

Article

Study on the Performance of High-Performance Mortar (HPM) Prepared Using Sodium-Silicate-Modified Graphite Tailing Sand

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Abstract: In order to rationalize the consumption of graphite tailing sand and reduce its pollution of the environment—with sodium silicate being a commonly used activator for improving the strength of concrete composites—in this study, the joint effects of sodium silicate (SS) and graphite tail sand (GT) on the strength and frost resistance of graphite tail sand high-performance mortar (GT-HPM) were investigated. Experiments were conducted to evaluate the bulk density, water absorption, compressive strength, speed of sound, and working performance status of GT-HPM before and after freezing and thawing at different SS dosages and different GT substitution rates. The microstructural properties of GT-HPM were also analyzed by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR), and scanning electron microscopy (SEM/EDS). The results showed that 4% SS doping improved the performance of GT-HPM more obviously. Moreover, with an increase in the GT substitution rate, the mechanical properties and frost resistance of GT-HPM increased firstly and then decreased, and the best performance of GT-HPM was obtained when the GT substitution rate was 20%. At 6% SS doping, the performance of GT-HPM gradually decreased with the increase in the graphite tailing sand substitution rate. FT-IR testing showed that there was no significant change with the type of hydration products used, and the Si–O–T absorption peak and average bond length of GT-4 were the largest. SS and GT promoted the generation of hydration products. Microstructural analysis showed that 4% SS promoted the hydration reaction; in addition, an appropriate amount of GT improved the pore structure of HPM, increased the strength and frost resistance, and provided fundamental insights for the subsequent comprehensive utilization of graphite tailing sand.

Keywords: graphite tailing; high-performance mortar; workability; mechanical properties; frost resistance



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1. Introduction

As engineering demands escalate and challenges from increasingly extreme environmental conditions arise, traditional concrete materials are proving to be inadequate for specific engineering applications [1]. High-performance mortar (HPM) improves conventional mortars by optimizing the mix design and adding auxiliary cementitious materials (e.g., silica fume) to achieve compressive strengths up to 60 MPa [2]. HPM usually has excellent mechanical properties and durability in comparison with ordinary cement mortar and concrete, and it has a very dense microstructure and better frost resistance [3–6]. It is particularly suitable for facilities that are subjected to extreme environmental stresses, such as sea bridges, earthquake-resistant structures, and nuclear facilities.

Liquid sodium silicate ($\text{Na}_2\text{O}-\text{SiO}_2$, abbreviated as NS) is an environmentally friendly, water-soluble, alkali metal silicate material [7]. It is non-toxic, harmless, and will not cause environmental pollution to soil and water. The ratio of the mole fractions of SiO_2 to Na_2O

in their molecular formula is called a modulus. The modulus of NS is generally 1–3.5, and it is often mixed with a certain proportion of sodium hydroxide (NaOH) to be configured into a composite excitant with a fixed alkali equivalence [8–10]. Due to the presence of $[\text{SiO}_2(\text{OH})_2]^{2-}$, the polymerization reaction in the system can be accelerated, and the NS gel can be attached to the cracks and pores of HPM and react with $\text{Ca}(\text{OH})_2$ to generate hydrated calcium silicate (C–S–H) gel. In addition, it can form a fibrous structure, within which the elements can intersperse with each other to form a network gel [11]. It can be combined with a high-efficiency water-reducing agent for compounding, dispersing the microscopic particles of the HPM [12–14], to effectively improve the performance of the mortar, improving its resistance to chlorine ion penetration and electrochemical corrosion resistance, and finally render it a high-strength and high-performance mortar.

As an important non-metallic material, graphite is widely used in the new materials industry and other emerging technologies [15]. Graphite ore is crushed and ground to dissociate graphite particles from the ore, resulting in a large amount of graphite tailings [16]. China's graphite reserves can be mined, and 1.2 billion tons are currently made available annually. The effective utilization rate of graphite is only about 10–15% [17], which means that billions of tons of graphite tailing waste are generated [18]. The accumulation of GT in large quantities not only encroaches on farmland and reduces the land utilization rate, but also damages the environment around the mines and the physicochemical properties of soil. Moreover, due to the small particle size of GT, it can easily generate dust, which seriously damages the ecological environment [19,20]. Therefore, in order to reduce environmental pollution as well as resource recycling, the treatment of graphite tailings has become particularly important.

In the field of building structural materials, academic research has focused on the use of GT as the main material for replacing fine aggregates in order to achieve resource recycling. Liu [21] found that the GT substitution rate had a significant effect on the overall performance of concrete, with GT with a low substitution rate having an ameliorating effect on the microstructures, pores, and cracks of concrete, and GT with a high substitution rate leading to the overgrowth of concrete defects and reduced performance. He [22] investigated the effects of different GT substitution rates and the addition of different basalt fiber dosages on the performance of cement mortar and found that a 20% GT substitution rate and the addition of a 0.1% basalt fiber dosage had the best performance. Zhang [23] studied the effects of different GT substitution rates on the chlorine erosion resistance of recycled aggregate concrete (RAC) and found that a 20% GT substitution rate was the best, which enhanced the chlorine erosion resistance of RAC. Quan [24] investigated the use of GT as a fine aggregate, as well as the use of natural crushed stone and gangue as the coarse aggregate for the preparation of a large admixture of GT concrete, and found that, with the increase in the tailings' substitution rate, the strength of the concrete gradually decreased.

Existing studies have mainly explored the effects of solid waste admixtures on the mechanical properties of concrete, its durability effects, its material modification, etc. The alkali excitation conditions and freeze–thaw cycle conditions of the common coupling of these materials have a role in the problem that is to be investigated here; the preparation of graphite tailings of concrete produces a more complex interfacial transition zone (ITZ) [25]. Graphite tailings' particles are smaller, with a high rate of water absorption [26], so freezing resistance can have a certain negative effect. Also, as graphite tailings contain sulfides and heavy metal ions, they exhibit a relatively acidic state. This reduces the total alkalinity of the alkali activator and is thus unfavorable for the alkali activation reaction. Therefore, this study is devoted to evaluating the feasibility of preparing high-performance graphite tailing mortar (GT-HPM) through sodium silicate modification. GT materials are used to replace river sand in HPM in equivalent masses, with substitution rates of 0%, 20%, 40%, and 60%, respectively. The properties and characteristics of GT-HPM are systematically investigated. Macroscopic properties include the mechanical properties at different curing ages, nondestructive testing, and freeze–thaw cycle resistance. Microscopic characteristics comprise mineral composition, infrared spectra, and microstructure. In addition, the

damage caused by freeze–thaw cycles to the performance of GT-HPM is comprehensively investigated.

2. Materials and Methods

2.1. Cementitious Materials and Additives

In this study, P·O 42.5 cement was employed; its properties are exhaustively presented in Table 1. Silica fume (SF) details are indexed in Table 2. The chemical compositions of the cementitious materials are shown in Table 3. During mixing and blending, local tap water was utilized, supplemented with polycarboxylic acid high-performance water reducer as an admixture with 25.3% solids content, which reduces the water content by 27%. An alkaline activator solution in the form of liquid sodium silicate with a Baume degree of 50 and a modulus of 2.5 was used, and its properties are shown in Table 4. Steel fibers in the form of end-hooked fibers were employed, and their properties are shown in Table 5.

Table 1. Basic properties of cement (P·O 42.5).

Setting Time (min)		Flexural Strength (MPa)		Compressive Strength (MPa)		Ignition Loss (%)
Initial	Final	3d	28d	3d	28d	
260	315	4.7	8.2	25.2	51.2	4.1

Table 2. Indicators of silica fume auxiliary cementitious materials.

Ignition Loss (%)	Density (kg/m ³)	Specific Surface Area (m ² /kg)	Moisture Content (%)
2.73	270	19,000	1.32

Table 3. Chemical composition of cementitious materials (wt%).

Substance	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O ₃	TiO ₂	SO ₃	LOI
Cement	22.68	4.7	4.07	59.64	4.03	1.15	—	—	3.08	4.1
Silica fume	97.27	0.07	0.15	0.01	0.01	0.01	0.01	0.27	—	2.73

Table 4. Liquid sodium silicate physical and chemical index.

Modulus	°Bé	W (Na ₂ O)%	W (SiO ₂)%	Density (g/cm ³ , 20 °C)
2.5	50	14.12	33.11	1.5

Table 5. Physical properties of hooked-end steel fibers.

