lithium slag on the setting time, mechanical properties, water resistance, phase composition, and microscopic morphology of MOC were analyzed, and the influence of lithium slag on the performance of MOC was explored to determine the optimal amount of lithium slag.

2. Materials and Experimental Procedure

2.1. Raw Materials

MgO powder with active MgO (MgO_a) content of 40.22 wt%, as measured by direct hydration at 105 °C [36], was supplied by Haicheng Magnesium Cement Mining in Liaoning, China. The chemical composition is shown in Table 2. The material consisted of light yellow or white powder, with some activity obtained by calcining magnesite (mainly MgCO_3) at 750–1100 °C. Its physical and chemical formula was MgO, and it had a melting point of 2850 °C and a boiling point of 3600 °C. The material was soluble in acid and ammonium salts, but insoluble in water and alcohol.

Table 2. Chemical composition of the MgO powder.

Component	MgO	MgO _a	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	Ignition Loss
Content (%)	85.92	40.22	1.24	1.31	6.03	0.55	4.84

Analytically pure magnesium chloride (MgCl_2) was purchased from Damao Chemical Reagent Co., Ltd., Tianjing, China. The appearance consisted of a colorless crystal or white crystalline powder, which was soluble in water and alcohol and slightly soluble in acetone. It could easily deliquesce in air and was soluble in water. In this experiment, magnesium chloride was configured as brine with a concentration of 24.49% and tap water was used in this experiment.

Lithium slag was incorporated as an additive and was obtained from Lithium Industry Co., Ltd., Qinghai, China. Its appearance was milky white, as shown in Figure 1a. The XRD diffraction pattern of lithium slag is shown in Figure 1b. The main mineral phases in lithium slag were β -spodumene ($\text{Li}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$), quartz (SiO_2), gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), and hydrotalcite ($\text{Mg}_4\text{Al}_2(\text{OH})_{12}\text{CO}_3 \cdot 3\text{H}_2\text{O}$). The chemical composition of lithium slag is shown in Table 1, indicating that SiO_2 , Al_2O_3 , and SO_3 were the most abundant, while CaO was the least abundant.

2.2. Specimen Preparation

Based on previous studies, the investigated MOC pastes were fabricated with using an active-MgO/ MgCl_2 molar ratio of 7 and an $\text{H}_2\text{O}/\text{MgCl}_2$ molar ratio of 15 [32,37,38]. The addition contents of lithium slag were 0%, 5%, 10%, 20%, and 30%.

In specimen preparation, according to the calculated amount of each substance, the required amounts of lightly fired magnesium oxide powder and lithium slag were first placed in a beaker, the required amounts of measured brine were poured into the beaker, and the stirred MOC slurry was injected into a 20 × 20 × 20 mm mold three times. After each injection of the slurry, it was necessary to gently stir the slurry in the mold with a spoon, to prevent incomplete preparation of the MOC specimens. The MOC slurry prepared in this experiment was relatively thin according to the utilized raw material ratio. To avoid shrinkage of the paste during the initial setting process, it was not necessary to clean up excess slurry after casting the mold, and the surface of the mold was covered with plastic preservative film. After initial setting of the slurry, we immediately removed

excess slurry on the mold surface with a blade, and maintained the specimens at room temperature (25 °C) until the mold could be demolded after molding. The demolded MOC specimens were cured in air for 3, 7, and 14 days. Finally, the specimens cured in air for 7 days were further soaked in water for 3, 7, and 14 days. We placed the samples into a sample bag and marked the samples according to the content of lithium slag, curing environment, and curing time (i.e., 30% air: 3 days, 30% water: 7 days).

2.3. Test Methods

The setting time of the MOC slurry was determined by a Vicat needle, according to the ASTM C191 standard [39]. The compressive strength of the specimens was tested by a CMT5105 microelectronic mechanical universal tester (CMT6104, Shanghai, China) with a loading speed of 1 ± 0.5 mm/min. For the water immersion test piece, the softening coefficient calculation formula is given by [37]

$$R = S_n / S_{7d} \quad (3)$$

where S_{7d} is the average compressive strength of the specimens cured for 7 days, and S_n is the average compressive strength of the specimens soaked for n days. A Rigaku (Tokyo, Japan) D/MAX-2550V diffractometer was used to analyze the phases of the samples. The scanning range was $5\text{--}90^\circ$, and the scanning rate was $12^\circ/\text{min}$. The dwell time was 15.50 s, the current was 30 mA, and the voltage was 40kV in the XRD analysis. In addition, a JSM-6390LV (JEOL, Tokyo, Japan) scanning electron microscope was used to analyze the micro morphologies of the samples mixed with 0%, 5%, and 30% lithium slag after curing in air for 7 days and soaking in water for 7 days.

