

Article

Chemical Mechanisms of Cement Stabilization and Durability Enhancement in High-Content Modified Phosphogypsum

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Abstract: Phosphogypsum, a byproduct of phosphate fertilizer production, accumulates in large quantities annually, posing significant environmental challenges due to harmful components such as fluorine, heavy metals, and acidic salts. To mitigate these issues, phosphogypsum is often combined with cement and single modifiers such as sodium silicate, hydrated lime, and defluorinating agents for use in pavement applications. However, concerns about the durability of unmodified or singly modified high-content phosphogypsum have hindered its widespread adoption. To address this issue, this study explored the use of sodium silicate, hydrated lime, and defluorinating agents as composite modifiers to enhance the durability of cement-stabilized phosphogypsum. The mechanisms of modification by individual and composite additives were investigated using X-ray diffraction (XRD) and scanning electron microscopy (SEM). Durability was evaluated through three-point bending fatigue, freeze–thaw, and drying shrinkage tests on both unmodified and modified cement-stabilized phosphogypsum. The results demonstrated that the composite modification of sodium silicate, hydrated lime, and defluorinating agents significantly improved the material’s density, strength, and stability by enhancing hydration products and stabilizing fluoride ions. The modified material exhibited superior fatigue and freeze–thaw resistance, with reduced mass loss and slower strength decline compared to unmodified phosphogypsum. Additionally, the modified material showed improved drying shrinkage performance due to enhanced hydration. However, caution is recommended when using these materials in regions with high moisture content and significant temperature fluctuations.

Keywords: cement-stabilized high-content modified phosphogypsum; durability; modifier; modification mechanism



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1. Introduction

In recent years, with the increasing emphasis on the sustainable use of resources, the recycling technology of waste materials has gained growing attention in the field of road engineering [1,2]. Phosphogypsum, a byproduct of phosphate fertilizer production, is generated in large quantities annually [3,4]. With the rising global demand for phosphate fertilizers, managing the discharge of phosphogypsum has become a pressing environmental concern. This byproduct contains harmful substances such as fluorine, heavy metals, and acidic salts, which contribute to significant environmental pollution and pose risks to ecosystems [5,6]. As a result, the effective utilization of phosphogypsum has become a key

area of focus in environmental research and resource recycling. Once regarded as a waste product, its potential is now being increasingly recognized in light of stricter environmental regulations and the growing push for sustainable development [7,8]. Research has shown that converting phosphogypsum into construction materials can mitigate its environmental impact while providing a cost-effective, high-performance alternative for pavement construction. In line with the global shift toward sustainable building practices, the use of phosphogypsum in road infrastructure is gaining attention as a viable and eco-friendly solution [9–12].

To promote the resource utilization of phosphogypsum, cement stabilization technology has gained significant attention as an effective treatment method [13–15]. This technology enhances the mechanical properties of phosphogypsum by mixing it with cement. Studies have demonstrated that cement-stabilized phosphogypsum exhibits strong compressive and flexural strength, making it suitable for various engineering applications, such as foundation filling and pavement construction [16,17]. Ou et al. [18] conducted an in-depth analysis of the unconfined compressive strength of cement-stabilized phosphogypsum, revealing that its strength exceeded 3 MPa after just 7 days of curing. In contrast to conventional semi-rigid base materials, cement-stabilized phosphogypsum exhibited no shrinkage strain; instead, it showed an increase in expansion strain with higher phosphogypsum content. Similarly, Wu et al. [19] investigated the mechanical properties of cement-stabilized roadbase materials incorporating both phosphogypsum and recycled concrete aggregate. Their results confirmed that cement-stabilized phosphogypsum exhibited excellent mechanical properties. Specifically, the unconfined compressive strength of cement-stabilized phosphogypsum containing 20% phosphogypsum reached 5.34 MPa after 7 days of curing. Peng et al. [20] examined the influence of particle fineness and pH on the mechanical properties of cement-stabilized phosphogypsum. Their tests on unconfined compressive strength and California bearing ratio values revealed that increasing the pH and reducing the particle fineness of phosphogypsum both contributed to enhanced mechanical properties. They found that when the pH exceeded 5, or the fineness was below 20%, the mechanical properties of cement-stabilized phosphogypsum remained stable. Wang et al. [21] compared the mechanical properties of cement-stabilized silty sand with and without additives, using red mud and phosphogypsum as binders. Their results indicated that the composite binder stabilized silty sand exhibited superior mechanical properties at early ages, although the rate of strength development was relatively slow during the curing period. These studies collectively demonstrate that cement-stabilized phosphogypsum prepared through cement stabilization technology possesses favorable mechanical properties. However, concerns about the material's durability in practical applications have limited the widespread use of phosphogypsum in cement stabilization, hindering its efficient reuse.

Compared to other pavement materials, cement-stabilized phosphogypsum exhibits notably inferior durability. This limitation is primarily due to the harmful constituents present in phosphogypsum, such as fluorine and heavy metals, which can migrate under moist conditions, negatively affecting the material's performance [20]. Additionally, the bonding reaction between cement and phosphogypsum may be suboptimal, particularly in high-humidity or freeze–thaw cycling environments, leading to the formation of microcracks that compromise the material's durability. Therefore, addressing these challenges is essential for improving the long-term performance of cement-stabilized phosphogypsum in various applications. To this end, some researchers have proposed enhancing the durability of cement-stabilized phosphogypsum by adjusting the pH value and particle fineness of the phosphogypsum [21]. Furthermore, incorporating modifiers into the material is also considered a promising approach to improving its durability [22].

To overcome these issues, modifiers can be incorporated to enhance the material's performance and eliminate harmful substances [23,24]. Among the various modifiers, sodium silicate is frequently employed as an alkaline activator in cement materials. Research has shown that adding sodium silicate accelerates the hydration reaction, significantly improves strength, enhances workability, and increases chemical resistance, thereby greatly enhancing the overall performance of the materials [24,25]. Hydrated lime is commonly used as an enhancer in cement materials because it can increase the pH of the mixture and promote the hydration reactions of certain minerals [26]. In addition, defluorinating agents are also widely used in engineering materials, as they can effectively remove fluoride ions from the materials, thereby reducing the negative impact of fluoride on the strength and durability of the materials. However, current research mainly focuses on single modifiers. The durability of high-content modified phosphogypsum with a single modifier still sometimes fails to meet the required standards for use.

Given this, the present study aims to enhance phosphogypsum by applying a composite modification using three different modifiers: sodium silicate, hydrated lime, and defluorinating agents. And the durability performance of cement-stabilized high-content modified phosphogypsum is evaluated through erosion resistance, freeze–thaw, drying shrinkage, and fatigue performance tests, with the aim of identifying the optimal additive ratios for the modifiers.

2. Materials and Methods

2.1. Materials

2.1.1. Phosphogypsum

The phosphogypsum applied in the present study is a byproduct generated during the production of phosphoric acid in phosphate fertilizer plants, and the pH of the selected phosphogypsum ranges from five to six. Based on the X-ray fluorescence (XRF) and X-ray diffraction (XRD), the chemical composition of the selected materials was tested, and the results are shown in Figure 1. Based on the test results, it can be inferred that the main chemical components of the phosphogypsum used in the study are $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and a small amount of SiO_2 , along with trace elements such as P_2O_5 , Al_2O_3 , Fe_2O_3 , K_2O , TiO_2 , and other impurities.

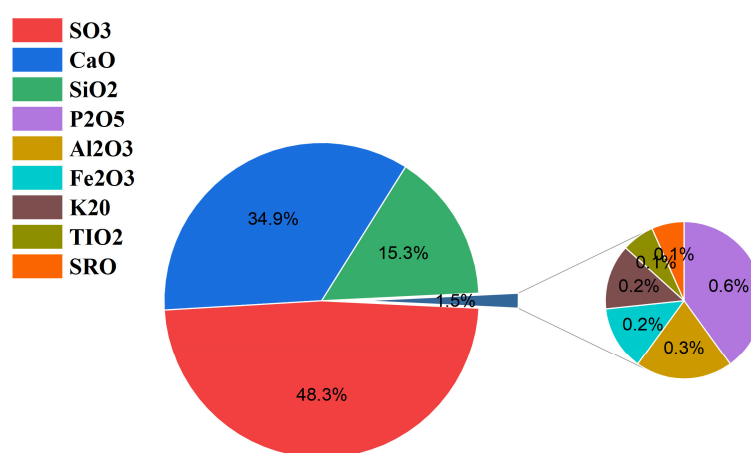


Figure 1. Chemical composition of the selected phosphogypsum.