Tensile Strength (MPa)	Diameter (mm)	Length (mm)	Aspect Ratio
1000	1	35	75

2.2. Aggregates

The particle size distribution of river sand and graphite tail sand was analyzed using a laser particle size meter. The particle size of river sand ranges from 0 to 1.4 mm, presenting a bright yellow appearance. The graphite tail sand is from Hegang City, Heilongjiang Province. It has a greyish-black color with a particle size distribution from 0 to 1.1 mm. The PH value is 8.8, indicating weak alkalinity. Its chemical composition, determined by XRF, is shown in Table 6. The suitability of these materials for construction sand was evaluated according to the relevant standards [27], and the results are detailed in Table 7. Figure 1 shows the particle size distribution of graphite tailings, fine sand, cement, and

silica fume. Cement and silica fume are mainly distributed between 20 μm and 40 μm . Graphite tailings are mainly distributed between 150 μm and 250 μm , which is slightly larger than the particle size of cementitious materials and belongs to the ultrafine tailings. The river sand is mainly distributed between 450 μm and 650 μm . The d_{50} particle sizes of them are 225 μm , 449 μm , 18.03 μm , and 10.5 μm , respectively.

Table 6. Chemical composition of graphite tailings (wt%).

Substance	SiO ₂	SO ₃	Al ₂ O ₃	CaO	Fe ₂ O ₃	K ₂ O	MgO	TiO ₂
	48.01	15.41	10.44	9.87	8.1	4.02	3.43	0.72

Table 7. Physical properties of aggregates.

	Fineness Modulus	Apparent Density (kg/m ³)	Packing Density (kg/m ³)	Categorization	PH
River Sand	2.4	2594	1606	Medium sand	7
Graphite tailings	0.89	2857	1450	Extra-fine sand	8.8

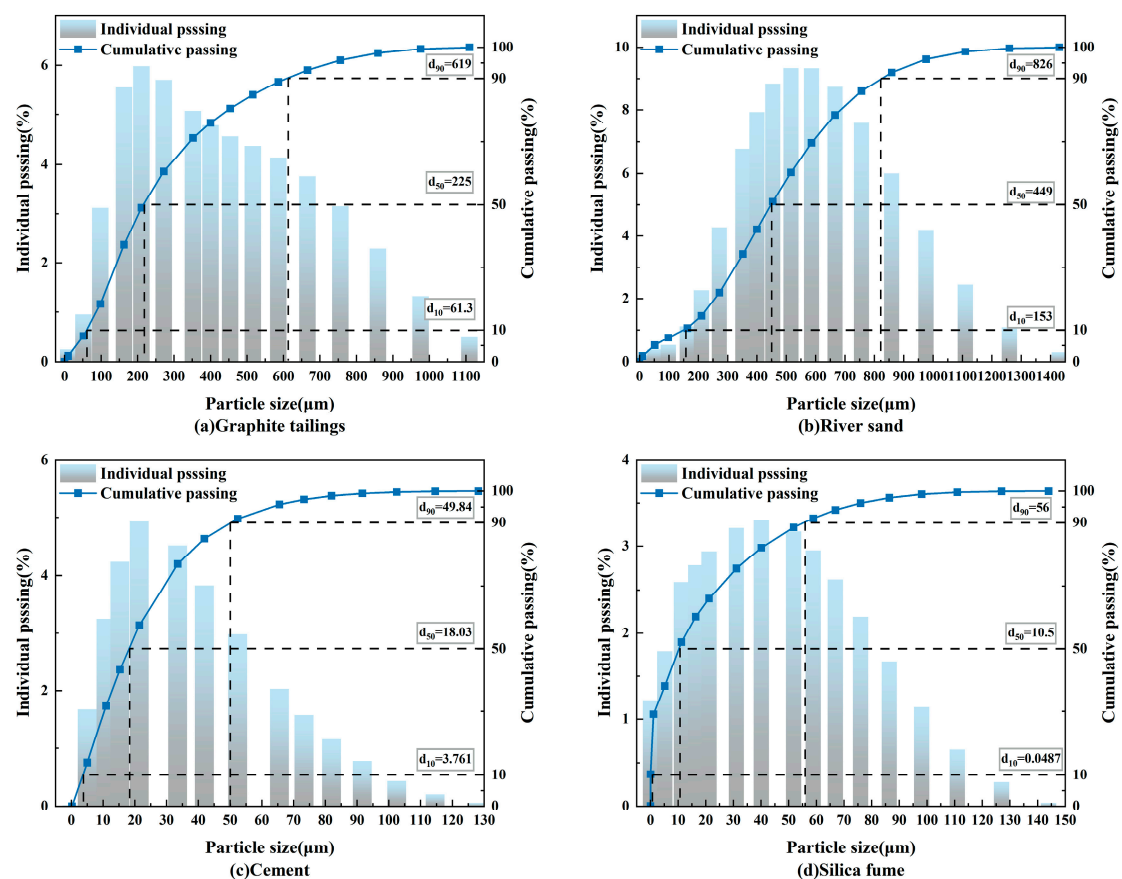


Figure 1. Particle size distribution of materials in GT-HPM.

2.3. Mixture Proportions and Preparation Process

After extensive preliminary proportioning tests, the baseline proportioning of high-performance mortar with river sand as fine aggregate was determined. By using GT to replace river sand with mass fractions of 0%, 20%, 40%, and 60%, HPM mix ratio designs with different GT substitution rates were developed, as shown in Table 8. The

dosage of polycarboxylic acid water reducing agent was stabilized at 2% of the mass of cementitious material. Here, RP—GT substitution rate; GT—graphite tailing sand; RS—river sand; C—cement; ST—steel fiber; SF—silica fume; W/B—water/cement ratio; WR—polycarboxylic acid high-performance water reducer dosage. According to a large number of studies, the water reducer dosage is controlled at an optimal level of 2% [28,29]. Note: SS—sodium silicate; SD—sodium silicate dosage. The literature review showed that the dosing of sodium silicate should be controlled between 4% and 6%, which can accelerate the hydration rate of cement and enhance the early strength [30]. The freshly mixed graphite tailing mortar body was stirred well and poured into 100 mm × 100 mm × 100 mm cubic plastic molds for the compressive strength test and 100 mm × 100 mm × 400 mm prismatic plastic molds for the freeze–thaw test. All mortar specimens were compacted using a vibrating table and then cured in an indoor environment (around 30 °C and above 50% relative humidity) until the planned pre-conditioning time. The test flowchart is shown in Figure 2.

Table 8. Mix proportion of prepared GT-HPM.

Mixture	RP (%)	GT	RS	C	ST	SF	W/B	WR	SS	SD (%)
GT-1	0	0	1200				0.2		0	0
GT-2	0	0	1200				0.2		38.4	4
GT-3	0	0	1200				0.2		57.6	6
GT-4	20	240	960				0.2		38.4	4
GT-5	40	480	720	960	96	240	0.2	19.2	38.4	4
GT-6	60	720	480				0.2		38.4	4
GT-7	20	240	960				0.2		57.6	6
GT-8	40	480	720				0.2		57.6	6
GT-9	60	720	480				0.2		57.6	6

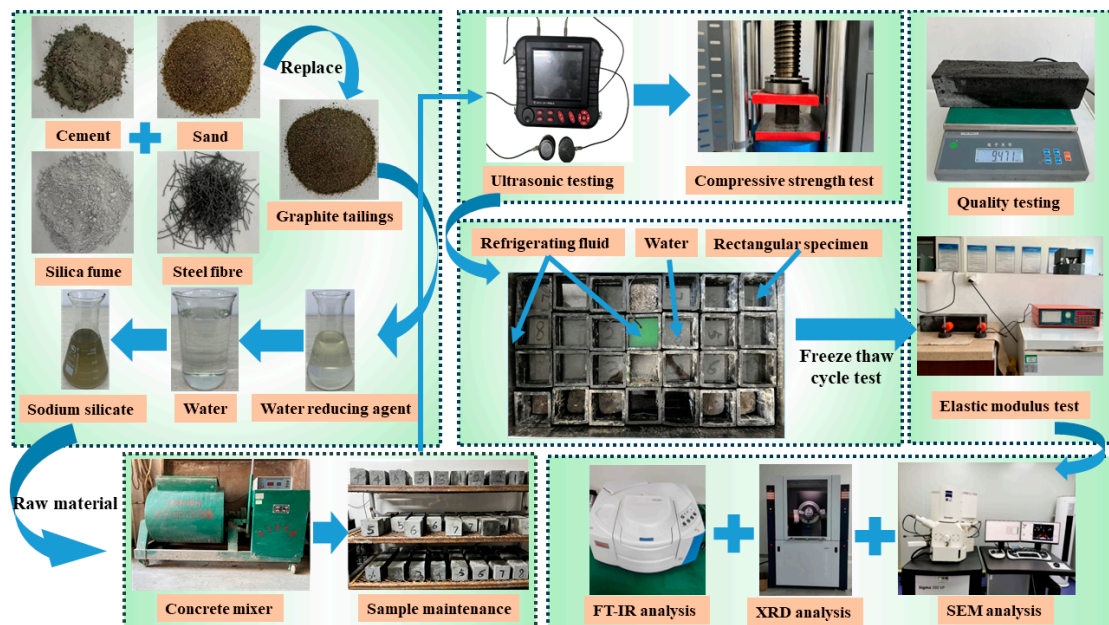


Figure 2. Flowchart of the test.