3. Results

3.1. Effect of Lithium Slag on the Setting Time

The setting time of MOC has been shown to be closely related to the hydration rate of MgO and the process of crystalline phase formation [29]. Figure 2 shows the effect of different lithium slag amounts on the setting time of MOC. As shown in Figure 1, the initial and final setting times of MOC increased with the increasing lithium slag content. Compared with the MOC specimen without lithium slag, the initial setting time and final setting time of the MOC specimen increased by 16.53% and 0.99%, respectively, when a 5 wt% content of lithium slag was added. When doped with a 10 wt% content of lithium slag, the initial and final setting times of MOC increased by 16.53% and 4.37%, respectively. When doped with a 20 wt% content of lithium slag, the initial and final setting times of MOC increased by 20.27% and 5.77%, respectively, and when doped with a 30 wt% lithium slag content, the initial and final setting times of MOC increased by 20.27% and 6.63%, respectively. According to the setting time test, we observed that the addition of lithium slag was similar to other solid wastes, which could prolong the setting time of magnesium cementitious materials. There were two reasons for the above results. The addition of lithium slag reduced the content of MgO, which hindered the reaction with water to a certain extent, and the lithium slag had a heat absorption and cooling effect on the MOC slurry, which slowed down the early hydration reaction and prolonged the setting time to some extent. The hydration of MOC is the result of the dissolution of MgO in the phosphoric acid solution. Then, the reaction kinetics are mainly controlled by the dissolution of the oxide [40]. When lithium slag was added, the total water consumption in the system was reduced, which reduced the amount of water required by the solvent of the MgO particles, restraining the dissolution rate of the MgO particles in the slurry to some extent, and prolonging the setting time of the slurry [4].

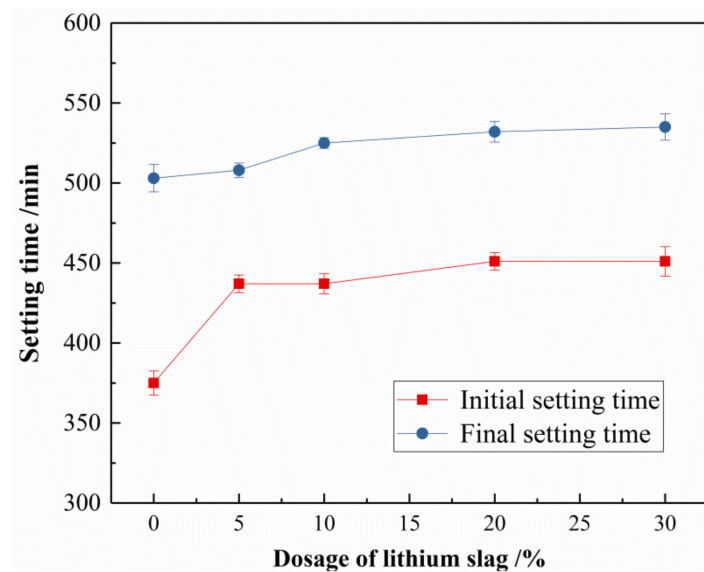


Figure 2. Effect of different dosage of lithium slag on the setting time of MOC.

3.2. Effect of Lithium Slag on the Compressive Strength

Figure 3 shows the compressive strength of MOC mixed with lithium slag in air (a) and water (b) at different curing times. During air curing (Figure 3a), with an increase in lithium slag content, the compressive strength of MOC generally shows a downward trend. When lithium slag is not added, the compressive strength gradually increased with an increase in the temporary period, reaching a peak at 14 days, which was 1.92 times and 2.85 times that of the compressive strength at 7 and 3 days, respectively. When the content of lithium slag increased from 0 to 5 wt%, the compressive strength values at 3 and 7 days showed an increasing trend, while the compressive strength at 14 days showed a decreasing trend. This phenomenon was possibly due to the continuous hydration reaction of MgO, which was not fully reacted in the system at 3 and 7 days, and increased the strength of the solid system. Compared with the sample with a 5 wt% lithium slag content, when the lithium slag content increased to 20 wt%, the compressive strength values of the MOC sample at 3, 7, and 14 days showed a downward trend, which decreased by 25.11%, 42.78%, and 26.69%, respectively. When the lithium slag content increased to 30 wt%, the compressive strength of the specimens aged for 3 days continued to decrease, while the strength of the specimens aged for 7 and 14 days remained relatively stable.