From a microscopic perspective, the microstructure of phosphogypsum was observed using scanning electron microscopy (SEM), and the results are shown in Figure 2. The original crystal structure of phosphogypsum predominantly exhibits a monoclinic configuration characterized by irregular shapes. Notably, the crystallization forms of phosphogypsum

can vary, appearing as plate-like, columnar, or needle-like structures, influenced by the specific storage environment and formation processes

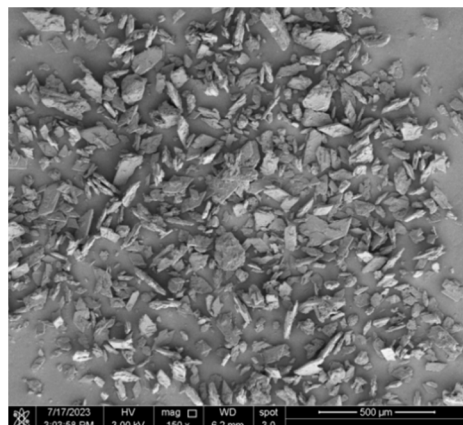


Figure 2. Microstructure of phosphogypsum.

2.1.2. Cement

The cement used in this study is P-I 42.5 Portland cement. It has high strength and durability, along with a moderate setting time and good workability, making it easy to use in concrete mixtures. Its basic properties are shown in Table 1.

Table 1. Basic properties of cement used in this study.

Density (g/cm ^{−3})	Specific Surface Area (m ² ·kg ^{−1})	Soundness	Setting Time (min)		Flexural Strength (MPa)		Compressive Strength (MPa)	
			Initial	Final	3 d	28 d	3 d	28 d
3.26	389	Qualified	207	258	6.7	9.4	35.2	60.4

2.1.3. Modifier

To prepare composite-modified phosphogypsum, three types of modifiers were used in this study, including sodium silicate, hydrated lime, and defluorinating agent.

The sodium silicate used in the study is a white powdery solid, which is commonly known for its properties as a binding agent and its ability to enhance the mechanical strength of materials [27]. It is soluble in water, forming a viscous solution that can aid in the modification of phosphogypsum by improving its stability and durability. Detailed information on the selected sodium silicate is shown in Table 2.

Table 2. Detailed information on the selected sodium silicate.

Chemical Formula	Molecular Weight	Na ₂ O Content	The Ratio of Na ₂ O to SiO ₃ Content	Sulfate	Impurity Content (%)		
					Chloride	Pb	Al ₂ O ₃ + Fe ₂ O ₃
Na ₂ SiO ₃ ·9H ₂ O	284.22	19.3	1.03	0.01	0.01	0.001	0.05

The hydrated lime used in the study is a soft white granular material known for its high reactivity and ability to improve the properties of construction materials [28]. It plays a crucial role in various applications, including soil stabilization and the modification of phosphogypsum. Hydrated lime reacts with moisture to form calcium hydroxide, which enhances the binding properties and increases the durability of the material. Its fine particle size allows for easy dispersion in mixtures, promoting uniformity and effectiveness in improving the overall performance of the final product. Additionally, hydrated lime is valued for its environmental benefits, as it can help reduce the carbon footprint of construction processes. Detailed information on the selected hydrated lime is shown in Table 3.

Table 3. Detailed information on the selected hydrated lime.

Chemical Formula	Molecular Weight	Ca(OH) ₂ Content	Chloride	Sulfate	Impurity Content (%)			Al ₂ O ₃ + Fe ₂ O ₃
					Fe	MgO	Pb	
Ca(OH) ₂	74.09	95.00%	0.01	0.2	0.01	0.5	0.002	0.25

The defluorinating agent selected for the study is the WDCF professional defluorinating agent, which is specifically designed to effectively remove fluoride ions from various materials. This agent is known for its high efficiency and reliability in reducing fluoride content, making it suitable for applications in construction and environmental remediation. The WDCF agent works through a chemical process that binds fluoride ions, facilitating their removal and ensuring compliance with environmental standards. Detailed information on the selected defluorinating agent is shown in Table 4.

Table 4. Detailed information on the selected defluorinating agent.

Morphology	Color	pH	Effective Content (%)
liquid	colorless	5.6	99

2.2. Preparation Procedure of Cement-Stabilized High-Content Modified Phosphogypsum

To investigate the effects of various modifiers on the durability of cement-stabilized phosphogypsum, both singly modified cement-stabilized phosphogypsum and composite-modified cement-stabilized phosphogypsum were prepared in accordance with the previous study [29].

For singly modified cement-stabilized phosphogypsum, sodium silicate, hydrated lime, and defluorinating agents were first mixed with water for 5 min to prepare the corresponding modifiers. Subsequently, the modifiers were evenly sprayed onto the pre-prepared phosphogypsum and sealed in plastic bags to allow for immersion for 1 day, resulting in modified phosphogypsum. Following this, water, cement, and modified phosphogypsum were mixed evenly for 5 min to prepare singly modified cement-stabilized phosphogypsum. Finally, after mixing, the mixture was subjected to static pressure molding using the digital hydraulic pressure testing machine (WE-1000B) within 1 h. In the singly modified mixture, the contents of sodium silicate, hydrated lime, and defluorinating agent were 1%, 3%, and 1%, respectively. For composite-modified cement-stabilized phosphogypsum, three modifiers were first mixed with water to prepare the composite modifier. The remaining preparation process is consistent with that of the singly modified mixture. In composite-modified cement-stabilized phosphogypsum, the contents of sodium silicate, hydrated lime, and defluorinating agent were 1%, 1.5%, and 1%, respectively. And regardless of the type of modifier used, the mass ratio of cement to phosphogypsum in cement-stabilized phosphogypsum is always 1:9. Table 5 presents the detailed preparation plan and the corresponding ID of the specimen. The preparation process of cement-stabilized modified phosphogypsum is shown in Figure 3.

Table 5. Detailed preparation plan and ID of the specimen.

Sodium Silicate	Modifier Content (%)		IDs
	Hydrated Lime	Defluorinating Agents	
0	0	0	P
1	0	0	S-1
0	3	0	S-2
0	0	1	S-3
1	1.5	1	C-1

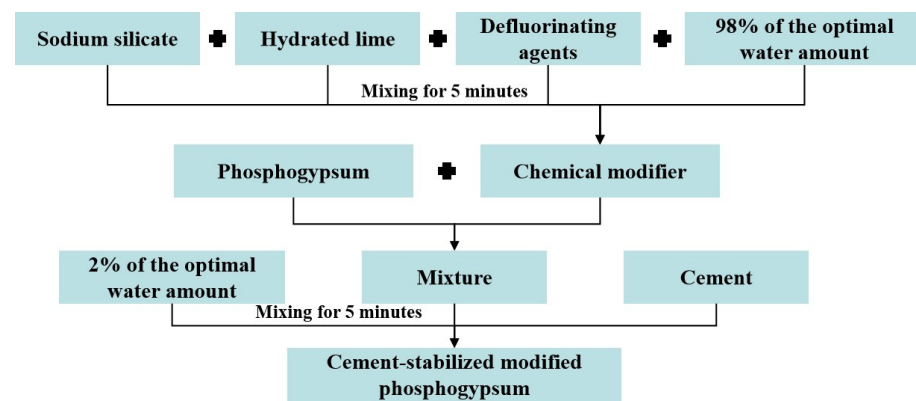


Figure 3. Preparation process of cement-stabilized modified phosphogypsum.

2.3. Methods

In this study, XRF and SEM tests were conducted to analyze the modification mechanism of the modifiers. In addition, three-point bending fatigue tests, freeze–thaw tests, and drying shrinkage tests were conducted to evaluate the durability of modified cement-stabilized phosphogypsum. The specific experimental procedures are as follows.

2.3.1. XRF Test

The XRF (X-ray fluorescence) test was conducted in this study to thoroughly investigate the compositional changes in cement-stabilized phosphogypsum before and after modification. The XRF technique is a powerful and non-destructive method for analyzing the elemental composition of materials, and it was chosen for its ability to provide precise and rapid results.

Initially, the specimens of cement-stabilized phosphogypsum were carefully prepared to ensure that the surfaces were smooth and uniform. This involved meticulous grinding or pressing of the samples to remove any irregularities and achieve the desired surface finish. Once the samples were prepared, the XRF instrument was configured and calibrated according to the specific characteristics of the cement-stabilized phosphogypsum. Calibration procedures were carried out using certified reference materials to ensure the instrument's accuracy and reliability. The samples were then placed on the analysis platform, where they were exposed to a controlled X-ray beam. Throughout the testing process, X-rays excited the elements within the samples, resulting in the emission of characteristic fluorescence. The instrument diligently collected these signals over a predetermined period, and advanced software was employed to analyze the data, allowing for precise determination of the elemental composition and concentrations in the cement-stabilized phosphogypsum.

2.3.2. XRD Test

In this study, X-ray diffraction (XRD) was utilized to analyze the mineralogical composition of cement-stabilized phosphogypsum both before and after modification. The testing process involved several steps, beginning with meticulous sample preparation and concluding with data analysis and interpretation. The first step involved preparing the cement-stabilized phosphogypsum samples, which were initially dried at 60 °C for 24 h to remove any moisture. The dried samples were then ground into a fine powder to ensure uniformity in particle size. During the XRD analysis, diffraction patterns were collected and analyzed to identify the mineral phases present in the samples. Finally, the data were processed using the accompanying analysis software, and quantitative phase analysis was conducted to determine the relative proportions of each mineral phase.