2.3.1. Water Absorption Test

The GT-HPM specimens, which were maintained until they reached the planned age, were immersed in water for 24 h. The specimens were taken out, and the surface moisture was dried with paper towels. They were weighed with a differential balance and the weight

was recorded until the weight of the specimens became stable. Calculations were made according to Equation (1) [31]:

$$W = \frac{M_2 - M_0}{M_2} \times 100\% \quad (1)$$

where M_2 is the mass of the saturated specimen in air in kg; M_0 is the mass of the dry specimen in air in kg; W is the water absorption in %.

2.3.2. Mechanical Performance Testing and Ultrasonic Testing

Mechanical properties and ultrasonic testing of GT-HPM were carried out at maintenance ages of 3 and 28 days. The specimen size was $100 \times 100 \times 100$ mm, and 3 replicate specimens were prepared for each age. Referring to the relevant provisions of GB/T 50081-2002 [32] “Standard of Test Methods for Mechanical Properties of Ordinary Concrete”, the basic mechanical properties were tested and the results were averaged. The instrument used for the test is ZT805-automatic pile tester to test the internal sound velocity value of GT-HPM. For the measurement of ultrasonic sound velocity, the pair measurement method is adopted. Before the ultrasonic value test, a layer of petroleum jelly should be uniformly coated as a coupling agent on the test point of the test surface of the specimen to ensure good coupling between the transducer and the test surface. The test should be conducted to ensure that the central axis passes through the midpoint of ultrasonic test point and the test surface is perpendicular to the central axis for sound velocity testing. The principle is as shown in Figure 3. For each test block, 3 groups of values are tested. In the same way, the average from its 3 groups of data is taken as the sound velocity representative value of the test block. This was performed in order to determine the effect of early and late sodium silicate and graphite tailing sand on the performance of GT-HPM.

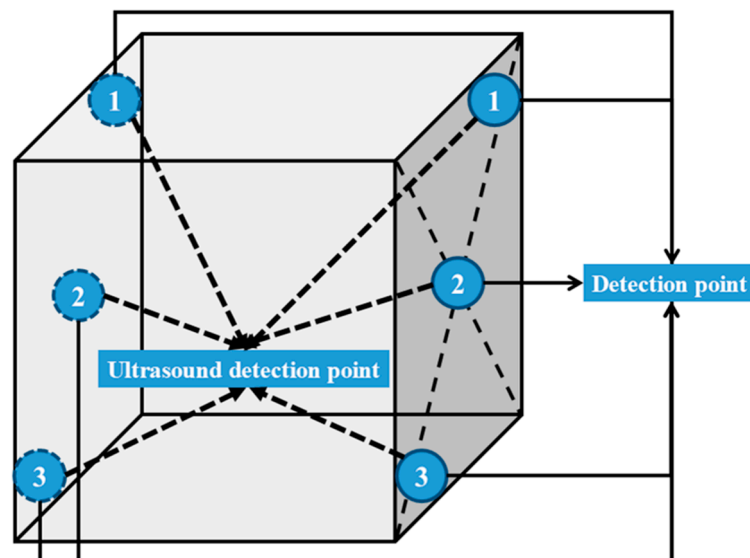


Figure 3. Velocity of sound test principle.

2.3.3. Freeze–Thaw Cycle Test

The freeze–thaw cycle test was conducted with reference to the test method in GB/T50082-2009 [33] “Standard for Test Methods of Long-term Performance and Durability of Ordinary Concrete”. Prior to the test, on day 28, nine cubic specimens were dried to constant weight in an oven at 60°C and then immersed in pure water until saturation was reached, at room temperature. Then, the 9 saturated specimens were placed in a rubberized container with pure water and subjected to freeze–thaw test on a TDR-28 rapid freeze–thaw tester. A cycle of every 4 h consisted of a 2.5-h freezing phase and a 1.5-h thawing phase. Four sensors were used to monitor the temperature inside the cooling box:

three sensors measured the temperature of the cooling box, and one sensor measured the core temperature of the specimen. The cooling box temperatures ranged from $-20 \pm 2^\circ\text{C}$ during freezing to $+20 \pm 2^\circ\text{C}$ during thawing, while specimen core temperatures ranged from $-17 \pm 2^\circ\text{C}$ to $+5 \pm 2^\circ\text{C}$. Repeated freeze–thaw cycles were designed to simulate field exposure and induce internal damage to GT-HPM. Time–temperature curves under freeze–thaw cycles for GT-HPM are shown in Figure 4.

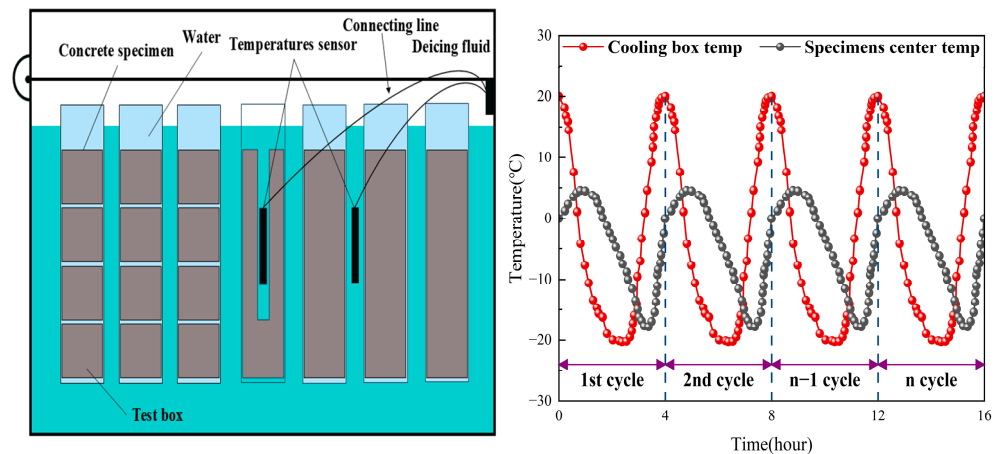


Figure 4. Freeze–thaw test chamber temperature–time curve.

After every 50 freeze–thaw cycles, the surface of the specimen was observed and its mass, relative modulus of elasticity, and compressive strength were measured. The freeze–thaw process was continued until the samples had undergone 200 cycles and the equation for the rate of mass loss was as follows:

$$\Delta W_n = \frac{w_0 - w_n}{w_0} \times 100\% \quad (2)$$

where ΔW_n is the mass loss rate after n F-T cycles (%); W_0 is the mass at 0 F-T cycle (kg); W_n is the mass after n F-T cycles (kg).

The relative modulus of elasticity equation is as follows:

$$R_{Ed} = \frac{E_n}{E_0} \times 100\% \quad (3)$$

where R_{Ed} is the RDM after n F-T cycles (%); E_n is the dynamic modulus of elasticity after n F-T cycles (MPa); E_0 is the dynamic modulus of elasticity at 0 F-T cycle (MPa).