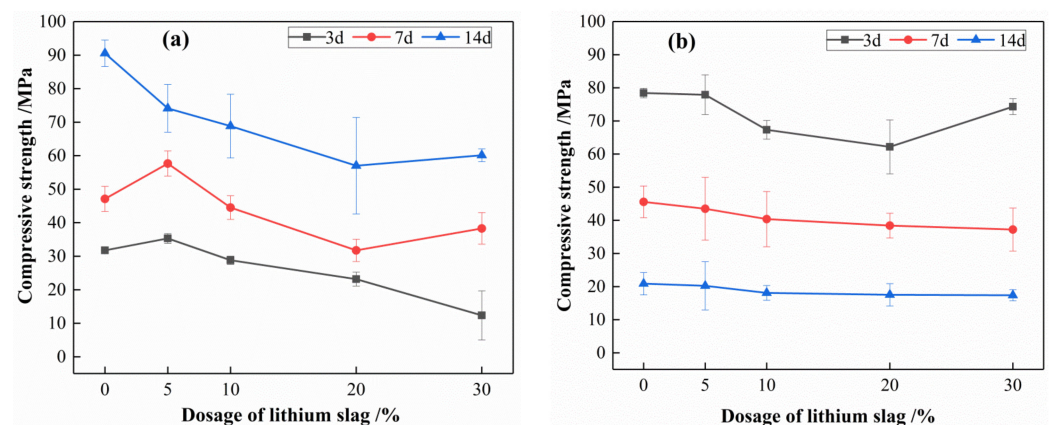


Figure 3. The compressive strength of MOC in air (a) and water (b).

The MOC specimens with different amounts of lithium slag were cured in water, as shown in Figure 3b. Figure 3b shows a decreasing trend for the different temporary

periods with an increase in the lithium slag content. However, compared with the air-cured specimens (Figure 3a), the decreased trend in the water was relatively stable, and the MOC specimens with a temporary period of 3 days showed a large change when the lithium slag content was greater than 5 wt%. With a 20 wt% lithium slag content, the MOC specimens showed a decrease of 20.74% compared with the neat paste. However, the 7- and 14-day MOC samples with 20 wt% lithium slag content decreased by 15.74% and 16.08%, respectively, compared with the samples without lithium slag.

3.3. Effect of Lithium Slag on the Water Resistance

Figure 4 shows the softening coefficient of the MOC specimens with different lithium slag contents in the water environment. As shown in Figure 4, the incorporation of lithium slag increased the softening coefficient of the MOC specimens, indicating that the water resistance of MOC improved. At the same age, with an increase in the lithium slag content, the softening coefficient generally increased first and then decreased. When the content of lithium slag was 20 wt%, the softening coefficient of the specimen reached a maximum value, and the softening coefficients at 3, 7, and 14 days were 1.47, 1.91, and 1.51 times that of MOC without lithium slag, respectively. With 30 wt% lithium slag content, the samples soaked in water for 7 and 14 days showed softening coefficient reductions of 23.04% and 48.62%, respectively, compared with the samples with 20 wt% lithium slag content. At the same time, the softening coefficient of the specimens with different lithium slag contents decreased significantly with increasing age. According to the above analysis, adding lithium slag could improve the water resistance of MOC, and the appropriate amount of lithium slag was about 20 wt%. Studies have shown that the strength retention rate of MOC mortar can be improved to varying degrees after mixing reinforced waste filler [41]. Similarly, lithium slag acts as a filler in the MOC system, which can fill some pores in the matrix after the solidification of MOC slurry, reducing the degree of water erosion. Therefore, MOC mixed with lithium slag shows relatively better water resistance.