2.3.3. SEM Test

The SEM tests conducted in this study aimed to investigate the microstructural changes in cement-stabilized phosphogypsum before and after modification. To facilitate this, the prepared specimens underwent a meticulous process of cutting, drying, and coating with a conductive layer, ensuring optimal conditions for high-quality imaging during electron beam scanning. The instrument parameters were carefully adjusted to scan the specimen surfaces with an electron beam, resulting in high-resolution secondary and backscattered electron images. These images provided valuable insights into the morphological characteristics and microstructure of cement-stabilized phosphogypsum, significantly enhancing our understanding of its performance in engineering applications.

2.3.4. Three-Point Bending Fatigue Test

The fatigue performance of different cement-stabilized modified phosphogypsum specimens was tested via three-point bending fatigue test in reference to the “Test Methods of Materials Stabilized with Inorganic Binders for Highway Engineering” (JTG E51-2009) [30]. The specimen dimensions used in three-point bending fatigue test were 50 mm × 50 mm × 200 mm, with six parallel specimens in each group. All tested specimens were cured for 90 days prior to testing. Before the fatigue tests, the applied load was determined based on the flexural strength tests, and two different stress levels were selected (0.6 and 0.7). Prior to applying the main load, a pre-compression load at 0.2 stress level was applied for 2 min. During the formal testing, a continuous sinusoidal load was applied at a frequency of 10 Hz. Throughout the test, the number of load repetitions on the specimen was recorded, indicating the fatigue life.

2.3.5. Freeze–Thaw Test

The freeze–thaw tests on various cement-stabilized modified phosphogypsum specimens were conducted in accordance with the “Test Methods of Materials Stabilized with Inorganic Binders for Highway Engineering” (JTG E51-2009). For each group of freeze–thaw tests, 18 cylindrical specimens with dimensions of $\phi 150$ mm × 150 mm were prepared. Of these, 9 specimens were subjected to freeze–thaw cycles, while the remaining 9 served as control specimens, which were not exposed to freeze–thaw conditions. The specimens intended for freeze–thaw testing underwent standard curing for 28 days. On the final day of curing, they were immersed in water for treatment. Following immersion, the unconfined compressive strength of the specimens was measured. The specimens in the freeze–thaw test group were numbered and placed in a low-temperature chamber, with the temperature set to -18 °C. They underwent freezing for 16 h, maintaining a 2 cm gap between each specimen. After the freeze–thaw cycle, the specimens were transferred to a constant temperature water bath at 20 °C for thawing, which lasted 8 h. Once thawing was complete, the specimens were dried. After completing the specified number of freeze–thaw cycles, the unconfined compressive strength of the specimens was re-measured. To assess the water stability of the specimens, the mass change rate and the breaking down ratio (BDR) were calculated using Equations (1) and (2) [30].

$$BDR = \frac{R_{DC}}{R_c} \times 100 \quad (1)$$

$$W_n = \frac{m_0 - m_n}{m_0} \times 100 \quad (2)$$

where BDR represents the compressive strength loss of the specimens after n freeze–thaw cycles, %; R_{DC} represents the unconfined compressive strength of the specimens after n freeze–thaw cycles, MPa; R_c represents the compressive strength of the control specimens,

MPa; W_n represents the mass loss rate of the specimens, %; m_n represents the mass of the specimens after n freeze–thaw cycles, g; and m_0 represents the mass of the control specimen, g.

2.3.6. Drying Shrinkage Test

The drying shrinkage performance of different cement-stabilized modified phosphogypsum specimens was tested in accordance with the “Test Methods of Materials Stabilized with Inorganic Binders for Highway Engineering” (JTG E51-2009). The drying shrinkage test was conducted using a shrinkage gauge and a controlled chamber. The relative humidity of the drying shrinkage chamber was maintained at 60%, with a temperature of $20\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$. The specimen dimensions were $50\text{ mm} \times 50\text{ mm} \times 200\text{ mm}$, with six specimens prepared for each type of cement-stabilized phosphogypsum. Three specimens were used to measure drying shrinkage deformation, while the remaining three specimens served as standard specimens to measure the water loss rate. The specimens were cured under standard conditions for 7 days. After curing, the initial length and mass of each specimen were measured using a caliper and a balance. A dial gauge was then fixed at both ends of the specimens, which were placed in the drying shrinkage chamber. Throughout the test period, changes in the shrinkage and mass of the specimens were recorded. Based on the test results, several key indicators were calculated [25].

$$w_i = (m_i - m_{i+1}) / m_p \quad (3)$$

$$\delta_i = (\sum_{j=1}^4 X_{i,j} - \sum_{j=1}^4 X_{i+1,j}) / 2 \quad (4)$$

$$\varepsilon_i = \delta_i / l \quad (5)$$

$$\alpha_{di} = \varepsilon_i / w_i \quad (6)$$

where w_i represents the water loss rate of the i -th test, %; δ_i represents the drying shrinkage of the i -th test, mm; ε_i represents drying strain of the i -th test, %; α_{di} represents the drying shrinkage coefficient of the i -th test, %; l represents the length of the specimen, mm; m_p represents the mass of the specimen, g; and $X_{i,j}$ represents the reading of the j -th dial gauge after the i -th test, mm.

3. Results

3.1. The Modification Mechanism of Different Modifiers on Cement-Stabilized Phosphogypsum

The main objective of this study is to extensively utilize the chemical byproduct phosphogypsum in an environmentally friendly manner. However, due to its inherent low strength, weak acidity, and the presence of trace harmful chemical elements, the content of phosphogypsum in cement-stabilized phosphogypsum is not high. To solve this, the present study aims to enhance the performance and eliminate harmful substances of cement-stabilized phosphogypsum via the addition of different modifiers. As such, the modification mechanism of modifiers was first analyzed.

3.1.1. Modification Mechanism of Sodium Silicate

Figure 4 illustrates the chemical composition of unmodified and sodium silicate-modified cement-stabilized phosphogypsum.

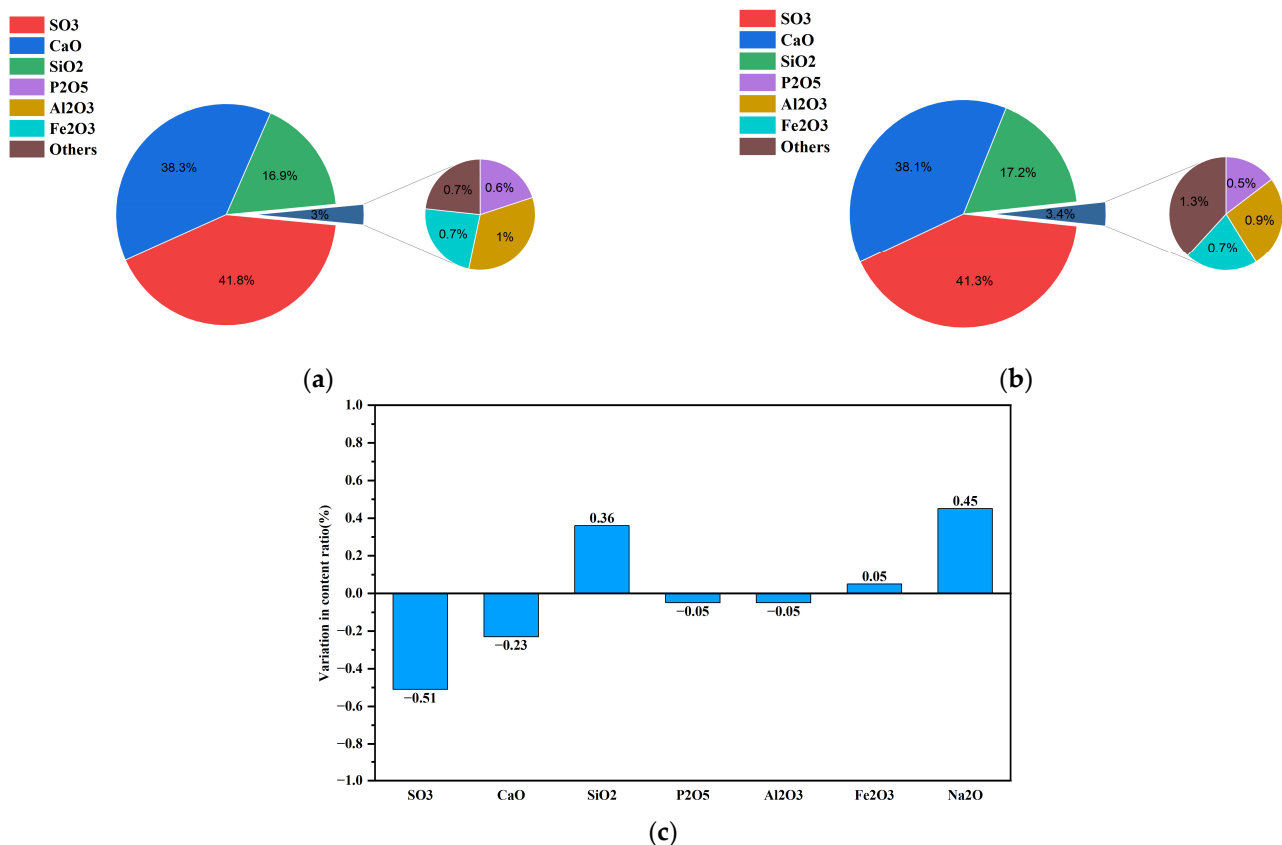


Figure 4. XRF test results of unmodified and sodium silicate-modified cement-stabilized phosphogypsum: (a) unmodified; (b) sodium silicate-modified; (c) variation in chemical composition compared with unmodified cement-stabilized phosphogypsum.