2.3.4. Microscopic Testing

Samples of GT-HPM were taken at a maintenance age of 28 days. These samples were pre-soaked in anhydrous ethanol for 24 h to terminate hydration. Then, the samples were dried in a vacuum-drying oven for 24 h at a temperature of 40°C . The samples were ground into a powdered form and collected through a 200-mesh sieve. The powdered specimens were placed on Cu targets and tested using a D8 advanced X-ray diffractometer with a scanning range of 5° to 90° and a scanning rate of $2^\circ/\text{min}$. The infrared spectra of the HPM specimens were analyzed using a Bruker ALPHA II. The terminally hydrated and dried HPM samples were ground with potassium bromide (KBr) powder abrasive (100–120 mg). The resulting mixture was then transferred to a mold and compacted under vacuum to form disk-shaped test samples [34]. The hydration mechanism was interpreted based on the change of characteristic peaks in the infrared spectra of nine HPM groups. Microstructural analysis of the HPM specimens was performed using a scanning electron microscope Sigma 300. The HPM samples were mechanically crushed after 200 cycles of freezing and thawing, and the surfaces of the samples were sprayed with gold to enhance

their electrical conductivity; this was conducted by selecting the samples with a particle size of 5–8 mm and a flat surface. Then, SEM-EDS analysis was performed to analyze the microstructure and chemical composition of the hydration products of GT-HPM after freezing and thawing.

3. Results and Discussion

3.1. Water Absorption and Wet-Packing Density Analysis

Figure 5 shows the Variation in water absorption and apparent density of GT-HPM. The wet-packing density serves as an indicator to gauge the compactness of fresh GT-HPM slurry. Higher values indicate a lower porosity within the slurry [35]. When the substitution rate of GT reaches 20% and the dosage of sodium silicate is 4%, the stacking density peaks, and the water absorption is at its lowest. This is because the density of graphite tailing sand is greater than that of river sand; using appropriate amounts of graphite tailing sand to substitute river sand will increase the density of the mortar. However, due to the high water-absorption property of graphite tail sand, its excessive use as a sand substitute in mortar can significantly increase the water absorption of the prepared mortar. Moderate amounts of sodium silicate can improve the surface microcracking of GT-HPM. However, the strong alkaline environment produced by an excess of sodium silicate results in a hydration reaction that is too rapid, leading to a short initial setting time, cracks on the surface of the sample [36], a significant increase in water absorption, and a decrease in density. The results show that a 20% GT substitution rate combined with sodium silicate can effectively inhibit the cracked porosity of GT-HPM. Among the substitution rates, 4% sodium silicate has a significant effect; however, with the gradual increase in the GT substitution rate, GT promotes a gradual increase in the water absorption of GT-HPM.

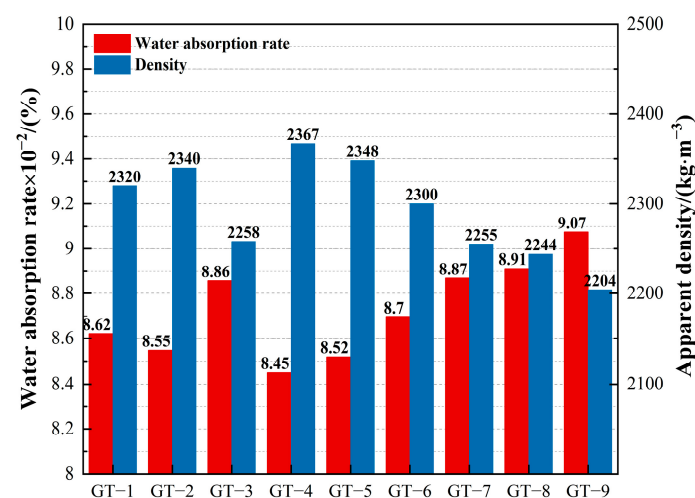


Figure 5. Water absorption and wet-packing density.

3.2. Compressive Strength and Speed of Sound Analysis

Figure 6 shows the damaged interior of the GT-HPM specimen. It can be found that there are fewer holes and cracks in the interior. This is because steel fibers enhance the adhesion and integrity between the interfaces of the GT-HPM matrix. In the case of compression, steel fibers and the matrix bear the loads simultaneously and inhibit the growth of macroscopic cracks, in addition to enhancing the ability of the cracks to create bridges [37]. The compressive strength and sound velocity values of GT-HPM at different maintenance ages are shown in Figure 7. The results show that the mechanical properties increase with the increase in the maintenance age due to the continuous hydration reaction of cement. The 3-day compressive strength of HPM can reach 65.3% to 78.52% of the 28-day compressive strength. This indicates that an appropriate proportion of sodium silicate alkaline stimulant has strong viscosity. In the internal pores of the test block, it can flow and

bond to the inner wall of the test block, playing the role of repairing cracks and leading to a denser structure, which is conducive to optimizing the strength of HPM mortar. However, excessive Na_2SiO_3 will lead to too high a concentration of hydroxide ions, resulting in the precipitation of aluminosilicate gel in the early stage and reducing the strength of the HPM [38]. Thus, 28-day compressive strength follows the order $\text{GT-2} > \text{GT-1} > \text{GT-3}$. Since the sodium silicate has a significant effect on the early strength, the 3-day compressive strength is $\text{GT-2} > \text{GT-3} > \text{GT-1}$. Because the specific surface area of graphite tailing sand is larger than that of river sand, and a small amount of graphite tailing sand plays the role of filling gaps, the compressive strength of GT-4 is the largest. At the same time, because graphite tailing sand contains sulfide and heavy metal ions and presents a relatively acidic state, it will reduce the total alkalinity of sodium silicate; thus, this is not conducive for the occurrence of the alkali activation reaction. As a result, GT-5, -6, -7, -8, and -9 all show a linear decrease in compressive strength with increasing graphite tailing sand substitution rate, which is consistent with the results of other studies [22]. Meanwhile, the higher the sound velocity, the denser the mortar interior, and the stronger the performance, which is consistent with the conclusion of the trend of compressive strength change.

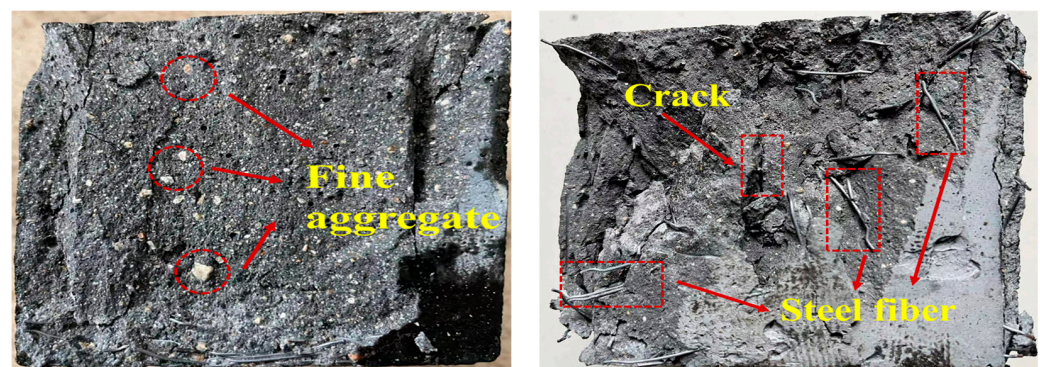


Figure 6. GT-HPM compressive damage section.

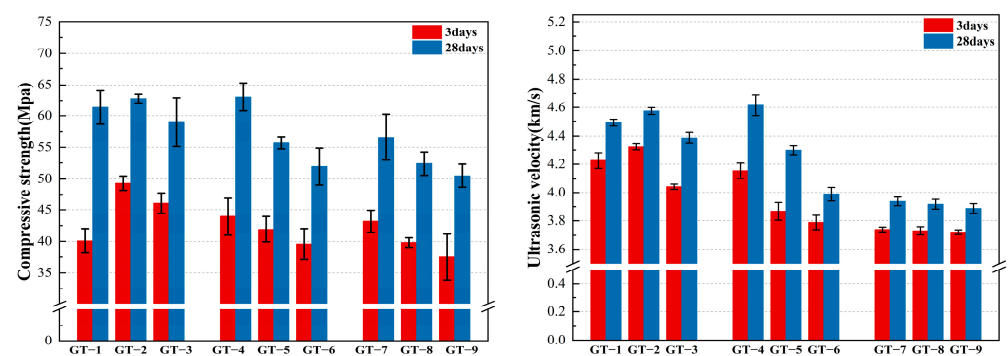


Figure 7. Variation in GT-HPM compressive strength and sound velocity values at different ages.