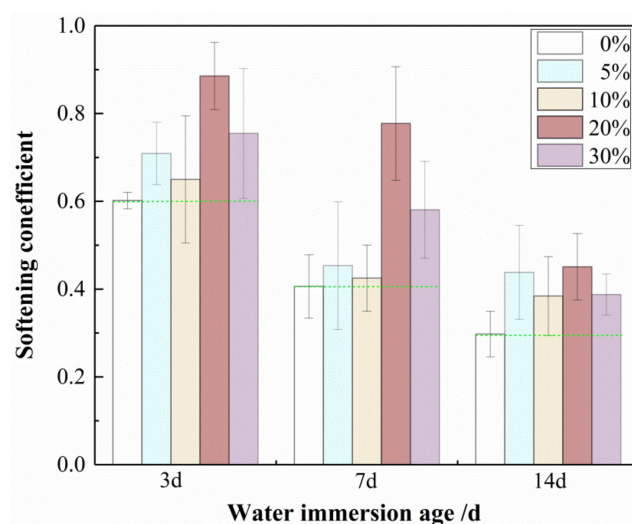


Figure 4. The softening coefficient of MOC with different contents of lithium slag.

3.4. Effect of Lithium Slag on the Phase Composition

Figure 5a,b shows the XRD diffraction patterns of the MOC samples with different lithium slag contents after curing in air for 7 days and soaking in water for 7 days. The analysis showed that the addition of lithium slag did not change the phase composition of MOC, which contains MgO, Mg(OH)₂, and 518 phases. As shown in Figure 5a, the sample contained a large amount of 518 phases (P5), and the diffraction peak of the 518 phases decreased slightly with increasing lithium slag content. As shown in Figure 5b, the diffraction peak area of the 518 phases in the sample after soaking in water decreased compared with the 518 phases in Figure 5a, while the diffraction peak of Mg(OH)₂ increased,

indicating that a small amount of the 518 phases hydrolyzed and generated $\text{Mg}(\text{OH})_2$ after soaking in water. Therefore, the dissolution of these two ions could better explain the state of MOC in water environments, as portrayed in Equations (1) and (2), where 318 phases and 518 phases were transformed into brucite ($\text{Mg}(\text{OH})_2$), followed by the dissolution of magnesium and chloride ions in water. The dissolution of 518 phases in water is the main reason for the decrease of MOC matrix strength. In addition, with an increase in lithium slag content, the diffraction peak area of $\text{Mg}(\text{OH})_2$ decreased, indicating that lithium slag inhibited the hydrolysis of the 518 phase. This was possibly because after lithium slag was added, silicon, aluminum, and other substances in the lithium slag-generated gel substances attached to the crystal surface, to a certain extent, protecting the 518 phases from water erosion and thus improving the water resistance of MOC. For another reason, due to the addition of lithium slag, which enhanced the compactness of the matrix, the cations (Mg^{2+}) had difficulty dissolving from the matrix in a short time [37].

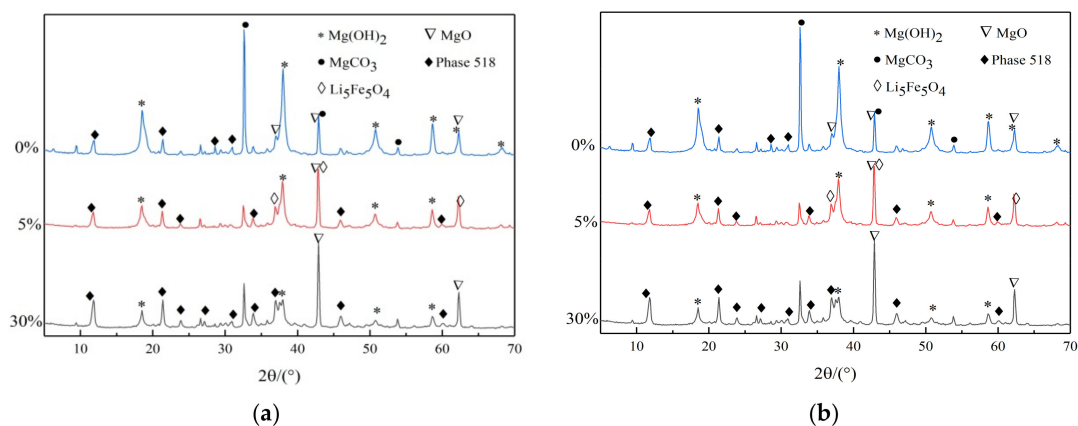


Figure 5. The XRD patterns of MOC with different contents of lithium slag. (a) Curing in air and (b) Soaking in water.