It is evident that the addition of sodium silicate, which contains silicon (Si), will directly increase the silicon content in the cement-stabilized phosphogypsum, resulting in an increase of 0.36% in SiO₂ compared to the original cement-stabilized phosphogypsum. At the same time, the addition of sodium silicate will also affect the content of other elements in the cement-stabilized phosphogypsum. For instance, the reaction between sodium silicate and calcium oxide in the cement will consume some of the calcium (Ca), thereby reducing the Ca content in the material. Additionally, the introduction of sodium silicate will alter the chemical environment of the material, affecting the dissolution and precipitation of other elements such as sulfur (S) and phosphorus (P), which in turn influences their content in the material.

Then, the XRD spectra of cement-stabilized phosphogypsum were modified with sodium silicate after 7 days of curing, and the original materials were collected. The results are shown in Figure 5.

Based on the intensity of the same diffraction peaks, it can be observed that after modification with sodium silicate, the diffraction peak intensity of CaSO₄·2H₂O in the material is significantly lower than that of the unmodified material, while the diffraction peak intensity of the hydration product ettringite (AFt) is higher than that of the unmodified material. This indicates that the addition of sodium silicate can enhance the hydration reaction of the cement. Although the hydration product C-S-H gel cannot be effectively detected by XRD due to its low crystallinity, the changes in the diffraction peaks of CaSO₄·2H₂O also suggest that more CaSO₄·2H₂O participates in the hydration reaction compared to the unmodified material. This implies that sodium silicate can promote the hydration reaction of

cement-stabilized phosphogypsum, resulting in the generation of more hydration products, thereby enhancing the bond strength between cement and phosphogypsum.

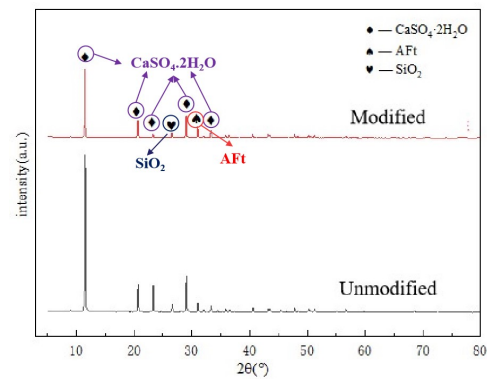


Figure 5. XRD spectra of unmodified and sodium silicate-modified cement-stabilized modified phosphogypsum.

Figure 6 presents the SEM images of sodium silicate-modified cement-stabilized modified phosphogypsum.

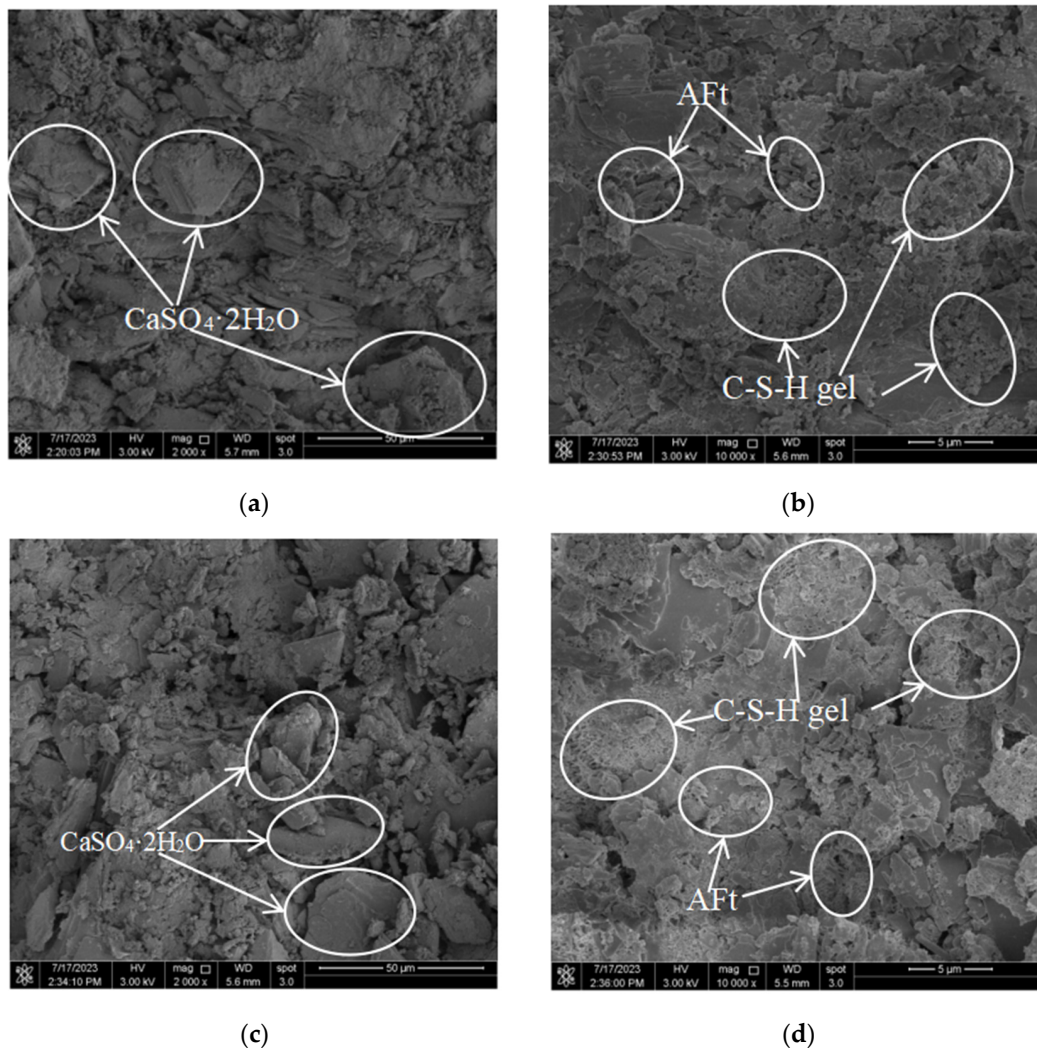


Figure 6. SEM images of unmodified and sodium silicate-modified cement-stabilized modified phosphogypsum: (a) unmodified materials at 2000× magnification; (b) unmodified materials magnified at 10,000× magnification; (c) modified materials at 2000× magnification; (d) modified materials at 10,000× magnification.

Using SEM, it can be observed that the addition of sodium silicate triggers a chemical reaction with water and carbon dioxide, resulting in the formation of sodium carbonate and silicate gel. This gel is subsequently reacted with calcium hydroxide to generate calcium silicate hydrate, a novel hardened product that significantly enhances the strength and erosion resistance of cement-stabilized phosphogypsum materials. The newly formed calcium silicate hydrate is seen in the form of fine flakes or granules intricately distributed throughout the material's microstructure. Moreover, the incorporation of sodium silicate promotes the development of a more uniform and dense microstructure within the cement-stabilized phosphogypsum, thereby improving its durability and overall performance [31].

3.1.2. Modification Mechanism of Hydrated Lime

Figure 7 illustrates the chemical composition of hydrated lime-modified cement-stabilized phosphogypsum.