Figure 8 shows the fitted relationship between the replacement rate of HPM graphite tailing sand with compressive strength and speed of sound at different sodium silicate dosages. Compared with the doping of 6% sodium silicate, the fitting of compressive strength and sound velocity to graphite tail sand substitution rate data for 4% sodium silicate doping is more satisfactory, with an average coefficient of determination, R^2 , greater than 0.95. This may be due to the excessive sodium silicate content in HPM specimens at 6% sodium silicate content, which provides more OH^- ions and forms a strong alkali solution. In this case, the generation rate of hydration products is much larger than the diffusion rate, and the specimen is not internally uniformly dense. Moreover, the internal defects are increased, which is prone to causing variations in the compressive strength and the speed of sound. This finding is basically consistent with the conclusions

of existing studies [39,40]. Overall, the proposed polynomial function clearly reveals the variation patterns of compressive strength and sound velocity with the graphite tail sand substitution rate.

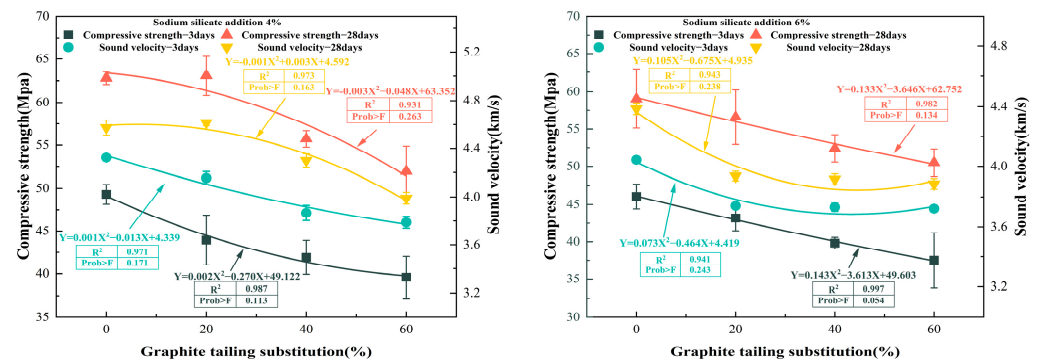


Figure 8. Graphite tailing substitution rate versus compressive strength and sound velocity fit.

3.3. Frost Resistance

3.3.1. Surface Erosion

Table 9 shows the typical surface etching patterns of nine groups of HPM specimens after 200 freeze–thaw cycles. The specimens showed different degrees of internal defects from the surface to the inside and from the outside to the inside after freezing and thawing. Macroscopically, the surfaces gradually became loose and porous, and the fine aggregates were slowly dislodged. GT-7, -8, and -9 showed severe freeze–thaw damage after 200 cycles, with the separation of the cement mortar on the surfaces and the loss of part of the aggregates at the ends of the specimens, accompanied by serious cracks, fissures, and spalling. GT-4, -5, and -6 showed relatively improved damage patterns after the freeze–thaw cycles compared with GT-7, -8, and -9. Although there was still a significant amount of aggregate loss at the ends of the specimens, no extensive disintegration was observed. This suggests that a moderate amount of sodium silicate further improves its resistance to the surface degradation that is induced by freezing and thawing. GT-1, -2, and -3 showed insignificant cracking and spalling after 200 cycles. All graphite tailing sand additions exhibited surface softening, and most of them experienced surface flaking, which could be attributed to water uptake from excess graphite tailing sand during thawing. It is evident that the addition of graphite tailing sand plays an inhibitory role in HPM frost resistance, and the graphite tailing sand in the slurry matrix effectively contributes to the capillary pore expansion stress that is induced by water freezing. The above results suggest that adding 4% sodium silicate and reducing the substitution with graphite tailings can lead to more favorable cycle times and surface degradation patterns.

During the freeze–thaw process, positive and negative temperature fluctuations can inflict two main types of damage on the mortar. Firstly, during freezing, the free water in the pore medium freezes, causing volume expansion. This makes it difficult for the freezing and expansion stresses within the mortar to be released. The irreversible tensile stresses generated by this volume expansion lead to microcracks and macrocracks in the mortar. Secondly, during thawing, the bonding properties between aggregates and cementitious materials will be weakened as the ice crystals in the mortar melt. This is manifested macroscopically as the loss of cementitious mortar on the surface of the mortar [41].

Table 9. Change of specimen surface before and after freeze–thaw cycle.

Designation	Typical Surface Image of 0 Freeze–Thaw Test Samples	Typical Surface Image of 200 Freeze–Thaw Test Samples	Typical Surface Alteration Descriptions
GT-1			Microcracks, softening
GT-2			Microcracks, micropores
GT-3			Microcracks, surface peeling, and cracks
GT-4			Microcracks
GT-5			Cracks, fissures, and spalling
GT-6			Cracks, fissures, and moderate spalling
GT-7			Cracks, fissures, severe spalling, and softening
GT-8			Fissures, moderate spalling, and softening
GT-9			Moderate crack, fissures, severe spalling, and softening

3.3.2. Mass Loss

The evolution of the mass loss rate of HPM is shown in Figure 9a. It was found that there was almost no change in the mass loss of HPM until 50 freeze–thaw cycles had occurred. This is because, at this stage, HPM mainly experiences water absorption and there is relatively little mortar spalling. When the number of freeze–thaw cycles is greater than 100, mortar spalling becomes the main component of mass loss for HPM, and the degree of damage is further intensified, which is in line with the results of previous studies. After 200 freeze–thaw cycles, the mass loss rates of GT-4, -5, and -6 were lower than those of GT-7, -8, and -9. Among them, GT-4 had the lowest mass loss rate. This is mainly due to the fact that an appropriate amount of sodium silicate in the mortar can increase the amount of active silica dissolved in the polymerization reaction. Consequently, it reacts with $\text{Ca}(\text{OH})_2$ to generate C-S-H, covering the cracks and pores and playing the role of repairing cracks. Similar findings have also been reported by Wang et al. [42]. At the same time, a small amount of graphite tailing sand acts as micro-aggregate filling, thus creating a superposition effect, effectively preventing free water from entering HPM and enhancing its frost resistance. Excessive sodium silicate cannot increase the dissolution amount of activated silica in polymerization reaction, which has an unfavorable impact on the specimens. Meanwhile, with the increase in the substitution rate of graphite tailing sand, the mass loss rate of the specimens gradually rises. This is because the particles of graphite tailing sand are finer than those of river sand, which absorbs water and expands, accelerating the damage caused by pore water expansion pressure on HPM.

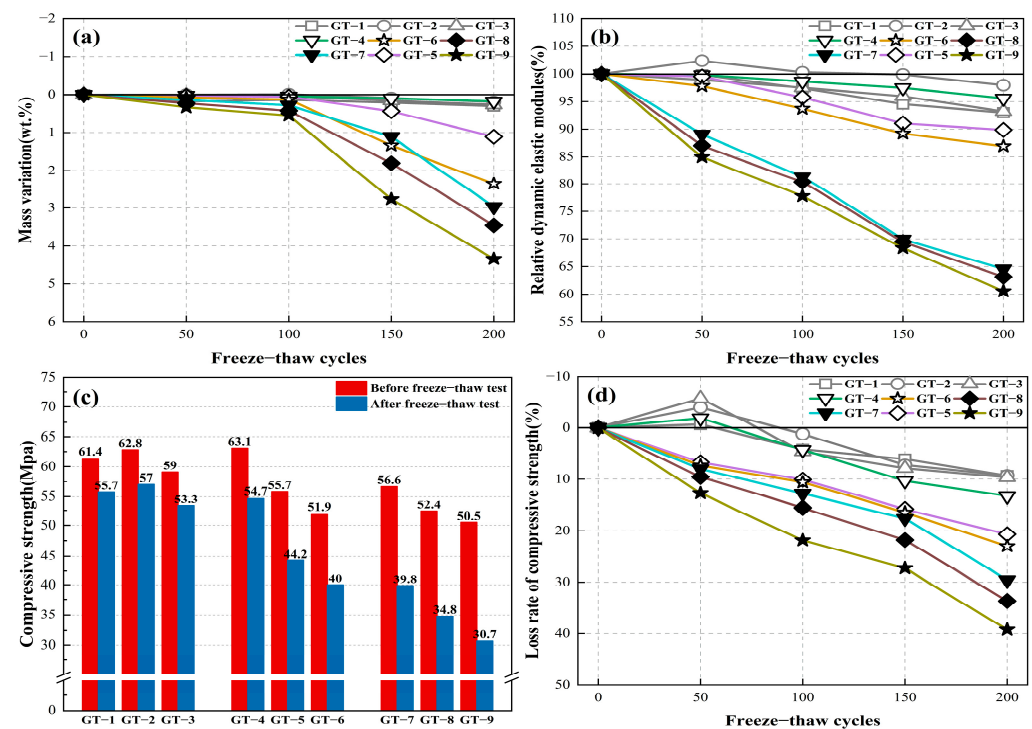


Figure 9. Changes in GT-HPM performance before and after freeze–thaw cycles. (a) The mass loss of GT-HPM after freeze–thaw test; (b) the RDME of GT-HPM after freeze–thaw test; (c) the change in the strength of GT-HPM before and after freeze–thaw; (d) the strength change of GT-HPM after freeze–thaw test.