3.5. Effect of Lithium Slag on the Micromorphology

Cement soundness refers to whether the volume change of cement is uniform during setting and hardening. The cracks, disintegration, or other flaws can be measured by a cement soundness test [42,43]. Figure 6 shows the microscopic morphology of the MOC specimens with different lithium slag contents cured in air for 7 days. As shown in Figure 6a, the MOC specimens without lithium slag contained a large number of needle-like or rod-like crystals, which were closely distributed and had a strong binding force. After adding 5 wt% of lithium slag (Figure 6b), the acicular crystals in MOC became thicker and thinner. With an increase in the lithium slag doping content (Figure 6c,d), the needle-like crystals in MOC gradually disappeared and changed into columnar or blocky structures, the crystallization range expanded, and the binding force weakened. This explains why the strength of the samples cured in air decreased with an increasing lithium slag content.

Figure 7 shows the micromorphology of MOC with different lithium slag contents after immersion for 7 days. As shown in Figure 7, the porosity of the MOC specimen increased and the structure was loose after 7 days of soaking in water. Meanwhile, cracks appeared on the surface, which explained the decrease in strength of the specimen after soaking in water. In addition, we observed that gelatinous substances were produced and attached to the surface of the 518 crystals, and when the content of lithium slag was 30%, needle-like crystals were observed, indicating that the addition of lithium slag inhibited the hydrolysis of the 518 phases to a certain extent. After lithium slag was added, silicon and aluminum in the lithium slag formed gelatinous substances that could attach to the crystal surface and protect the 518 phases from water erosion, and the lithium slag acts as a filler in the MOC system, which can fill part of the pores in the matrix after the solidification of MOC slurry, thus improving the water resistance of MOC.

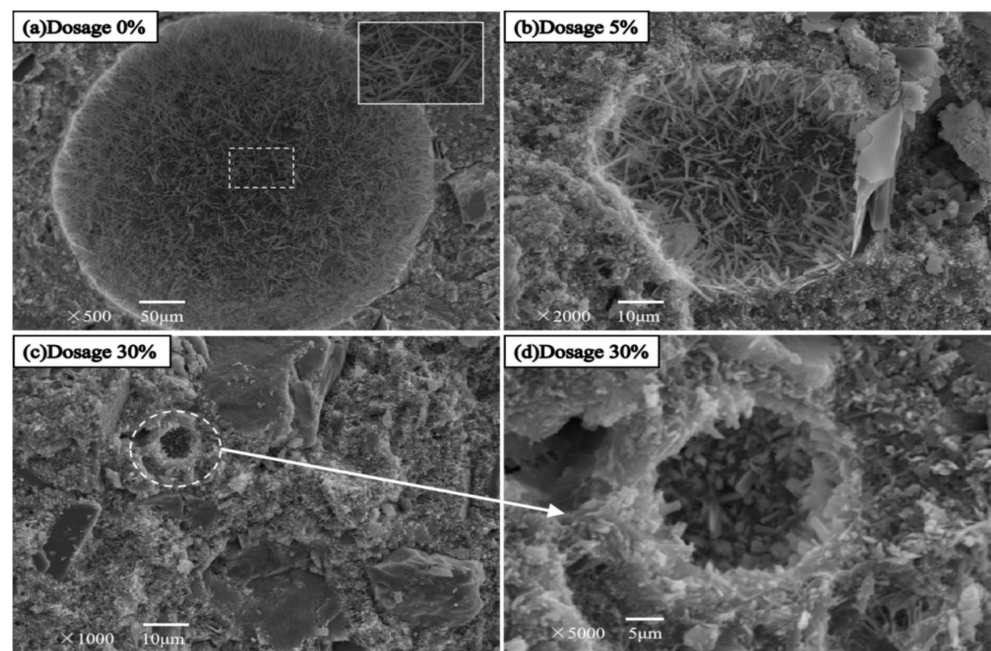


Figure 6. SEM images of MOC in air ((a–d) in the figure represent the incorporation amount of lithium slag as 0%, 5%, 30% and 30% respectively).