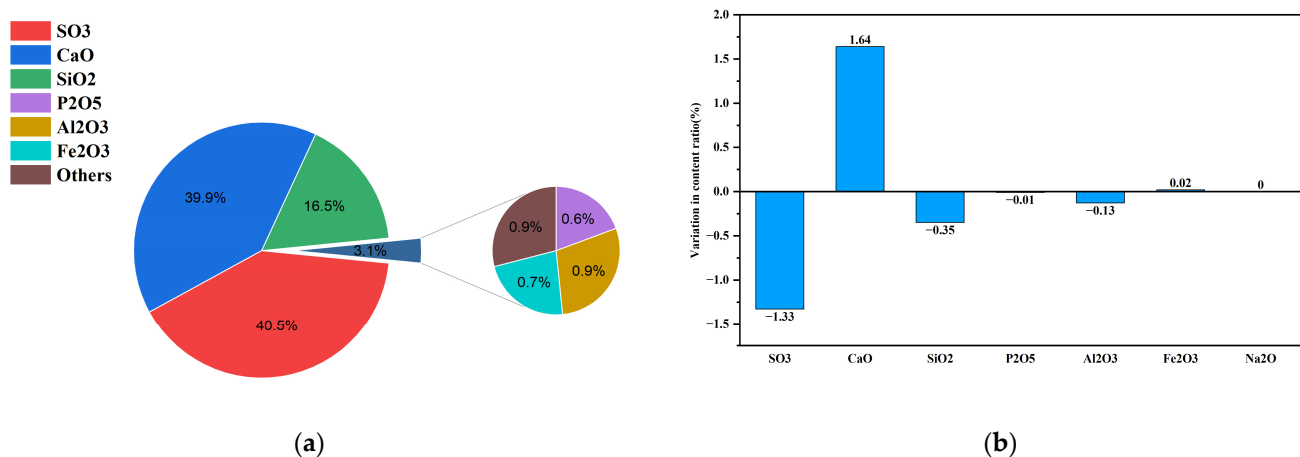


Figure 7. XRF test results of hydrated lime-modified cement-stabilized phosphogypsum: (a) chemical composition; (b) variation in chemical composition compared with unmodified cement-stabilized phosphogypsum.

It can be observed from the figure the calcium hydroxide in hydrated lime will increase the calcium content in the modified cement-stabilized phosphogypsum. These additional Ca elements can react with phosphate ions (PO_4^{3-}) to form the sparingly soluble $\text{Ca}_3(\text{PO}_4)_2$, thereby enhancing the strength and stability of the material. In addition, the addition of hydrated lime will also lead to the precipitation of sulfur (S), reducing the proportion of SO₃ content from the original 41.8% to 40.5%. The impact of hydrated lime on other elements is minimal, and the proportions of other metal oxides remain almost unchanged. Figure 8 presents the XRD spectra of cement-stabilized phosphogypsum modified with hydrated lime after 7 days of curing and the original materials.

As illustrated in the figure, the intensity of the $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ diffraction peak decreases following the addition of hydrated lime, while the diffraction peak of Aft shows an increase. This observation suggests that hydrated lime not only promotes the cement hydration reaction, leading to the formation of C-S-H gel but also directly participates in the hydration process to enhance the content of the hydration product Aft. Concurrently, the amount of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ diminishes progressively as the reaction advances.

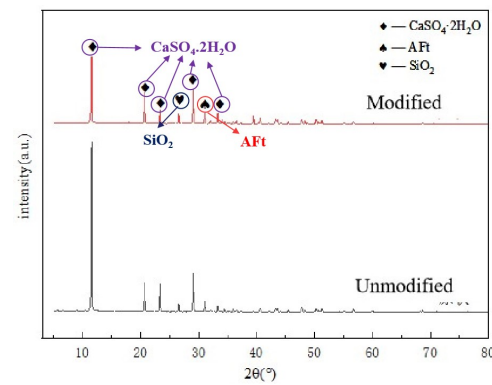


Figure 8. XRD spectra of unmodified and hydrated lime-modified cement-stabilized modified phosphogypsum.

Figure 9 presents the SEM images of hydrated lime-modified cement-stabilized modified phosphogypsum.

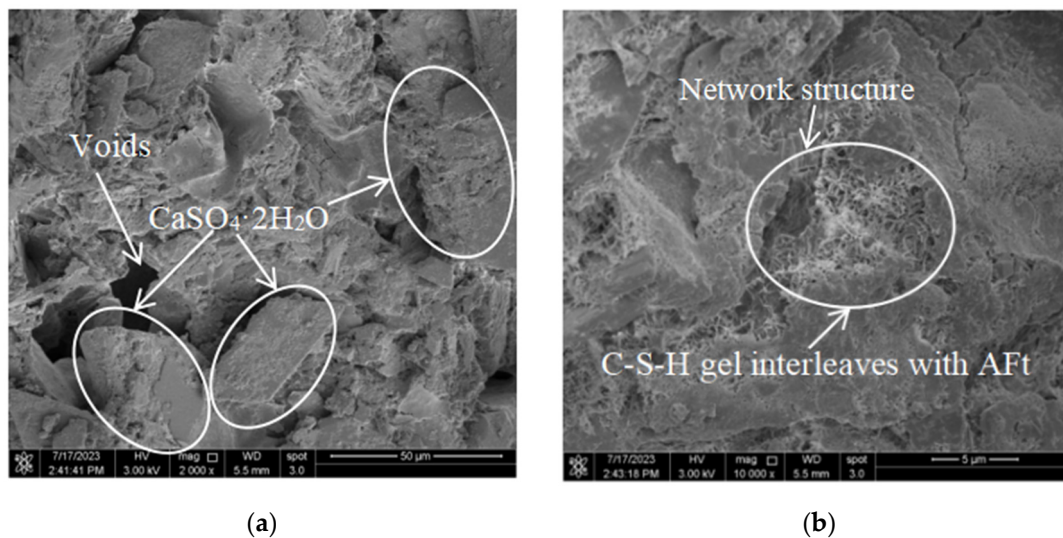


Figure 9. SEM images of hydrated lime-modified cement-stabilized modified phosphogypsum: (a) magnified 2000 times; (b) magnified 10,000 times.

The SEM images of cement-stabilized modified phosphogypsum reveal an interlocking gel-AFt structure, indicating that the incorporation of hydrated lime has significantly altered the material's microstructure. This interlocking network likely results from the interaction between hydrated lime and the components of phosphogypsum, forming a more intricate and resilient structure. Such a network not only enhances the bonding strength of the material but also improves its overall compressive and tensile properties. Furthermore, this interlocking configuration may be crucial in enhancing durability and resistance to erosion, as it facilitates better stress distribution and reduces localized stress concentrations, ultimately contributing to a longer service life for the material.

3.1.3. Modification Mechanism of Defluorinating Agent

Figure 10 illustrates the chemical composition of defluorinating agent-modified cement-stabilized phosphogypsum.

Compared to the chemical composition of the unmodified cement-stabilized phosphogypsum and the materials modified with the other two agents, the defluorinating agent modification resulted in the highest proportion of SO_3 at 42.1%, an increase of 0.31% relative to the unmodified material. It also led to the highest proportion of Al_2O_3 at 1.2%,

an increase of 0.22%. Conversely, the proportions of CaO and SiO₂ decreased by 0.18% and 0.32%, respectively, with minimal impact on the proportions of Fe₂O₃ and P₂O₅. Figure 11 presents the XRD spectra of the cement-stabilized phosphogypsum modified with the defluorinating agent after 7 days of curing alongside the spectra of the unmodified material.

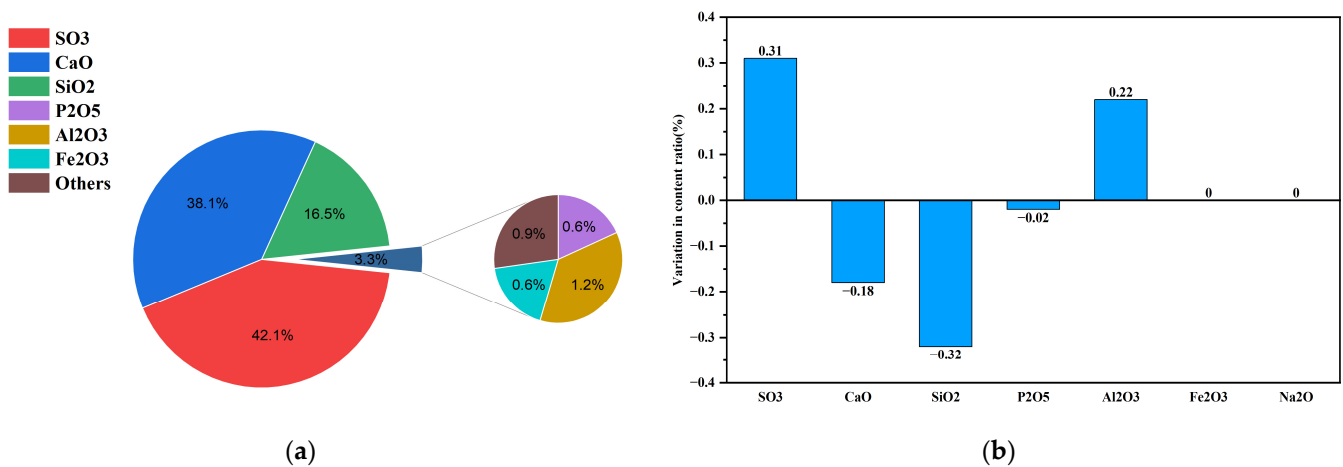


Figure 10. XRF test results of defluorinating agent-modified cement-stabilized phosphogypsum: (a) chemical composition; (b) variation in chemical composition compared with unmodified cement-stabilized phosphogypsum.