3.3.3. RDME

The change curve of the relative dynamic elastic modulus of the specimen during the freeze–thaw cycle is shown in Figure 9b. The relative kinetic elastic modulus can reflect the internal damage of the specimen through the speed of acoustic wave transmission inside the frost-resistant specimen. The larger the relative kinetic elastic modulus is, the smaller the internal damage of the specimen will be. The relative dynamic elastic modulus of the specimens decreased with the increase in the number of freeze–thaw cycles, and all of them showed microcracks. Among them, the relative elastic modulus of GT-7, -8, and -9 changed very significantly. When the number of freeze–thaw cycles reached 200, the specimens were structurally damaged. The relative dynamic elastic modulus of GT-7, -8, and -9 were 64.6%, 63.1%, and 60.5%, respectively. However, all of them met the critical value of freeze–thaw for concrete, and the RDME was higher than 60%. This indicates that, under the alkaline environment of excess sodium silicate, although graphite tailing sand shows relative neutrality, it still cannot compensate for the excess hydroxide ions to generate cementitious products, resulting in a rapid decrease in the properties of HPM under freeze–thaw conditions.

3.3.4. The Strength Change

The compressive strength and compressive strength loss rate of the specimens before and after freezing and thawing are shown in Figure 9c,d. When the number of freezing and thawing cycles is less than 50, the compressive strength of GT-1, -2, and -3 increases slowly. This is due to the fact that active Na_2SiO_3 enters the inside of the HPM specimen through diffusion, infiltration, and capillarity. It fills up the internal porosity of the specimen through secondary hydration with the cementitious material that has not been completely hydrated, increasing the compactness of specimen. However, excessive sodium silicate leads to a rapid alkali excitation reaction, causing the cementitious materials to rapidly dissolve and polymerize into clumps. These clumps will wrap other unreacted cementitious

materials, hindering further hydration reaction and leading to a reduction in compressive strength. At the same time, due to the incorporation of graphite tailings, heavy metals hinder the hydration reaction, resulting in a reduction in the compressive strength of GT-7, -8, and -9 with the increase in the replacement rate of graphite tailings under freeze–thaw cycle. This is consistent with previous findings on mass loss rate and relative modulus of elasticity.

3.4. Freeze–Thaw Damage Mechanism

The effect of freeze–thaw damage on HPM and the sodium silicate reaction mechanism are more clearly represented in Figure 10a,b. The freeze–thaw damage of HPM is essentially the accumulation of fatigue damage that occurs when initial defects such as internal pores and cracks are subjected to cyclic freezing and expansion forces [43]. Meanwhile, the performance strength of HPM is mainly formed by three materials: silica fume, cement, and sodium silicate. The polymerization reaction occurs through alkali excitation to form a reticulated polymer, which provides a prerequisite for the formation of C–A–S–H gels and N–A–S–H gels. The polymerization reaction mainly involves the dissolution, diffusion, and reorganization of aluminosilicate surfaces in the presence of alkaline activators to form flocculated structures. These flocculated structures are coalesced, diffused, and further transformed from a solid to a hard solid [44].

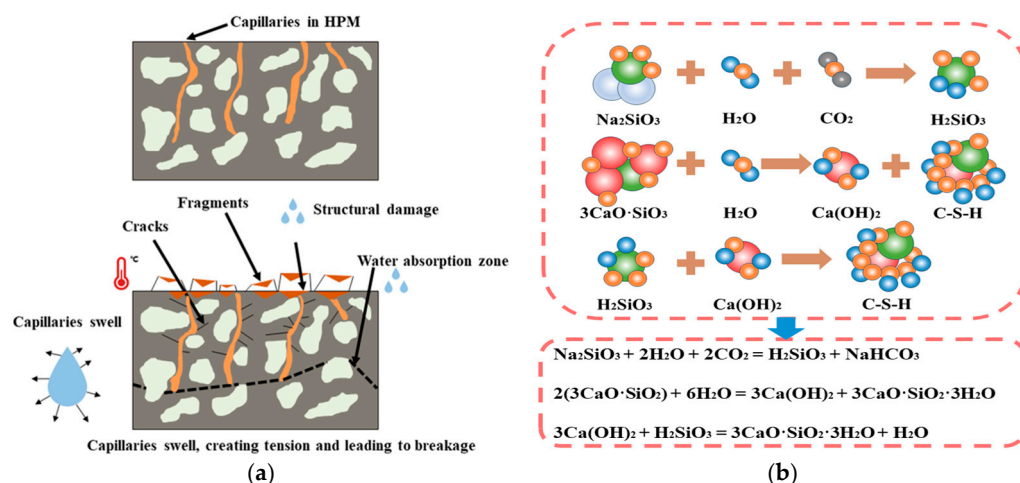


Figure 10. GT-HPM reaction mechanism diagram. (a) Freeze–thaw cycle damage mechanism diagram; (b) alkali exciter reaction mechanism diagram.

3.4.1. FTIR Analysis

Figure 11 and Table 10 show absorption peaks for all HPM samples that can be attributed to various chemical bond vibrational motions: in-plane bending vibration of Si–O near 462 cm^{-1} , symmetric telescopic vibration of the internal Si–O tetrahedral unit at 1117 cm^{-1} , telescopic vibration of O–C–O at 1427 cm^{-1} , and O–H telescopic vibration in $\text{Ca}(\text{OH})_2$ at 3639 cm^{-1} corresponding to O–H stretching vibration [45]. There are no obvious characteristic absorption peaks of other groups in the spectra, indicating that the characteristics of the groups in the nine groups of HPM are essentially the same. This implies that the hydration products did not change significantly due to the addition of SS or GT during the hydration process of the cement. With the increase in the graphite tailing sand substitution rate, the number of absorption peak waves caused antisymmetric telescopic vibration (wavelengths $784, 795, 794, 793, 791\text{ cm}^{-1}$) of the internal Si–O–T tetrahedra in 4% sodium-silicate-doped HPM first increases and then decreases. This shift indicates that the degree of polymerization of silica–oxygen tetrahedra within the C–S–H gel first increases and then decreases. Some chain structures develop into double-chain or lamellar structures, and the average bond length first increases and then decreases. This is because the active Si and Al in the graphite tailings will promote the C–S–H to generate

more complex chain structure or reticular structure, making the C-S-H gel structure more dense. The chemical reaction equations are shown in Equations (4)–(6) [46]. However, excessive graphite tailings will form insoluble substances adhering to the surface of the cement particles due to the presence of heavy metal ions inside. At the same time, under the alkaline conditions of high concentration, they will also form insoluble substances, adhering to the surface of cement particles, blocking the contact between cement and water and thus hindering the hydration reaction. Therefore, compared with 4% sodium silicate dosage, the absorption peak wave number of 6% sodium silicate dosage (wavelength 784, 791, 786, 782, 781 cm^{-1}) is reduced. A sodium silicate hydrolysis reaction occurs, resulting in silicate ions (SiO_3^{2-}) and hydroxide ions (OH^-). Silicate ions with a certain degree of activity can further undergo condensation reaction and form polymerized silicate ions. With excessive sodium silicate doping, the poly-silicate ion grows continuously, which eventually leads to sodium silicate self-polymerization phenomenon. This is similar to the conclusion that Wu is prone to self-polymerization when the alkali concentration is too high, which leads to a decline in performance [47].