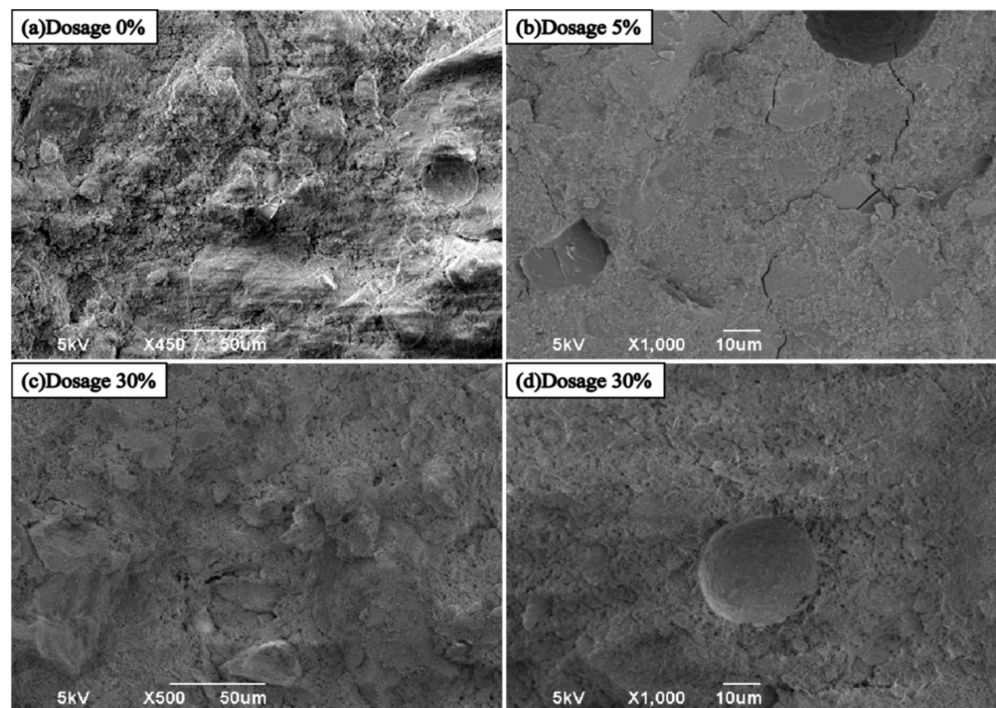


Figure 7. SEM images of MOC after water immersion for 7 days ((a–d) in the figure represent the incorporation amount of lithium slag as 0%, 5%, 30% and 30% respectively).

4. Conclusions

Extracting lithium from spodumene will produce lithium slag. The utilization of lithium slag by other industries is difficult due to the presence of sulfate ions. The stacking of lithium slag not only occupies farmland but also causes serious environmental pollution. Thus, utilizing the high strength, good workability, and other performance characteristics of MOC to comprehensively use lithium slag and study the performance of composite materials is of positive significance for the preparation of lithium extraction from ore and

the treatment of solid waste as a by-product solid waste in the industry. Adding lithium slag to the MOC system showed a potential for obtaining building composite materials with a good performance, and the performance parameters also showed large deviations under different doping conditions.

- (1) Lithium slag had a retarding effect on the MOC system, where the compressive strength of MOC decreased with the increasing content of lithium slag.
- (2) Although lithium slag could not significantly improve the water resistance of the MOC system, compared with MOC without lithium slag, the water resistance of MOC with lithium slag still showed better performance at different ages. When the lithium slag content was 20 wt%, the softening coefficient reached its peak value for each age.
- (3) Adding lithium slag into the MOC system did not change the phase composition of the system, and the addition of lithium slag could inhibit the hydrolysis of the 518 phases in the MOC system, thus improving the water resistance of MOC.
- (4) Lithium slag could significantly change the microstructure of the MOC system. The addition of lithium slag resulted in needle-like crystals in the air-cured MOC sample, which gradually changed into columnar or blocky structures, while the porosity of the MOC sample after soaking in water increased, the structure was loose, and cracks appeared on the surface.

Author Contributions: All authors discussed and agreed upon the idea and made scientific contributions: writing—original draft preparation, S.L. (Shitong Li) and W.Q.; experiment designing, Q.H.; experiment performing, S.L. (Siru Liu); data analysis, Y.D. and W.Z.; writing—review and editing, Q.H. and W.Z. All authors have read and agreed to the published version of the manuscript.

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