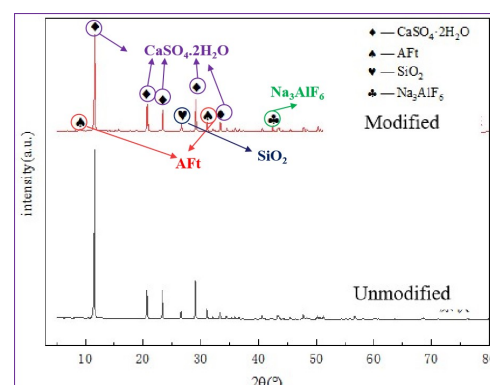


Figure 11. XRD spectra of unmodified and defluorinating agent-modified cement-stabilized modified phosphogypsum.

The figure clearly highlights the key difference between the defluorinating agent-modified cement-stabilized phosphogypsum and the unmodified material: the presence of Na₃AlF₆ in the modified phosphogypsum. This observation further suggests that the fluoride removal agent effectively precipitates free fluoride ions, forming complexes that immobilize the soluble fluoride ions within the material, thereby mitigating their potential environmental impact. Additionally, the XRD diffraction peaks indicate that the defluorinating agent exerts a much smaller effect on cement hydration compared to sodium silicate and hydrated lime. The intensity of the CaSO₄·2H₂O diffraction peaks is also higher than those of the other two agents, indicating that the amount of CaSO₄·2H₂O in the modified material is greater than that in the sodium silicate and hydrated lime-modified materials. This suggests that among the three agents—the defluorinating agent, sodium silicate, and hydrated lime—when added individually, the defluorinating agent leads to the least amount of hydration products generated in the modified material.

Figure 12 presents the SEM images of defluorinating agent-modified cement-stabilized phosphogypsum. The microstructural images at various magnifications reveal a notable

observation: the microvoids within the cement-stabilized phosphogypsum are filled with a viscous substance, a novel compound formed through the interaction between the defluorinating agent and fluoride ions. This newly formed fluoride compound effectively occupies the microvoids, significantly enhancing the material's density and stability. By filling these voids, it contributes to a more cohesive structure, ultimately improving the material's overall performance and durability.

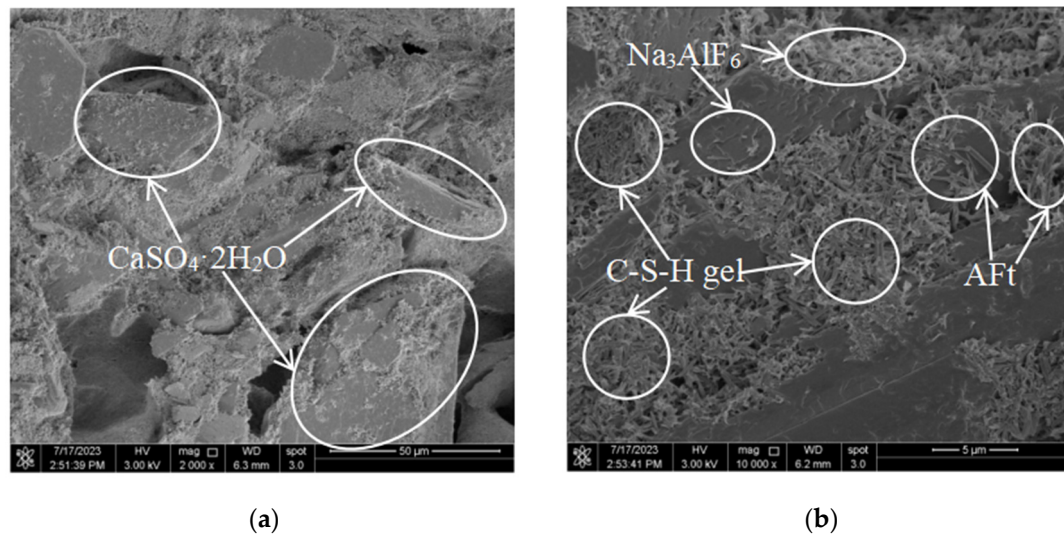


Figure 12. SEM images of defluorinating agent-modified cement-stabilized modified phosphogypsum: (a) magnified 2000 times; (b) magnified 10,000 times.

3.1.4. Modification Mechanism of Composite Modifier

Figure 13 illustrates the chemical composition of composite-modified cement-stabilized phosphogypsum. As can be seen from the figure, compared to the chemical composition after the addition of three single modifiers, the composite-modified cement-stabilized phosphogypsum has the lowest SiO_2 content, while the SO_3 content ratio is only lower than that when the defluorinating agent is added alone. The difference in the CaO content ratio is relatively small.

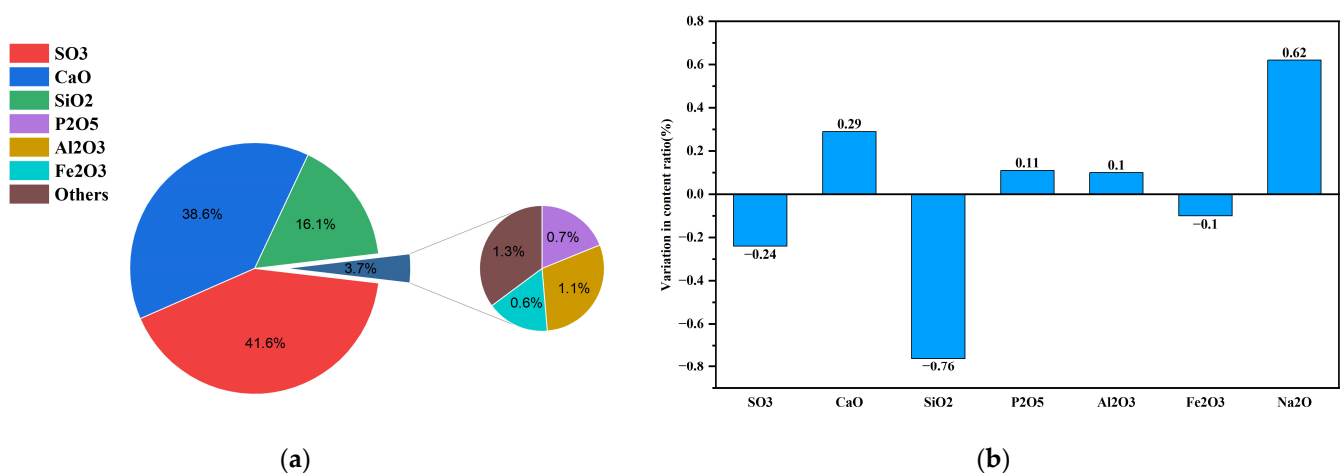


Figure 13. XRF test results of composite-modified cement-stabilized phosphogypsum: (a) chemical composition; (b) variation in chemical composition compared with unmodified cement-stabilized phosphogypsum.

Figure 14 presents the XRD spectra of composite-modified cement-stabilized phosphogypsum after 7 days of curing and the unmodified materials. It can be concluded from the

figure that the main products of the composite-modified material are $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, SiO_2 , AFt, and Na_3AlF_6 . The composite modification combines the characteristics of sodium silicate, hydrated lime, and defluorinating agent, which not only promotes the participation of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration reaction to generate more C-S-H gel and hydration products such as AFt but also effectively fixes the free soluble fluoride ions in the cement-stabilized phosphogypsum.

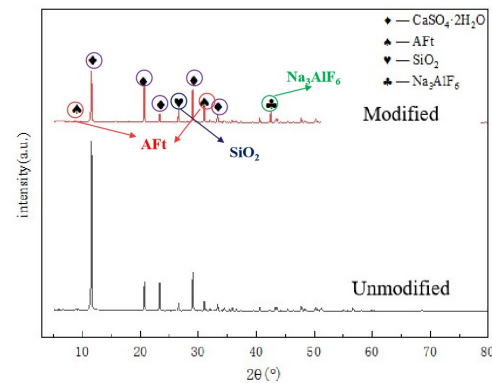


Figure 14. XRD spectra of composite-modified cement-stabilized modified phosphogypsum.

Figure 15 presents the SEM images of composite-modified cement-stabilized modified phosphogypsum.