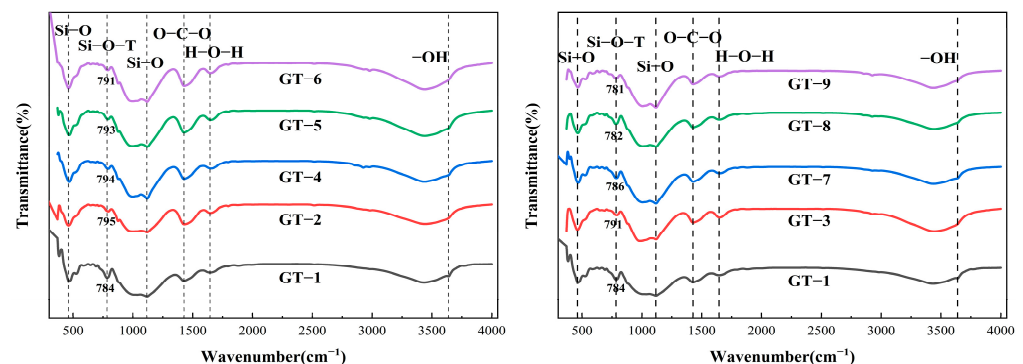
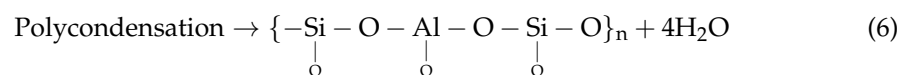
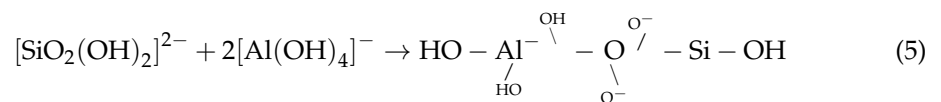
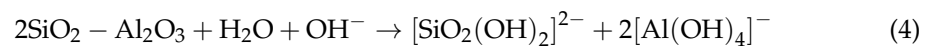


Figure 11. FTIR spectra of GT-HPM after freezing and thawing.

Table 10. Functional groups present in the GT-HPM specimens.

Species	Wavenumber	Type of Vibration
-OH	3639 cm^{-1}	Stretching vibrations
H-O-H	1645 cm^{-1}	Bending vibrations
O-C-O	1427 cm^{-1}	Stretching vibrations
Si-O-T (T: Si or Al)	$784\text{--}795 \text{ cm}^{-1}$	Stretching vibrations
Si-O	1117 cm^{-1}	Symmetric stretching vibration
Si-O	462 cm^{-1}	In-plane bending vibration

3.4.2. XRD Analysis

The results in Figure 12 show that the main physical phases of HPM are C-S-H gel, $\text{Ca}(\text{OH})_2$, SiO_2 , calomel, and a small amount of cement clinker (C_2S , C_3S). Compared with the control GT-1, similar hydration products were exhibited, indicating that the incorporation of graphite tailing sand and sodium silicate does not change the hydration products and types of HPM. With the increase in graphite tailing sand substitution rate, the intensity of SiO_2 diffraction peaks near 27 nm first weakened and then strengthened.

The intensity of SiO_2 diffraction peaks was the weakest when the substitution rate reached 20%. This phenomenon occurs because the overall gradation of graphite tailing sand is finer than that of river sand. When graphite tailing sand is added to HPM, the fine particles in the mixture play a “nucleation effect”, accelerating the hydration reaction, resulting in a weakening of the SiO_2 peaks and generating more CSH gel and $\text{Ca}(\text{OH})_2$. These, together with SiO_2 , can continue to undergo the pozzolanic reaction, generating more C-S-H. When the excess amount of graphite tailing sand reaches 20%, the SiO_2 peaks are the weakest, generating more C-S-H to fill HPM [48]. When excessive graphite tailings are added to HPM, the heavy metals in the graphite tailings attenuate the alkali excitation reaction, slowing down the hydration rate and subsequently enhancing the SiO_2 peak.

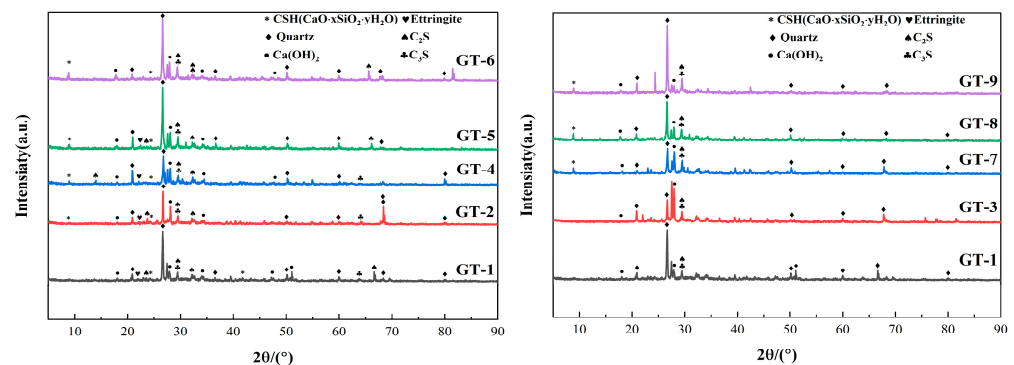


Figure 12. XRD pattern of GT-HPM samples after freeze–thaw cycles.

3.4.3. SEM/EDS Analysis

Figures 13 and 14 show the SEM micromorphology and EDS analysis results of GT-HPM. As shown in Figure 13a–d, a relatively dense structure is presented. It is mainly characterized by the various morphologies of hydration products. The microstructures of GT-1, -2, -3, and -4 are relatively homogeneous. The hydration products are densely piled up, the pores between the particles become smaller, and the hydration products encapsulate the particles into a whole, making the microstructures denser. There is no obvious interfacial transition zone or crack, and there are small amounts of spherical or flocculent hydration products on their surface. The EDS analysis results indicated that the flaky and flocculent hydration products were CH and C-S-H gel or their mixed products. SEM images showed that GT-7, -8, and -9 had obvious pores and localized extensive longitudinal cracks. There was a high amount of Aft and $\text{Ca}(\text{OH})_2$, and the C-S-H gel was less. Non-hydrated cement particles could be found at the same time, which affected the strength of GT-HPM. The EDS analysis of GT-HPM hydration products in Figure 14 and Table 11 mainly consisted of O, Ca, Si, and C, indicating that the hydration products are mainly a mixture of C-S-H gels, which act as a binder for HPM pores and cracks [49]. The Ca/Si variation in GT-HPM is found to be similar to the previous findings. GT-4 has the smallest Ca/Si and forms a hydrated calcium silicate that looks finer. This is due to the fact that a moderate amount of sodium silicate provides reactive SiO_2 doped into the HPM for a secondary hydration reaction with $\text{Ca}(\text{OH})_2$ to produce more C-S-H gels [50]. Also, due to the small particle size and large specific surface area of graphite tailing sand, it can fill the internal pores of C-S-H gels. However, excessive graphite tailing sand may adsorb onto the active sites on the surface of cement particles due to the presence of heavy metals inside. For example, Fe^{2+} may adsorb on the active sites on the surface of the cement particles, preventing the formation of hydration products such as hydrated calcium silicate [51]; Cr^{3+} may enter into the structure of hydrated calcium silicate, leading to an increase in structural defects and a decrease in the strength of the hydrated calcium silicate [52]; Pb^{2+} may form a hydroxide precipitation that fully encapsulates the cement particles and leads to a decrease in the hydration reaction properties of cement [53]. The resulting microscopic cracks and pores on the surface of GT-7, -8, and -9 are the main cause of the decrease in HPM strength.

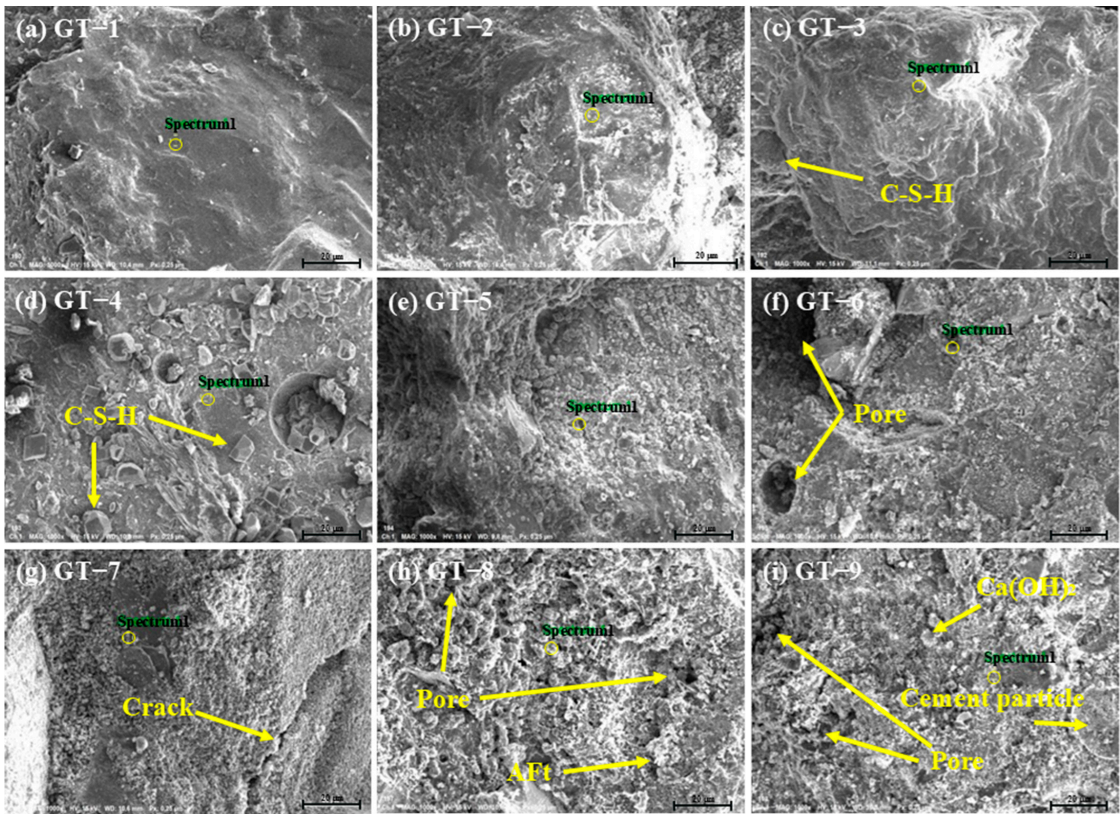


Figure 13. SEM images of GT-HPM samples after freeze–thaw cycles.

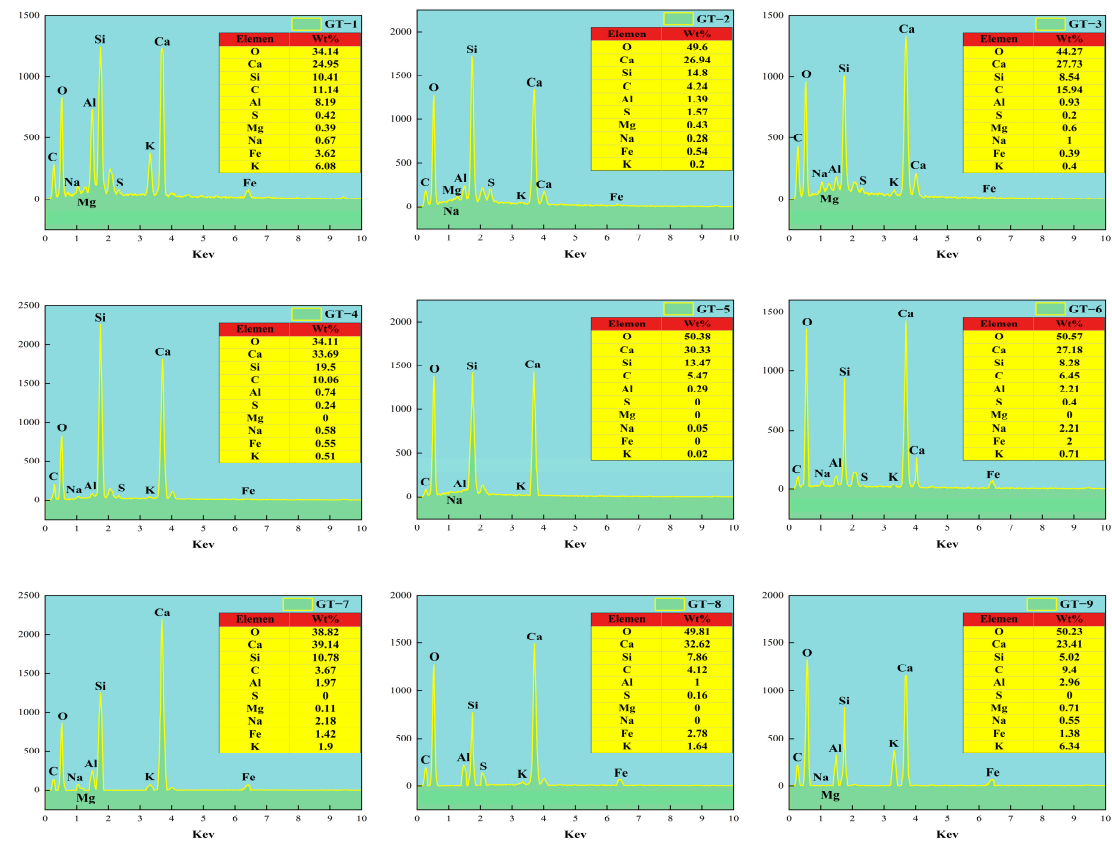


Figure 14. EDS results of GT-HPM samples after freeze–thaw cycles.

Table 11. GT-HPM EDS element results.

Mixture	O	Ca	Si	C	Al	S	Mg	Na	Fe	K	Ca/Si
GT-1	34.14	24.95	10.41	11.14	8.19	0.42	0.39	0.67	3.62	6.08	2.40
GT-2	49.6	26.94	14.8	4.24	1.39	1.57	0.43	0.28	0.54	0.2	1.82
GT-3	44.27	27.73	8.54	15.94	0.93	0.2	0.6	1	0.39	0.4	3.25
GT-4	34.11	33.69	19.5	10.06	0.74	0.24	0	0.58	0.55	0.51	1.73
GT-5	50.38	30.33	13.47	5.47	0.29	0	0	0.05	0	0.02	2.25
GT-6	50.57	27.18	8.28	6.45	2.21	0.4	0	2.21	2	0.71	3.28
GT-7	38.82	39.14	10.78	3.67	1.97	0	0.11	2.18	1.42	1.9	3.63
GT-8	49.81	32.62	7.86	4.12	1	0.16	0	0	2.78	1.64	4.15
GT-9	50.23	23.41	5.02	9.4	2.96	0	0.71	0.55	1.38	6.34	4.66

4. Conclusions

The performance of high-performance mortars prepared by using sodium-silicate-inspired graphite tailings to replace river sand was investigated. The following major conclusions were drawn:

1. The effect of SS (sodium silicate) and GT (graphite tailings) on the compressive capacity and internal densification of GT-HPM (graphite tailing high-performance mortar) was obvious. The optimum SS doping was 4%, and the GT substitution rate was 20%. The compressive strength of GT-4 reached a maximum of 63.05 Mpa, and the value of the sound velocity reached a maximum of 4.61 km/s. The compressive strength and the value of the sound velocity showed a quadratic polynomial relationship with the substitution rate of graphite tailing sand, and the fitting effect was good.
2. Incorporation of 4% SS and 20% GT substitution rate significantly retarded the destructiveness of GT-HPM under freeze–thaw cycling. After 200 freeze–thaw cycles, the loss of compressive strength was 13.28%, the mass loss reached 0.17%, and the relative modulus of elasticity reached 95.57%, which improved the frost durability of GT-HPM.
3. According to the microscopic mechanism, it was found that the microstructure of GT-4 was relatively dense without obvious cracks and pores. This was attributed to the fact that an appropriate amount of SS and GT would provide active Si for the hydrate gelling material for the second time, fill the pores and cracks, and optimize its pore structure. Excessive SS will lead to a high concentration of OH^- and the formation of silica gel precipitation, which will affect the hydration reaction. While GT will hinder the hydration reaction due to its internal heavy metal content, reducing the performance of GT-HPM.

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