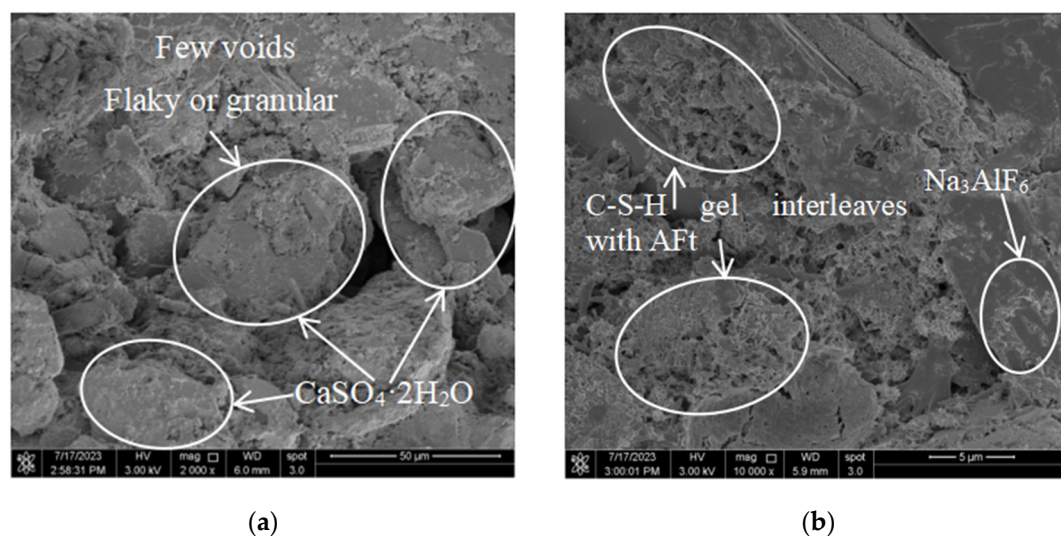


Figure 15. SEM images of composite-modified cement-stabilized modified phosphogypsum: (a) magnified 2000 times; (b) magnified 10,000 times.

As illustrated in the figure, the individual addition of sodium silicate, hydrated lime, or defluorinating agents can enhance the microstructure of the material; however, the extent and scope of improvement are limited. In contrast, the combined use of these three modifiers results in the simultaneous enhancement of multiple microstructural parameters, including density, strength, and stability. Specifically, the reaction products formed from sodium silicate, hydrated lime, and the defluorinating agent—such as C-S-H gel, AFt, and fluorides—effectively fill the microvoids within the material, thereby increasing its density. Furthermore, these reaction products can chemically interact to form more stable crystalline structures, which further contribute to the material's improved strength and stability.

3.2. Fatigue Performance of Cement-Stabilized Modified Phosphogypsum

Fatigue failure is one of the primary modes of pavement deterioration, making fatigue performance a critical criterion for assessing a material's ability to withstand repeated traffic loads. This resilience is crucial as it directly affects both the service life and safety of pavement materials. Fatigue testing simulates the effects of real-world traffic loads by applying varying frequencies and amplitudes. This approach not only quantifies the material's fatigue life under different stress intensity ratios but also provides a comprehensive evaluation of its overall fatigue performance, ensuring optimal reliability and durability in pavement applications. In this study, a three-point bending fatigue test was conducted on unmodified, singly modified, and composite-modified cement-stabilized phosphogypsum to investigate its deformation characteristics and fatigue life under varying loading conditions.

Before conducting the three-point bending fatigue test, the flexural strength of the specimens was measured. The flexural strengths of the unmodified, sodium silicate, hydrated lime, defluorinating agent-modified, and composite-modified cement-stabilized phosphogypsum were found to be 1.02 MPa, 1.16 MPa, 1.10 MPa, 1.01 MPa, and 1.23 MPa, respectively. Following a 90-day curing period, the three-point bending fatigue test was performed on the specimens, with stress levels controlled at 0.6 and 0.7. The results are presented in Figure 16.

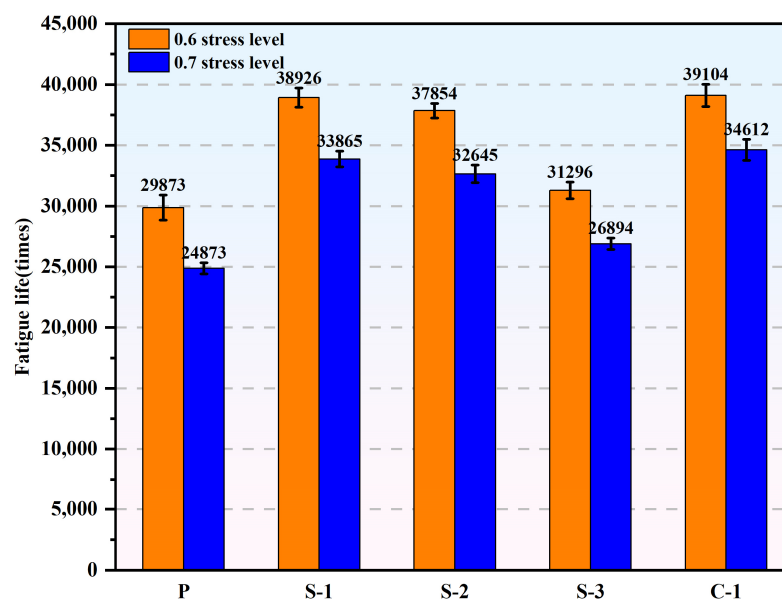


Figure 16. Fatigue life of the specimen.

The figure clearly shows that the fatigue life of cement-stabilized phosphogypsum materials decreases as the stress level increases. Moreover, for the same stress level, the fatigue life of the materials improves to varying degrees after modification. Notably, the composite modifier, consisting of sodium silicate, hydrated lime, and defluorinating agents, provides the most substantial improvement in fatigue performance, surpassing the effects of individual modifications. Among the single additives, sodium silicate proves to be the most effective, followed by hydrated lime, while the defluorinating agent alone has a relatively limited impact.

In conclusion, both sodium silicate and hydrated lime significantly enhance the fatigue resistance of cement-stabilized phosphogypsum. The composite modifier, in particular, not only further amplifies these benefits but also reduces the rate at which fatigue life declines as stress intensity increases. Therefore, utilizing composite-modified cement-stabilized

phosphogypsum in road bases can extend their service life and greatly improve the overall fatigue durability of the road structure, providing a durable solution for long-lasting infrastructure.

3.3. Water Stability of Cement-Stabilized Modified Phosphogypsum

Freeze–thaw cycles pose a significant challenge for road subgrade materials, especially in regions with cold climates. These cycles can severely impact the structural integrity of the subgrade, leading to cracking and deterioration, which ultimately shorten the lifespan and compromise the safety of the pavement. Assessing compressive strength after freeze–thaw cycles is crucial for evaluating the suitability of cement-stabilized phosphogypsum as a base layer material in pavement construction. Based on the results obtained, freeze–thaw tests were conducted only on unmodified and composite-modified cement-stabilized phosphogypsum in this study. Figure 17a presents the relationship between the mass loss rate and strength of the specimens under standard curing for 28 days as the number of freeze–thaw cycles increases.

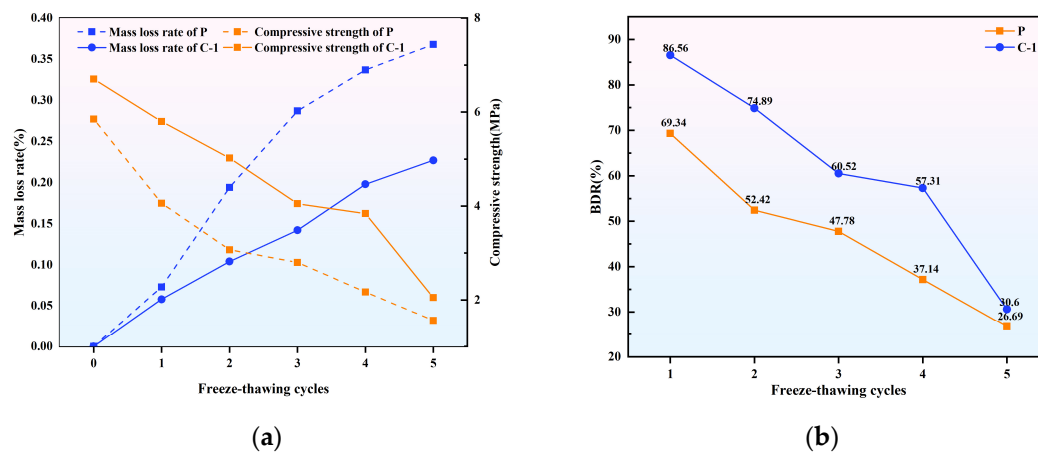


Figure 17. Results of freeze–thawing test: (a) mass loss rate and compressive strength; (b) BDR.

From the figure, it is evident that the mass loss rate of both specimen types gradually increases with the number of freeze–thaw cycles. For the unmodified material, the mass loss rate rises from 0.07% to 0.35% after undergoing one to five freeze–thaw cycles, representing a fourfold increase. In contrast, the modified material exhibits a mass loss increase from 0.06% to 0.23%, reflecting a 2.8-time increase. This suggests that the rate of increase in mass loss for the composite-modified material is 1.2 times lower than that of the unmodified material. During the initial two freeze–thaw cycles, the mass loss of the unmodified material is higher than in the subsequent three cycles, while the mass loss of the modified material remains relatively stable, with minimal fluctuations. After five freeze–thaw cycles, both materials exhibit an increase in mass loss, but the modified material shows a clear deceleration in this rate. This indicates that the composite modifier—comprising sodium silicate, hydrated lime, and a defluorinating agent—effectively enhances the resistance of cement-stabilized phosphogypsum materials to water-induced damage.

When considering compressive strength, the modified specimens show a notable improvement, with both pre- and post-freeze–thaw compressive strengths surpassing those of the unmodified materials. However, as the number of freeze–thaw cycles increases, the compressive strength of both specimen types declines. After five freeze–thaw cycles, the compressive strength of the unmodified material decreases from 5.85 MPa to 1.56 MPa, representing a reduction of 73.3%. In contrast, the compressive strength of the modified material decreases from 6.7 MPa to 2.05 MPa, a decrease of 69.4%. Notably, the rate of change

in compressive strength for the modified material after five freeze–thaw cycles is reduced by 3.9%. This finding suggests that the composite modifier plays a beneficial role in enhancing the post-freeze–thaw compressive strength of cement-stabilized phosphogypsum materials.

Furthermore, the BDR of the unmodified and composite-modified materials after the first~fifth freeze–thaw cycles was determined; results are shown in Figure 17b. As expected, the compressive strength of cement-stabilized phosphogypsum is significantly influenced by the number of freeze–thaw cycles, with both types of materials exhibiting a marked reduction in strength. However, the modified materials demonstrate superior performance compared to the unmodified ones. For instance, the BDR of the unmodified material decreases from 69.34% to 26.69% after one to five freeze–thaw cycles, a reduction of 42.65%. In contrast, the BDR of the modified material decreases from 86.56% to 30.6%, a reduction of 55.96%. The unmodified material shows a rapid strength decline during the first two freeze–thaw cycles, while the modified material experiences a sudden strength drop only after the fifth freeze–thaw cycle. This suggests that the addition of the modifier delays the onset of strength loss due to freeze–thaw cycles in cement-stabilized phosphogypsum materials.

Although the inherent freeze–thaw resistance of cement-stabilized phosphogypsum materials is relatively weak, the composite modifier—comprising sodium silicate, hydrated lime, and a defluorinating agent—significantly enhances the durability and water stability of these materials. These findings are consistent with those of previous studies, which reported that composite modifiers such as sodium silicate and hydrated lime could improve the freeze–thaw resistance of similar materials by promoting the formation of more stable hydration products and filling microvoids in the matrix. Based on these results, however, it is not recommended to use cement-stabilized phosphogypsum materials in regions with significant diurnal temperature variations and high surface moisture content due to their inherent sensitivity to freeze–thaw damage.

3.4. Drying Shrinkage Properties of Cement-Stabilized Modified Phosphogypsum

The drying shrinkage properties of cement-stabilized phosphogypsum impact the durability and fatigue life of the pavement. As such, the drying shrinkage test of the unmodified and composite-modified cement-stabilized phosphogypsum was performed after the standard curing process. Results are shown in Figure 18.

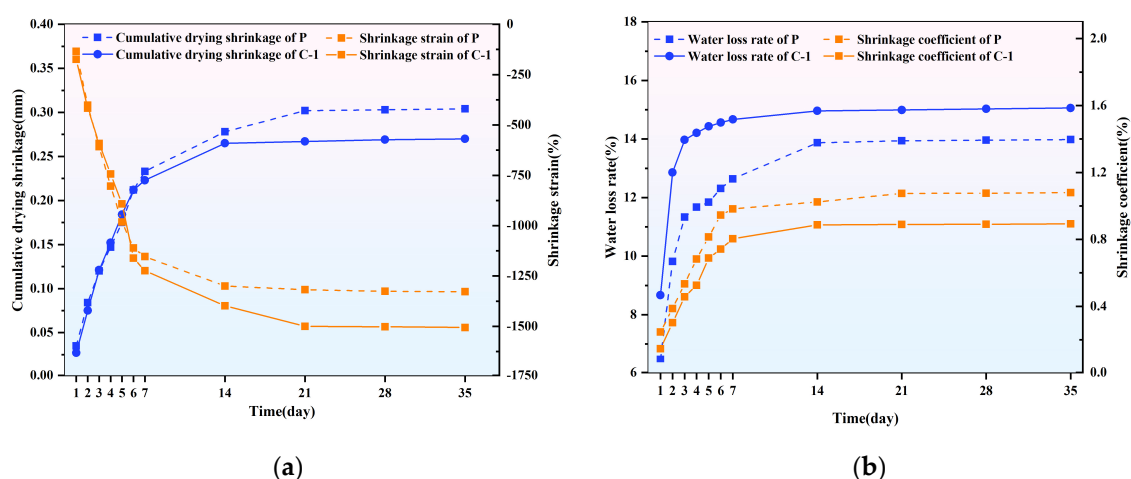


Figure 18. Results of drying shrinkage test: (a) cumulative drying shrinkage and shrinkage strain; (b) water loss rate and shrinkage coefficient.

As shown in the figure, the water loss rate of the modified cement-stabilized phosphogypsum is generally higher than that of the unmodified material. However, its drying shrinkage performance is relatively better. This phenomenon can be attributed to the role

of the modifier in promoting the hydration process in the early stages of cement-stabilized phosphogypsum. The modifier facilitates a more extensive participation of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in the hydration reactions, leading to the release of a significant amount of water molecules. In a drying environment, these water molecules evaporate, causing the modified material to lose more moisture compared to the unmodified material.

However, this hydration process also generates additional hydration products and some gel-like substances, which significantly improve the microstructure of the material. These new phases enhance the density of the cement-stabilized phosphogypsum by filling in the voids and reducing the porosity of the material. As a result, the modified material exhibits reduced drying shrinkage, as the denser structure helps limit the extent of moisture loss. The improved microstructure also acts as a barrier to further moisture evaporation, effectively controlling the drying shrinkage deformation.

The reduction in drying shrinkage strain is thus attributed to the combined effects of accelerated hydration and the formation of more robust hydration products. These hydration products fill the pore spaces within the material, resulting in a more compact structure that reduces the potential for shrinkage cracks. This controlled hydration process ensures that the modified cement-stabilized phosphogypsum retains its dimensional stability during the drying phase, ultimately leading to a significant decrease in drying shrinkage and a reduction in the occurrence of cracking caused by shrinkage.

4. Conclusions

The present study applied three types of modifiers—sodium silicate, hydrated lime, and defluorinating agent—to improve the durability of cement-stabilized high-content phosphogypsum. First, the modification mechanisms of each modifier, both individually and in combination, are analyzed using XRF and SEM tests. Subsequently, the durability performance of cement-stabilized high-content phosphogypsum is validated through three experiments labeled three-point bending fatigue test, freeze–thaw test, and drying shrinkage test. Based on the observed findings, the following conclusions were arrived at:

1. The addition of sodium silicate increases silicon content and promotes additional hydration products, improving bond strength and durability. Hydrated lime enhances calcium content, boosting the formation of C-S-H gel and ettringite, resulting in a more resilient microstructure. The defluorinating agent immobilizes fluoride ions, improving stability and density. The composite modification leverages all three agents, enhancing density, strength, and overall performance through stable hydration products that fill microvoids;
2. The combination of sodium silicate and hydrated lime significantly enhances the fatigue performance of cement-stabilized phosphogypsum. Compared to single modifications, the composite modifier provides the greatest improvement in fatigue durability, effectively mitigating fatigue life decline under various stress levels. These findings demonstrate the potential of composite-modified cement-stabilized phosphogypsum to extend the service life and durability of road infrastructure;
3. The freeze–thaw process significantly impacts the compressive strength of cement-stabilized phosphogypsum. The composite modifier, including sodium silicate, hydrated lime, and defluorinating agent, reduces mass loss and improves compressive strength after freeze–thaw cycles compared to unmodified materials. The modified materials exhibit enhanced resistance to water-induced damage and a slower rate of strength decline under freeze–thaw conditions, highlighting the potential for pavement applications. However, caution is advised when applying high-content modified phosphogypsum in regions with high moisture levels and significant temperature fluctuations;

4. The modified cement-stabilized phosphogypsum shows a higher water loss rate than the unmodified material but demonstrates superior drying shrinkage performance. This improvement is due to the modifier's enhancement of the hydration process, forming additional hydration products that increase material density and reduce voids, effectively managing drying shrinkage and minimizing cracks;
5. Laboratory studies have shown the promising performance of composite-modified phosphogypsum, yet its practical application in road construction requires further validation. Future research should prioritize evaluating the feasibility of using this material in large-scale projects, investigating its performance across different road types (e.g., highways, urban roads), and particularly assessing its long-term durability under real-world conditions. Additionally, conducting a thorough cost–benefit analysis and optimizing construction processes will be crucial to fully understand the material's potential and its practical viability in road construction.

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